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Tesi di Laurea Magistrale

Experimental characterization of a novel, multistage, countercurrent, cavitational solid-liquid extractor and its applicability to agro-food industrial waste valorization

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Ai miei compagni di viaggio

Alla mia famiglia

A Simona

A me

Sommario

Negli ultimi anni si è registrata una sempre maggiore attenzione, in particolar modo da parte delle aziende e delle filiere produttive, verso tematiche correlate all'intensificazione di processo e alla riduzione e valorizzazione degli scarti che inevitabilmente vengono prodotti. In ambito industriale, adottare provvedimenti mirati alla riduzione dell'impatto ambientale implica adottare una diversa gestione generale degli impianti, che deve necessariamente partire già dalla fase di progettazione nel caso di impianti di nuova generazione. Bisogna spingersi verso l'adozione di tecnologie all'avanguardia, con elevate efficienze e bassi consumi energetici, cercando di impiegare per buona parte energia prodotta sfruttando fonti rinnovabili.

Tra le varie tipologie di rifiuti prodotti nel mondo, una di quelle che negli ultimi decenni ha attratto maggiormente l'interesse di scienziati e imprenditori è la frazione di rifiuti di origine agro-industriale. Trattasi in generale di matrici vegetali residue da processi di lavorazione che puntano alla valorizzazione di prodotti che sono inevitabilmente connessi ad esse. Un esempio sono la sansa e le foglie di ulivo che risultano essere i principali scarti correlati alla produzione dell'olio di oliva. Nonostante questi siano classificabili come sottoprodotti, questo non significa che non possano essere sfruttati ulteriormente: basti pensare all'olio di sansa, ormai diffuso sul mercato da diversi anni, che viene estratto utilizzando esano dalla sansa residua del processo di macinazione delle olive.

Come si può facilmente intuire pensando alla varietà di prodotti di origine vegetale che giornalmente arrivano sulle nostre tavole, i volumi in gioco relativamente agli scarti agroindustriali prodotti annualmente sono davvero notevoli. Purtroppo, nella maggior parte dei casi questi vengono ancora smaltiti come rifiuti, o eventualmente valorizzati bruciandoli per la produzione di energia termica, nonostante spesso rappresentino un fonte di composti che, intrappolati al loro interno, hanno un elevato valore aggiunto.

Tra di essi si annovera un ampio spettro di comporti polifenolici, lipidi, proteine e fibre vegetali, in grado di essere sfruttati per diverse applicazioni industriali che spaziano dal settore alimentare, il settore farmaceutico, cosmetico ma anche interessanti per il sempre più diffuso settore delle bioraffinerie. Di sottoprodotti agro-industriali con una concentrazione interessante di sostanze ad alto valore aggiunto non sono pochi: si annoverano le foglie di tè esauste ancora ricche di catechine, le foglie di ulivo ricche di oleuropeina, i raspi di vino ricchi di polifenoli e il *brewer's spent grain* ricco di proteine e fibre.

Il processo necessario per ottenere tali composti è generalmente un processo di estrazione solido liquido, dove si sfrutta la loro affinità chimica con specifici

solventi per permetterne la solubilizzazione in una corrente liquida che viene successivamente concentrata fino all'ottenimento del prodotto in polvere o comunque ad alta concentrazione.

La percezione comune circa gli impianti di estrazione può essere a primo impatto considerata più "*green*" se comparata con le tradizionali industrie chimiche e petrolchimiche. In realtà, l'impatto ambientale di questa tipologia di impianti dipende sensibilmente da numerosi parametri, correlati alle modalità con cui viene effettuato il processo di estrazione, al tipo di solvente utilizzato, alla provenienza delle materie prime e a come vengono in seguito gestiti gli scarti prodotti.

Negli ultimi anni, a causa dell'incremento del bacino di utenze di un mercato sempre più interessato a prodotti di origine naturale, si è registrato un incremento dell'impatto ambientale ad essi correlato. Infatti, nei processi di estrazione condotti in maniera tradizionale sono generalmente necessari grandi volumi di solvente, ottenendo rese di estrazione per singolo stadio che spesso sono nell'ordine delle unità. Per riuscire a migliorare in questo campo risulta quindi sempre più necessaria una stretta collaborazione tra le diverse discipline scientifiche per ricercare e mettere a punto macchinari e metodi di estrazione più efficienti e dal ridotto impatto ambientale.

I parametri che influenzano prevalentemente la resa di un generico processo estrattivo sono essenzialmente quattro: l'affinità tra soluto e solvente, la temperatura, la superficie di contatto tra le fasi e il coefficiente di trasporto di massa. Tra le varie tecnologie, che mirano ad incrementare uno o più di questi parametri, proposte e studiate negli ultimi si annoverano tecnologie estrattive ad ultrasuoni, ad alta pressione, con fluidi supercritici, che sfruttano le microonde o anche assistiti da fenomeni di cavitazione controllata.

Tra tutte, per lo sviluppo di questo progetto di tesi, l'attenzione ricade verso un macchinario che intensifica la resa del processo cercando di massimizzare tutti o quasi i parametri che entrano in gioco nel processo di estrazione: l'estrattore cavitazionale idrodinamico TURBEX. Il macchinario è in grado di indurre al suo interno fenomeni di cavitazione controllata, generata da cavitatori idrodinamici di tipo "rotore-statore" dove l'elemento rotante permette al fluido di raggiungere velocità tali da generare fenomeni di cavitazione in una regione confinata a più alta pressione.

Si definisce cavitazione il fenomeno di transizione di fase liquido-vapore causata, in condizioni isoterme, dall'abbassamento della pressione sino al valore della tensione di vapore del liquido. Durante il processo, le bolle di vapore che si formano collassano in un tempo dell'ordine dei microsecondi, generando grandi incrementi locali di temperatura e pressione.

L'estrattore cavitazionale TURBEX, grazie ai suoi organi cavitatori, è in grado di impartire al liquido alti sforzi di taglio direzionati, che generando dei micro-getti ad altissima velocità e causano l'implosione asimmetrica delle bolle formatesi durante il processo di cavitazione. Inoltre, l'implosione delle bolle e i micro-getti di liquido ad alta velocità causano fenomeni di rottura delle pareti cellulari della matrice trattata, favorendo così il rilascio dei soluti di interesse nella corrente di estraente, causando la generazione di nuova area interfacciale e aumentando la porosità del solido.

Il regime fluidodinamico all'interno dell'estrattore è quindi fortemente turbolento, cosa che incrementa notevolmente i coefficienti di trasporto di materia solido-liquido rispetto ad altri estrattori. Il TURBEX è in grado di realizzare un mescolamento a livello microscopico tra le due fasi, massimizzandone il contatto, evitando la formazione di agglomerati solidi e migliorando così l'accessibilità dell'estraente all'interno della matrice solida.

Altro importantissimo vantaggio della tecnologia che sta alle spalle del TURBEX è che il macchinario permette l'alimentazione in continuo del solido e del liquido in controcorrente. Questo garantisce, alternando sezioni in regime cavitazionale a sezioni in regime di grande mescolamento, la possibilità di performare in un singolo passaggio più stadi di equilibrio e con un tempo di permanenza di qualche secondo, che è notevolmente inferiore rispetto alle ore necessarie per le tradizionali tecnologie.

La tecnologia del TURBEX promette di ottenere rese di estrazione superiori del 20-30% rispetto alle tecnologie tradizionali. Lo scopo di questa tesi è quello di dimostrare che tale risultato è effettivamente ottenibile, analizzando le performance di estrazione del prototipo TURBEX EX30, attualmente in fase di test. Ci si propone dunque di valutare non solo l'efficienza di estrazione, ma anche il numero di stadi di estrazione teorici che il macchinario è in grado di garantire con un singolo passaggio.

Durante il periodo di svolgimento della tesi, partecipando ad un tirocinio con Andritz è stato possibile prendere parte a diversi test funzionali effettuati sul prototipo nella sua fase di messa a punto. I test funzionali sono stati effettuati utilizzando acqua corrente come solvente e passiflora in taglio polvere e tisana. È stato dunque possibile collezionare due campioni di estratto, uno per ciascuna matrice testata, e dei campioni di matrice in taglio tisana prima e in seguito all'estrazione.

I risultati sperimentali ottenuti dall'analisi degli estratti e i parametri funzionali dell'estrattore ricavati, vengono infine utilizzati per il dimensionamento di due impianti di valorizzazione di due residui agro-industriali prodotti in grandi quantità a livello europeo e globale: le foglie di ulivo e il *brewer's spent grain*.

Le analisi sulle matrici solide vengono condotte presso i laboratori DISAT del Politecnico di Torino e hanno come obiettivo quello di calcolare la variazione di alcuni parametri della matrice in seguito all'estrazione. In particolare, viene calcolata la variazione di densità di cumulo, il contenuto di umidità, l'angolo di riposo statico e la distribuzione granulometrica.

Le analisi sui due estratti vengono invece condotte presso i laboratori di chimica dell'Università di Torino, con lo scopo di determinare l'efficienza di estrazione del TURBEX comparandola con un'estrazione di riferimento in singolo stadio.

- Variazione della densità di cumulo

Il calcolo della densità di cumulo dei campioni di matrice solida viene effettuato utilizzando dei piccoli recipienti in vetro graduati e una bilancia di laboratorio con risoluzione di 0,1 milligrammi. Vengono analizzati in totale quattro campioni di matrice solida, due per la matrice prima dell'estrazione e due per la matrice dopo l'estrazione. Come prima cosa sono stati pesati i recipienti da vuoti per segnarne la tara, poi sono stati riempiti fino ad un valore stabilito di volume e successivamente pesati di nuovo. Il peso finale epurato della tara permette di ottenere il quantitativo in grammi di matrice presente all'interno del recipiente. Il rapporto tra la massa del campione e il volume occupato restituisce la densità di cumulo. I risultati, elencati nella tabella seguente, hanno mostrato che in seguito all'estrazione si verifica una riduzione media della densità di cumulo pari a circa il 10%. Le pesature sono poi state effettuate nuovamente in seguito ad una lieve mescolata dei campioni, con lo scopo di simulare una parziale compattazione della matrice. Anche in questo caso i risultati mostrano una diminuzione della densità di cumulo di circa il 7,5%.

	SAMPLE A			SAMPLE B			AVERAGE
	Volume [mL]	*Weight [g]*	*Density [g/mL]*	*Volume [mL]*	*Weight [g]*	*Density [g/mL]*	*Density [g/mL]*
Raw tea cut	10	1,6468	0,1647	10	1,5885	0,1589	0,1618
Exhaust tea cut	10	1,4642	0,1464	10	1,4484	0,1448	0,1456
Raw tea cut A.S.	7,8	1,6468	0,2111	7,6	1,5885	0,2090	0,2101
Exhaust tea cut A.S.	7,4	1,4642	0,1979	7,6	1,4484	0,1906	0,1942

- Variazione del contenuto di umidità

La variazione del contenuto di umidità è stata valutata secondo una semplice analisi termogravimetrica. Sono stati analizzati quattro campioni di matrice solida, due per tipo che, in seguito a taratura, sono stati inseriti all'interno di quattro piccoli contenitori in vetro aperti, pesati e posti in forno a circa 110 °C per 12 ore. In seguito al fenomeno di essiccamento i campioni sono stati sottoposti ad un'ultima pesatura. Il peso epurato della tara riconsegna il valore del peso della matrice essiccata. Sottraendo tale valore al peso registrato per la matrice umida si ottiene, con buona approssimazione, il peso corrispondente alla massa di acqua evaporata. I risultati, elencati nella tabella successiva, mostrano che la matrice in seguito all'estrazione presenta un contenuto di acqua notevolmente inferiore rispetto alla matrice prima dell'estrazione.

	TARE *[g]*	$WEIGHT_{wet}$ *[g]*	$WEIGHT_{dryTOT}$ *[g]*	$H2O_{loss}$ *[g]*	MOISTURE *[%]*	AVERAGE *[%]*
Raw tea cut (A)	34,3800	0,9444	35,2275	0,0969	10,26%	10,14%
Raw tea cut (B)	33,4213	1,0577	34,3731	0,1059	10,01%	
Exh. Tea cut (A)	49,5679	1,0374	50,5772	0,0281	2,71%	2,70%
Exh. Tea cut (B)	62,3440	1,0142	63,3310	0,0272	2,68%	

- Variazione dell'angolo di riposo statico

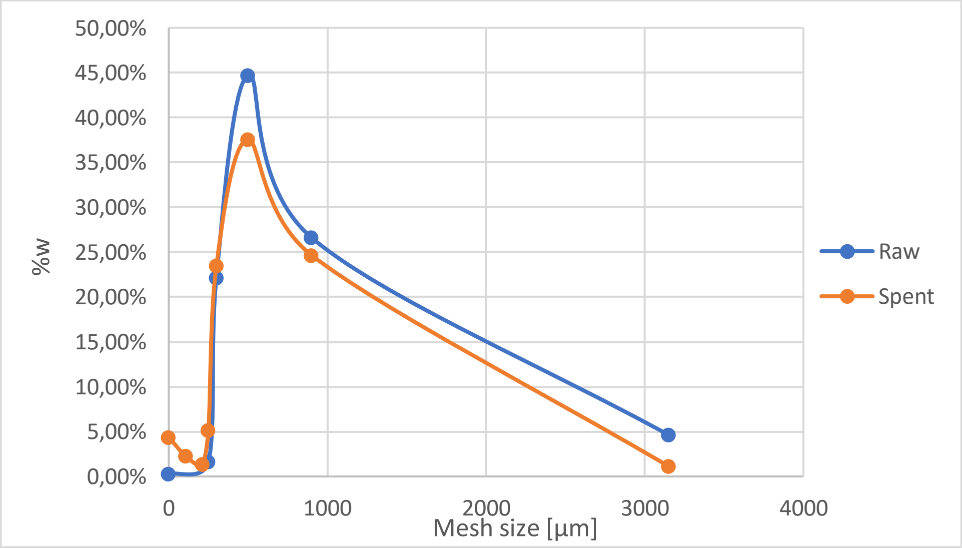
Per valutare l'angolo di riposo statico sono stati creati due cumuli costituiti dalle matrici analizzati. Un imbuto in vetro è stato fissato sulla cima di un sostegno verticale ad un'altezza fissata. Il solido, alimentato dall'estremità superiore, è stato lasciato cadere fino a formare un cumulo di materiale con altezza prossima allo sbocco dell'imbuto. Per ciascun cumulo sono poi state scattate delle fotografie nelle quali figurava anche un righello graduato per avere un termine di paragone delle misure prese. Tramite l'immagine, viene quindi stimata l'altezza del cumulo e la larghezza della circonferenza che questo forma con il piano su cui poggia. Si calcola dunque l'altezza e la base del triangolo che il cumulo forma sul piano con vista frontale e per mezzo di correlazioni trigonometriche vengono calcolate l'ipotenusa e l'angolo che restituisce una buona approssimazione dell'angolo di riposo statico. I risultati mostrano una diminuzione di circa 5° per il cumulo costituito dal solido post estrazione rispetto al cumulo di matrice fresca.

SAMPLES	H *[cm]*	B *[cm]*	HYP *[cm]*	θ *[°]*
Raw passionflower	5,7	4,77	7,43	50,08
Spent passionflower	3,81	3,79	5,37	45,17

- Variazione della distribuzione granulometrica

Per il calcolo della distribuzione granulometrica viene utilizzata una pila di sette setacci aventi luce pari a 3150 μm, 900 μm, 500 μm, 300 μm, 250 μm, 212 μm e 106 μm, impilati con luce crescente dal basso verso l'alto su di un piatto di raccolta di fondo. Ciascun setaccio, incluso il fondo, viene prima pesato per segnarne la tara e successivamente i campioni di solido vengono alimentati dall'alto della pila che viene agitata manualmente per circa cinque minuti. Al termine del periodo di agitazione della pila, i setacci vengono staccati e pesati singolarmente insieme al solido rimasto intrappolato in essi. I valori di peso registrati per ciascun piatto, epurati del peso della tara, rappresentano la quantità in massa di particelle che hanno un diametro medio compreso tra la luce del superiore e quello in cui si trovano. I risultati ottenuti, riportati di seguito sia in forma numerica che grafica, mostrano una riduzione della dimensione media delle particelle in seguito all'estrazione.

MATRIX	MESH *[μm]*	W_{matrix} *[g]*	[%]
Raw tea cut passionflower	0	0,0304	0,30%
	250	0,1674	1,63%
	300	2,2705	22,14%
	500	4,5831	44,68%
	900	2,7302	26,62%
	3150	0,475	4,63%
	W_{tot} [g]	10,26	
Spent tea cut passionflower	*0*	0,4392	4,39%
	106	0,2285	2,29%
	212	0,1353	1,35%
	250	0,5149	5,15%
	300	2,3468	23,47%
	500	3,7554	37,55%
	900	2,4661	24,66%
	3150	0,1138	1,14%
	Wtot [g]	10	



- Analisi degli estratti

Per analizzare l'efficienza di estrazione del TURBEX, per ciascuno dei due estratti raccolti in impianto, si effettua un processo di liofilizzazione con lo scopo di eliminare tutta l'acqua presente e ottenere un estratto solido in forma di polvere contenente prevalentemente le sostanze zuccherine estratte dalla passiflora durante i test funzionali. Inoltre, per avere dei risultati comparativi, le matrici solide in forma di polvere e taglio tisana, vengono sottoposte ad un'estrazione in singolo stadio all'interno di un serbatoio agitato. Per effettuare questi stadi di estrazione in batch, si utilizza lo stesso rapporto di liquido su solido alimentato che si è utilizzato durante i test con il TURBEX (10/1) e la matrice viene lasciata in estrazione per circa 24 ore. Al termine del periodo di estrazione, l'estratto viene filtrato e viene recuperato anche il liquido di imbibizione effettuando spremitura della matrice solida. Gli estratti e i liquidi di imbibizione vengono dunque sottoposti a liofilizzazione per valutare il contenuto di soluti che presenta la matrice e l'efficienza di estrazione di un singolo stadio di equilibrio operante nelle stesse condizioni in cui ha operato il TURBEX.

Nel caso dell'estratto di passiflora in polvere si ottiene che in 100 ml di estratto, avente un peso di 94,328 g, si recupera una quantità di estratto solido in forma di polvere pari a 1,732 g. Di seguito si riporta una foto dell'estratto ottenuto.

Per lo stadio comparativo effettuato con la polvere si preleva un campione di estratto di 100 ml che ha il peso di 96,473 g dal quale, in seguito a liofilizzazione, si recuperano 1,077 g di soluto. Da 100 ml di liquido di imbibizione, del peso di 93,768 g, si recuperano invece 1,903 g di soluto.

Nel caso dell'estratto ottenuto dalla passiflora in polvere, si trova che 100 ml di estratto, dal peso di 94,415 g, contengono 1,399 g di soluti. Invece, dallo stadio di equilibrio comparativo si trova che 100 ml di estratto, dal peso di 97,341 g, contengono 0,812 g di soluti, mentre 23 ml di liquido di imbibizione, dal peso di 21,786 g, contengono 0,387.

Inoltre, si effettuano anche delle prove di imbibizione della matrice per valutarne il rapporto tra il liquido assorbito e il solido secco. Questo parametro è di fondamentale importanza per la risoluzione dei bilanci di massa che sono poi stati impostati circa il calcolo del numero di stadi teorici del TURBEX EX30 Prototype.

Dai risultati delle analisi si riescono quindi a calcolare le concentrazioni di soluto nelle varie correnti di liquido analizzato. I risultati ottenuti sono consultabili in forma più immediata all'interno della tabella seguente.

	VOLUME *[mL]*	WEIGHT *[g]*	WEIGHT$_{DRY}$ *[g]*	SOLUTE CONC. *[g/mL]*
Turbex extract A	100	94,328	1,732	0,0173
Single stage extract A	100	96,473	1,077	0,0107
Retentate A	100	93,768	1,903	0,0190
Turbex extract B	100	95,415	1,399	0,0134
Single stage extract B	100	97,341	0,812	0,0081
Retentate B	23	21,786	0,387	0,0168

	WEIGHT$_{DRY}$ *[g]*	WEIGHT$_{WET}$ *[g]*	WEIGHT$_{H2O}$ *[g]*	L/S wetting ratio
Passionflower powder	100	374	274	2,74
Passionflower tea cut	209	762	553	2,65

Note le concentrazioni di soluto nei vari liquidi di interesse, risolvendo i bilanci di massa del processo di estrazione si riescono a calcolare la concentrazione massima di soluto presente nelle matrici solide che può essere solubilizzata nelle condizioni in cui si è effettuata l'estrazione, l'efficienza di estrazione del singolo stadio comparativo e del passaggio all'interno del TURBEX EX30. I risultati, schematizzati nella tabella seguente, mostrano che per l'estrazione della passiflora in polvere il singolo stadio di equilibrio ha un'efficienza di estrazione del 59,3%, mentre il TURBEX ha un'efficienza di estrazione del 97,5%. Nel caso della passiflora in taglio tisana il singolo stadio presenta un'efficienza di estrazione del 56,6% mentre il TURBEX registra un'efficienza di estrazione del 99,4%. Si ribadisce che questi valori di efficienza non sono relativi al contenuto totale di sostanze solubilizzabili all'interno della matrice, bensì al contenuto di sostanze solubili che riescono ad essere solubilizzate in equilibrio nel liquido alle condizioni operative nelle quali sono stati condotti i test.

	$\eta_{\text{single stage}}$	η_{TURBEX}	$\eta_{\text{increment}}$
Powder passionflower	59,3%	97,5%	+64,4%
Tea cut passionflower	56,6%	99,4%	+75,6%

Altro parametro molto importante che si ricava da questi dati è il valore della costante di equilibrio di solubilizzazione dei soluti nei due casi. Viene calcolata come il rapporto tra la concentrazione di soluto nell'estratto e quella nel liquido di imbibizione relativamente all'estrazione effettuata in singolo stadio *batch*. Si ottengono i valori di 0,55 e 0,47 rispettivamente per i soluti ottenuti dalla passiflora in polvere e da quella in taglio tisana. Per il calcolo del numero di stadi di equilibrio in controcorrente performati dal TURBEX, si stima che il valore di questa costante rimanga invariato in seguito a ciascuno stadio.

Impostando i bilanci di massa relativi allo scambio di massa tra solvente e soluto, viene effettuato un calcolo iterativo per riuscire a determinare il numero di stadi teorici che permettono di ottenere i risultati registrati dall'analisi degli estratti ottenuti. In particolare, si effettuano i calcoli ipotizzando il numero di stadi e calcolando così la concentrazione dei soluti nell'estratto in uscita e nel liquido di imbibizione del solido. Il calcolo viene reiterato aumentando il numero degli stadi ipotizzato sino a quando non si raggiunge un valore di concentrazione di soluti residui nel liquido di imbibizione che è uguale o inferiore rispetto a quello calcolato empiricamente. Così facendo si trova che, nel caso della passiflora in taglio tisana, il TURBEX è stato in grado di effettuare l'estrazione performando quanto 7 stadi di equilibrio in controcorrente, mentre nel caso della passiflora in taglio polvere quanto 15 stadi in controcorrente.

I risultati ottenuti risultano quindi in accordo con le previsioni: il TURBEX presenta delle efficienze di estrazione notevolmente superiori rispetto al singolo stadio tradizionale di estrazione, avendo l'efficienza equivalente a quella di almeno sette stadi di equilibrio in controcorrente, permettendo di effettuare il processo in tempi brevi e volumi contenuti. Anche i cambiamenti subiti dalla matrice solida in seguito al passaggio all'interno dell'estrattore sono in accordo con quanto atteso: la matrice solida dopo l'estrazione risulta avere una granulometria più fine e un contenuto di umidità minore rispetto al precedente, frutto della rottura delle membrane cellulari causata dagli elevati sforzi di taglio e dai fenomeni di cavitazione ai quali la matrice è andata incontro.

Alla luce di questi risultati, il lavoro di tesi trova completamento nella proposta di due impianti di valorizzazione di scarti industriali che vedano il TURBEX EX30 come estrattore cardine: un impianto di valorizzazione delle foglie di ulivo e un impianto di valorizzazione del *brewer's spent grain*.

- Impianto di valorizzazione delle foglie di ulivo

Il mercato mondiale dell'olio di oliva è abbastanza vasto da rendere il quantitativo di scarti agro-industriali ad esso associato molto interessante per un impianto di valorizzazione. Le foglie di ulivo vengono prodotte in grandi quantità durante il processo di potatura degli alberi di ulivo, ma anche durante il processo di raccolta e spremitura delle olive. Nonostante queste vengano generalmente smaltite come rifiuti o bruciate, le foglie di ulivo contengono quantitativi molto interessanti di polifenoli, in particolare di oleuropeina che trova impieghi all'interno di diversi settori dell'industria alimentare, farmaceutica, nutraceutica e cosmetica.

Viene dunque progettato un impianto in Turchia, nella citta di Içel, dove le coltivazioni di alberi d'ulivo per la produzione di olio sono molto diffuse.

L’impianto è in grado di processare 25.000 tonnellate l’anno di foglie, lavorando in un periodo di venti settimane l’anno in prossimità dei mesi della raccolta delle olive e della potatura degli alberi. Si divide in tre sezioni: pretrattamenti e stoccaggio, estrazione e concentrazione. Considerata l’elevata stagionalità della matrice prima da trattare, le tre sezioni dell’impianto lavorano in maniera indipendente tra di loro alternate da opportuni sistemi di stoccaggio.

La matrice in arrivo all’impianto viene dapprima essiccata, poi macinata, raffreddata e stoccata all’interno di sacche in poliestere poste in magazzino. Da lì viene in seguito preparata per essere alimentata inumidita alla batteria di estrattori cavitazionali TURBEX. Per effettuare una scelta più sostenibile, l’estrazione viene effettuata utilizzando solamente acqua come solvente, alla temperatura di 50 °C. Il TURBEX viene simulato, in maniera cautelativa, come un estrattore in grado di effettuare sette stadi di equilibrio in controcorrente.

Il solido esausto, in seguito a spremitura viene inviato ad un generatore combinato di energia termica ed elettrica, con lo scopo di ridurre l’impatto ambientale del suo smaltimento e i costi energetici associati all’impianto. L’estratto che contiene ancora una concentrazione molto bassa di polifenoli, viene inviato ad un evaporatore a ricompressione meccanica del vapore che concentra la soluzione fino al 30% in peso di soluti. L’essiccamento dell’estratto viene poi ultimato per mezzo di uno specifico essiccatore a tamburo rotante.

L’impianto porta dunque alla produzione di un estratto in forma di polvere con una capacità di circa 2,5 tonnellate anno e una concentrazione di oleuropeina pari al 33%. Il prodotto così ottenuto ha un valore commerciale di circa 20/25 euro al chilo. Le caratteristiche del prodotto sono elencate nella tabella di seguito, dove sotto la voce di sostanze estraibili si annoverano prevalentemente composti di tipo zuccherino

Oleuropein	32,90%
Extractable	65,10%
Water	2,00%
Unextractable	0,00%

- Impianto di valorizzazione del *brewer’s spent grain*

La produzione annua a livello globale di birra è si attesta su valori di 1,9 milioni di ettolitri l’anno, portandosi dietro una grande quantità di scarti di origine vegetali. Tra di essi vi è il *brewer’s spent grain* (BSG) che rappresenta tipicamente il residuo di orzo e altri cereali prodotto in seguito ai primi step del processo di produzione della birra. Il BSG è stato oggetto di diversi studi negli ultimi anni, che hanno dimostrato il suo interessante contenuto in sostanze quali proteine, polifenoli, lipidi e fibre. Visti i grandi volumi con i quali questo viene prodotto, risulta sicuramente interessante prevedere la progettazione di impianti che ne vedano un recupero.

Viene quindi progettato un impianto che ha sede in Siberia, nella provincia di Novosibirsk, nei pressi della quale sono presenti diverse birrerie in grado di fornire a prezzo vantaggioso il BSG. L'impianto tratta 20.000 tonnellate anno di BSG e mira alla produzione di quattro prodotti distinti: una frazione lipidica che può trovare diversi impieghi in varie sezioni della chimica, una frazione polifenolica, una frazione proteica e una frazione di fibre vegetali composta prevalentemente da arabinossilani che trovano invece impieghi in diversi settori tra cui l'industria alimentare, farmaceutica o cosmetica.

Vista la possibilità di reperire il BSG durante tutto l'anno, l'impianto opera per circa 49 settimane l'anno e si divide in quattro sezioni operanti in continuo: sezione di pretrattamenti e stoccaggio, estrazione dei lipidi e dei polifenoli, estrazione delle proteine e degli arabinossilani e sezione di recupero dell'etanolo e dell'acido citrico.

Nella prima sezione il BSG viene essiccato e stoccato all'interno di silos, per essere poi macinato e inviato alla prima sezione di estrazione. Lì, il BSG viene prima opportunamente inumidito e poi alimentato alla prima batteria di estrattori TURBEX operante con n-esano a 67°C come solvente. Sia l'estratto che il solido residuo vengono sottoposti a processi per il recupero dell'esano, ottenendo così la frazione lipidica con una portata di circa 45 tonnellate anno e un valore commerciale di circa 1 euro al chilogrammo, pronta per essere immessa nel mercato con le seguenti caratteristiche:

Water	2,262%
Polyphenols	6,075%
Lipids	91,659%
Hexane	0,004%

La successiva estrazione viene invece effettuata utilizzando una soluzione acquosa a 50 °C di etanolo al 60% in volume e porta alla separazione della frazione polifenolica ad alto contenuto di acido ferulico e *p*-cumarico. Il solido viene lavato con acqua per recuperare l'etanolo contenuto in esso ed inviato alla successiva sezione di estrazione. Dalla corrente di estratto viene prima separato l'etanolo e poi effettuata la disidratazione che porta all'ottenimento di un prodotto in forma di polvere con le caratteristiche elencate nella seguente tabella e una capacità di 93,6 tonnellate anno, con un valore commerciale di circa 50 euro al chilogrammo.

Water	2,00%
Cellulose	2,71%
Proteins	42,06%
Polyphenols	53,22%

Il solido, dopo le due estrazioni, è costituito prevalentemente da una matrice ligno-cellulosica, con un grande contenuto di proteine su base secca. Si effettuano dunque 4 estrazioni in serie: le prime tre sono estrazioni alcaline con soluzioni di idrossido di sodio a concentrazione crescente (0,1 M, 1 M e 5M) seguite da un'estrazione con acqua che ha lo scopo di estrarre altra frazione di arabinossilani e neutralizzare il solido prima inviarla ad un'unità di generazione combinata di energia termica ed elettrica, riducendo l'impatto ambientale associato al suo smaltimento e i costi energetici dell'impianto. Le correnti di estratto vengono prima acidificate per favorire la precipitazione delle proteine, poi nuovamente acidificate e diluite con una soluzione di acqua ed etanolo al 70% in volume per favorire la precipitazione degli arabinossilani evitando la precipitazione di citrati. Per decantazione vengono dunque ottenuti prima gli estratti diluiti contenenti le proteine e poi quelli contenuti gli arabinossilani, che vengono convogliati presso due serbatoio di reparto distinti per essere poi sottoposti a finale essiccamento con appositi essiccatori a tamburo rotante Anche la frazione di arabinossilani, previa concentrazione con sistema a membrane e concentrazione per evaporazione , viene convogliata nel serbatoio di reparto dell'estratto liquido contenente arabinossilani.

Si ottengono quindi i due prodotti finiti in forma di polvere. Il primo è rappresentato da una polvere ad elevata concentrazione di proteine prodotta con una capacità di 1245 tonnellate anno ed un valore commerciale di circa 2,5 euro al chilogrammo. Le caratteristiche sono elencate nella tabella di seguito:

Water	1,96%
Cellulose	0,68%
Hemicellulose	6,00%
Insoluble lignin	0,41%
Proteins	90,60%
Polyphenols	0,01%
Ash	0,32%

Il secondo è anch'esso un prodotto in forma di polvere avente elevata concentrazione di arabinossilani. Viene prodotto con una capacità di 1161 tonnellate l'anno e un valore commerciale di 0,10 euro al chilogrammo.

Water	1,92%
Cellulose	8,54%
Hemicellulose	83,10%
Insoluble lignin	5,63%
Proteins	0,60%
Polyphenols	0,08%
Ash	0,13%

In conclusione, lo sviluppo del progetto di tesi ha portato ad una prima fase di test del prototipo industriale dell'estrattore cavitazionale TURBEX. Tale studio ha permesso di avere una prima idea delle potenzialità del macchinario, confermando i risultati attesi e aprendo la strada a future valutazioni. È stato anche dimostrato che la macchina è in grado di essere inserita in maniera efficace all'interno di processi industriali, ottenendo eccellenti performance.

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Chapter 1

Introduction

Since its inception, mankind has always experienced a deep connection with plants, not only as a source of food, but also for their flavour, fragrance and colours. It is reasonable to assume that the beginning of the medicinal plants' usage was instinctive, as it is for animals (1). At the time, there was not enough knowledge neither to understand the reasons for the illnesses nor to understand which plant and how it could be utilized as a cure, everything was based on experience. The most ancient testimony about the use of herbs for drug preparations is a 5.000 years old Sumerian clay slab found at the archaeological site of Nagpur, western India. It contains 12 full recipes for drug preparation and over 250 references to various plants (2). Since then, mankind has never abandoned the use of plants for food or healing purposes.

There are many reported medical practices that have developed over the years by exploiting the "healing power" of leaves, fruits and seeds of some plants, used alone or in combination to create for example healing ointments for the healing of external wounds or preparations to be ingested to cure fever.

Even the use of medicinal plants exploited for their aroma has spread on a large scale: just think of the considerable number of aromatic herbs used nowadays in our kitchens or the alcoholic preparations typical of monks in medieval times. The leaves of Camellia Sinensis or Yerba Mate left to infuse in hot water for the preparation of tea and mate, the lemon peels left to macerate in alcohol for the preparation of Limoncello, are just some of the solid-liquid extraction practices widespread in all over the world for the extraction of aromatic molecules present in plant matrices.

Until modern times, the use of this or that plant was based only on empirical observations: over the centuries man has been able to experiment on his own skin, often with not exactly pleasant consequences, the effects related to use of different plant matrices, choosing them instinctively for their taste, aroma, texture or colour. Plants have therefore often been used in their entirety, or following extraction processes in various solvents, without knowing exactly which molecule could be responsible for the properties attributed to the plant.

Thanks to the development of technology, today there are many laboratory practices that allow us to analyse how many and which molecules are contained within a given substance, allowing us to isolate and study them and their effects on humans. For example, today we know that the antioxidant action of tea leaves is attributable to its catechin content or that the anti-inflammatory properties of the olive leaf infusion can be attributed to their high content of ferulic acid. Although many molecules can be designed to be synthesized, this can be much more expensive than the direct extraction from plant matrices, also bringing the benefit of being considered healthier by consumers.

The common perception about the extraction plants can at first glance be considered “greener” when compared with the traditional chemical and petrochemical industries, but actually the environmental impact of this type of plant depends significantly on numerous parameters, related to the methods used to perform the extraction process, the type of solvent used, the origin of the raw materials and how the waste produced is subsequently managed. In recent years, due to the increasingly number of consumers interested in the consumption of natural products, there has been an increase in the environmental impact related to the industrial extraction plants. In fact, to perform the extraction process in a traditional way, large volumes of solvent are generally required, also obtaining extraction yield for each stage that are often very low.

In most cases, the raw materials used for extraction are characterized by considerable concentrations of the compounds of interest and the plants that contain them are grown specifically for this purpose. Furthermore, due to the low extraction efficiency achievable with traditional extractors, the matrices cannot be exploited to their full potential. Usually only 50-60% of the solutes of interest are extracted, leading to the production of large volumes of matrix still containing discrete quantities of interesting compounds, but considered as a waste to be managed.

1.1. Agro-food industrial wastes valorization

In recent years, there has been an increasing attention, especially by companies and production chains, towards issues related to process intensification and reduction and enhancement of the produced wastes. In order to reduce the environmental impact of the production lines, it is necessary to move towards the adoption of cutting-edge technologies, with higher efficiencies and lower energy consumption, also trying to use a good part of the energy produced by exploiting renewable sources.

A fundamental role in reducing the environmental impact is assumed by the reduction of wastes. It is estimated that around 2.5 billion tons of waste are produced every year within the European Union. New regulations are still being developed to promote a shift to a more sustainable model known as circular economy: *a model of production and consumption which involves sharing, leasing, reusing, repairing, refurbishing and recycling existing materials and products as long as possible*, thus extending the life cycle of the products (3). The widespread application of this model implies reducing waste to a minimum, reusing products as much as possible and trying to enhance all the waste related to them, creating new added value.

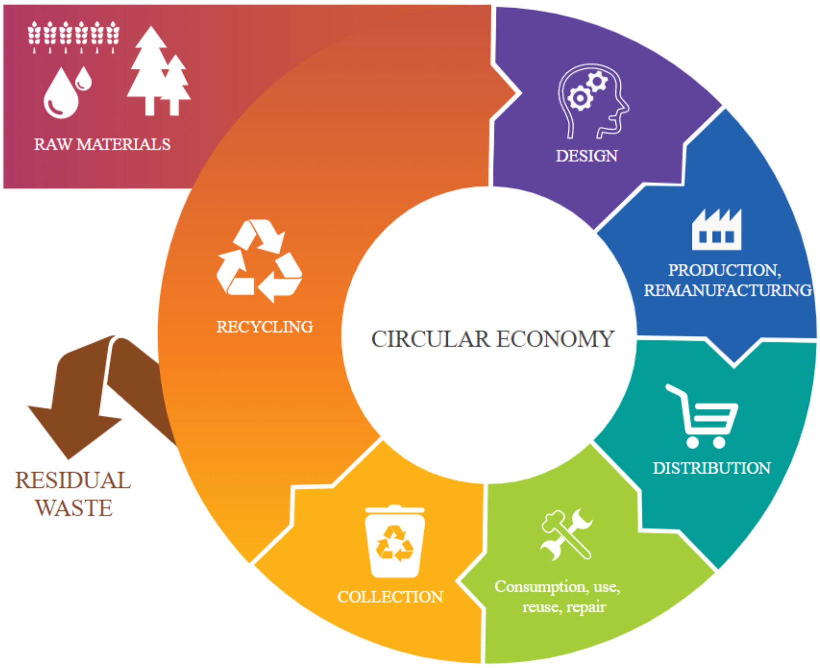


Figure 1.1 - Schematic representation of the circular economy model (3)

Among the various types of waste produced in the world, one of those that in recent decades has attracted the interest of scientists and entrepreneurs the most is the fraction of waste of agro-industrial origin. Due to the demographic increase, agricultural activities aimed at the production of food are constantly expanding. According to several reports, the process of food manufacturing is currently emission intensive, multifaceted and inefficient, characterized also by the loss of approximately 30-40% of all food produced for human consumption (4). In the countries of the European Union the increase in resource consumption, together with an inadequate waste management, causes the production of about 700 million tons of waste associated with the industrial agro-food chain (5).

Many of these wastes are made up of residues of vegetable matrices that cannot be immediately used in the human or animal food chain, but which contain a wide spectrum of substances with high added value, the extraction of which can lead to a substantial gain both in terms of economic and environmental terms. An example are pomace and olive leaves which are the main waste related to the production of olive oil. Although these are classifiable as by-products, this does not mean that they cannot be further exploited. For example, the olive pomace oil, now widespread on the market for several years, represents a well-known attempt to valorise the main residue obtained from the production of olive oil. It is obtained by extracting it from the olive pomace using hexane as solvent.

A growing awareness and attention to the potential of the reuse of agro-industrial residues has led in recent decades to conduct numerous researches on the subject. It has been pointed out that there are many plant residues that contain a fair amount of substances such as, for example, lipids, polyphenols, proteins or dietary fibers, that can be exploited for various industrial applications ranging from the food sector, the pharmaceutical and cosmetic sectors but also interesting for the increasingly widespread sector of biorefineries. There are not few agro-industrial by-products with an interesting concentration of high added value substances: there are exhausted tea leaves still rich in catechins, olive leaves rich in oleuropein, wine stalks rich in polyphenols and brewer's spent grain rich in protein and fiber.

Considering the potential of waste of this type, more and more efforts are made to be able to exploit these matrices as much as possible. Often however, considering their lower content of substances that can be exploited, traditional extraction technologies do not allow the development of processes that can be considered effectively profitable or with a reduced environmental impact.

1.2. Green chemistry for a “greener” world

The challenge to be able to obtain substances with high added value from waste it’s more and more widespread during the last years. The goal is to obtain these products using innovative technologies, which allow to reduce costs and the environmental impact of the process. With these goals, the concept of Green Chemistry has increasingly taken hold all over the world. Under the name of Green Chemistry are enclosed a series of guidelines that could help scientists and engineers to fine-tune more sustainable processes, maximizing yields and process intensification.

A branch of this discipline, increasingly widespread, is dedicated to the Green Extraction of Natural Products, defined as follows: “*Green Extraction is based on the discovery and design of extraction processes which will reduce energy consumption, allows use of alternative solvents and renewable natural products, and ensure a safe and high quality extract/product”* (6).

To provide a clear direction towards the Green Extraction of Natural Products, six fundamental principles have been defined to be understood not as absolute rules, but as practical advice developed by scientists and subsequently applicable in industry (6).

1. *Innovation by selection of varieties and use of renewable plant resources.*
2. *Use of alternative solvents and principally water or agro-solvents.*
3. *Reduce energy consumption by energy recovery and using innovative technologies.*
4. *Production of co-products instead of waste to include the bio- and agro-refining industry.*
5. *Reduce unit operations and favour safe, robust and controlled processes.*
6. *Aim for a non-denatured and biodegradable extract without contaminants.*

References are often made to more efficient technologies and the use of alternative solvents. These two concepts are closely related to each other: the efficiencies of single traditional extraction processes are already low enough using traditional solvents, their replacement with less efficient alternative solvents would lead to a further decrease in extraction efficiency and an increase in the volumes of liquids necessary to obtain the same extraction yield, possibly leading to a consequent increase in energy consumption. Therefore a highly efficient and selective extraction process, in compliance with the six principles of Green Extraction, must necessarily make use of process intensification technologies (7).

1.3. Process intensification techniques

The extraction of substances with high added value from vegetable matrices is very widespread in the world, especially for those substances with high commercial value that are used in sectors such as nutraceuticals and pharmaceuticals. The technology used to perform the solid-liquid extraction in most of the plants is very simple and consists in the use of percolation extractors or stirred vessels, which however are able to perform only a single equilibrium stage reaching at maximum extraction yields in the order of 50- 60%.

The extraction takes place under the effect of the driving force given by the solute concentration gradient between the extracting solution and the wetting solution containing the solutes. The extraction therefore goes on as long as there is a concentration gradient between the two phases, not allowing to go further. To overcome this limit, several extractors have been designed to guarantee the continuous feeding of the solid and the solvent in countercurrent to keep the solute concentration gradient between the two phases high during the whole process. Despite this, all these systems are characterized by long extraction times with large solvent hold-up times, not allowing to bring the fed matrix to exhaustion in a reasonable time.

In recent years, numerous studies have been conducted with the aim of researching new and efficient process intensification techniques capable of improving the efficiency of solid-liquid extraction processes for the recovery of bioactive substances from plant matrices. The common aim was to develop processes able to favour the increase of the mass transfer coefficients, of the turbulence in the vicinity of the solid surface and the ability of the solid to release the solutes contained in it. Among the various phenomena that have been studied, the most promising ones seem to be those related to extraction assisted by cavitation or high shear stresses.

Cavitation is defined as the phenomenon of liquid-vapor phase transition caused, in isothermal conditions, by lowering the pressure up to the value of the vapor pressure of the liquid. During the process, the vapor bubbles that are formed collapse in a time of the order of microseconds, generating hot spots characterized by large local increases in temperature and pressure.

Cavitation is able to create condition for intense heat and mass transfer in solid-liquid systems: during the microbubbles collapse the diffusion boundary layer at solids becomes turbulent and the solids are crushed, enhancing the dissolution of solutes under diffusion control (8). During the collapse of the asymmetrical bubbles, violent micro-jets of liquid are generated, imposing mechanical stresses on the surface of the solids. Thanks to the great mechanical stresses to which the biomass solid particles are subjected, the cell walls tend to break, facilitating the release of the compounds contained within them. Furthermore, the increase in porosity related to this phenomenon also increases the ability of the solvent to penetrate the solid more easily.

Similarly, also the generation of high shear stresses within the solid liquid system allows to obtain advantages on the increase of the extraction yield. It was demonstrated that the use of high-shear mixers as the advantage to intensify chemical reaction processes that have good reaction rates, but relatively slow mass transfer rates (9). The use of a rotor rotating with very high tip velocity allows to obtain a considerable increase in turbulence near the solid particles, increasing the mass transfer coefficients, favouring the mixing between the phases, also breaking the solid particles increasing their specific surface available for solute exchanges between the phases.

Several intensified extractors have been designed over time, able to exploit the principle of cavitation generated by ultrasounds, hydrodynamic cavitation generated by rotor-stator groups or even extractors that exploit the high shear stresses. All these extractors, however, are still limited to being used on a laboratory scale and, although they all can reduce the overall extraction times, none of these is still able to perform more than one theoretical stage of equilibrium.

The first and only extractor in the world that allows to exploit in a combined way all the principles of process intensification previously described in a continuous countercurrent multistage configuration is the TURBEX extractor. The intellectual property rights of TURBEX technology as well as TURBEX equipment is protected by the international PCT patent application n. WO 2018/146647 A1 (10), as well as by its national/regional patent extensions family.

The machine is made with a multirotor-multistator configuration where one or more discs of suitable geometry alternate each other connected to a motor shaft which, rotating inside a confined chamber, generate high friction with the fluid with which they come into contact and therefore creating cavitation. Moreover, according to the invention at least one first high-turbulence mixing stage, realized with radial pin mounted on the rotor, and one stage of cavitation and high-shear-stress follow each other inside the machine (10).

The TURBEX extractor allows the counter-current feeding of liquid phase and solid phase, thus allowing to operate in a continuous multistage configuration where for each physical rotor-stator group there are more than one extraction stages. Representing a novel unique technology, the TURBEX extractor aims to be a disruptive technology characterized by high extraction efficiency, low energy consumption, reduced extraction times and the possibility of carrying out solvent-free extractions with low ratios between liquid and solid fed. The equipment has all the features to be used for Green Extractions processes, promising to reduce both operating costs and the environmental impacts of the process.

1.4. Purpose of the thesis

The main purpose of the thesis project was to test, during an internship with the Andritz company, the TURBEX EX30 Prototype extractor, the first prototype suitable for industrial-scale applications of the TURBEX hydrodynamic cavitator. Furthermore, the thesis aims to design two industrial plants with the aim of extracting bioactive compounds with high nutraceutical value from residues of the industrial agri-food chain, using the TURBEX EX30 as the main extractor.

During the period of development of the thesis, which lasted from October 2019 until August 2020, it was possible to follow together with Andritz the development of the TURBEX EX30 Prototype, gaining direct experience in the industrial field. Among the various functional tests to which the extractor was subjected during the commissioning phase, samples of extracts were collected, obtained by processing flowering tops of passionflower, in powder and tea cut, and tap water as the extracting solvent. Moreover, sample of the two raw solid matrixes where collected, together with samples of residual tea cut passionflower after the extraction process.

The extract samples were analysed, with the aim of obtaining the extraction yield of process and number of equilibrium stages performed, by the Department of Pharma Science and Technology (DSTF) of the University of Turin. The tea cut passionflower samples before and after the extraction were instead analysed, with the aim of determining the morphological variations undergone by the matrix during the process, in the laboratories of the Department of Applied Sciences and Technology (DISAT) of the Turin Polytechnic.

Based on the results obtained from the empirical analyses, it was possible to confirm the theoretical expectations on the operation of TURBEX EX30 and to determine the parameters necessary to design the two solid-liquid extraction plants operating continuously using the TURBEX EX30 as the extractor. It was developed a plant for the recovery of polyphenols from olive leaves and a plant for the recovery of lipids, polyphenols, proteins and dietary fibers from the brewer's spent grain.

Chapter 2

Description of the experimental activity

In carrying out the thesis project, it was possible to experience the first steps and the development of the TURBEX EX30 Prototype during a series of functional tests performed between December 2019 and August 2020. The TURBEX EX30 functional tests were performed using passionflower tops as feeding to the extractor in two different size: powder and tea cut. It was possible to collect samples of raw passionflower, obtained extract and spent (after extraction) passionflower which were subsequentially analysed to evaluate the extraction efficiency, the theoretical number of stages of the extractor and how the solid matrix is transformed during the extraction process.

The experimental activity was realized thanks to the support of the equipment and laboratory technicians of the DISAT laboratory of the Turin Polytechnic (POLITO) and the DSTF (Department of Pharma Science and Technology) laboratory of the Turin University (UniTO).

2.1. Characterization of the solid matrix

Raw and exhaust tea cut passionflower samples were delivered to the DISAT laboratory of the Turin Polytechnic. The exhausted passionflower sample, before being delivered, was subjected to drying in order to remove the imbibition solvent and delivery a dry solid matrix ready to be analysed. The main purpose of the analysis carried out in this laboratory was to evaluate how the morphology of the solid matrix fed is modified in the passage inside the TURBE EX30.

Considering that inside the extractor the matrices are subject to cavitation phenomena and high shear stress, a size reduction of the solid particles constituting the analysed matrixes is expected. This phenomenon can lead to an increase in the specific surface, improving also the contact between solids and liquid extractant. This result can be useful to partially justify the increase in extraction efficiency related to the machine. To prove this, compatibly with the availability of the DISAT laboratory, the variation of four physical characteristics of the tea cut passionflower were evaluated: bulk density, moisture content, static angle of repose and particle size distribution. The instrumentation and methods used to evaluate these quantities will be described below together with the results obtained.

Typically, property evaluations in laboratory tests are carried out on as many samples as necessary to consider the statistical variability associated with them, in order to increase the precision of the results. Being not interested in precise estimations of the properties, but mainly interested in evaluating the differences between the properties before and after the passage inside the extractor, for all the tests it was decide to do not measure the various properties a high number of times, using only a couple of samples per solid matrix.

The various tests conducted will be described, together with the methodologies used and the results obtained, in the next paragraphs.

2.1.1 Bulk density

Bulk density, also called volumetric or apparent density, is a typical property of powder, granules or generally of the masses of corpuscular or particulate matter. It is evaluated as the mass of many particles divided by the total volume they occupy, considering that the total volume includes particle volume, inter-particle void volume and internal pore volume. (11) For our purpose, the analysis can be interesting to evaluate if particle shape and internal pore volume vary significantly because of the extraction process.

Conducting density measurements is quite simple and does not require complex equipment. The necessary equipment consists of a graduated beaker and a precision balance. Once the mass of the graduated container is known, it is possible to fill it with the matrix to be analysed up to a predetermined volume value, indicated by one of the volume notches of the beaker. The accuracy of the result obtained depends to a large extent on the accuracy of the person conducting the tests, for this reason the measurements must be carried out with due attention. At the DISAT laboratory, the weighing was done using a laboratory balance with a sensitivity of 0,001 grams. The step-by-step procedure was:

1. Small graduated glass beakers were selected, one for each sample to be analysed. Each of them was marked with an identification number and subsequently weighed. The weights of each beaker were then noted.
2. Each beaker was delicately filled with the passionflower samples to be analysed until the 10 ml volume mark was reached.
3. The beakers were weighted again, and the weights of each sample were noted.
4. By simply subtracting the weight of the glass beaker from the final weight noted, it’s possible to find the weight of each sample occupying the defined volume. The bulk density it’s easily measured as the ratio between the weight of the passionflower sample and his occupied volume.
5. For each type of sample analysed, an average density between the results for the two samples was evaluated in order to have a single reference value.

6. Samples were slightly shaken to simulate a better densification, reducing the volume occupied by the samples. Hence, the new occupied volume value was measured by eye referring to the marks of the graduated beaker and the obtained values were noted.

The data obtained from the analysis are shown in Table 2.1, while Figure 2.1 shows a picture of the graduated beakers used during the measurements.

Table 2.1 - Bulk density data results

	SAMPLE A			**SAMPLE B**			**AVERAGE**
	Volume [mL]	***Weight [g]***	***Density [g/mL]***	***Volume [mL]***	***Weight [g]***	***Density [g/mL]***	***Density [g/mL]***
Raw tea cut	10	1,6468	0,1647	10	1,5885	0,1589	**0,1618**
Exhaust tea cut	10	1,4642	0,1464	10	1,4484	0,1448	**0,1456**
Raw tea cut A.S.	7,8	1,6468	0,2111	7,6	1,5885	0,2090	**0,2101**
Exhaust tea cut A.S.	7,4	1,4642	0,1979	7,6	1,4484	0,1906	**0,1942**

Comparing the measured average bulk density, it's clear that after the extraction the bulk density of the solid matrix is reduced. The relative reductions in both cases were evaluated as:

$$\varepsilon = \frac{\rho_{exh} - \rho_{raw}}{\rho_{raw}} = \begin{cases} not\ shaken & \dfrac{0{,}1456 - 0{,}1618}{0{,}1618} = -9{,}97\% \\ shaken & \dfrac{0{,}1942 - 0{,}2101}{0{,}2101} = -7{,}54\% \end{cases}$$

Results shows that the bulk density is reduced by 9,97% in the first case and by 7,54% after a slight densification. The density reduction can be explained considering what happens to the leaf during the extraction phenomenon inside the TURBEX EX30. One could expect that two main phenomena occur: the decrease in size of the passionflower particles due to cavitation and high shear stress phenomena that can break them forming smaller ones, and the removal of extractable substances by the extracting solvent.

The first phenomenon leads to an increase in bulk density: with the same mass, if the size of the solid particles constituting a heap is reduced, the volume of the inter-particles void tends to decrease and this leads to a smaller overall volume occupied by the solid matrix.

The second phenomenon is a bit more complicated to analyse. During the extraction inside the TURBEX EX30 obviously not only the size of the particles is reduced, but extractive phenomena occur which therefore lead the plant matrix to the loss of numerous soluble substances. Imagining the structure of plant matrices such as the tops of passionflower, we can surely say that these are largely formed by cellulose and hemicellulose, forming a fibrous network within which the above soluble molecules are variously distributed. We can assume that the fibrous structure of the plant material is changed only to a small extent, thus leading to a decrease in the specific density of the plant matrix.

Imagine we are following the path of a single and intact leaf of passionflower inside the extractor, ignoring any possible mechanical phenomenon that would lead to a breakage of the structure that composes it. We also assume that, following extraction, the particle is dried to neglect the phenomena of imbibition of the solvent. The volume of the particle, due to its characteristic structure, will have changed little, while its mass should be considerably reduced due to the extractive phenomena. The specific density of the single leaf will therefore be reduced. Moreover, the internal voids left by the solubilization of the extracted substances increase the internal pore volume of the matrix, effect that is added to the mass decrease of the leaf.

If this reasoning is true, considering the results obtained in the laboratory, we can say that between the two phenomena, in this case, the second prevails and this can be the reason that causes the bulk density of the solid to decrease after the extraction process.

2.1.2 Moisture content

The moisture content was measured using the thermo-gravimetric analysis method. The principle on which the method is based is to determine the weight loss of mass that occurs as the material is heated. The sample weight is taken prior to heating and again after reaching a steady-state mass after drying. The drying was carried out using a laboratory hoven, leaving the samples in the hoven at 100 °C for approximately 12 hours. The step-by-step procedure applied is as follows:

1. For each sample to analyse, it was selected a small cylindrical glass beaker that was weighted empty. The weight was noted and tared on the used laboratory balance.
2. Beakers were then filled with passionflower samples and weighted again so that it was possible to determine the weight of the wet sample to be analysed simply by subtracting the tare weight.
3. Samples were placed in the hoven in the late afternoon. The temperature has been set at 100°C and the hoven was closed.
4. The following morning, using suitable safety gloves, samples were collected from the hoven and allowed to cool.
5. As soon as the samples reached about room temperature, they were reweighed. The weight obtained was noted.

The difference between the initial and the final weight of the sample, without the tare, is equal to the weight of the water that was loss during the drying process. The moisture content was then evaluated as the ratio between the weight of removed water and the weight of the wet passionflower sample. Results are shown in Table

2.2. Results show that after the extraction process the moisture content in the tea cut passionflower is reduced by about 7,44%.

Table 2.2 - Results moisture determination

	TARE [g]	WEIGHT$_{wet}$ [g]	WEIGHT$_{dryTOT}$ [g]	H2O$_{loss}$ [g]	MOISTURE [%]	AVERAGE [%]
Raw tea cut (A)	34,3800	0,9444	35,2275	0,0969	10,26%	10,14%
Raw tea cut (B)	33,4213	1,0577	34,3731	0,1059	10,01%	
Exh. Tea cut (A)	49,5679	1,0374	50,5772	0,0281	2,71%	2,70%
Exh. Tea cut (B)	62,3440	1,0142	63,3310	0,0272	2,68%	

Figure 2.1 - Picture of the hoven used in the DISAT laboratory to perform the moisture content analysis. (Picture of Giuseppe Pipitone)

Within the plant matrices, water is found in three different forms:

- Free or imbibition water: water contained in the cell lumina held only by capillary forces, located in the macropores of the matrix. It usually represents the greatest contribution of water present within the plant matrix.
- Bond or saturation water: it’s bond to the molecular structure of the biomass via hydrogen bonds. It’s located in the micropores, mainly cellulose and hemicellulose.

- Vapour: it’s a part of free water, not bond with the molecular structure and present in the cell lumina. Its contribution is usually negligible compared to the others.

Leaves, as well as any other biomass, start to dry immediately from the moment they are cut down. First, free water evaporates from the largest pores of the biomass and only after it’s completely evaporated, the bond water begins to slowly evaporate until it reaches a dynamic balance with the outward moisture (*fibre saturation point*). After drying, the final moisture content depends on the equilibrium condition reached inside the hoven and surely the only water eventually present must be bond water. When the matrix is exposed again to the atmosphere, it starts to slowly accumulate water as free water until it reaches the equilibrium condition.

The moisture content of the plant matrix is intrinsically influenced by the exposed surface of the matrix analysed. One of the consequences of the cavitation phenomena that occur inside the TURBEX is that it creates surfaces on the processed solid particles. The increase in surfaces involves a better exchange of free water between the plant matrix and the atmosphere. Furthermore, the mechanical stress to which the matrix is subjected inside the extractor could lead to a modification of the molecular structure constituting the solid, favouring the breaking of the hydrogen bonds that keep the water bound. This would also lead to a decrease in the water bounded inside the solid.

It should also be considered that the thermo-gravimetric analysis method does not measure directly the moisture content of the sample, but it measures the mass difference between the wet and the dry matrix. During the drying process it’s not only water that it’s leaving the solid matrix, but also all the substance volatile under the drying conditions. These volatile substances loss contribution it’s expected to be higher for the raw passionflower samples, because it is possible that a part of them it’s extracted by the solvent during the extraction process to which the spent passionflower samples were subjected. The occurrence of this phenomenon means that the loss of mass recorded, without considering other factors, should be greater for the samples of raw material. The above appear to be in line with the results obtained from the experimental tests.

The sum of the two contributions could be the cause of the lower humidity recorded for the spent passionflower samples.

2.1.3 Static angle of repose

The static angle of repose is an important basic property of non-cohesive materials. It represents the maximum slope angle at which the material is at rest: above this slope angle, the material starts to flow; below this angle, the material is stable. (12) It depends on density, shape, surface roughness, surface area and coefficient of friction of the solid particles and it can vary from 25° for smooth spherical particles to 45° for rough angular particles. (13) The static angle of repose it's useful to design equipment for the processing of solid particles, such us solid particle conveyors or hoppers.

To measure the static angle of repose of raw and spent tea cut passionflower it was used the fixed funnel method: the material is poured inside a paper funnel placed at a fixed height and it is allowed to flow until the height of the cone approaches that of the funnel outlet. A picture that contains the heap of material formed and a meter is then taken. Measuring the base and the height of the heap, it is possible to determine the hypotenuse of the right triangle formed and then the static angle of repose is measured as the arc sin of the ratio between the height and the hypotenuse of the right triangle.

Table 2.3 - Static angle of repose data

SAMPLES	H *[cm]*	B *[cm]*	HYP *[cm]*	θ *[°]*
Raw passionflower	5,7	4,77	7,43	50,08
Spent passionflower	3,81	3,79	5,37	45,17

Not having the possibility to carry out the measurements directly in the laboratory, the measurements were taken remotely by processing the photographs taken in the laboratory using the measuring instrument of the Microsoft Visio software. From the photo on the other, with reference to the ruler included in the image, the measurement of the base of the heap is taken. From the horizontal photo, instead, the measurement of the height of the heap is taken, tracing the imaginary lines that continue the outer side of the heap and meet at the central peak. With H we mean the height of the heap, with B the base of the right triangle formed by the representation on the plane of the heap and with HYP its hypotenuse. The photographs used are shown at the end of the chapter.

The results obtained show a decrease in the static angle of rest following the extraction process, but very little can be said about the validity of the results: to obtain more accurate values, it is necessary to repeat the analyses on a greater number of samples, taking the measurements needed directly in the laboratory.

The angle of repose presents a big variability with the shape of the particles that make up the heap. However, nothing can be said about this, as it would be necessary to conduct an analysis under an optical microscope to see how the morphology of the solid particles varies following extraction. However, the static angle of repose also has an important dependence on the water content of solids constituting the heap: the presence of water can increase the attraction phenomena between the particles, leading to an increase in the static angle of repose. This statement is in agreement with what was recorded during the analysis of the water content of the matrices examined. The greater value of the static angle of repose for the non-extracted matrix could therefore be related to its greater moisture content.

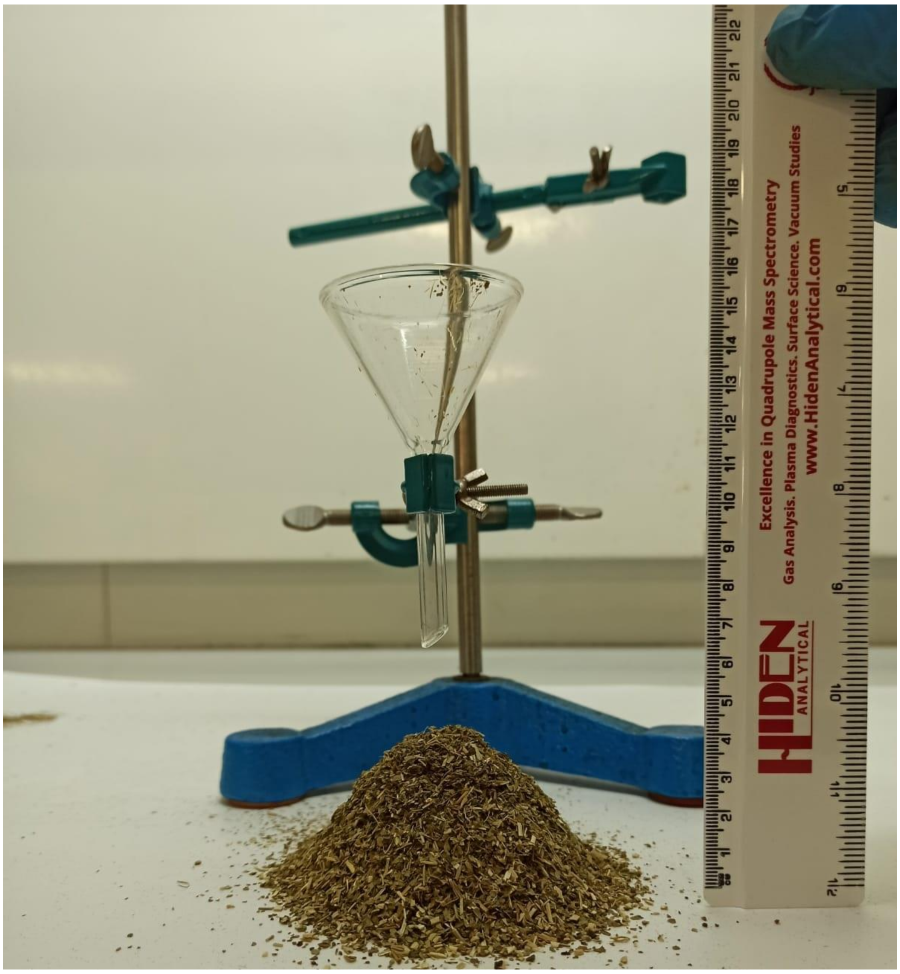


Figure 2.2 - Vertical picture of the raw passionflower heap. (Picture taken by Giuseppe Pipitone)

Figure 2.3 - View from above of the raw passionflower heap. (Picture taken by Giuseppe Pipitone)

Figure 2.4 - Vertical picture of the spent passionflower heap. (Picture taken by Giuseppe Pipitone)

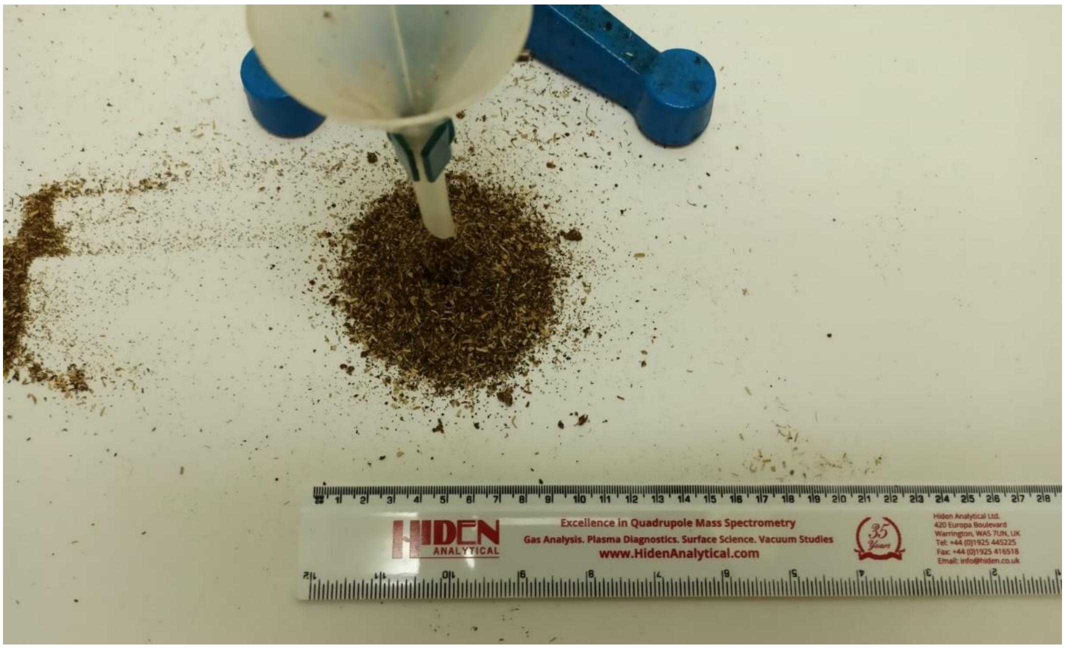


Figure 2.5 - View from above of the spent passionflower heap. (Picture taken by Giuseppe Pipitone)

2.1.4 Particle size distribution

Sieve analysis was used to determine the particle size distribution. The method consists in allowing a sample of solid particles to pass through a series of laboratory sieves progressively smaller mesh size and then weighing the amount of material that remain trapped in each sieve as a fraction of the whole mass. Sieves are displaced one upon the other to form a nested column of sieves, placing a round pan, called the receiver, at the base with the function of collecting the particles that have smaller size than the mesh size of the last sieve. Each sieve, including the receiver, is weighed as a first step of the analysis and the measured weight are noted.

A weighed sample of material is poured into the top sieve which has the largest mesh openings, then the sieve column is shacked until particles are well distributed throughout the column, reaching even the receiver if possible. Finally, proceeding from the top to the bottom, each sieve is removed from the column and weighed again. The recorded weight, net of the weight of the sieve itself, returns the weight of the portion of particles having a diameter greater than the opening of the sieve. Results are then noted and provided in graphical form.

Five sieves useful for this purpose were available at the DISAT laboratory, listed here in order of decreasing mesh size: 3150 µm, 900 µm, 500 µm, 300 µm, 250 µm, 212 µm, 106 µm. Figure XX show the sieve columns used to perform the sieve analysis. The analysis was performed for 10,26 g sample of raw tea cut passionflower and a 10 g sample of spent tea cut passionflower. The sieve column was shaken manually for approximately five minutes at the beginning and it was shaken again every time a sieve is removed, just to make sure that all visible material distributed on the top sieve is sufficiently sifted. If coarse particle agglomerates became visible, they were manually broken to avoid distorting the results of the analysis.

The obtained results are listed in numerical form in Table 2. and Table 2.5 and in graphical form in Figure 2.3.

Table 2.4 - Sieve analysis results fot raw tea cut passionflower.

MATRIX	MESH *[µm]*	W_{matrix} *[g]*	[%]
Raw tea cut passionflower	0	0,0304	0,30%
	250	0,1674	1,63%
	300	2,2705	22,14%
	500	4,5831	44,68%
	900	2,7302	26,62%
	3150	0,475	4,63%
	W_{tot} *[g]*	10,26	

Table 2.5 - Sieve analysis results for the spent tea cut passionflower.

MATRIX	MESH *[µm]*	W_{matrix} *[g]*	[%]
Spent tea cut passionflower	0	0,4392	4,39%
	106	0,2285	2,29%
	212	0,1353	1,35%
	250	0,5149	5,15%
	300	2,3468	23,47%
	500	3,7554	37,55%
	900	2,4661	24,66%
	3150	0,1138	1,14%
	W_{tot} [g]	10	

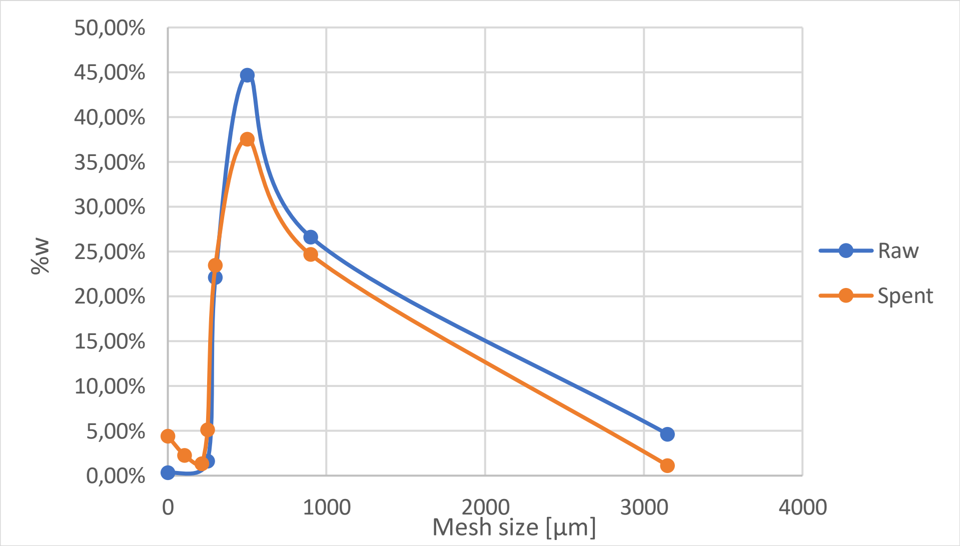


Figure 2.3 - Particle size distribution for raw and spent tea cut passionflower.

The results obtained confirm the expectations: the particle size of the solid fed to the TURBEX is reduced following the extraction process. In particular, a percentage decrease is noted already for the coarser dimensions, but with a more significant increase in particles below 106 microns. This could be due to the fact that the mechanical phenomena which the solid particles undergo lead both to the breaking in the bulk of the solid, but maybe in a larger part to the detachment of small particles from the outer surface of the particles, which is the most exposed. In both cases the prevailing fraction is that which has particle size between 900 and 500 microns.

Figure 2.6 - Picture of the sieves columns used to perform the sieve analysis. (Picture taken by Giuseppe Pipitone)

Figure 2.7 - Picture of the sieves before weighing (Picture taken by Giuseppe Pipitone)

2.2. Extract analysis

Analysis were performed to determine the extractive yield and the equivalent number of theoretical stages of TURBEX EX30 Prototype. The base case is related to the extraction of sugar-based solute from powder and tea cut passionflower using tap water as extraction solvent. Extract samples obtained after TURBEX extraction were compared with extract samples obtained in laboratory with a single stage equilibrium extraction. The main purpose of these analyses is to demonstrate that using the TURBEX extractor is possible to exceed the extraction efficiency that can be reached using a common single stage extraction operating in the same condition of temperature and feeding.

Two samples of liquid extract were collected during two different TURBEX EX30 commissioning functional tests carried out at the manufacturer workshop. The first extract sample (*sample A*) was collected at TURBEX liquid extract nozzle during February 21st functional test, when the machine was operating using powder cut passionflower as feed. The second extract sample (*sample B*) was collected during May 18th functional test, when the machine was operating using tea cut passionflower. Functional tests were both performed using tap water at 20 °C as extraction solvent with liquid over solid ratio (L/S) equal to 10/1 and with an estimated residence time of 80 s. The extract samples were stored at -18°C just after collection to maintain their property and avoid fermentation. Afterwards, together with sample of raw powder and tea cut passionflower, they were delivered to the UNITO laboratory to be analysed.

In the laboratory of the University of Turin UNITO, a freeze-drying process was used to evaluate the content of sugar-based solutes in the extract samples. Freeze-drying, also known as lyophilization or cryodesiccation, consist in a dehydration process in which the product is frozen lowering the pressure and then the ice is removed by sublimation. The process is typically used in the pharmaceutical and food industries, especially on biological materials, as it is a process that allows the removal of water minimizing the deterioration of the structure and components of the raw material to be dehydrated. The freeze-drying process is mainly divided in three stages: freezing stage, primary drying and secondary drying. (14)

During freezing stage, the material, distributed inside vials or metal trays, must be cooled below its triple point to assure that sublimation instead of melting will occur during the following steps. The rate of heat removal is a fundamental parameter: it influences the size of ice crystals that will be formed. Rapid cooling promotes the formation of numerous small intercellular crystals, making the sublimation process more complicated. Slow cooling promotes the formation of large crystals, easier to sublimate, but which could cause the cell walls of the material to break. A compromise must be found case-by-case according to the desired results.

The second stage is the primary drying: the pressure is lowered to the range of few millibars and enough eat is supplied by conduction or radiation to the material so that the ice can sublimate. This drying phase can remove up to 95% of the water contained in the material.

Finally, the secondary drying is carried out with the aim of removing the non-frozen water still present in the material. The temperature is raised and usually the pressure is decreased to enhance water desorption. When this stage is completed, the vacuum is broken with an inert gas, such as nitrogen. The final residue contains up to 1-2% of residual water.

Figure 2.8 - Extract sample for the extraction performed using powder passionflower as feed. Picture taken by Giuliano Cavaglià

2.2.1 Passionflower powder extract analysis

A 100 ml sample of powder passionflower liquid extract (*A*) was weighted to determine density. The weight of 100 ml of liquid extract resulted to be 94,328 g, thus showing to have a calculated density equal to 943,28 g/m^3. After, the liquid extract sample has been freeze-dried to remove the water contained and the remaining dry extract has been weighted: the weight of the recovered sugar-based dry extract is 1,732 g.

Figure 2.9 - Dry solid extract (after freeze drying). Picture taken by Giuliano Cavaglià.

Another liquid extract sample was collected during a comparative single stage test on a conventional extraction system. The mixing between 1 litre of tap water and 100 g of powder cut passionflower was performed in a cylindrical glass beaker, equipped with a 3-blade propeller type stirrer. Again, the (L/S) ratio was equal to 10/1 and tap water at 20°C was used as liquid extractant. The stirrer rotational velocity was of 25 rpm and the residence time was 12 hours. Finally, the exhaust solid residue has been separated with laboratory paper filter and the filtered liquid was collected.

As in the previous case, a 100 ml of liquid extract sample was weighted to determine the density. The weight of 100 ml of liquid extract resulted to be 96,473 g. After that the liquid extract sample has been freeze-dried and the remaining dry extract was weighted: the weight of the recovered dry extract is 1,077 g.

To evaluate the liquid/solid wetting ratio a third test was performed using 100 g of powder cut passionflower. The powder was completely wetted immersing it in tap water at 20°C. The wetted solid has been drained and lightly squeezed. The remaining wet solid was weighted: the weight of the wet solid was measured as 374 g. The measured liquid/solid wetting ratio is 2,74.

Another important test was carried out during week 13 (March 23rd - 27th), 2020. A conventional batch single stage was performed exactly as before, but this time the exhaust solid was squeezed to collect the soaking liquid. A 100 ml sample of this soaking liquid has been collected and weighted to determine density: the weight of 100 ml of imbibition (or soaking) liquid resulted to be 93,768 g. After that the sample has been freeze dried and the remaining sugar-based dry extract was weighted: the weight of the recovered dry extract is 1,903 g.

2.2.2 Passionflower tea cut extract analysis

A 100 sample of liquid tea cut passionflower extract (B) was weighted to determine density. The weight of 100 ml of liquid extract resulted to be 95,415 g, thus showing to have a calculated density equal to 954,1 g/m^3. After that, the liquid extract sample has been freeze-dried and the remaining dry extract was weighted: the weight of the recovered dry extract is 1,399 g.

Another liquid extract sample was collected during the comparative single stage test on a conventional extraction system. The mixing between 1 litre of tap water and 100 g of tea cut passionflower was performed in a cylindrical glass beaker, equipped with a 3-blade propeller type stirrer. Again, the (L/S) ratio was equal to 10/1 and tap water at 20°C was used as liquid extractant. The stirrer rotational velocity was of 25 rpm and the residence time was 12 hours. Finally, the exhaust solid residue has been separated with laboratory paper filter and the filtered liquid was collected.

As in the previous case, 100 ml of liquid extract sample was weighted to determine the density. The weight of 100 ml of liquid extract resulted to be 97,341 g. After that the liquid extract sample has been freeze dried and the remaining dry extract was weighted: the weight of the recovered dry extract is 0,812 g.

To evaluate the amount of extractable solute, the solid residue was squeezed with high pressure press recovering 23 ml of wetting liquid, with a weight of 21,786 g. The said liquid has been freeze-dried and the residual dry extract was weighted. The weight of the recovered dry extract is: 0,387 g.

This time, to evaluate the liquid/solid wetting ratio a sample of wet solid coming out from the TUERBEX EX30 squeezing press was weighted. The weight of the wet sample resulted to be 762 g. The sample was oven-dried to measure the weight of the dry solid, that resulted to be equal to 209 g. The weight of the wetting water was calculated simply by subtracting the dry weight of the solid matrix from the wet weight. The measured weight of the wetting water is 553 g. Finally, the liquid/solid wetting ratio was calculated as follows:

$$L/S_{wetting\ ratio} = \frac{553}{209} = 2{,}65\ g_{water}/g_{solid}$$

2.3. Olive Leaves wetting ratio

A very important parameter for the sizing of the olive leaf valorisation plant, part of this thesis project, is the value of water that the dried olive leaves can absorb. To achieve this result, post-pruned olive leaves were collected in Sicily and left to dry for three weeks under the sun until they reached the typical appearance and consistency of dry leaves. During the drying period, the leaves were manually stirred daily to ensure uniform drying.

The analyses were conducted in home environment, using what could be done to carry out the tests as precisely as possible. Once dried, the leaves were ground using a common tomato press, reaching few millimetres in size. Five samples of ground dry leaves were weighed and placed inside a beaker, into which water was added and weighed. The leaves were then left to soak in water for about 24 hours. At the end of the wetting period, the leaves were filtered with a cotton cloth and weighed. They were then manually squeezed to allow part of the excess liquid to escape and weighed again. The results of the analysis are shown in the Table 2.6. An average value is therefore calculated between the results obtained, taking 0,724 as the final liquid over solid wetting ration of the dry leaves.

Table 2.6 - Results of the liquid over solid wetting ratio of dry olive leaves.

Sample	1	2	3	4	5
$W_{\text{Dry Leaves}}$ [g]	5,0	5,0	5,0	5,0	5,0
W_{water} [g]	50,5	50,7	50,5	51,1	49,9
W_{TOT} [g]	55,5	55,7	55,5	56,1	54,9
$W_{\text{filtered leaves}}$ [g]	11,00	11,3	10,8	12	10,8
$W_{\text{squeezed leaves}}$ [g]	8,8	8,2	9	8,6	8,5
L/S wetting ratio	0,76	0,64	0,8	0,72	0,7

Figure 2.10 - Samples of olive leaves after wetting and before weighing.

Chapter 3

Processing of the experimental results

The empirical experiments, described in the previous chapter, allowed to obtain the data explained in the following table.

Table 3.1- Extract analysis results

	VOLUME *[mL]*	**WEIGHT** *[g]*	**WEIGHT$_{DRY}$** *[g]*	**SOLUTE CONC.** *[g/mL]*
Turbex extract A	100	94,328	1,732	0,0173
Single stage extract A	100	96,473	1,077	0,0107
Retentate A	100	93,768	1,903	0,0190
Turbex extract B	100	95,415	1,399	0,0134
Single stage extract B	100	97,341	0,812	0,0081
Retentate B	23	21,786	0,387	0,0168

	WEIGHT$_{DRY}$ *[g]*	**WEIGHT$_{WET}$** *[g]*	**WEIGHT$_{H2O}$** *[g]*	**L/S wetting ratio**
Passionflower powder	100	374	274	2,74
Passionflower tea cut	209	762	553	2,65

Thanks to the processing of these results, it is possible to easily calculate the quantities that characterize the extraction process with the TURBEX EX30 carried out for the two processed plant matrices. In particular, the logical and mathematical steps that lead to the evaluation of the extraction efficiency and the number of theoretical stages of the TURBEX will be explained in the following paragraphs of this chapter.

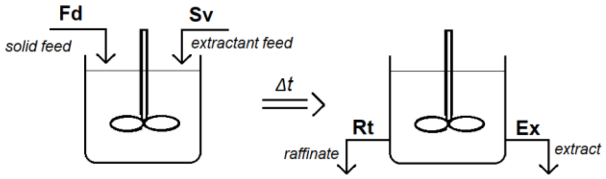
3.1 Schematic representation of extraction processes and terminology

Extraction processes, in a schematic way, take place through exchanges of matter between three distinct substances: a carrier A, a solute B and a solvent C. In an ideal leaching equilibrium stage, all the solute is dissolved by the solvent, leaving the carrier unchanged. (15) The process however is not always ideal and, depending on the case, it can be quite complex to understand. Generally, the process can be considered as constituted of three parts: first the change of phase of the solute as it dissolves in the solvent, secondly its diffusion through the solvent in the pores of the solid to the outside particle and thirdly the transfer of the solute from the solution in contact with the particles to the main bulk of the solution. (15) Anyone of this three process can be responsible for limiting the extraction rate, even if usually the first process is the fastest and has a negligible effect on the overall extraction rate.

For our purpose, it’s convenient to define a series of mass currents of interest and assign a symbolic reference to each of them. It follows, in Figure 3.1, a schematic representation of the currents involved for both a single stage and a counter-current multistage extraction.

- *Solid feed* (*Fd*): raw solid matrix fed to the process.
- *Inert solids* (*In*): solid phase constituted by the inert substances that do not take part in the extraction stage.
- *Solvent feed* (*Sv*): fresh liquid solvent fed to the extractor.
- *Extract* (*Ex*): liquid solution of solvent and extracted solute, constituting the main bulk of the solution. It can be considered as the desired product or as the extracting liquid if a subsequent counter-current extraction stage is performed.
- *Soaking Liquid* (*Sk*): liquid solution of solvent and extracted solute trapped inside the porosity of the solid from which the solute is extracted.
- *Raffinate* (*Rt*): outlet solid phase constituted by the sum of the inert solids plus the soaking liquid trapped inside the porosities of the solid.

Single stage equilibrium extraction



Counter-current multistage extraction

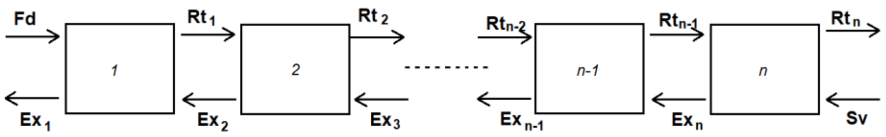


Figure 3.1 - Schematic of a single stage and a counter-current multistage leaching process.

We assume that all the solute that can be extracted in the set conditions of pressure and temperatures is dissolved inside the soaking liquid. Equilibrium occurs between the solute present in the soaking liquid and the solubilized solute in the extract. It is necessary to refer to the solid-liquid thermodynamic equilibrium equation, that describes the extraction phenomena in each stage. It can be written as follows:

$$y = k \cdot x$$

Where y means the solute concentration in the liquid extract, x means the solute concentration in the soaking liquid (*Sk*) present in the raffinate solids (*Rt*) and k is the equilibrium approach constant. From now on, the letter y will be used to represent the solute concentration in a liquid stream, usually in the extract streams (*Ex*), while the letter x will be used to represent the solute concentration in a solid stream or in the soaking liquid (*Sk*) carried by the solids. This equation is valid for each equilibrium stage, even if the value of the equilibrium approach constant may vary from stage to stage.

3.2 Extraction efficiency

To evaluate the extraction efficiency of a leaching process is necessary to know the solute concentration in the extract and the maximum amount of solute that is solubilized under the temperature and pressure conditions at which the process is performed. Thanks to the extract analysis described in Chapter 2, it’s possible to obtain the required data. In particular, the freeze-drying of the extracts and of the soaking liquids obtained with a single stage equilibrium extraction, make it possible to evaluate the total amount of solute present in the raw passionflower. The calculation, that will be explained below in the equations, will always report the value both for passionflower in tea cut (*TC*) and powder cut (*PC*).

Under the assumption that the liquid/solid wetting ratio will remain constant through the stages, it is possible to calculate the mass of the soaking liquid carried by the solids as the product between the mass of the solid fed and the liquid/solid wetting ratio:

$$y = Fd \cdot L/S_{wr} = \begin{cases} PC \rightarrow \quad 100 \cdot 2{,}74 = 274\ g \\ TC \rightarrow \quad 100 \cdot 2{,}65 = 265\ g \end{cases}$$

The liquid solvent used to perform the extractions was tap water at 20°C. Considering that the density of tap water at 20°C is approximately 1000 g/L, it is possible to calculate the total solute content in the initial solid matrix as follows:

$$Fd \cdot x_{Fd} = y_{Ex} \cdot (Sv - Sk) + x_{Rt} \cdot Sk = \begin{cases} PC \rightarrow & 13{,}66\ g \\ TC \rightarrow & 10{,}839\ g \end{cases}$$

Where the first term represents the amount of solute contained in the liquid extract stream, the second represents the amount of solute in the soaking liquid. This equation is true if we consider that solutes are completely solubilized in the liquid and no residue extractable solute is present in the solid matrix.

Considering that the amount of solute contained in extract is equal to:

$$y_{Ex} \cdot (Sv - Sk) = \begin{cases} PC \rightarrow & 0{,}011 \cdot (1000 - 274) = 8{,}104\ g \\ TC \rightarrow & 0{,}011 \cdot (1000 - 274) = 6{,}131\ g \end{cases}$$

The resulting extraction yield for the single conventional extraction stage is the ratio between the solute present in the extract and the solute in the raw passionflower:

$$\eta_{S.S.} = \frac{Ex \cdot y_{Ex}}{Fd \cdot x_{Fd}} = \begin{cases} PC \rightarrow & {}^{8{,}140}\!/_{13{,}66} = 59{,}3\% \\ TC \rightarrow & {}^{6{,}131}\!/_{10{,}839} = 56{,}6\% \end{cases}$$

The same calculations can be repeated using the data obtained from the analysis of the extract obtained from the TURBEX:

$$y_{Ex,1} = \frac{w_{dry\ solute}}{w_{extract}} = \begin{cases} PC \rightarrow & {}^{1{,}732}\!/_{94{,}328} = 0{,}0183 \\ TC \rightarrow & {}^{1{,}399}\!/_{95{,}415} = 0{,}01466 \end{cases}$$

$$\eta_{TX} = \frac{(Sv - Sk) \cdot y_{Ex}}{Fd \cdot x_{Fd}} = \begin{cases} PC \rightarrow & {}^{13{,}2858}\!/_{13{,}66} = 97{,}5\% \\ TC \rightarrow & {}^{10{,}7751}\!/_{10{,}839} = 99{,}4\% \end{cases}$$

Table 3.2 - Calculated extraction efficiencies and increment using TURBEX EX30

	$\eta_{\text{single stage}}$	η_{TURBEX}	$\eta_{\text{increment}}$
Powder passionflower	59,3%	97,5%	+64,4%
Tea cut passionflower	56,6%	99,4%	+75,6%

3.3 Number of theoretical stages

Since TURBEX EX30 is a counter current multistage extractor, it is necessary to refer to the thermodynamic equilibrium equation, that describes the extraction phenomena in each stage, in order to determine the equivalent number of equilibrium theoretical stages for a single passage through the machine. Using the solid-liquid equilibrium equation and knowing the solute concentration in the Liquid Extract stream y_{Ex}, the solute concentration in the imbibition liquid coming out with the exhaust solid stream and their respective flowrates, it is possible to evaluate the equilibrium approach constant as:

$$k = \frac{y_{Ex}}{x_{Rt}} = \begin{cases} PC \rightarrow & \dfrac{1{,}077/96{,}473}{1{,}903/93{,}768} = 0{,}55 \\ \\ TC \rightarrow & \dfrac{0{,}812/97{,}341}{0{,}387/21{,}786} = 0{,}47 \end{cases}$$

The equilibrium approach constant depends on many factors and it may vary from stage to stage, but for our purpose it's necessary to assume that it remains always constant. This is a reasonable assumption according solid-liquid extraction fundamentals, and the error resulting from this hypothesis is negligible. Now it is necessary to solve a mass balance for each stage constituting the system. To better understand the calculations done, it is convenient to consider the wet solid stream as its breakdown into single components:

$$Solid\ feed\ (wet) = Fd \cdot (1 + L/S_{wr}) = \begin{cases} PC \rightarrow & 374\ kg/h \\ \\ TC \rightarrow & 365\ kg/h \end{cases}$$

$$Inerts\ amount = Fd \cdot (1 - x_{Fd}) = \begin{cases} PC \rightarrow & 86{,}33\ kg/h \\ \\ TC \rightarrow & 89{,}16\ kg/h \end{cases}$$

$$Solute\ amount = Fd \cdot x_{Fd} = \begin{cases} PC \rightarrow & 13{,}67\ kg/h \\ \\ TC \rightarrow & 10{,}84\ kg/h \end{cases}$$

$$Water\ amount = Fd \cdot L/S_{wr} = \begin{cases} PC \rightarrow & 274\ kg/h \\ \\ TC \rightarrow & 265\ kg/h \end{cases}$$

$$Total\ liquid = Fd \cdot (x_{Fd} + L/S_{wr}) = \begin{cases} PC \rightarrow & 287{,}67\ kg/h \\ \\ TC \rightarrow & 275{,}84\ kg/h \end{cases}$$

Table 3.3 - Wet solid feed components distribution

WET SOLID FEED *[kg/h]*	INERT	SOLUTE	WATER	TOT LIQ.	TOT
Powder passionflower	86,33	13,67	274	287,67	374
Tea cut passionflower	89,16	10,84	265	275,84	365

Knowing the extract flowrate and its solute concentration, the amount of extracted solute can be easily calculated as the product between the two:

$$Ex_1 \cdot y_{Ex,1} = \begin{cases} PC \rightarrow & (1000 - 274) \cdot 0{,}0183 = 13{,}33\ g \\ TC \rightarrow & (1000 - 265) \cdot 0{,}01466 = 10{,}78\ g \end{cases}$$

It is possible to evaluate the amount of residual solute present in the exhaust solid coming out from the last stage of the TURBEX EX30 Prototype:

$$Sk \cdot x_{Rt} = Fd \cdot (1 - \eta_{TX}) = \begin{cases} PC \rightarrow & 0{,}335\ g \\ TC \rightarrow & 0{,}062\ g \end{cases}$$

Thus, it can be evaluated its concentration as:

$$x_{Rt} = \frac{Sk \cdot x_{Rt}}{Sk} = \begin{cases} PC \rightarrow & 0{,}0012 \\ TC \rightarrow & 0{,}00023 \end{cases}$$

Considering the liquid/solid wetting ratio as a constant, then the liquid extract flowrate (*Ex*) circulating in each stage can be considered constant and equal to:

$$Ex = Fd \cdot (L/S_{wr}) - Sk = \begin{cases} PC \rightarrow & 726\ kg/h \\ TC \rightarrow & 735\ kg/h \end{cases}$$

To evaluate the number of equivalent stages it's necessary to solve a mass balance for each stage starting from the first where the solid matrix is fed. In general, the mass balance for the stage "*n*" can be written as follows:

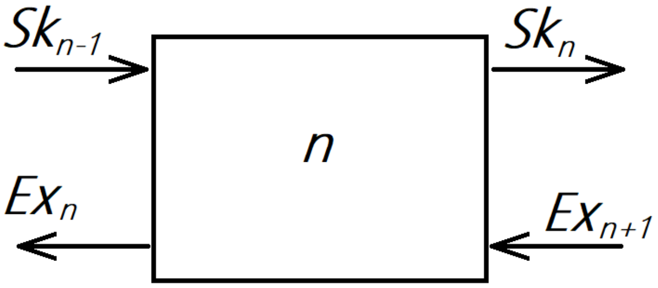


Figure 3.2 - Streams in a generic stage "n" for a counter-current multistage extractor

$$Ex \cdot y_{Ex,n+1} + Sk \cdot x_{Rt,n-1} = Ex \cdot y_{Ex,n} + Sk \cdot x_{Rt,n}$$

The solute concentration in the n+1 stage can be evaluated solving the previous equation:

$$y_{Ex,n+1} = \frac{Ex \cdot y_{Ex,n} + Sk \cdot x_{Rt,n} - Sk \cdot x_{Rt,n-1}}{Ex}$$

Using the equilibrium equation to correlate the solute concentration in the extract $y_{Ex,n}$ and the solute concentration in the soaking liquid $x_{Rt,n}$, it is necessary to proceed calculating all the concentrations involved in each stage. The calculation must go on until it's reached a value of $x_{Rt,n}$ that is equal or lower than the experimentally measured one. The hereinbelow tables portray stage-by-stage calculation results for both the solid matrix tested.

Table 3.4 - Results of the calculation of the number of theoretical stages performed by the TURBEX EX30. Case of powder cut passionflower

MATRIX	N° STAGE	$y_{Ex,n}$	$x_{Rt,n}$	$x_{Rt,n}$ exp.
Powder Passionflower	1	0,01836	0,03338	> 0,00122
	2	0,01214	0,02206	> 0,00122
	3	0,00786	0,01430	> 0,00122
	4	0,00493	0,00897	> 0,00122
	5	0,00292	0,00532	> 0,00122
	6	0,00154	0,00281	> 0,00122
	7	0,00060	0,00109	< 0,00122

Table 3.5 - Results of the calculation of the number of theoretical stages performed by the TURBEX EX30. Case of tea cut passionflower

MATRIX	N° STAGE	$y_{Ex,n}$	$x_{Rt,n}$	$x_{Rt,n}$ exp.
Tea cut Passionflower	1	0,01836	0,03122	> 0,00023
	2	0,01117	0,02379	> 0,00023
	3	0,00894	0,01809	> 0,00023
	4	0,00644	0,01371	> 0,00023
	5	0,00486	0,01035	> 0,00023
	6	0,00365	0,00776	> 0,00023
	7	0,00272	0,00578	> 0,00023
	8	0,00200	0,00426	> 0,00023
	9	0,00145	0,00309	> 0,00023
	10	0,00103	0,00219	> 0,00023
	11	0,00071	0,00151	> 0,00023
	12	0,00046	0,00098	> 0,00023
	13	0,00027	0,00057	> 0,00023
	14	0,00012	0,00026	> 0,00023
	15	0,00010	0,00002	< 0,00023

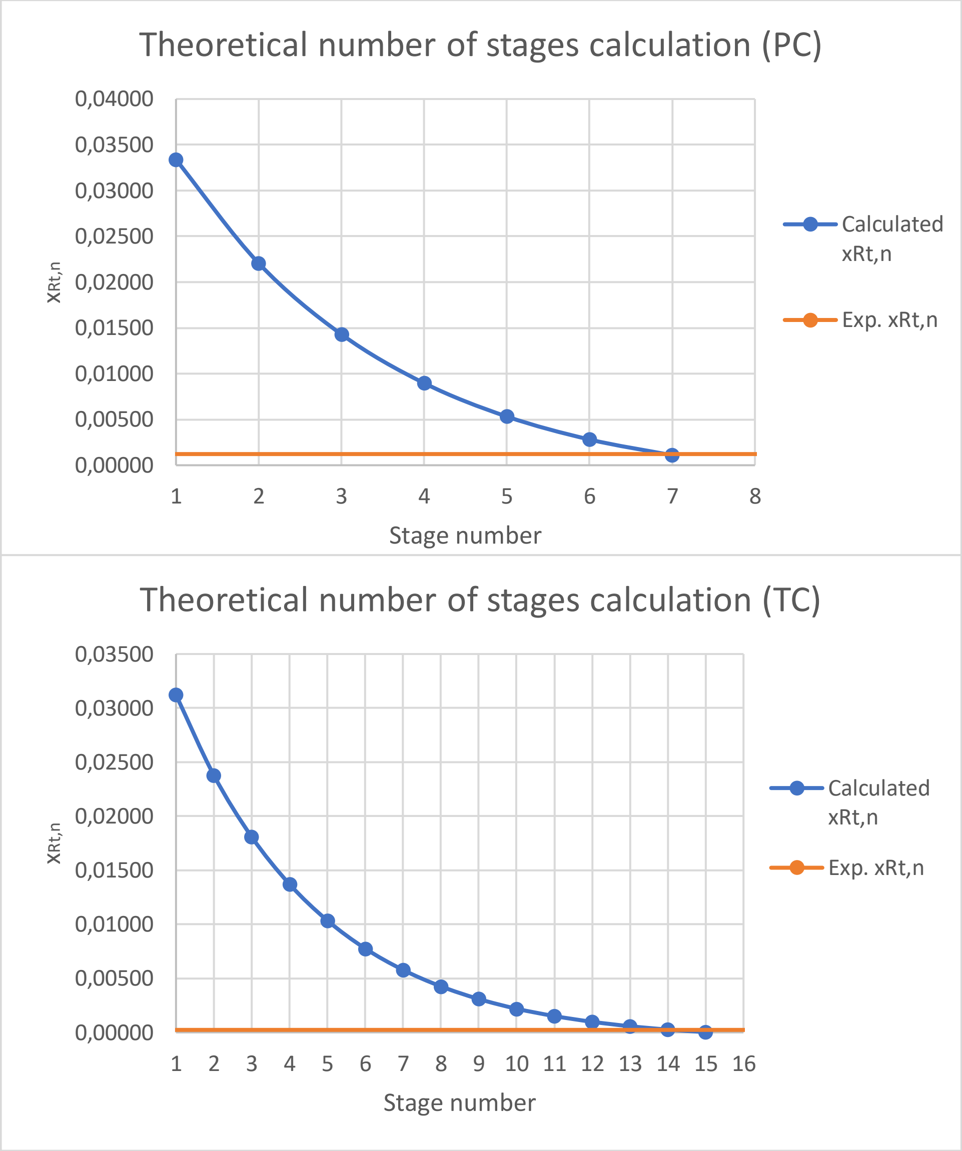


Figure 3.3 - Graphical representation of theoretical number of stage calculations

3.4 Discussion of the results

The results obtained during this experimental test and related calculation confirm that the TURBEX EX30 prototype extractor can perform the solid-liquid extraction with a higher efficiency compared to a conventional single stage extractor. Table 3.2 clearly shows that using the TURBEX there was a significant increase in the extraction efficiency: it increased by 64,4%, rising from 59,3% to 97,75%, in the case of powder passionflower, and it increased by 75,6%, growing from 56,6% to 99,4%, in the case of tea cut passionflower.

It’s necessary to underline again that the extraction efficiency is related to the maximum amount of solute that can be extracted in the operating condition of temperature and pressure. Considering that, in the cases analysed, the extracted solute is constituted by sugar-based substances, saying that with TURBEX EX30 it was experienced an extraction efficiency of 99,4% it doesn’t mean that the 99,4% of the sugar-based substances present in raw passionflower is extracted. Instead, it means that the 99,4% of the sugar-based substances dissolved in the soaking liquid was extracted. The amount of solute dissolved in the soaking liquids depends on the temperature and the pressure at which the process is conducted.

The most interesting results of this analysis are those concerning the calculation made to determine the number of theoretical equilibrium stages that it is possible to achieve using the TURBEX EX30 Prototype. Tables 3.4 and 3.5 show that using passionflower powder the machine performed 7 theoretical equilibrium stages, with tea cut passionflower it performed 15 theoretical equilibrium stages. Results are very promising: the machine has only three physical cavitation stages, but as far as we know at the present it can perform up to 15 stages of equilibrium, perhaps even more.

It must be considered that the tests were performed in conditions that could be different from the usual one: when the analysed extract samples were collected, the Prototype was still in final phase of commissioning, and these specific tests have been carried out to have a first ballpark idea of TURBEX performance. In fact, extraction tests were performed using raw passionflower and tap water at room conditions (20°C), whereby passionflower extraction is usually performed using water-ethanol (60/40) solution at 45-50°C.

The results obtained in this chapter represent the starting point on which the extraction simulations carried out for the sizing of the two plants described in chapters 5 and 6 will be based. In particular, the most useful data is the theoretical number of equilibrium stages that the TURBEX EX30 can perform. Considering that we are not yet aware of the effectiveness of TURBEX with solvents traditionally used by industry, as a precaution it will be assumed that this will be able to guarantee 7 stages of counter-current equilibrium. However, considering that it has proved to be very efficient using only water as a solvent, it is certainly able to obtain similar, if not better results even with traditional solvents such as hydroalcoholic solutions or hexane.

Chapter 4

TURBEX hydrodynamic cavitational extractor

The undisputed protagonist of this thesis project is the TURBEX cavitational extractor, which is tested in the version of the first prototype of this technology suitable for industrial use. To understand why this machine is so interesting, it is first necessary to understand the principles underlying the extraction processes, then investigating what the two phenomena that characterize it entail: cavitation and the high shear stresses to which the liquid and the solid matrix are subject in the passage inside the machinery.

The purpose of this chapter is precisely to investigate how the process intensification phenomena that led to the invention of TURBEX influence the extraction phenomena. The purpose of this chapter is precisely to investigate how the process intensification phenomena that led to the invention of TURBEX influence the extractive phenomena, correlating the theoretical assumptions with the results obtained from the experimental analyses described in the previous chapters.

Furthermore, some general details are also provided on the main characteristics of the machinery tested during the commissioning phase. The intellectual property rights of TURBEX technology as well as TURBEX equipment is protected by the international PCT patent application n. WO 2018/146647 A1 (10), as well as by its national/regional patent extensions family. Specific construction details of the rotor-stator series active parts are not disclosed on this thesis document since the IPR owner (i.e. BOB Service Srl) considers them as “proprietary know-how”.

4.1. Solid liquid extraction: principles and state of the art

The most common method for the extraction of bioactive components from natural matrices is certainly the liquid solid extraction, where the solute or solutes of interest are removed from the matrix that contains them thanks to the extracting power of a liquid solvent or a supercritical fluid.

The mass transfer between two immiscible phases, in the absence of chemical reactions, is that of the double layer. We can imagine that on both sides of the interface there are two very thin layers, named boundary layer, in which the mass transfer occurs only by diffusion. By moving from the surface of the solid particle towards the bulk of the solvent that surrounds it, the mass transfer is limited by the turbulence of the fluid.

The solid-liquid extraction process involves five fundamental phenomenological steps:

1. Diffusion of the solvent from the bulk of the solution to the surface of the solid;
2. Penetration of the solvent into the micro or macro porosity of the solid particles, forming a continuous soaking phase inside them;
3. Dissolution of the solute present in the liquid in the solvent contained within the strength of the solid;
4. Diffusion of the solute towards the surface of the solid, generated by the difference in concentration of the solute in the solution which is more concentrated inside the matrix than outside;
5. Diffusion of the solute through the boundary layer towards the bulk of the solution.

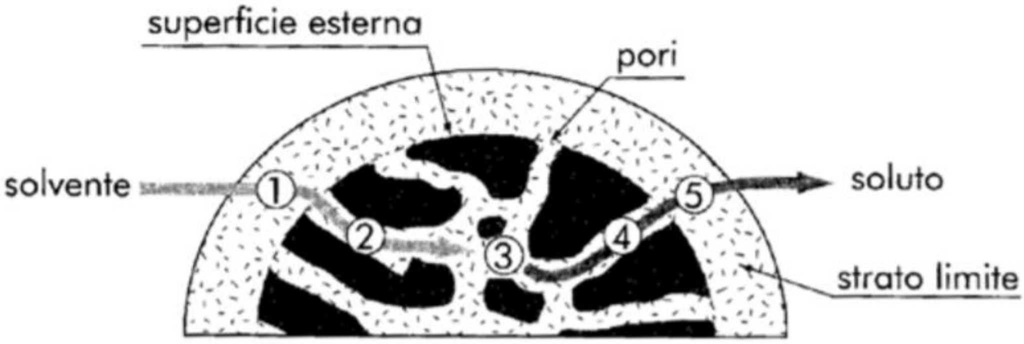


Figure 4.1 - Schematic representation of the five solid-liquid extraction steps. (16)

The diffusion mechanism, cornerstone of the extraction process, intrinsically depends on the thermal energy of the molecules in solution. Inside a stagnant fluid, the molecules due to their intrinsic thermal oscillation are subject to what are called Brownian motions: they tend to move randomly, colliding with each other and constantly changing their direction of motion. If in a certain volume the molecules are more concentrated, these will diffuse towards lower concentration areas until they are evenly distributed throughout all the available space. The phenomenon of this microscopic motion of the molecules towards an established direction with a lower concentration is called diffusion.

The diffusion mechanism is regulated by Fick's law, which can be expressed mathematically, by highlighting the diffusion speed of the molecules, with the following mathematical expression:

$$v = \frac{D \cdot A \cdot \Delta c}{x}$$

Where v indicates the diffusion rate of the solute molecules between two points of the solution, D is the diffusivity coefficient, A is the surface through which diffusion occurs, Δc is the difference in concentration between the two points under consideration placed at distance x. The diffusivity coefficient intrinsically depends on the chemical characteristics of the couple solute/solvent and on the temperature.

In general, the extraction ends as soon as the diffusion process of the solute towards the bulk of the solution ends, therefore when an equilibrium condition is reached between the solute concentration in the solution that imbibes the solid and that of the solution in the fluid bulk. The key parameter to perform the extraction efficiently is the diffusion speed of the solute molecules: in order to try to decrease the extraction times, it’s necessary to increase the diffusion speed of the solutes by trying to intervene on one or more of those five steps of the extraction process.

As can be guessed by looking at the mathematical expression of Fick's law, the parameter that, under the same thermodynamic conditions, most influences the speed of extraction is the realization of an intimate contact between the solid particles and the extracting fluid. Traditional extraction technologies, such as static or agitated maceration, try to improve this aspect by subjecting the matrix to be extracted to initial grinding. In fact, by reducing the matrix to powder, the surface of the solid directly in contact becomes much greater, but the grinding process, generally carried out with a hammer or blade mill, consumes as much energy as the smaller the particle size required.

However, traditional techniques have two limitations that cannot be further overcome: the impossibility of carrying out solvent-free extractions and of obtaining mass transfer coefficients beyond certain levels. This entails the need for longer residence times of the extraction phase, not less than one or two hours, with consequent not negligible hold-ups of solvents inside the equipment (7).

From the theoretical studies and experimental evaluations carried out in recent years by the Green Technologies Development Platform of the DSTF (Dipartimento di Scienza e Tecnologia del Farmaco of the University of Turin), it is shown that among the innovative and emerging process intensification technologies, the most promising for the extraction of micro-components with high nutraceutical value from vegetable matrixes is the "Cavitation Assisted Extraction" (7).

The TURBEX rotating hydrodynamic cavitator aims to improve the extraction efficiency trying to promote a more intimate contact between the phases involved, by exploiting the principles behind the cavitation and high-shear stress phenomena. How these two phenomena can influence the extraction process will be described in the next two paragraphs.

4.2. Cavitation

Cavitation is defined as the phenomenon of liquid-vapor phase transition caused, in isothermal conditions, by lowering the pressure up to the value of the vapor pressure of the liquid. During the process, the vapor bubbles that are formed collapse in a time of the order of microseconds, generating hot spots characterized by large local increases in temperature and pressure.

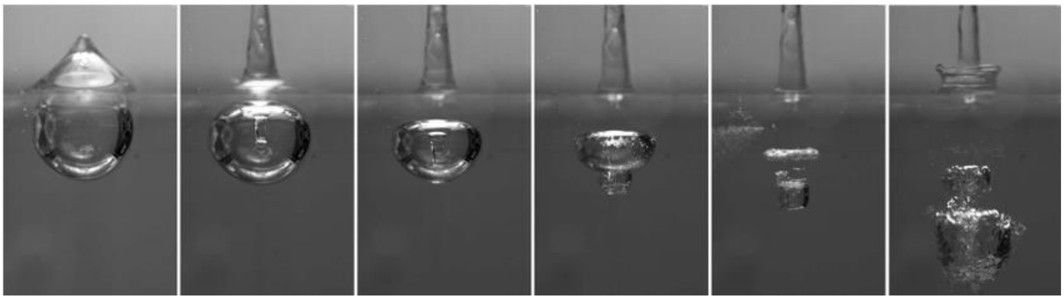


Figure 4.2 - The collapse and rebound of a laser-induced cavitation bubble collapsing near a free surface. (17)

Cavitation tends not to be well seen in the industrial world. In fact, it is usually a phenomenon to be prevented and avoided as it can cause mechanical deterioration of pipes or equipment: the energy released by the collapse of the bubbles is high and it dissipates greatly increasing the turbulence of the area in which the phenomenon occurs, creating high-speed micro-jets that impact against the solid walls of the devices. Fortunately, when uncontrolled cavitation phenomena occur these are easily recognizable due to the loud noise they generate. Among the devices that are most affected by the problem of cavitation are the control valves: to regulate the flow of the fluid that passes through them, the valves impart a pressure gradient which, if too marked or poorly designed, can lead to phenomena of cavitation which deteriorate the valve, considerably reducing its life time.

Despite the widespread distrust in the industrial world towards cavitation, the phenomenon can bring significant benefits in various applications: many studies on innovative cavitation assisted equipment have been done in recent years. When performed in a controlled way, in a confined and suitably designed area of the machinery, cavitation can be exploited as an advantage. Cavitation-based extractions can be divided in three main groups: ultrasound-assisted extraction (UAE), negative pressure cavitation (NPC) and hydrodynamic cavitation extraction (HCE) (18). In any case, the controlled cavitation mechanism can be generated through two distinct mechanisms: acoustic cavitation and hydrodynamic cavitation.

In the case of acoustic cavitation, the phenomenon occurs due to the passage of ultrasound waves that propagate in the fluid medium, causing the generation of micro-jets of the order of microns and of high frequency (between 20 and 40 thousand oscillations per second) (7). The propagation of an ultrasonic wave in a fluid generates a pressure oscillation with a sinusoidal trend, triggering the formation and oscillation of small gas filled cavitied, called microbubbles, in the fluid (19). In particular, the first part of the wave is of tension and leads to the formation of vapor bubbles with an average diameter ranging between 10 and 200 microns, while the following second part is of compression and lead to the collapse of the bubble (7).

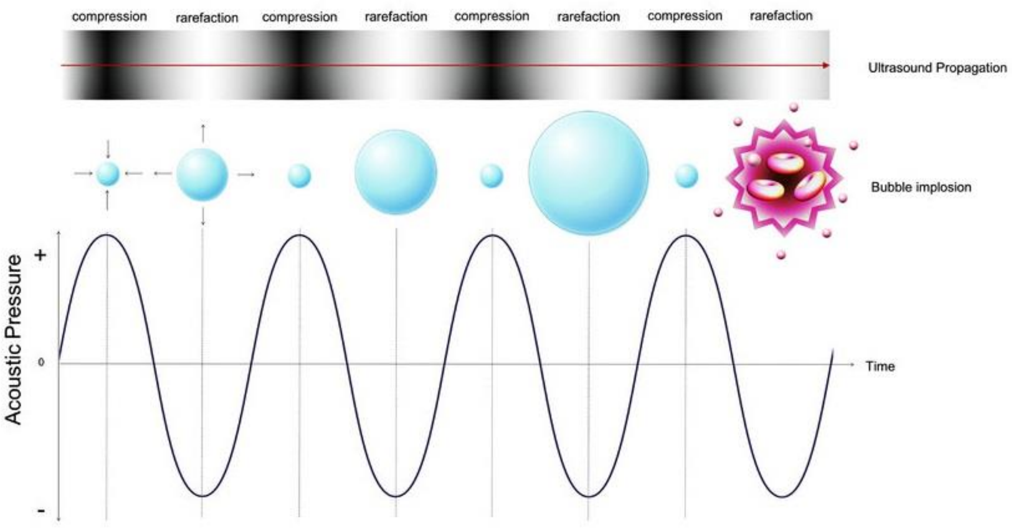


Figure 4.3 - Schematic rapresentation of phenomenon of compression and rarefaction at the base of cavitation in a liquid. (19)

In the case of hydrodynamic cavitation, this is generated by change in geometry, cavitational elements or cavitators of the rotor-stator type, in which the rotating element imposes a speed on the fluid to generate the cavitation process. In general, the fluid is passed at high speed within geometric constrictions of the machinery. When the fluid passes through the constrictions, the velocity of the liquid further increases at the expense of local pressure, letting the pressure around the point of vena contracta to fall below the vapor pressure of the medium at the operating pressure, resulting in the formation of cavities filled with gas (18). At the downstream of the restriction the liquid jests are subjected to expansion due to the pressure recovery, resulting in the collapse of cavities.

Although the cavitation can be generated in different ways, the chemical and physical principles behind the phenomenon of formation and collapse of microbubbles, as well as their consequences, are the same. Cavitation is able to create condition for intense heat and mass transfer in solid-liquid systems: during the microbubbles collapse the diffusion boundary layer at solids becomes turbulent and the solids are crushed, enhancing the dissolution of solutes under diffusion control (8).

The increase in extractive yield in cavitation assisted extractions can be largely attributed to the intensification of mass transfer and to the facilitation of solvent access to plant cells. If cavitation occurs in isolation within a liquid, the bubbles that form tend to have a spherical shape for the duration of the process until they collapse. However, the presence of solids in the vicinity of the bubble alters its expansion volume, so the bubble is unable to maintain spherical symmetry and must change its shape. During the collapse of the asymmetrical bubbles, violent micro-jets of liquid are generated, imposing mechanical stresses on the surface of the solids. Thanks to the great mechanical stresses to which the biomass solid particles are subjected, the cell walls tend to break, facilitating the release of the compounds contained within them. Furthermore, the increase in porosity related to this phenomenon also increases the ability of the solvent to penetrate the solid more easily.

All these effects are directly linked to the increase in the pressure and temperature of the liquid. In particular, according to the cavitation theory, during the phenomenon the temperature in the central part of the microbubbles increases and, at the moment of collapse, reaches the highest critical value (8). It is reported that the bubble core can reach pressure of the order of 1000 atmospheres and temperature of the order of 5000 °C (20), causing also the production of highly reactive radicals resulting from the dissociation of the molecules constituting the solvent able to enhance the extraction (18) (8). From the point of view of an external observer moving in solidarity with the micro bubbles, it is as if the system is in conditions of temperature and pressure much higher than those of the system average. Due to the small dimension of the microbubbles, the phenomenon of temperature and pressure increase is exclusively punctual and very fast, not affecting the average temperature and pressure of the system: the phenomenon of formation and collapse of micro bubbles is estimated to be around 450 microseconds and a generated heat dissipation in the order of 10 billion degrees per second (20).

4.3. High-shear mixing

Fluids undergoes shear stress when one area of fluid travels with a different velocity relative to an adjacent area. This phenomenon can be used for the realization of mixing devices called high-shear mixers: they use rotating impellers or high-speed rotors to let the fluid rotate with high velocity creating flow and shear.

High-shear mixers are devices typically used in different applications with the principal aim of dispersing a compound or a phase within a continuous liquid medium. They are characterized by high rotor tip speed, very high shear rates, highly localized energy dissipation rates and relatively higher power consumption compared to conventional mechanically stirred vessels (21). These characteristics are attributed to the centrifugal forces generates from the relative high-speed motion between the rotor and the stator equipped with narrow spacing. The velocity of the fluid at the outside diameter of the rotor, named tip velocity, is higher than the rotation velocity of the rotor.

High-shear mixers can also be used to perform chemical reactions to produce fine chemicals or intermediate. They have the advantage to intensify chemical reaction processes that have good reaction rates, but relatively slow mass transfer rates (9). This improvement is linked to the locally intense turbulence generated in the small shear gap with a short residence time (21): mass and heat transfer can be facilitated in multiphase reactors tanks to the large interphase areas provided by the high-shear mixing. Some companies are already owners of patents relating to fine chemical production processes using high-shear mixer reactors.

Their use in solid-liquid extraction processes has also been considered. There are several studies related to the extraction efficiency of the lipid fraction from algal biomass using high-shear extractors (22). These devices have a further advantage if used for the solid-liquid extraction of substances contained within vegetable matrices: the shear stresses they generate is applied not only to the liquid, but also to the solids rotating in the close proximity to the rotor. This cause the cell walls of the plant matrix to break, allowing easier infiltration of the solvent and the consequent solubilization of the substances trapped inside the cells.

A study was conducted by researchers from the Korea Advanced Institute of Science and Technology to investigate the extraction efficiency of lipids contained in samples of wet aurantiochytrium sp. KRS101 using a high-shear mixer as an extractor (22). To verify the effect of rupture of the cell walls, the scientists subjected to extraction several samples of algal matrix for an increasing time, and then they analysed the particle size distribution of every solid residue. The results obtained, shown in Figure 4.3, confirm a decrease in the solid particles with increasing residence time inside the mixer, confirming that the mechanical stresses to which the solid particles are subjected lead to its consequent breakage.

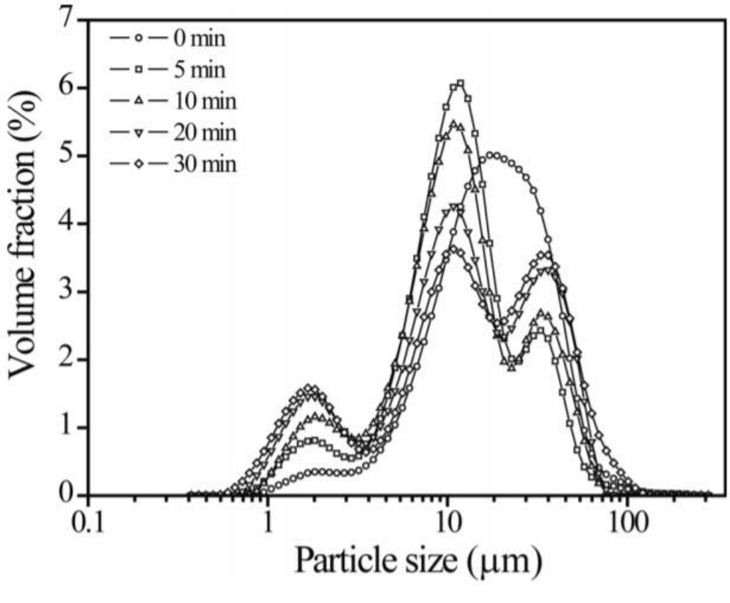


Figure 4.4 - Particle size distribution of Aurantiochytrium sp. KRS101with time course analysis (0 min: circle, 5 min: square, 10 min: triangle, 20 min: inverted triangle, 30 min: diamond)

Moreover, they investigated also the effectiveness of the high-shear mixers for the lipids extraction using various organic solvents, comparing the results to that of a conventional extraction stage performed with a conventional mixer. They found that the high-shear mixer yielded far better overall results with all the tested solvents (22).

The enormous advantage of this technology certainly lies in the ability of the high-shear mixers to perform mixing at a microscopic level, favouring the intimate contact between the phases involved, also reducing the particle size of the treated solid matrix. However, the real effectiveness of these machines is related to the phenomenon of cavitation: it is reported in a previous study that when the high-shear mixer operate at rotor speed higher than 8000 rpm, it gives rise to the cavitation phenomenon (23).

The combination of the high mixing and the destructive energy correlated to the collapse of the micro bubbles created by cavitation is therefore responsible for the high efficiency of these machines still mainly limited to use for research purposes in laboratories. Nonetheless, this technology has considerable applicability on an industrial scale, also because the scape-up of a high-shear mixer can realize all the described performances even operating with lower rpm: the value of the centrifugal force and shear stress are proportional to the rotor diameter (22).

4.4. Multistage solid-liquid extractions

To understand the concept of extraction theoretical stage, it’s necessary to start from the definition of the extraction equilibrium condition. As explained previously, the solid-liquid extraction process occurs due to the ability of a solute of interest to dissolve within the solvent with which it is placed in contact. The dissolution of the solute in the solvent takes place in the soaking solution, that is the continuous phase of solvent distributed within the porosities of the solid particle. The extraction process leads to the removal by diffusion of solute from the wetting solution towards the bulk of the solution, and it stops as soon as the solute concentration in the bulk and the solute concentration in the soaking solution reach the same value.

As soon as this condition is reached, the extraction process stops and the solid is separated from the liquid. The separation between solid and liquid is typically not perfect: a part of the liquid is inevitably trapped inside the porosity of the solid, wetting it completely, together with the portion of the solutes dissolved within it. From this condition comes the concept of extraction yield, defined as the ratio between the quantity of solutes contained in the solution and that of solutes contained within the wetting solution trapped inside the solid (named refined phase).

If it were possible to perfectly separate the solution from the solid following extraction, the yield of the process would be unitary. However, it is possible to separate the two phases and mix again the wet solid with fresh solvent. In this way a new extraction process will be triggered, leading to the extraction of further solute, until a new equilibrium condition is reached. This process can be repeated for a reasonable number of times until the desired extraction yield is reached.

The concept of stage refers to a portion of the solution which has reached the equilibrium condition, or which is very close to that. It can therefore be considered a purely theoretical concept for two reasons: the condition of equilibrium is not achievable in practice as it requires an infinite time and furthermore the concept of stage does not find a representation with a physical stage of extraction. Referring to the second statement, it is possible to carry out an extraction with a single operation which is equivalent to the yield of several equivalent equilibrium stages. So, even if the real physical stage is only one, it may be able to have the performance of more than one theoretical stage.

Based on these considerations, three extraction mechanisms are distinguished: single stage extraction, multistage cross-current extraction and multistage countercurrent extraction.

The single stage extraction consists of a simple extraction stage where the solvent is left in contact for a sufficiently long interval of time to get as close as possible to the equilibrium conditions. Then the solution is separated from the solid particles and the process is considered concluded. The process in industrial applications is generally carried out in batch extractors consisting of large static or agitated tanks which must be emptied and refilled at the end of each operation.

The cross-current extraction process consists in repeating several single-stage extraction processes in series, separating at the end of each stage the extract from the refined phase which is sent to the next extraction together with new fresh solvent. As in the previous case, even this extraction methodology is not well suited to be applied in continuous operations. With the succession of stages, the overall yield of the process increases, but the continuous decrease of the equilibrium concentration leads to an ever-lower extraction efficiency of the single stage, gradually making more and more stages necessary to significantly increase the yield. Moreover, the volumes of the solutions obtained tend to become quite high and the concentrations lower and lower, making the subsequent concentration process of the extracts much more onerous.

The counter-current extraction is the only one that is carried out continuously, making the solvent flow in the opposite direction to the solid containing the solute of interest. In this way, after an extraction stage, the extract solution moves towards a solid that contains a higher solute concentration, managing to perform a further extraction stage and so on until it reaches the fresh solid with the highest concentration of solute. On the other hand, the incoming solid meets the more concentrated solution at the inlet, and it moves toward the exit where it reaches the fresh solvent supply with the lowest solute concentration.

The counter-current feeding allows to ensure that the driving force of the process, given by the difference in solute concentration between the wetting solution and the bulk of the solvent, remains on average high throughout the process. A further great advantage is that the extraction times are shorter compared to the single conventional batch extraction stage, very attractive feature for industrial scale applications.

A schematic representation of the main advantage deriving from the counter-current feeding of solvent and solid can be obtained by looking at the Figure 4.5. The figure clearly shows how for the same extraction process, carried out for the same time interval, the concentration of solute inside the final extract is higher for the counter-current case, as well as the lower the concentration of residual solute in the exhausted matrix. An important parameter to consider, highlighted in red in the diagrams, is the difference in solute concentration between the wetting solution and the solution in the bulk.

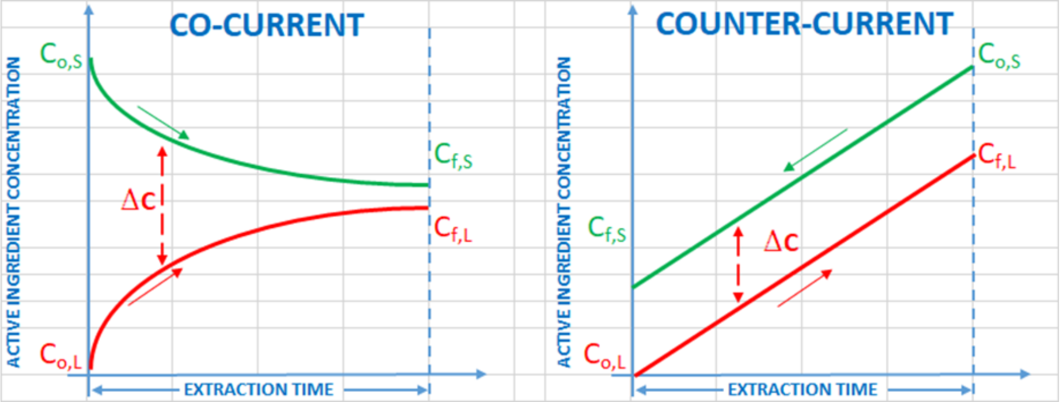


Figure 4.5 - Variation in solute concentration between soaking solution and extractant solution during a co-current and a counter-current extraction process

During co-current extraction the gradient tends to decrease as the biphasic mixture approaches the exit. This means that the extraction, even if continued for an infinite time, can never lead to the complete exhaustion of the solid matrix. In fact, feeding solute and solvent in co-current allows to obtain at the most an equilibrium stage.

On the other hand, during the counter-current extraction the concentration gradient between the wetting solution and the extracting solution remains practically constant. As can be seen by comparing the diagrams, the counter-current extraction can lead to a greater enrichment of the extracting solution and a greater exhaustion of the solid. Moreover, if the extraction is continued for sufficiently long times, there is no intermediate limit in the reduction of the concentration of the soaking solution, meaning that the complete exhaustion of the matrix is achievable.

Over the years, several models of continuous counter-current extractors have been proposed, most of which have never been accepted or used, are obsolete or have only been used for short periods of time (24). They are generally large machines, with relatively long solvent hold-up times, difficult to control and manage. Despite this, different models of counter-current extractors with traditional technology not intensified find application today, but to date there is still no intensified extractor in the world that is used on an industrial level.

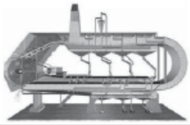
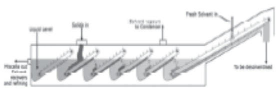
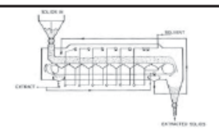
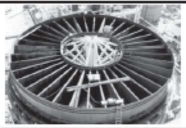
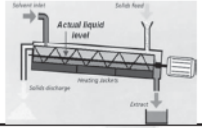
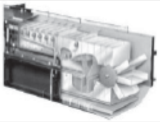
Extractors			**Capacity [tpd]**	**Yield [–]**	**Solid-liquid ratio [–]**	**Resi-dence time [min]**	**Equilibrium stages [–]**	**Corn size [mm]**	**Source [–]**
BMA, Germany	**BMA tower extractor**		4,000 – 17,000	99	3 – 5	90 – 150	n/a	5 – 100	[25]
Crown Iron Works, USA	**Model III™ Percolation type**		Up to 12,000	80 – 99	0.8 – 8	30 – 180	n/a	1 – 20	[16]
	Model IV™ Immersion type		Up to 800	80 – 99	0.8 – 8	30 – 300	n/a	1 – 20	[16]
	Model V™ (speciality materials) Percolation type	**n/a**	Up to 800	80 – 99	0.8 – 8	30 – 300	n/a	1 – 20	[16]
De Smet, Belgium	**LM™ extractor**		500 – 5,000	99	approx. 1	60 – 120	5 – 10	0.3 – 15	[28]
	Reflex™ extractor		500 – 12,000	99	approx. 1	60 – 120	5 – 10	0.3 – 15	[28]
GEA Niro, Denmark	**Contex™ extractor**		12 – 24	> 90	approx. 6	30 – 120	3 – 5	0.9 – 50	[25]
Harburg-Freuden-berger, Germany	**Carousel™ extractor**		50 – 5,000	92 – 98	0.85 – 1	30 – 90	n/a	n/a	[29]
Lurgi, Germany	**Sliding Cell™ extractor**		100 – 5,000	98 – 99	n/a	n/a	8 – 12	0.5 – 20	[30]

Figure 4.6 - List of the main counter-current solid-liquid extractors for industrial applications on the market (24)

To date, there are many plants that deal with solid-liquid extractions, especially to obtain products that have a high commercial value and that find applications in sectors such as pharmaceutical, nutraceutical or to produce aromas. However, most of these plants use extraction systems by single percolation stage or stirred vessels, obtaining a degree of exhaustion of the solid matrix that is around 50-60%, throwing away most of the product initially contained in the matrix.

4.5. The TURBEX EX30 Prototype

The TURBEX cavitational extractor was born from the idea of intensifying the extraction process trying to intervene on a series of factors that influence the efficiency of the operation. It represents a device that exploits controlled cavitation, capable of imparting high shear stresses to the liquid passing through it, generating numerous micro-jets in the liquid and causing the asymmetrical explosion of the microbubbles formed during the cavitation process (7).

The invention is described in the patent WO 2018/146647 as: "*an apparatus and a method for enhancing phase contact, and hence matter and heat transport, and for enhancing chemical reaction kinetic*" (10). It is also emphasized that the invention can be applied to treat different multiphase mixtures, in particular solid-liquid, gas-liquid, gas-liquid-solid and liquid-liquid, thus showing a spectrum of potential applications in different fields of chemistry.

The machine is made with a multirotor-multistator configuration where one or more discs of suitable geometry alternate each other connected to a motor shaft which, rotating inside a confined chamber, generate high friction with the fluid with which they come into contact and therefore creating cavitation. Furthermore, the extractor guarantees an excellent degree of mixing at a microscopic level (micromixing), allowing to break up the solid agglomerates and making the matrix much more accessible to the solvent.

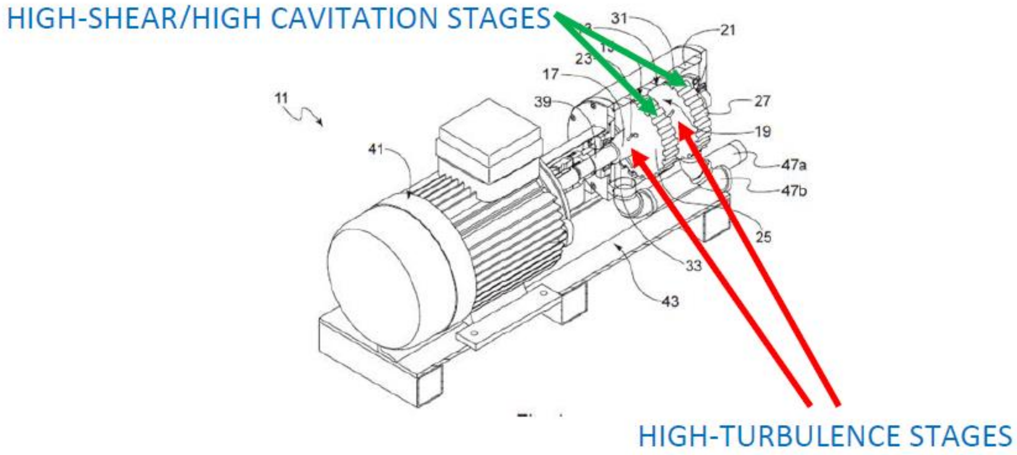


Figure 4.7 - Schematic representation of the TURBEX rotating hydrodynamic cavitatori (10) (7)

In particular, according to the invention at least one first high-turbulence mixing stage and one stage of cavitation and high-shear-stress follow each other inside the machine (10), as shown in Figure 4.7. Both stages share the central rotating shaft, placed in the middle of the extraction chamber, but are obviously made differently. For the stages of high turbulent mixing, metal devices, such as radial pin, are mounted radially to the rotor and arranged in such a way to cause turbulent motion in the substances present inside the chamber defined by stator. Instead, the high-shear-stress and high-cavitation stages are constituted by rotors and stators provided with toothed peripheral surface facing each other (10). Every time a rotor tooth, rotating, overlaps a stator tooth, a dynamic "venture" geometry is formed which accelerates the liquid flow to speeds of the order of 100-200 m/s, generating shear stress and cavitational event. Taking into account that there are 15-30 teeth and that the rotor at 500 - 200 rpm, there are thousands of overlapping teeth per second and therefore thousands of cavitational events per second.

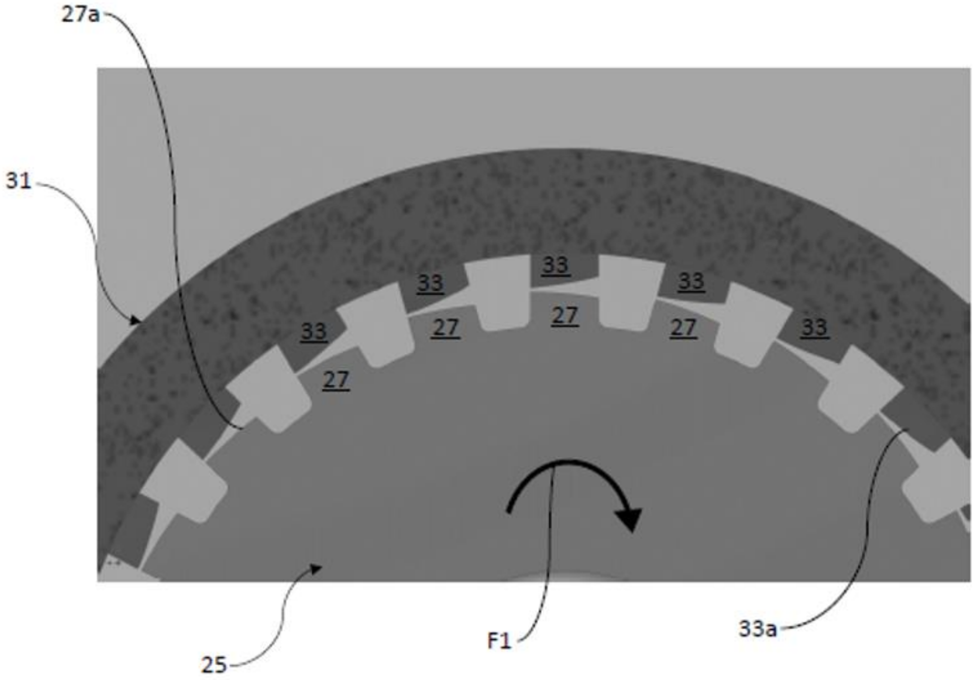
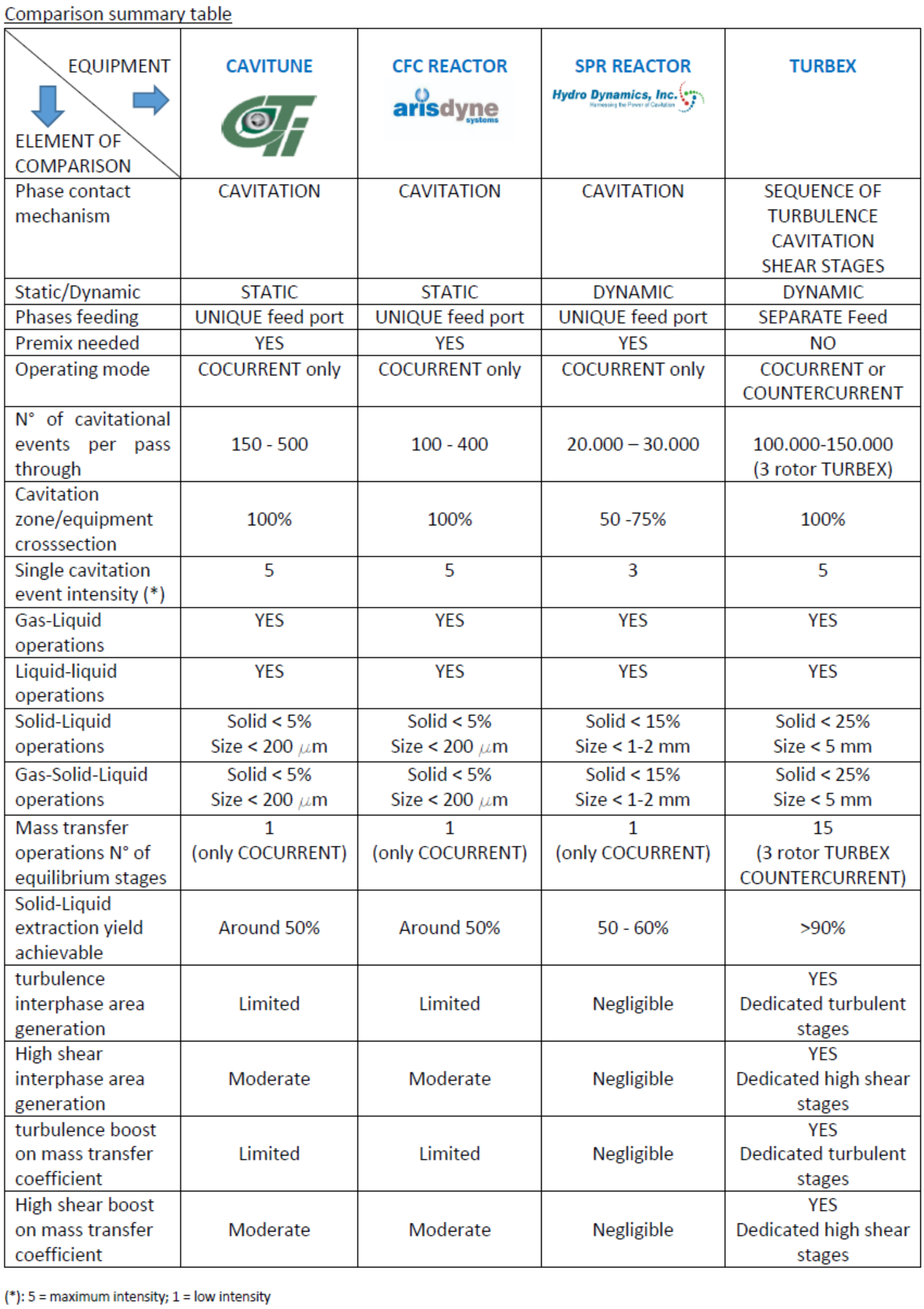


Figure 4.8 - Schematic representation of the geometry that is generated during the superimposition of rotor and stator teeth (10).

The TURBEX extractor manages to efficiently exploit the advantages deriving from cavitation-assisted extraction. Feeding the solid matrix and the liquid solvent inside the hydrodynamic cavitator guarantees a disintegration effect of the matrix, with associated generation of interfacial area, increase in porosity, rupture of the cell walls of the plant matrix releasing the most difficult to access solutes, high turbulence near the surface of the solid, excellent degree of mixing at the microscopic level, ensuring the optimization of the extraction yield and all by carrying out the process continuously.

In fact, the TURBEX extractor allows the counter-current feeding of liquid phase and solid phase, thus allowing to operate in a continuous multistage configuration where for each physical rotor-stator group there are more than one extraction stages. Thanks to this characteristic, the machine can bring the solid matrix fed towards exhaustion, minimizing the consumption of solvent and the required time.

Other extractors assisted by hydrodynamic cavitation generated by rotor-stator groups already exist on the market, as shown in Figure 4.9.



Comparison summary table

EQUIPMENT → ELEMENT OF COMPARISON ↓	CAVITUNE	CFC REACTOR arisdyne systems	SPR REACTOR Hydro Dynamics, Inc.	TURBEX
Phase contact mechanism	CAVITATION	CAVITATION	CAVITATION	SEQUENCE OF TURBULENCE CAVITATION SHEAR STAGES
Static/Dynamic	STATIC	STATIC	DYNAMIC	DYNAMIC
Phases feeding	UNIQUE feed port	UNIQUE feed port	UNIQUE feed port	SEPARATE Feed
Premix needed	YES	YES	YES	NO
Operating mode	COCURRENT only	COCURRENT only	COCURRENT only	COCURRENT or COUNTERCURRENT
N° of cavitational events per pass through	150 - 500	100 - 400	20.000 – 30.000	100.000-150.000 (3 rotor TURBEX)
Cavitation zone/equipment crosssection	100%	100%	50 -75%	100%
Single cavitation event intensity (*)	5	5	3	5
Gas-Liquid operations	YES	YES	YES	YES
Liquid-liquid operations	YES	YES	YES	YES
Solid-Liquid operations	Solid < 5% Size < 200 μm	Solid < 5% Size < 200 μm	Solid < 15% Size < 1-2 mm	Solid < 25% Size < 5 mm
Gas-Solid-Liquid operations	Solid < 5% Size < 200 μm	Solid < 5% Size < 200 μm	Solid < 15% Size < 1-2 mm	Solid < 25% Size < 5 mm
Mass transfer operations N° of equilibrium stages	1 (only COCURRENT)	1 (only COCURRENT)	1 (only COCURRENT)	15 (3 rotor TURBEX COUNTERCURRENT)
Solid-Liquid extraction yield achievable	Around 50%	Around 50%	50 - 60%	>90%
turbulence interphase area generation	Limited	Limited	Negligible	YES Dedicated turbulent stages
High shear interphase area generation	Moderate	Moderate	Negligible	YES Dedicated high shear stages
turbulence boost on mass transfer coefficient	Limited	Limited	Negligible	YES Dedicated turbulent stages
High shear boost on mass transfer coefficient	Moderate	Moderate	Negligible	YES Dedicated high shear stages

(*): 5 = maximum intensity; 1 = low intensity

Figure 4.9 - Comparison between TURBEX and other rotor-stator cavitational extractors (Courtesy to Giuliano Cavaglià)

As you can see by looking at the table, TURBEX is the only device that allows solid and liquid to be fed in countercurrent from two separate feed holes. In fact, while the other extractors allow to carry out only one equilibrium stage, the TURBEX allows to carry out up to 15 equilibrium stages. It can also be appreciated that only the TURBEX is able to exploit the phenomena of high-shear-stress, generation of high-turbulence, generation of new interface surface and increase of the material transport coefficients in a dedicated and effective way. In fact, the peculiarity of the TURBEX consists in the fact that it is the first extractor in the world to exploit in a combined way high turbulence, high shear stress and multistage cavitation in countercurrent.

The "disruptive" purpose that moved the development of TURBEX in the solid-liquid field of application was precisely the challenge of being able to make the solid travel in counter-current in an ultra-intensified device in which the liquid phase travels with a velocity of 100-200 m/s.

The development of a model suitable for industrial scale has gone through tests on small prototypes. A TURBEX cavitational hydrodynamic extractor model was tested by researchers from the DSTF (Department of Drug Science and Technology) of the University of Turin on olive leaves and pomace to test the extraction efficiency of the polyphenols contained within these plant matrices. The results of the tests have shown that the machinery is very efficient and allows to obtain from a ton of grape pomace about 20 kilograms of dry extract rich in polyphenols, with a commercial value of 40 €/kg in bulk, and from a ton of olive leaves about 80 kilograms of dry extract that can be valued at 30 €/kg sold in bulk (7).

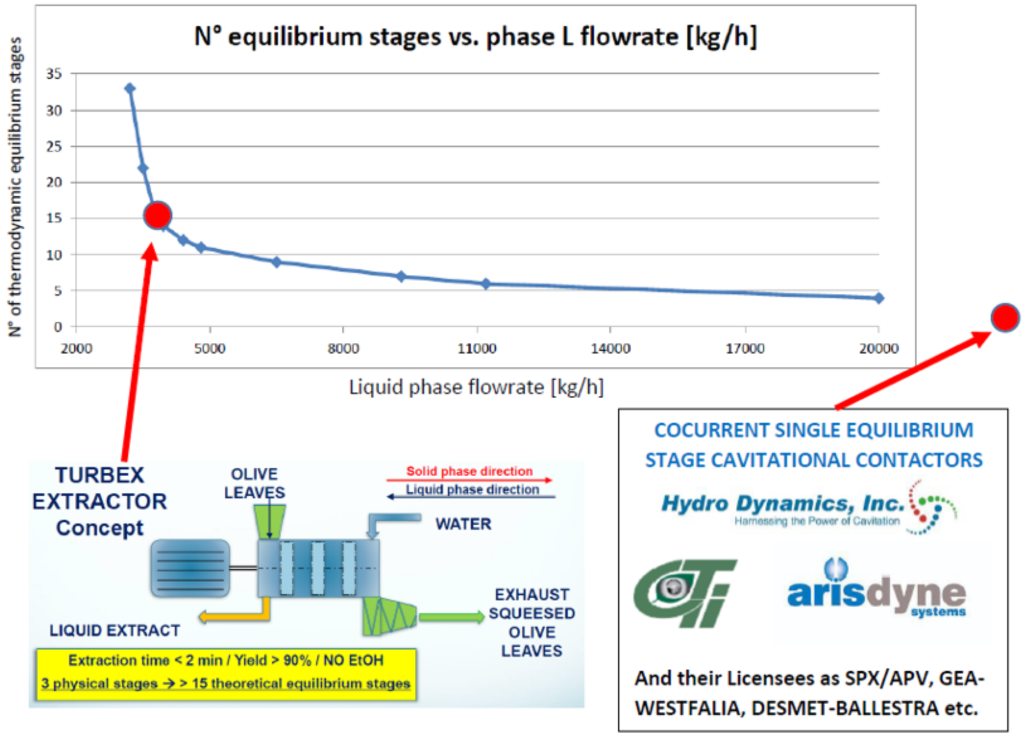


Figure 4.10 - Results of comparative analysis between the number of equilibrium stages performed by the TURBEX and the other cavitational extractor varying the liquid phase flowrate (Courtesy to Giuliano Cavaglià)

An interesting comparative analysis, schematized in the Figure 4.10, was carried out between TURBEX and the other hydrodynamic cavitator extractors mentioned for the base case of the extraction of polyphenols from olive leaves. it was possible to show that to reach extraction efficiencies higher than 90%, it was considered necessary to feed the solvent with a liquid over solid ratio of approximately seven, which proved to be considerably lower than the ratios required by the other equipment. Moreover, the ultra-intensified extractor allows the process to be carried out with residence times inside the machinery of the order of a few minutes and using tap water as the extracting solution, thus performing a solvent-free extraction. Given the very short residence time required and the limited solvent-free liquid phase flowrate required, TURBEX is expected to bring a huge CAPEX and OPEX benefits either for the extraction step or for downstream separation, purification and drying one.

These results were among the first confirmations that allow TURBEX to be presented as a disruptive technology in the context of the valorisation of waste from the agro-food chain. Considering the obtained results, further developments of the invention, born from the collaboration of the inventor Giuliano Cavaglià with the Andritz group, led to the creation of the first prototype of the machinery suitable for industrial applications: the TURBEX EX30 Prototype, protagonist of this thesis project.

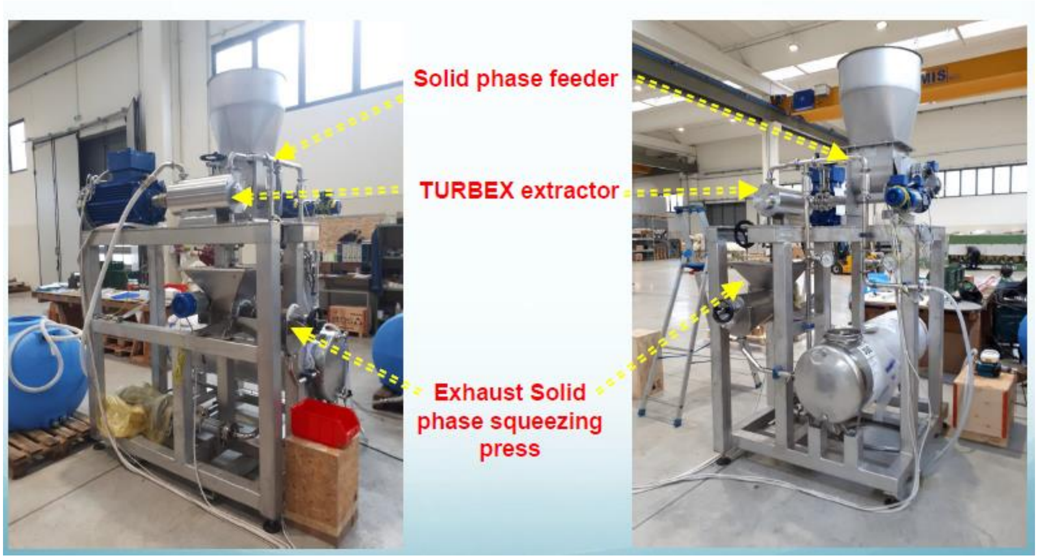


Figure 4.11 - View of the TURBEX EX30 Prototype extraction unit. (Courtesy to Giuliano Cavaglià)

The machine contains a succession of three physical stages of high-shear-stress and high cavitation, alternated by four physical stages of high-turbulence mixing. In anticipation of the gradual comminution of the solid as it passes through the TURBEX EX30, the three stages of high cavitation and high shear stresses have been designed with a rotor-stator gap descending along the direction of the solid between inlet and outlet. Because of the different gap in the three physical stages, a different cavitation starting point occurs for each stage. The cavitation starting point is located around 600 rpm for the rotor-stator pair closest to the solid outlet, around 800 rpm for the intermediate rotor-stator pair and 1100-1200 rpm for the rotor-stator pair closer to the solid entrance.

The confirmation of these values was obtained from the data that were collected during one of the first cold tests carried out on the machinery, performed feeding only water inside it. Data relating to the required power and the torque of the motor recorded as the rotor rotational speed varies. By plotting the function that describes the variation of the incremental torque as the rotation speed varies, the three distinct points of cavitation trigger are recognized as the maximum points of the curve.

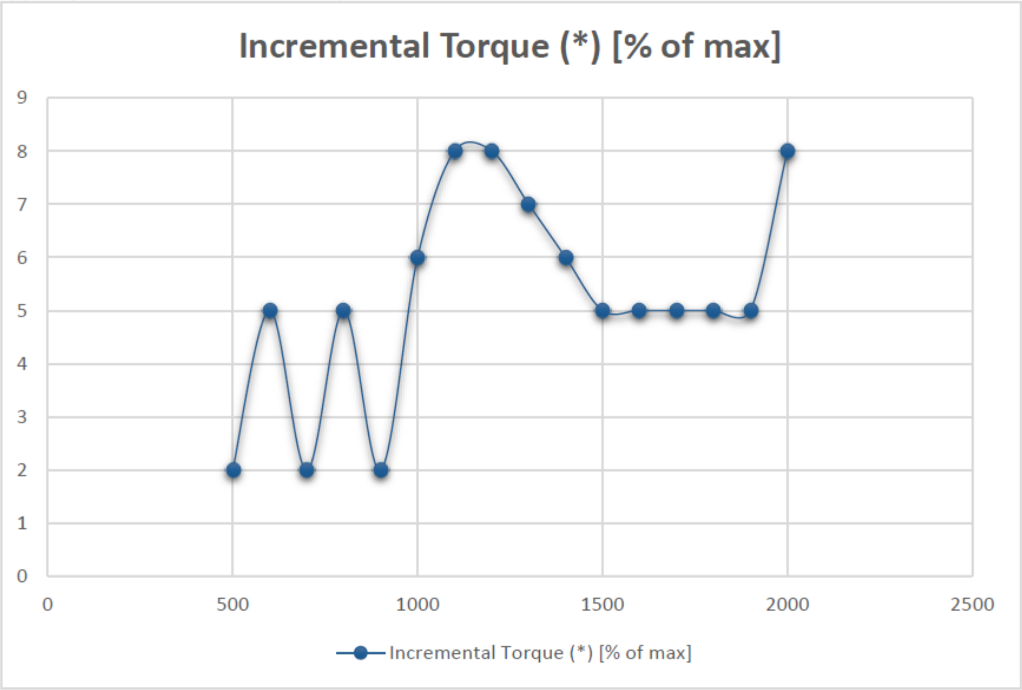

(*) Torque increase for the last +100 rpm increase

Figure 4.12 - Incremental torque variation as the rotor speed varies. Results obtained in one of the first cold tests carried out on the machine (Courtesy to Giuliano Cavaglià)

It has been designed to process up to 100 kilograms per hour of dry solid matrix and rotor rotation speeds of up to about 2000 rpm. At present the extraction unit includes: a TURBEX EX30 extractor, a dosing hopper for the dry solid, a progressive cavity feeder to fed wet solid inside the extractor, a filtering system for the extract with two bag filters working on parallel, a squeezing press equipped with a hopper for squeezing the exhaust solid, a horizontal tank for the storage of the squeezed liquid and a volumetric pump for sludge for the recirculation of the squeezed liquid.

During the commissioning phase of the machinery it was possible to gain experience with all those tuning procedures that are necessary when a prototype is put into motion for the first time. Several tests were carried out on the machinery between the period of October 2019 and August 2020, significantly slowed down also by the sanitary emergency that characterized this last year. Numerous tests were performed to fine-tune especially the solid feeding system and the extract filtering system, which showed the greatest criticalities. However, since the first tests, the thing that most worried all the production staff was confirmed: the machinery can operate the extraction continuously and in countercurrent, making the solid and the liquid flow in opposite directions.

The functional tests of the extractor were conducted by feeding tap water and flowering tops of passionflower in tea and powder cut. This made it possible to collect the samples that have been described in the previous chapters and to conduct the tests necessary to have a first ballpark idea of the extraction efficiency of the machinery and of the morphological changes that it is capable of imparting on the fed plant-based matrix.

The results confirmed all the expectations that were had on the machine. As described in the previous chapter, the extractor showed an extraction efficiency in both cases higher than 97%, confirming that it is therefore capable of bringing the solid matrix fed until the solutes contained in it are exhausted. Furthermore, confirmation was found of the fact that, with the succession of only three cavitational physical stages, the TURBEX EX30 can perform in a single passage a greater number of equilibrium stages, in particular at least between 7 and 15 according to the results.

The theoretical reasons that allow to justify the high efficiency of TURBEX are also confirmed in the experimental analyses carried out to characterize the changes undergone by the matrix following its passage inside the machinery. In particular, the reduction of the particle size and the moisture content are signs attributable to the breaking effect of the solid particles and the cell walls of the matrix deriving from the high-shear stresses and cavitational phenomena to which the matrix is subjected inside the extraction chamber. Even if a direct measurement has not been carried out, it is also plausible to suppose that the imbibition ratio of the solid matrix is drastically decreased following the breaking phenomena to which it is subjected. This further increases the yield of the extraction process because, as explained in the previous chapter, the separation of the higher solute concentrated soaking liquid from the solid residue becomes more effective.

Undoubtedly, these represent the results of a first preliminary analysis of TURBEX EX30, carried out with samples obtained during the commissioning phase. In the following months the equipment will be tested at the Greenfield factory, located in San Damiano d'Asti (AT), where different types of solid matrices will be fed and the extraction yields evaluated, with the aim of also point out the optimal operating parameters of the machinery. However, the results obtained were enough to set up two plants for the recovery of substances with high added value from residues of the agro-food chain. The projects of the two plants, relating to the recovery of polyphenols from olive leaves and of lipids, polyphenols, proteins and dietary fibers from brewer's spent grain, will be described in the next two chapters.

Chapter 5

Olive leaves valorisation plant

In the first century after Christ, Columella, roman writer of agriculture, in his "*De Rustica*" wrote "*Olea prima omnium arborum est*", which means "The olive tree is the first of all trees". The populations of the Mediterranean have always had a strong bond with the olive tree, exploiting its fruits since ancient times both to be eaten as it is and to produce the precious olive oil. In different parts of Europe and the Middle East there are traces of olive growing since prehistoric times (25). A good purpose in modern times could be to take this bond to the next level, enhancing not only the fruits offered by the tree, but also something that has always accompanied the collection and production of olives for millennia without being appropriately enhanced: the leaves.

Among the agro-industrial residues related to the cultivation of olive trees, in addition to pomace obtained after squeezing the fruits, there are the leaves of the tree, produced in large quantities during the periods of tree pruning and olive harvesting. The leaves traditionally represent a waste to be disposed of or possibly used on site, after drying, to produce thermal energy. In recent years, with a view to process intensification and sustainability, research is increasingly widespread with the aim of enhancing them rather than perceiving them as a waste.

The low incidence of cardiovascular diseases and cancer in Mediterranean countries has drawn the attention of numerous scientists and investigators, and the Mediterranean diet, in which the consumption of olive-based products has an important role, has been thought to be the reason of it (26). In recent times, in order to obtain the maximum benefits from the plant, many studies on the components of the olive leaves have been conducted, highlighting their richness in functional bioactive molecules. The most interesting molecules are flavonoids and phenolic compounds such as oleuropein and hydroxytrysol, that have shown an interesting antioxidant activity (27) (28). Oleuropein can be considered the substance with the greatest economic interest and as the main representative of the polyphenolic fraction of the olive leaf.

It was demonstrated that oleuropein helps prevents cardiac disease by protecting membrane lipid oxidation, acting on coronary dilatation and through antiarrhythmic action improves lipid metabolism (29). Moreover, it helps in obesity related problems, prevent hypertensive cell death in cancer patients, protect enzymes, and exhibits antiviral properties (30) (31). Even the hydroxytrysol, a metabolite of oleuropein, has interesting functional bioactive properties: it has effects like that of oleuropein, but also it prevents diabetic neuropathies and protects against atherosclerosis (26) (31). Other studies, still to be carried out, are pushing towards the anti-HIV effects of oleuropein ed hydroxytrysol (32) (33).

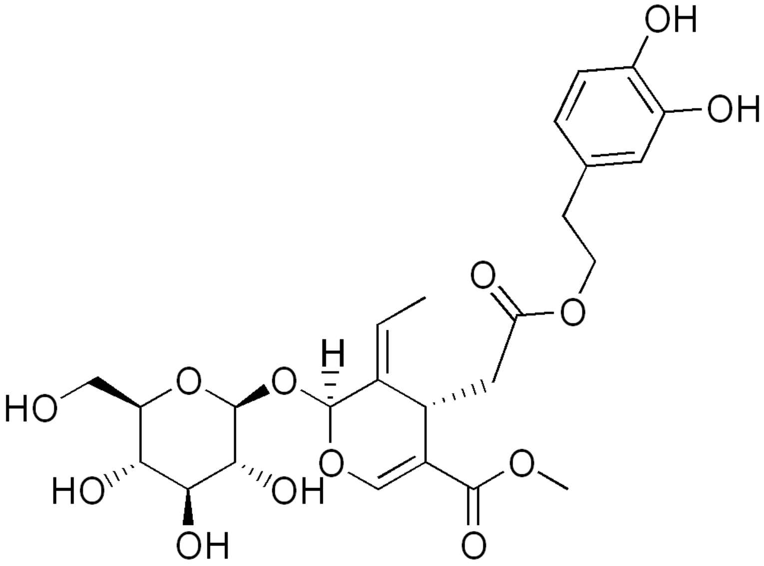


Figure 5.1 - Oleuropein molecular structure

To date, the global market of olive leaves extract is widespread in different countries: North America and Europe hold a significant market share owing to the increasing awareness of its benefits and applications (34). Olive leaves extracts are mainly used as active ingredients in the preparation of dietary supplements, various cosmetics, beverage and pet food. During the last six years, among the main manufacturers producing olive leaves extracts we can include, just to name a few, companies like Nutra Green, DSM, La Gardonnenque, Martin Bauer Group (35).

All these reasons led to the choice of designing a plant that treat olive leaves, setting the goal of obtaining an extract in the form of a powder with a high oleuropein content. The main purpose is to develop an innovative process where the TURBEX EX 30 Prototype represents the heart of the extraction process, thus demonstrating that it is possible to insert it in a production line of a plant, bringing significant benefits in terms of process sustainability, efficiency and versatility.

5.1. Process description

5.1.1. Targets

The purpose of the process is the recovery of high added value substances from one of the agro-industrial residues related to the production of olive oil: olive leaves coming from tree pruning and fruit harvesting. The plant aims to carry out the extraction of polyphenols from olive leaves, with a particular regard to oleuropein which is the most valuable bioactive compound, to obtain a powder with a high concentration of polyphenols that can be mainly used in the food and nutraceutical industry.

It was decided to carry out the extraction using the TURBEX EX30 cavitational extractor, which, compared to conventional extractors, increases the material transport coefficients by promoting the release of compounds and their solubilization in water.

Among the various targets, one of the most important is that of realize the extraction without the use of solvents: the extracting fluid consists of water only at a temperature of 50 ° C. This solution is made possible tanks to the high extraction efficiency of TURBEX EX30 and it guarantees a reduced environmental impact and lower costs while maintaining efficiencies comparable, if not superior, to those of a traditional extraction technology.

5.1.2. Plant overview

The plant was designed to process 25.000 tons per year of olive leaves, working for a total of twenty weeks per year, close to the pruning period of olive trees and the period of olive harvest. The project relates to a plant located in Turkey, in the city of Içel in whose region the cultivation of olive trees to produce oil is widespread.

The entire plant can be divided in three main sections:

- Pre-treatment and storage
- Extraction
- Concentration

The first section is considerable independent from the others: leaves are collected during the first month of the activity period, then they must be dried and stored to maintain their properties without risking fermentation phenomena. The second section is the main one, constituting the beating heart of the process: the TURBEX EX30 Prototype is used as the extractor, allowing to solubilize the oleuropein in the extraction water. In the third part, the solvent is separated from the solutes of interest and the final product id obtained.

The plant was designed to operate with a turn-down between 60% and 110% compared to the project conditions. It was decided to operate keeping the pre-treatment and product concentration sections in operation 7 days a week in order to avoid frequent start-ups and shutdowns, while keeping always high efficiency of the equipment. Moreover, concerning the pre-treatment section, it must be considered that the supply of fresh leaves will be available only during the period of their harvest: for this reason it has been assumed that the section is active only during the first three months of operation. The extraction section has no problems of dead times related to shut down and start-up, so it was considered that it could operate 5 days a week maintaining high efficiencies.

Table 5.1 - Olive leaves Plant Characteristics

Supply type	Olive leaves	
Plant capacity	25000	*ton/year*
Plant operation period	20	*weeks/year*
Section 1 operation period	12	*weeks/year*
Days a week	7	*days/week*
Hours a day	24	*h/day*
Hourly feeding	12,40	*ton/h*
Hourly feeding MAX	13,64	*ton/h*
Hourly feeding MIN	7,44	*ton/h*
Section 2 operation period	20	*weeks/year*
Days a week	5	*days/week*
Hours a day	24	*h/day*
Hourly feeding	5,81	*ton/h*
Hourly feeding MAX	6,39	*ton/h*
Hourly feeding MIN	3,48	*ton/h*
Section 3 operation period	20	*weeks/year*
Days a week	7	*days/week*
Hours a day	24	*h/day*
Hourly feeding	9,46	*ton/h*
Hourly feeding MAX	10,48	*ton/h*
Hourly feeding MIN	5,67	*ton/h*
Turndown Ratio MIN	0,6	
Turndown Ratio MAX	1,1	

SUPPLY:		
Wet Olive Leaves Feedstock	25000	ton/y
Dry Olive Leaves Feedstock	13936	ton/y
PRODUCTS:		
Polyphenols powder	2487,4	ton/y
RESIDUES:		
Spent wet Olive Leaves	20209	ton/y
Destination:	Feed to CHP unit	

5.1.3. Matrix characterization

The supply consists of green leaves with an average composition equal to that shown in Table 5.1 (26). Leaves have length between 0,04 and 0,10 meters and width between 0,01 and 0,03 meters.

The estimated leaves temperature is of 17 °C and the relative humidity is of 56%, considerations based on the average atmospheric conditions of the collection site, assumed to be in the same region where the plant is located.

Table 5.2 - Olive leaves composition

Proteins	5,45	*g/100g wt*
Carbohydrates	6,54	*g/100g wt*
Oils	27,58	*g/100g wt*
Crude Fiber	7	*g/100g wt*
Ash	3,6	*g/100g wt*
Water	49,83	*g/100g wt*

In Figure 5.1, the oily fraction of the leaves is highlighted: the polyphenols, including oleuropein, are found right within this fraction. It accounts for 6% of the total weight of green leaves, increasing up to 13% on a dry basis.

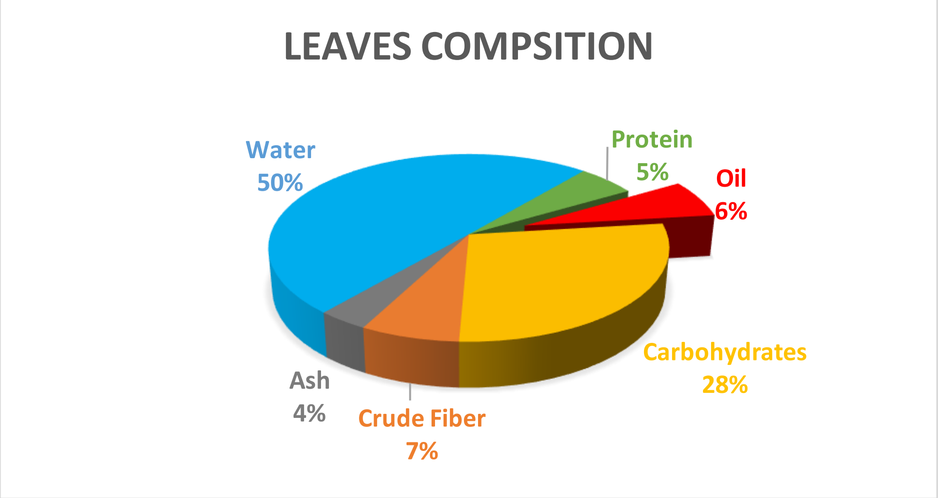


Figure 5.2 - Olive leaves composition

In order to identify in a more schematic way the various classes of compounds present inside the leaves, simplifying also the elaboration of mass balances, it was decided to subdivide the composition into pseudo-components as follows:

Table 5.3 - Pseudo-components composition of olive leaves

Oleuropein	3,51	*g/100g wt*
Extractable	32,24	*g/100g wt*
Water	49,83	*g/100g wt*
Unextractable	14,415	*g/100g wt*

This classification leads to a distinction between extractable substances, non-extractable substances, water and oleuropein, which represents the most valuable polyphenol within the extractable fraction and therefore it deserves to be considered individually. The concentration of available oleuropein was evaluated considering that the 7% of leaves, on dry base, is made up on it (36). The extractable fraction was evaluated integrally including sugar-based substances, a fraction of proteins (30%) and the oils fraction without oleuropein.

5.1.4. Product characterization

The final product obtained from the process is a powder, with an oleuropein content of about 33% and a moisture content of about 2%. The residual part is constituted of water-soluble substances of low value consisting mainly of sugars, carbohydrates and proteins. The product has uses in the food sector, mostly nutraceuticals: it can be used as it is as a fundamental component of food supplements. The estimated commercial value of the solid extract product is around 20 euro per kilogram.

Table 5.4 - Olive leaves solid extract composition

Oleuropein	32,90%
Extractable	65,10%
Water	2,00%
Unextractable	0,00%

Table 5.5 - Commercial value of the products obtained and annual turnover resulting from their sale

PRODUCTS COMMERCIAL VALUE:		
32% w/w Polyhenols powder	20	€/kg
PRODUCTS SALES TURNOVER:		
Polyphenols powder	49.700.000	€/y

5.1.5. By-product characterization

The main by-product of the process consists of ground exhausted olive leaves with a moisture content of about 50% which can be delivered to combined heat and power generator. This operation contributes to the intensification of the process, reducing wastes.

Table 5.6 - Residue solid composition

Oleuropein	0,30%
Extractable	31,87%
Water	50,00%
Unextractable	17,83%

Considering the residue oleuropein in the spent solid, it is possible to calculate a total extraction yield of the whole process equal to about 93,2%.

5.1.6. Pre-treatment and storage section

Olive leaves are harvested in large quantities only in a limited period; therefore, it is not possible to ensure their continuous supply over time. Moreover, green leaves risk undergoing fermentation phenomena that could alter their properties. For this reason, they need to be dried and correctly stored to assure a continuous flow of leaves during the operating period of the plant. In addition, the drying process is also a fundamental part of the extraction process: it enhances the extraction of phenolic compounds (36).

It must be considered that, during the drying process, the product's quality characteristics can change considerably because of the applied heat. Drying can affect the activity and stability of bioactive molecules, causing the loss of them due to volatilization or thermal degradation (36). Among the substances that degrade more easily with the temperature we find polyphenols. In the specific case of olive leaves, there is a substantial risk of losing substances such as peroxidase, polyphenol oxidase and beta-glucans. The best choice to preserve the quality of herbs is to let them dry naturally, exposing them to the air at ambient temperature. However, this process cannot be controlled, and it always requires a lot of time.

The choice of the most suitable drying method was based on taking into consideration that it must be rapid, but without raising the temperature of the leaves too much. It was chosen to operate the drying with a hot air stream heated using an air vein burner. This solution, widely adopted industrially, allow to heat the air stream and easily control its temperature, also it allows to carry out the drying operation very quickly. Using an air vein burner, it's possible to control the air temperature, setting it high enough to allow a rapid drying, but not too high to avoid the degradation of the substances of interest. For this plant it was decided to send air at an inlet temperature of 200 °C.

Leaves arrive processed with a nominal capacity of 12,4 tons per hour with a water mass concentration of about 50%. The drying process aims to bring the leaves to a water content of 10%, thus removing approximately 13 tons per hour of water. The final composition of the leaves at the end of the drying section of the plant is shown in Table 5.5.

Table 5.7 - Dried leaves composition

Oleuropein	6,30%
Extractable	57,84%
Water	10,00%
Unextractable	25,86%

The leaves are then ready to be sent to the second section of the process: the polyphenol extraction section.

5.1.7. Extraction section

The flow rate of leaves to be treated is 5,81 tons per hour. Considering the intrinsic characteristics of the TURBEX EX30, in order to make the most of all the process intensification phenomena that take place inside it, it is desirable to split the feeding into several machines. It has been chosen a size of the machine that allow to process 500 kilograms per hour of solids, thus leading to the design of a battery of 14 TURBEX, twelve in operation and two available to be used to maintain the continuity of the process in the case of any maintenance interventions.

The leaves are manually unloaded from the bags into the receiving hoppers of each TURBEX EX30. A stream of hot water at a temperature of 50 °C, constituting the extracting fluid, is sent into the TURBEX in counter current with the solids, using a liquid/solid ratio equal to 3.

The liquid extract stream, coming out from the battery of TURBEX with a total flow rate of approximately 13.2 tons per hour, is sent to a bag filter system to remove any traces of residue solids entrained during the passage through the machinery. The bag filters must be periodically emptied and the solid recovered from them can be reinserted into the receiving hopper of the TURBEX. A small part of the extract must be recirculated inside the progressive cavity feeder to favour the partial fluidification of the solids and guarantee their correct feeding. The remaining part of the liquid extract is sent to the concentration section.

The spent solids coming out from the TURBEX are collected by gravity inside a hopper connected to a squeezing press. The press, adjusted manually, has the purpose of gradually compress the solid recovering part of the imbibition liquid trapped inside it, thus reducing its water content. The liquid that percolate from the external holes of the press is conveyed and sent to a collection tank to be subsequently recirculated as part of the extracting water stream. The squeezed spent solid is instead collected inside a transport cart placed on the floor just below the exit from the press and periodically changed by an operator.

5.1.8. Concentration section

The goal of this section is to reduce the water content of the liquid extract until a powder with a moisture content of 2% is obtained. This goal is achieved by evaporating a water flowrate of 8,7 tons per hour, first using a series of three multiple effect evaporators and finally an indirect dryer. The choices made are consistent with the intrinsic nature of the product to be obtained: it’s necessary to avoid as much as possible the thermal degradation of the polyphenolic compounds.

The first step is to concentrate the liquid extract up to a mass fraction of water of 70% using a mechanical vapour recompression evaporator (MVR). This solution is widely adopted in industrial plants: they can perform the concentration of liquid liquors minimizing the energy costs related to water and steam consumption. The evaporated vapour is recompressed with a low speed fan and after the fan vapour can be used as heating steam in the same unit.

To give to the final extract the desired characteristics, the concentrated liquid extract is subjected to a final drying stage inside a drum drier type dryer. This type of dryer works ensuring that the heat exchange between the process stream and the utility take place indirectly, thus avoiding contact between them. The drum dryer is equipped with two rotating cylinders inside which the steam is sent. The material in the form of a paste is distributed evenly on the surface of the rotating drums, heated by the contact with the steam.

By directly sending the low-pressure steam available in site, the extract paste can overheat too much, thus causing the degradation of a part of the polyphenols present in it. In particular, the degradation temperature of oleuropein is about 120 ° C, for this reason it was decided to send steam with a pressure of 2 bar instead of 6 bar, also trying to guarantee very short contact times in the order of some seconds.

The product obtained in the form of powder is finally collected from the cylinders by means of the mechanical action of special scraping knives installed at the solid outlet of the dryer.

5.1.9. Plant Flowchart

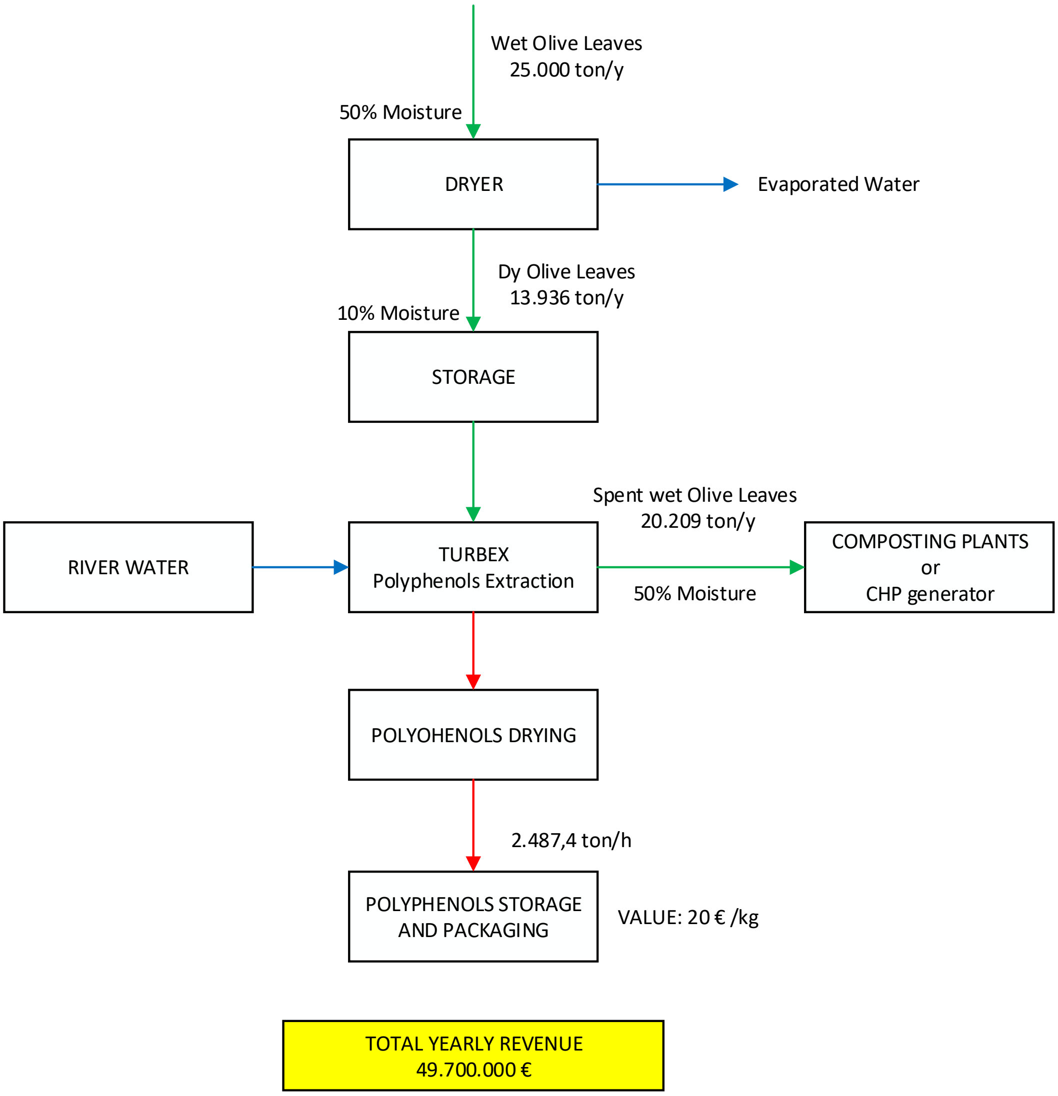


Figure 5.3 - Olive leaves valorization plant flowchart

5.2. Design basis

The description of the main equipment making up the plant and the related design criteria will be described in this paragraph. In order to better understand what is reported in this section, it is recommended to consult the process flow diagram in Appendix A. It should be emphasized that the design of this plant stops at the basic conceptual design phase, for the sole purpose of proposing a possible plant solution. The sizing of the individual devices was therefore not considered necessary and only the design criteria relating to their operation will be dealt in this paragraph.

5.2.1. Utilities characteristics

The utilities used in the plant are river water, low pressure steam, natural gas, and electric energy. Their main characteristics are described in Table 5.7.

Table 5.8 - Utilities Characteristics

Utility	Characteristics		
River Water	Temperature	15	*[°C]*
	Pressure	1	*bar*
Low Pressure Steam	Pressure	0,5	*[Mpag]*
	Temperature	158	*[°C]*
Natural Gas	Pressure	0,35	*[Mpag]*
	LHV	32,49	*[MJ/Nm^3]*
Air	Temperature	10	*°C*
	Relative humidity	70%	
Electric Energy LT	400V (±10%)-3Ph-50 ± 0,5 Hz		
Electric Energy MT	6000V (±10%)-3Ph-50 ± 0,5 Hz		

5.2.2. Utilities specific consumptions

In calculating the consumption of the various utilities, two versions are proposed: both the consumption of utilities actually required by the plant and the consumption considering the recovery of thermal and electrical energy, generated by the CHP generator, are indicated. In fact, the residual matrix from the extraction process is sent to a combined generation unit of thermal and electrical energy. According to experimental data, it is estimated that 1.25 kg/h of wood chips with 20% humidity can produce approximately 2,5 thermal kWh/h and 1 kWh/h of electricity in CHP units. Furthermore, it is assumed that the matrix in question has a calorific value equal to approximately 2/3 of that of the wood chips.

Table 5.9 - Utilities hourly consumptions

Utility	Hourly consumption (without CHP)	Unit	Hourly consumption (with CHP)	Unit
River water	11,3	*[ton/h]*	11,3	*[ton/h]*
Low Pressure Steam	2,0	*[ton/h]*	0,0	*[ton/h]*
Natural Gas	304,2	*[m^3/h]*	60,8	*[m^3/h]*
Electric Energy	659,8	*[kWh]*	-951,0	*[kWh]*

Table 5.10 - Utilities yearly consumptions

Utility	Yearly consumption (without CHP)	Unit	Yearly consumption (with CHP)	Unit
River water	38048	*[ton/y]*	38048	*[ton/y]*
Low Pressure Steam	6872	*[ton/y]*	0	*[ton/y]*
Natural Gas	1022101	*[m^3/y]*	204243	*[m^3/y]*
Electric Energy	2217	*[MWh/y]*	-3195	*[MWh/y]*

Table 5.11 - Utilities specific consumptions

Utility	Specific Consumption (without CHP)	Unit	Specific Consumption (with CHP)	Unit
River water	15,3	*[ton/$ton_{PROD.}$]*	15,3	*[ton/$ton_{PROD.}$]*
Low Pressure Steam	2,8	*[ton/$ton_{PROD.}$]*	0,00	*[ton/$ton_{PROD.}$]*
Natural Gas	410,9	*[m^3/$ton_{PROD.}$]*	82,1	*[m^3/$ton_{PROD.}$]*
Electric Energy	891,3	*[kW/$ton_{PROD.}$]*	-1284,6	*[kWh/$ton_{PROD.}$]*

Since the sizing of the equipment was not carried out, nor their spatial arrangement assumed, it was not possible to evaluate the electrical consumption relating to pumps. Since the latter is not very relevant to the overall electrical consumption of the system, it was decided to neglect it in this first analysis stage. The consumption of electricity shown in the previous tables is therefore to be understood as decreased by the contribution of the pumps.

5.2.3. Pre-treatment and storage section

5.2.3.1. Underground storage tank S-101

Olive leaves arriving from the collection areas are temporarily stored in the underground collection tank **S-101**. Thanks to a bucket elevator, green leaves are collected from the underground tank and sent, with a capacity of 12,4 tons per hour, to the miller M-101 to reduce their size up to few millimetres.

5.2.3.2. Mill M-101

Olive leaves, collected from the tank **S-101**, are subjected to a grinding stage using the hammer mill **M-101** which reduce their size up to few millimetres. The milling operation is done in order to facilitate the following operations and reduce the volume occupied by the sheets during storage.

5.2.3.3. Hoppers H-101 and H-102

The grounded leaves are then sent by gravity to a receiving hopper **H-101**, equipped with a lump breaker to avoid the formation of agglomerates and facilitate the unloading. The receiving hopper must have at least one or two hours of hold-up and it is connected to the dosing hopper **H-102** with a vertical pipe. The discharge from the receiving hopper to the dosing hopper takes place by gravity thanks to the opening and closing of a servo-assisted gate valve, controlled by the high- and low-level switch of the dosing hopper. The dosing hopper rests on load cells and is connected to the rotary feed valve **FE-101** that assure the green leaves feeding to the Rotary Drum Dryer **DD-101** with continuity.

5.2.3.4. Rotary valve feeder FE-101

The rotary feed valve **FE-101** is regulated by an inverter motor whose rotational speed is controlled by the weight control loop, deriving from the signal of the weight taken from the load cells on which the dosing hopper rests. The rotary feeding valve has a second important function: it avoids the rising of the hot fumes coming from the dryer.

5.2.3.5. Rotary Drum Drier DD-101

The leaves are dried inside a drum dryer using a hot air stream. The flow of air required for drying is evaluated considering the water content of the green leaves, the temperature and relative humidity of the external air and the operating temperatures of the process, following a dryer standard design procedure (37). The evaluated air stream, resulting from the sum of the air leaving the solid cooler **E-102** and the air pre-heater **E-102**, is heated up to 200°C using an air vein burner powered by methane and the hot gases are then sent to the Rotary Drum Dryer **DD-101** in counter current with the solid stream.

The dryer is a single tube type and the drying process takes place by direct contact between the hot gas stream and the wet solid stream and. This operational choice ensures lower investment and operating costs, without compromising the quality of the raw material: no problems have been recorded relating to direct contact between the hot gas stream and the leaves.

5.2.3.6. Hot gas purification system CY-101, PF-101/102

The gases leaving the Drum Dryer **DD-101** need to be purified before their discharge to the atmosphere. The first step is the passage inside the cyclone **CY-101** for powders, in order to eliminate traces of any small particles dragged during the drying process. The fumes coming out of the cyclone are finally sent to a bag filter system constituted by two filters **PF-101** and **PF-102** working alternately, in order to eliminate traces of particulate still present in the stream.

The solids removed are collected and disposed of in landfills in accordance with local regulations.

5.2.3.7. Open-back air draught burner B-101

The open-back air draught burner **B-101** has the purpose of supply the necessary heat to the air current used for drying to allow it to reach the temperature of 200 °C. It is fed with methane and allows to heat air currents quickly, effectively and with low emissions. Open-back air draught burners are also easy to install, start and use.

5.2.3.8. Blowers BL-101 and BL-102

The air flow required for the drying process is quite high: because of this it was decided to use axial fans, which can guarantee the delivery of higher air flow rates than centrifugal fans. The total air flow to be sent to the dryer is obtained by mixing two distinct streams: the first is sent by blower **BL-102** inside the solid cooler **E-102**, while the remaining part is sent by blower **BL-102** and preheated inside the economizer **E-101**.

5.2.3.9. Economizer E-101

The hot gas stream leaves the drum dryer with a temperature of 110 °C and, after purification, it can be used to pre-heat the cold air stream. The Economizer E-101 is used for this purpose: it heats up the air stream exchanging the amount of heat necessary to bring the temperature of the fumes from 110 °C to 80 °C. The outlet hot gas stream temperature of 80 °C is chosen to ensure that the stream has a sufficiently high temperature to allow its easy expulsion into the atmosphere.

The exchanger is of the tubular type, specially designed to guarantee gas / air exchange. This type of exchangers is typically used to recovery the sensible heat of post combustion gases.

5.2.3.10. Solid cooler E-102

The dried olive leaves reach a temperature of about 80 °C after the drying process and need to be cooled before their densification. They are collected by gravity from the Drum Dryer **DD-101** and, by means of a conveyor belt, they are fed to the **FBE-102** heat exchanger. It consists of a fluid bed heat exchanger where the solids are suspended and mixed by a stream of cold air. Maximum contact occurs between the solid particles and the air stream, resulting in high heat transfer efficiency and uniform cooling of the solids.

The outlet solids temperature is fixed to 40°C, to assure a safe packaging inside big bags to be stored inside the warehouse **S-102**. The outlet air stream pass trough a purification section constituted by a cyclone and a battery of filter bags, then it is recycled to be used as part of the hot air stream needed to perform the drying process.

5.2.4. Extraction Section

5.2.4.1. Hoppers H-201 and H-202

The dried leaves bags are manually unloaded into the receiving hopper **H-201**, equipped with a lump breaker to avoid the formation of agglomerates and facilitate the unloading. The receiving hopper must have at least one or two hours of hold-up and it is connected to the dosing hopper of the screw conveyor **SC-201** with a vertical pipe. The discharge from the receiving hopper to the dosing hopper takes place by gravity thanks to the opening and closing of a servo-assisted gate valve, controlled by the high- and low-level switch of the dosing hopper.

The dosing hopper of the screw conveyor rests on load cells and the weight signal control the rotational speed of the screw conveyor, assuring a continuous controlled fed of solids to the mixer MX-201, defining the gear of the extraction section.

5.2.4.2. Mixer MX-201

The feeding of the solid to the TURBEX, during various tests carried out in the commissioning phase, proved to be a critical part of the entire extraction phenomenon. Especially in the case of non-powdered solids, the lack of wetting of the solid can lead to the formation of agglomerates that tend to plug and block the screw feeder, interrupting the continuity of the process. For this purpose, it is necessary to carry out a complete wetting of the solid before feeding it, but in order not to change the counter-current operation of the machinery, this is done using the extract obtained rather than fresh liquid.

The mixing is therefore carried out inside the stirred tank **MX-201**, equipped with a paddle stirrer that guarantees continuous mixing between the solid and the extract. The output of the obtained slurry, with a concentration of about 92% by weight of water, is then sent to the DC-201 centrifugal decanter.

5.2.4.3. Decanter centrifuge DC-201

In order to separate the wet solid stream from the extract, the **DC-201** centrifugal decanter is used. This type of equipment is widely used in the industrial field for the dewatering of slurry streams, effecting the separation of the solids contained in them.

The operating principle of the machinery is based on the separation of substances with different densities by exploiting the centrifugal force generated by high rotational speeds, increasing the settling speed of the solid particles contained in the slurry. The slurry stream is pumped inside the decanter centrifuge through an inlet placed in the middle of the machinery. The feed goes into a horizontal rotating bowl composed of a cylindrical part and a conical part. The separation take place inside the cylindrical part: since the solids has a higher density, they tend to collect on the wall of the drum. A screw conveyor collects the solids and pushes them towards the conical part of the machinery where they are also subjected to an increasing compression, facilitating the release of additional liquid. The solid therefore comes out of the exit of the conical section, while the liquid extract is collected on the opposite side.

In the case under analysis it is not necessary to separate the solid with a low moisture content, but rather the separation efficiency of the centrifugal decanter must be set in such a way as to ensure that the solid is perfectly soaked. The outlet liquid content is taken slightly higher than the imbibition value of the solid. The solid is therefore fed by gravity perfectly soaked inside the dosing hoppers of the PCF-20X progressive cavity feeders, with a moisture content of about 60%.

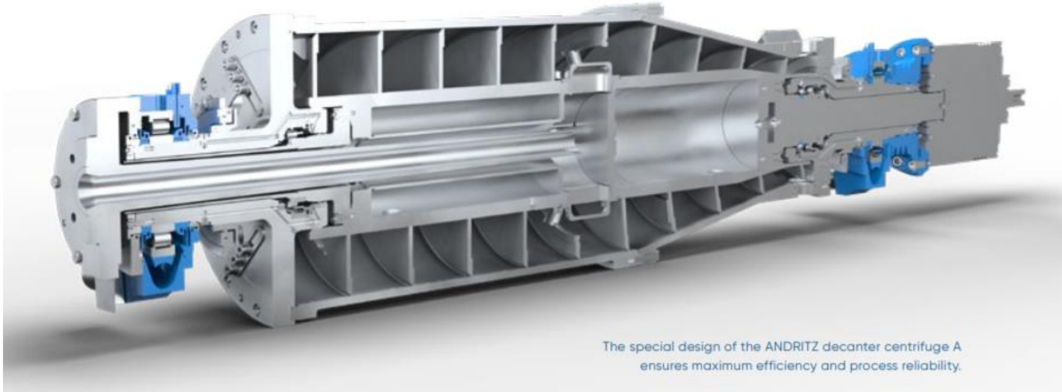

The special design of the ANDRITZ decanter centrifuge A ensures maximum efficiency and process reliability.

Figure 5.4 - View af the internals of an ANDRITZ decanter centrifuge A (38).

5.2.4.4. Progressive cavity feeder PCF-20X

The wet solid coming from the decanter centrifuge is collected by gravity into the collection hoppers of the feeding system of the extractors. The collection hopper must be sized to have a hold-up of 2-3 hours. For safety reasons, the collection hopper must be equipped with an upper closure able to avoid the accidental fall of solid bodies which, getting stuck between the stator and rotor of the TURBEX, could cause serious damage to the machine. A metal detector is also added in order to preventively identify the presence of metal bodies, such as screws or bolts accidentally dragged into the feed.

The feeder **PCF-201** consists of a food grade progressive cavity pump. This type of pumps is a kind of positive displacement pump, mainly constituted by a helical stainless-steel rotor which turns inside a helical stator made of a resilient elastomer.

The rotor tightly seals against the stator while it rotates, creating a series of small, fixed shape, discrete cavities. The pumping of the fluid takes place tanks to the formation of this cavities, leading to a volumetric flow rate proportional to the rotation rate of the motor.

It was chosen a Nova Rotors DX Series, which as the following characteristics: the drive is coupled directly to the pump via a flange. This solution is extremely cost effective and compact, considerably reducing installation costs and simplifying maintenance. The forces generated by the hydraulic part are supported by the drive itself. Each drive used is adequately selected based on their specific technical parameters and are subject to numerous duration tests with heavy loads (39).

To ensure the correct functioning of the feeder, it is necessary to send a small liquid current inside the pump in order to obtain a partial fluidification of the solids. Adding fresh water could alter the flow-dynamic operating principles of the extractor, also diluting the extract obtained: since the extracting liquid is fed in counter current with the solid, the outlet of the extract is located near the solid entrance. Therefore, if fresh water is fed together with the solids, it risks escaping from the extractor without taking part, if not to a limited extent, in the extraction process, leading to a decrease in the total efficiency of the TURBEX. To avoid this, it was decided to recirculate a small flow rate of liquid extract inside the feeder, to maintain the flow-dynamic conditions and the efficiency of the extractor almost unaltered.

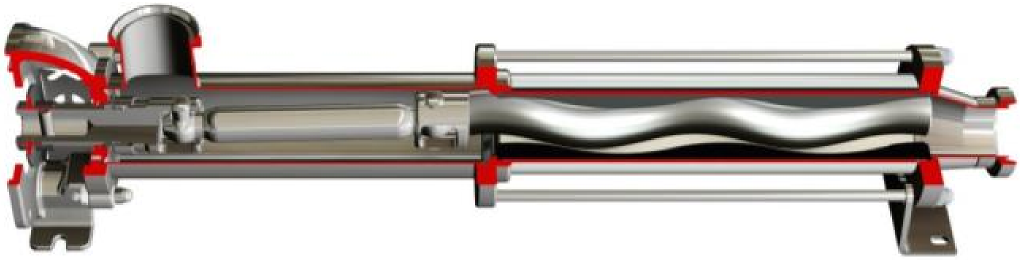


Figure 5.5 - Nova Rotors Progressive cavity pump DX series. (Picture from Nova Rotors "BROCHURE VANTAGGI DIAMOND" (39))

5.2.4.5. TURBEX EX30 extractors EX-2XX

To model the extraction process, equilibrium data relating to the extraction of oleuropein and extractable substances, using water at a temperature of 50 ° C as solvent, were used as a reference. The choice to use only water as a solvent is a choice aimed at reducing the environmental impact and costs related to the process.

As described in the previous chapters, typically the extraction processes are carried out using hydroalcoholic solutions or other solvents that have a better affinity with the solutes of interest and therefore make the extraction process more efficient. Thanks to the high extraction efficiency of the TURBEX EX30 comparable, if not better, results can also be achieved simply by using only water as the extraction solvent.

The extraction process has been modelled considering that the TURBEX is able to perform, in a single step, seven stages of counter current equilibrium. After having calculated the equilibrium approach constant from the data on the single conventional stage, it was therefore possible to carry out the mass balances for the seven stages of the machinery assuming that equilibrium approach constant doesn't vary from stage by stage. The used procedure is very similar to that one discussed in Chapter 3.

Once the mass balances were set, it was possible to evaluate the optimal L/S ratio for the process, keeping the extraction yield high, but trying to use the least amount of water possible. The extraction yield of the process is based on the quantity of oleuropein extracted in the liquid extract, compared to the initial amount present in the leaves. The optimal value chosen is a liquid over solid ratio equal to 3, which assure an extraction yield of about 93%.

To keep the TURBEX efficiency high, exploiting all the process intensification mechanisms that characterize the machine, it was decided to use extractors with a capacity of 500 kg/h. Considering that the flow rate of leaves to be treated is about 5,8 tons per hour, this leads to the need of using a battery of 12 TURBEX operating in parallel. Moreover, it was deemed necessary to add one more TURBEX to be kept in stand-by and ready to use if there is the need to carry out maintenance operations, assuring so the continuity of the process.

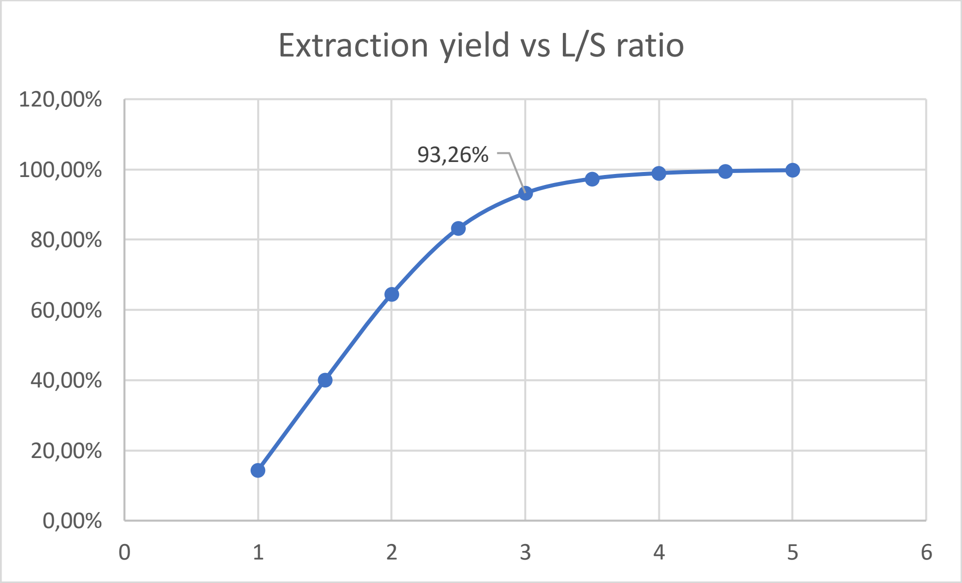


Figure 5.6 - Trend of the extraction yield as the L / S ratio varies

5.2.4.6. Squeezing presses **P-20X**

Spent leaves leaving the extractors from the solid outlet holes are collected by gravity inside hoppers connected to a food grade squeezing press **P-20X**. Each TURBEX is equipped with its own squeezing press. They consist of a screw press that has the purpose of recovery a part of the soaking liquid trapped inside the solids, reducing their water concentration up to a value of 50%. The degree of squeezing of each press is manually adjusted by opening a handwheel valve located near the solid outlet.

The solid squeezed out of the squeezing press is collected by gravity inside transport cart and subsequently collected manually to be then transferred to the CHP generator.

5.2.4.7. Squeezed liquid collection tank TK-201

The liquid recovered from each press is conveyed to the collection tank **TK-201**, from where it will be recirculated to the extractor and used as part of the extractant water. The liquid is pumped out of the tank by means of the volumetric sludge pump **P-201**.

The liquid outlet pipe of the tank must be protected by inserting a static filter that avoids the possible entrainment of solid bodies that could cause serious damage inside the extractor.

5.2.4.8. Liquid extract filters PF-201 and PF-202

The extracts of each TURBEX are collected and conveyed to a filtering system consisting of two bag filters (**PF-201** and **PF-202**) working alternatively on parallel, which have the function of capturing the solid particles entrained by the liquid during extraction.

The bags work one at a time: as soon as the liquid passing through it undergoes a pressure drop limit value, a signal is received that certifies that the filter needs to be changed. At this point a manual valve must be activated, directing the flow of extract to the other filter bag. The filter bag containing the solid separated from the extract must be emptied manually, returning the solid inside the collection hoppers of the feed system of the extractors.

5.2.4.9. Water heating system FH-201, B-201 and BL-201

The water required to perform the extraction, constituted by the squeezed water recycled and a stream of fresh river water, need to be heated up to a temperature of 50 °C. The heating of this water is carried out in the natural gas-fired boiler **FH-201**. It is constituted by the burner **B-201** and a centrifugal fan **BL-201**.

5.2.5. Concentration section

5.2.5.1. Mechanical Vapour Recompression Evaporator (MVR) EV-301

The liquid extract obtained in the extraction section is collected inside the **TK-301** insulated tank. The first stage of the concentration section is carried out inside a mechanical vapor recompression evaporator, which has the purpose of reducing the water content of the extract, taking it from 92,33% up to 70%, so as to allow an easy distribution inside the DD-301 Drum Dryer.

The evaporator consists of a vertical heat exchanger with short tubes (calandria type), inside which the liquid to be concentrated is made to flow. The tubes are heated externally by a current of steam. At the outlet from the exchanger, the biphasic mixture obtained is sent to a separation chamber, where the steam produced is separated from the concentrated extract. Mechanical vapour evaporators operate on a "heat pump" principle. The evaporated water vapor is recompressed with a simple, low-speed centrifugal fan which increases the saturation temperature of the vapor. After the fan, vapor can be used as heating steam in the same unit (40).

This technology allows to reduce the operating costs of the machinery, considerably reducing the environmental impact of the evaporation process and the amount of steam and cooling water to be used compared to conventional evaporative technologies.

The inlet temperature of the current to be concentrated is very important to allow the evaporator to self-sustain. For this purpose, the **E-301** heater is used, which brings the current fed to the evaporator to the target temperature.

5.2.5.2. Economizer E-301

The E-301 economizer has the purpose of recovering part of the sensible heat available from the condensate stream collected from the evaporator **EV-301**. The recovered heat is used to pre-heat the diluted extract stream fed to the extractors up to a temperature of 65°C. The design of the heat exchanger is carried out by ensuring a water outlet temperature of 100°C, so as not to reduce too much the temperature of the hot stream sent to the condenses recovery system.

5.2.5.3. Drum Drier DD-301

At the outlet of the **EV-301** evaporator, the extract stream needs to be further concentrated until it reaches a mass water content of 2% and the appearance of a powder. To carry out this operation, it was decided to use a low-pressure steam powered Drum Dryier, in which the contact between the utility and the process current occurs indirectly.

The chosen drum dryer configuration is equipped with two rotating cylinders inside which the steam is sent. The material in the form of a paste is distributed evenly on the surface of the rotating drums, heated by the contact with the steam.

Oleuropein has a degradation temperature of about 120 ° C, therefore to prevent the temperature of the extract from rising beyond this value, it was decided to send saturated steam to the drum with a pressure of 2 bar instead of 6 bar, guaranteeing however a time of limited contact in order to avoid possible thermal degradation of the product.

5.3. Process control logic

Pre-treatment and storage section:

- High- and low-level control of the **H-102** hopper manipulated by opening or closing the servo-assisted valve located in the vertical drop line of the solid from the **H-101** dosing hopper;
- Weight control in the **H-102** dosing hopper by means of the load cells on which it rests by acting on the rotation speed of the rotary feed valve **RFV-101**;
- Control of the temperature of the output gas current from the **DD-101** Rotary Drum Dryer by manipulating the methane flow rate sent to the open-back air draught burner **B-101**;
- Control of the air flow fed to the burner **B-101** manipulating the air flow draught from the outside through the blower **BL-101**;
- Control of the cooled solid temperature coming from the fluidized bed cooler **FBE-102** by manipulating the air flow draught from the outside through the blower **BL-102**;
- Control of the temperature of the hot air stream outgoing from the economizer **E-301** by manipulating the bypass valve of the stream;
- Weight control in the **H-103** dosing hopper by means of the load cells on which it rests by acting on the rotation speed of the rotary valve which doses the quantity of leaves to be inserted in each big bag.

Extraction section:

- High- and low-level control of the screw-conveyor **SC-201** hopper manipulated by opening or closing the servo-assisted valve located in the vertical drop line of the solid from the **H-101** dosing hopper;
- Weight control of the hopper of the screw-conveyor **SC-201** by regulating the rotational speed of the motor of the screw;

- Control of the level of the stirred tank **MX-201** by manipulating the number of revolutions of the pump **P-201**;
- Control of the weight of the collecting hopper of the progressive cavity feeders **PCF-20X** acting on the rotational speed of its motor;
- Control of the ratio between the solid flow and the extractant flow feed to the extractors **EX-20X** by manipulating the flow of the river water fed;
- Control of the temperature of the water fed to the extractors by manipulating the methane flow rate fed to the burner **B-201**;
- Control of the methane flow rate fed to the burner **B-201** to regulate the flow of air to be fed through the blower **BL-201**.

Concentration section:

- Flow control of the liquid outlet stream of the tank **TK-301** by manipulating the number of revolutions of pump **P-301**;
- Control of the temperature of the outgoing hot water stream from the economizer **E-301** by manipulating the bypass valve of the stream.
- Temperature control of the inlet extract stream of the evaporator **EV-301** by manipulating the inlet flow rate of the steam;
- Level control of the liquid inside the separation chamber of the evaporator EV-301 by manipulating the number of revolutions of the pump **P-302;**
- Flow control of liquid outlet stream of the tank **TK-302** by manipulating the number of revolutions of the pump **P-303;**
- Temperature control of solid extract by manipulating the drum dryer **DD-301** inlet flow rate of steam.

Chapter 6

BSG valorisation plant

Beer is one of the oldest drink humans have produced: the first established sources date back to 7000 years ago in Iran and in the following centuries, around 4500 BC, the first written records were found that explicitly mentioned beer and the craft of the brewer in Mesopotamia (41). In these cultures, there were already different varieties of beer, and each people had developed their own recipe, starting from germinated and cooked barley loaves from which the fundamental ingredient was obtained: malt. The importance of beer was such that it was considered a national drink in Egypt, while in Italy it acquired increasing importance after the Etruscans introduced it into Roman culture.

As the centuries advanced, the production of beer found decisive innovations, especially during the first industrial revolution, in which tools were invented that, although simple, were fundamental in the industrialization of beer production (42). Among these are mentioned: the thermometer, the densimeter, with which it became possible to evaluate the use of different types of malt even more valuable, and the coffee roaster. In addition to technological studies, microbiology studies were relevant, around the mid-nineteenth century the importance of yeasts for the control of fermentation was discovered, but the most important advances are seen starting from the mid-twentieth century with improvements in the field of refrigeration systems, which have enabled the production and distribution of beer on a global scale.

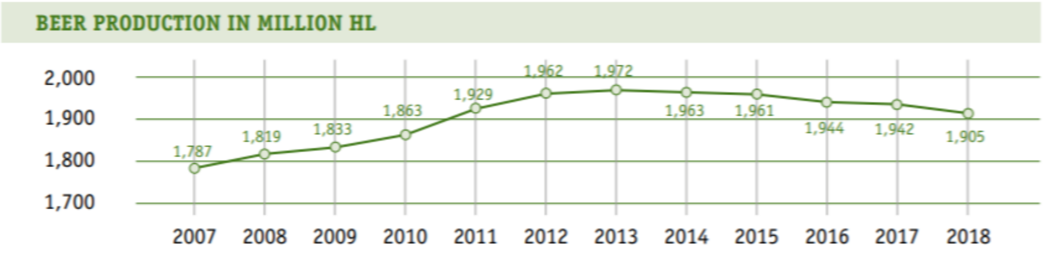


Figure 6.1 - Trend of beer production in the world from 2007 to 2018 (43)

Nowadays, beer is highly widespread, according to data from The Barth Report, published in 2019, world beer production in 2018 was 1,905 million hectolitres (43). From 1998 to today, this figure has had a strongly growing trend, with a slight decrease in recent years due to the lower production recorded in the largest importing country: China, with a decrease of 59 million. The European countries, the United Kingdom, Romania and Russia, maintain an increasing trend, with figures that average between one and three million hl, Mexico with 9.7 million hectolitres and Brazil with 1.4 million hectolitres (43).

Given the numbers involved, it is not difficult to imagine that the brewing industry generates relatively large amounts of by-products and residues, in particular spent grains, spent hops and yeast are the most common (44). Spent grain represents the most abundant brewing by-product, accounting to a total of 85% mass based of total by-products generated (45). It was demonstrated that spent grain constitutes approximately the 30% of the total weight of the malt, representing approximately 20 kilogram per 100 litres of beer produced (46). It has been estimated that the production of BSG in the world is in the order of 40 million tons per year (47).

During the preparation phase for brewing, harvested barely is cleaned, graded and subjected to a dormancy period that can vary from four to six weeks. After that, barely is malted in a controlled germination process to increase the enzymatic content of the grain (44). Malted barley is milled, mixed with water inside a mash tun and subjected to a gradual and slow increase of temperature to promote enzymatic hydrolysis of its constituents. This stage is named mashing and produces a sweet liquid known as wort. The insoluble part of the malted barley is allowed to set inside the mash tun and the sweet worth is filtered through it. The filtered worth is collected and sent to the fermentation section to carry on the brewing process process, while the residual solid part is what takes the name of brewer’s spent grain.

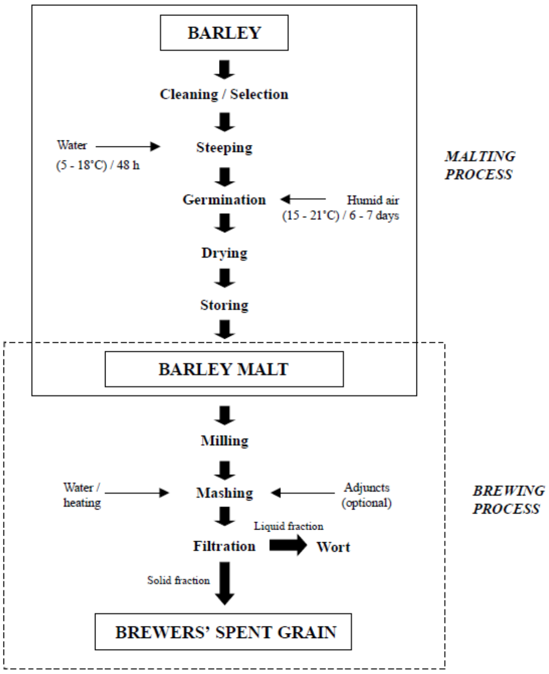


Figure 6.2 - Schematic representation of the process to obtain BSG from natural barely (44).

BSG represents a by-product readily available in large volumes, at very low or no cost, available almost everywhere in the world: about 3,4 million tonnes of BSG are produced from the European brewing industry every year (48). It basically consist of the husk-pericarp-seed coat layers that covered the original barely grain, which are rich in cellulose (17%) and non-cellulosic polysaccharides (28%) and lignin (28%), containing also some proteins (18-30%) and lipids (10%). The husk also contains silica and a considerable part of the polyphenolic components of the barely grain (44). Due to its composition, it represents a potentially valuable resource for industrial exploitation.

During the last years several attempts have been made in utilizing BSG as potential source of high added value substances, instead of considering it just a waste to manage. For several years it was used as feed for animals, in particular for ruminants, due to its low cost and interesting residue nutritional value. Anyway, since it is derived from raw materials and processes utilized for food-like products for humans, it can find more valuable application in the human food industry recovering valuable constituents, reducing also the environmental impact of the brewing industries.

The most interesting components present in BSG are lipids, polyphenols, proteins and arabinoxylans.

Lipids constitute approximately the 10% of the brewer's spent grain, consisting mostly of triglycerides, with an important amount of free fatty acids (49). They found interesting applications as a valuable source of phytochemicals to be used in the pharmaceutical, food, cosmetic and biorefinery industries.

Phenolic compounds are of considerable interest to scientists, manufacturers and consumers due to their influence on food quality and their protective and preventive roles in some chronic diseases (50). The two most important representative components of the polyphenolic fraction of BSG are Ferulic and *p*-coumaric acids.

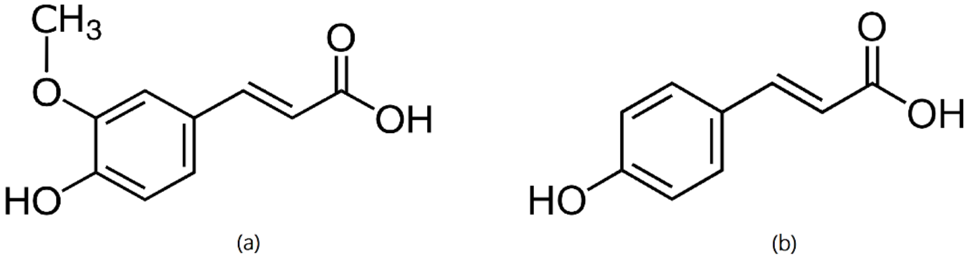


Figure 6.3 - Ferulic (a) and p-coumaric (b) acids molecular structures

Ferulic acid is considered as one of the most important phenolic acids: it exhibits many physiological functions such as anti-oxidant, anti-microbial, anti-inflammatory, anti-thrombosis and anti-cancer activities (50). Because of these properties, it is receiving increasing attention from the pharmaceutical, cosmetic and food industries. Like Ferulic, also the *p*-Coumaric is a phenolic acid of great interest due to its antioxidant and chemoprotectant properties (51).

The BSG protein composition can vary from about 18% to 30% depending on the raw materials, adjuncts and brewing process used by the industry (52). The trend of global protein demand, because of the growing global population, has increased continuously during the last decades and more attention is paid to the search for alternative protein sources to traditional ones. For this reason, several studies have been conducted during the last years to determine the protein content of BSG and find the best ways to extract a protein fraction with high added value. The protein fraction of the BSG consists of about 50% of hordeins, followed by glutenins (53).

Arabinoxylans AX constitute the major hemicellulose present in brewer's spent grains, very complex molecules composed by a backbone of β-(1→4)-linked xylopyranose in part substituted with single units of arabinofuranose at position 2, 3 or both (54). They are considered as dietary fibers due to their resistance to hydrolysis by enzymes present in the digestive tract (53) and are particularly interesting for the food industries to produce dietary fibers supplements. It has been suggested that dietary fibers can aid in numerous disease such as gastrointestinal disorders, cancer, diabetics and coronary heart disease (55).

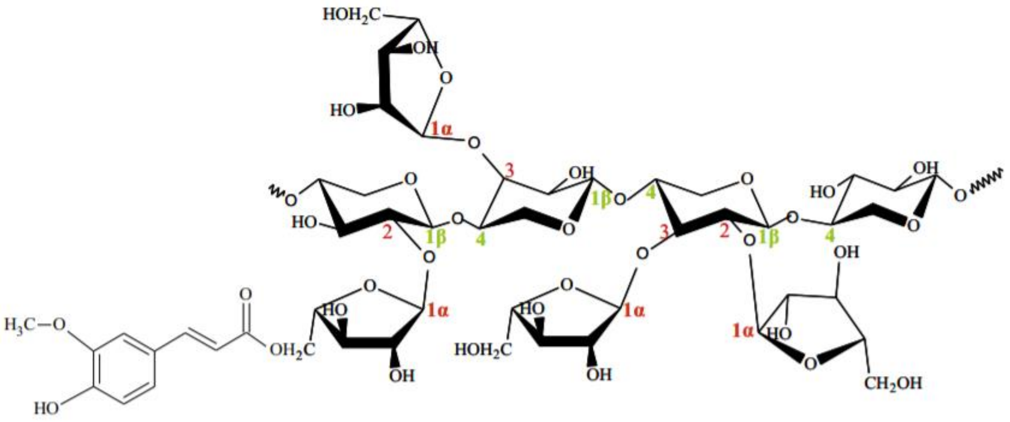


Figure 6.4 - Arabinoxylans molecular structure (56)

In light of the interesting substances contained in the BSG and the growing interest that the scientific community is showing in the last years, it was considered interesting to design a plant for the valorisation of BSG using TURBEX EX30 as the main extractor, with the aim of extracting a lipid fraction, a rich polyphenolic fraction, a protein fraction and an arabinoxylans fraction. The designed extraction plant will be described in the following paragraphs.

6.1. Process Description

6.1.1. Targets

The purpose of the process is the valorisation of the Brewer's Spent Grain (BSG), performing a series of extraction processes with the aim of recovery four added value components and put them on the market. In order it will be recovered a lipidic fraction, a polyphenolic fraction with high concentration of Ferulic and *p*-Coumaric acid, a protein and an arabinoxylans fraction.

With a view to designing a plant adopting more innovative technologies than traditional plants, exploiting also new perspectives of process intensification, it was decided to carry out the extraction processes using the TURBEX EX30 as the main extractor.

6.1.2. Plant Overview

The plant was designed to process 20.000 tons per year of brewer's spent grain. The production of beer is not subject to seasonality, so it is estimated that the BSG is available during the whole year and it is supposed that the plant can work continuously for a total of approximately 49 weeks. The project relates to a plant located in Siberia, in the city of Novosibirsk in whose areas there are several nearby breweries.

The entire plant can be divided in three main sections:

- Pre-treatment and storage
- Extraction of lipids and polyphenols
- Extraction of proteins and arabinoxylans
- Ethanol and citric acid recovery

In the first section, the drying process of the raw BSG is carried out to allow its correct storage with no risk of occurring any fermentation phenomena. In the second section it's performed first the extraction of lipids using n-hexane as solvent and then the extraction of polyphenols using a water-ethanol solution. In the third section is carried out the extraction of proteins and arabinoxylans through a series of three extraction performed using a solution of sodium hydroxide with increasing concentration plus a final extraction with water. The purpose of the last section is instead to recover the ethanol and citric acid used in the various stages of the process. The plant was designed to operate with a turn-down between 60% and 110% compared to the project conditions.

Table 6.1 - BSG valorization plant characteristics

Supply type	Brewer's Spent Grain	
Plant capacity	20000	*ton/year*
Plant operation period	49,0	*weeks/year*
Days a week	7	*days/week*
Hours a day	24	*h/day*
Hourly feeding MAX	2,66	*ton/h*
Hourly feeding MIN	1,45	*ton/h*
Turndown Ratio MIN	0,6	
Turndown Ratio MAX	1,1	

SUPPLY:			
Wet BSG Feedstock		20000	ton/y
Dry BSG Feedstock		5454	ton/y
PRODUCTS:			
Lipids		442,6	ton/y
Polyphenols powder		93,6	ton/y
Proteins powder		1245	ton/y
Arabinoxylans Dietary Fibers powder		1160,9	ton/y
RESIDUES:			
Spent wet BSG		5085	ton/y
	Destination:	CHP generator	
6% NaCl brine		26558	ton/y
	Destination:	Desalination units	

6.1.3. Matrix Characterization

The supply consists of wet brewer’s spent grain with the following average composition:

Table 6.2 - BSG supply composition

Water	76,000	$g/100g_{wet}$
Cellulose	5,208	$g/100g_{wet}$
Hemicellulose	7,104	$g/100g_{wet}$
Insoluble lignin	2,304	$g/100g_{wet}$
Proteins	5,904	$g/100g_{wet}$
Polyphenols	0,456	$g/100g_{wet}$
Lipids	2,064	$g/100g_{wet}$
Ash	0,960	$g/100g_{wet}$

The estimated BSG temperature is of 10 °C and the water content is of 76%, considerations based on the average atmospheric conditions of the collection site, assumed to be in the same region where the plant is located.

6.1.4. Product Characterization

The plant produces four products of valid commercial interest: lipidic, polyphenolic, protein and arabinoxylans fractions.

The lipidic fraction is obtained as a viscous liquid with a purity of 91,6%, a water content of approximately 2% in weight and a residue of hexanol lower of the 0,004%. The mass composition is shown in Table 6.3.

The process can obtain an amount of around 54,6 kilograms per hour of lipids (45 tons per year). The estimated commercial value of this product is of about 1 €/kg. It can be used in interesting applications as a valuable source of phytochemicals for the pharmaceutical, food, cosmetic and biorefinery industries.

Table 6.3 - Oil fraction weight composition

Water	2,262%
Polyphenols	6,075%
Lipids	91,659%
Hexane	0,004%

The polyphenolic fraction consists of a powder, with a purity of about 53,22% and a moisture content of about 2%. It's estimated that approximately the 74,3% of the polyphenols contained in the extract is constituted by Ferulic acid, thus leading to a concentration of the 39,54% of the weight of the product, characteristic that gives to the extract a greater commercial value. The residual part is constituted of water-soluble substances of low value consisting mainly of sugars, carbohydrates and proteins.

The process can obtain an amount of around 11,35 kilograms per hour of polyphenols powder (93,6 tons per year). The product has an estimated value of 50 €/kg and it can be used in the food sector as a fundamental component of food supplements, or in many other applications such as in the pharmaceutical or cosmetic companies.

Table 6.4 - Polyphenols fraction weight composition

Water	2,00%
Cellulose	2,71%
Proteins	42,06%
Polyphenols	53,22%

The protein product consists of a powder with a purity of approximately 90% and a water content lower than 2%. The remaining part is constituted by hemicellulose, cellulose and ashes.

The process can obtain an amount of around 150,92 kilograms per hour of protein powder (1245 tons per year). This product, with this high purity, ranks in a high price phase in the protein market, with a commercial value of about 2,5 €/kg. It finds applications mainly in the food sector for the preparation of protein supplements, snacks or food in general.

Table 6.5 - Protein fraction weight composition

Water	1,96%
Cellulose	0,68%
Hemicellulose	6,00%
Insoluble lignin	0,41%
Proteins	90,60%
Polyphenols	0,01%
Ash	0,32%

The arabinoxylans product fraction is obtained also in powder form, with a purity of 83% and a water content of approximately 2%. The remaining part is mainly constituted by cellulose and insoluble lignin, which are however part of the contribution of dietary fibers provided by the product. Proteins, polyphenols and ash are present in small amounts.

The process can obtain an amount of around 140,72 kilograms per hour of arabinoxylans (1161 tons per year). The commercial value of the product is of about 10 €/kg and it can be used in the food industry to produce vegetable dietary fibre supplements.

Table 6.6 - Arabinoxylans fraction weight composition

Water	1,92%
Cellulose	8,54%
Hemicellulose	83,10%
Insoluble lignin	5,63%
Proteins	0,60%
Polyphenols	0,08%
Ash	0,13%

According to the price estimates made and the productivity obtained by the plant, an annual value of products equal to 105.454 €/y for the lipid fraction, 4.679.955 €/y for the polyphenols powder, 3.112.647 €/y for the protein powder and 11.609.000 €/y for the arabinoxylans powder, account for a total of 19.844.100 €/y.

Table 6.7 - Commercial value of the products obtained and annual turnover resulting from their sale

PRODUCTS COMMERCIAL VALUE:		
Lipids	1	€/kg
High Ferulic acid concentration PPs powder	50	€/kg
Proteins powder	2,5	€/kg
Arabinoxylans Dietary Fibers powder	10	€/kg
PRODUCTS SALES TURNOVER:		
Lipids	442.600	€/y
Polyphenols powder	4.680.000	€/y
Proteins powder	3.112.500	€/y
Arabinoxylans Dietary Fibers powder	11.609.000	€/y
TOT	19.844.100	€/y

6.1.5. By-products Characterization

The main by-product of the process consists of exhaust brewer’s spent grain with a moisture content of about 65%. It is send to a combined heat and power generator to produce energy, increasing the intensification of the process, reducing the volumes of waste and recovering energy.

Table 6.8 - Spent BSG weight composition

Water	65,12%
Cellulose	16,07%
Hemicellulose	7,27%
Insoluble lignin	7,65%
Proteins	0,11%
Polyphenols	0,11%
Lipids	0,00%
Ash	3,68%

Another by-product that can be considered is sodium chloride resulting from the neutralization reaction that takes place between hydrochloric acid and sodium hydroxide. It is obtained in a 6% by weight aqueous stream after treatment in a decanter used for the recovery of citric acid.

6.1.6. Pre-treatment and storage section

The raw material supply consists of wet BSG with a nominal capacity of 2,42 tons per hour and a moisture content of 70% in weight. Due to its high moisture and fermentable sugar contents, it is a very instable material and it is subject to deteriorate rapidly due to microbial activity (44). To allow the temporary storage of the BSG without incurring deterioration phenomena of the matrix, it must first be dried up to a moisture content of 10%.

It was decide to perform the drying in two steps: first the moisture content is reduced up to about 60% in weight by pressing, and then it is dried using a rotary drum dryer up to a moisture concentration of 10% (57). Furthermore, drying is a process that also leads to the secondary benefit of reducing the volume of the raw material, reducing also storage costs.

It was chosen to operate the drying with a hot air stream heated using an air vein burner. This solution, widely adopted industrially, allow to heat the air stream and easily control its temperature, also it allows to carry out the drying operation very quickly. Using an air vein burner, it’s possible to control the air temperature, setting it high enough to allow a rapid drying, but not too high to avoid the degradation of the substances of interest. For this plant it was decided to send air at an inlet temperature of 200 °C.

The whole drying process leads to the removal of approximately 1,7 tons per hour of water, reducing the BSG capacity from 2,42 tons per hour wet to 661 kilograms per hour dry. The final composition of BSG at the end of the drying section of the plant is shown in Table 6.8.

Table 6.9 - Dried BSG composition

Water	12,00%
Cellulose	19,10%
Hemicellulose	26,05%
Insoluble lignin	8,45%
Proteins	21,65%
Polyphenols	1,67%
Lipids	7,57%
Ash	3,52%

The dried BSG is stored inside a silo, at the exit of which it is finally subjected to a grinding process using a hammer mill until it reaches an average diameter of 0.5 mm. The ground and dried BSG is then sent to the first extraction section by means of a screw conveyor.

6.1.7. Lipids and Polyphenols extraction section

The first class of components that is separated are lipids. The extraction is performed using n-hexane as extractant solvent. Hexane is a non-polar, immiscible with water and very volatile molecule with a boiling temperature of 69 °C. For these reasons, it is very easy to recovery both from the solid residue and the liquid extract, allowing to reach very high recovery yields. Due to its high affinity with lipidic compounds and its intrinsic characteristics it is a widely used solvent in the extraction industry.

The extraction is carried out using the TURBEX EX30 as the extractor. The capacity of dry BSG to be extracted is of about 660 kilograms per hour. Considering the intrinsic characteristics of the machinery, in order to make the most of all the process intensification phenomena that take place inside it, it is desirable to split the feeding into two separate TURBEX, each with a nominal capacity of 500 kg/h of dry solid to be treated.

The feeding of the solid to the TURBEX represents a critical part of the entire extraction phenomenon. Especially in the case of non-powdered solids, the lack of wetting of the solid can lead to the formation of agglomerates that tend to plug and block the screw feeder, interrupting the continuity of the process. For this purpose, it is necessary to carry out a complete wetting of the solid before feeding it, but in order not to change the counter-current operation of the machinery, this is done using the extract obtained rather than fresh liquid.

The dry BSG is then unloaded directly into a stirred tank where the liquid extract is also conveyed. From there the slurry stream containing the extract and the solid is sent to a decanter centrifuge which separates on one side a stream of completely wetted solid and on the other side a stream of extract. The wetted solid is then sent to the extractor, while the liquid extract is filtered inside a filter bag system and send to the following equipment.

The extraction process with n-hexane is not perfectly selective: hexane not only removes the lipid fraction present in the BSG, but also a fraction equal to about 30% of the polyphenols present in the solid matrix is dissolved in the extract.

The hexane is removed from the extract thanks to a distillation process followed by a stripping process with a stream of low-pressure steam. The stripper bottom stream is the lipid product ready to be placed on the market.

The hexane is recovered also from the after-extraction solid by the means of a nitrogen stream sent at 115 °C inside a flash desolventizer. The solid is then send to the second extraction devices while the gaseous stream is sent first to a rotoconcentrator and then to a direct condensation column of the hexane to allow the recovery of both nitrogen and hexane.

In the same section is performed also the extraction of polyphenols using a solution of water-ethanol 70% by volume at a temperature of 60 °C. The solid BSG coming from the desolventizer must be wetted with the solution before being fed to the extractor. To perform this operation is used a system with a stirred tank and a decanter centrifuge similar to the one used in the previous extraction process.

The extraction solution does not selectively extract the polyphenols only, but also a small amount of reducing sugars (22,8 mg/g_{BSG}) and proteins (8,06 mg/g_{BSG}). In the extract stream obtained, the polyphenolic fraction is lower in concentration than the sugar fraction (11,45 mg/g_{BSG}). For this reason, before the dewatering of the stream, it is necessary to carry out a process of concentration of polyphenols using a membrane filtration system. In particular, the liquid extract is compressed up to a pressure of 5 atmospheres and pumped first through an ultra-filtration membrane system and then to a nano-filtration membrane system. Even if the process leads to a small loss of polyphenols, at the end of the membrane system the concentration of polyphenols results to be the highest compared to that of the other solutes and the finished product will reach a higher commercial value.

Table 6.10 – Concentration expressed in mg/g_{BSG} of the solutes present in the extract after filtration with membranes

Polyohenols	9,93	*mg/g (BSG)*
Sugar	0,51	*mg/g (BSG)*
Proteins	7,85	*mg/g (BSG)*

The extract stream is sent to a system of two co-current evaporators to remove the remaining part of the solvent, recovering the 99,64% of the ethanol used and increasing the solutes concentrations up to 22%. The final drying of the product is carried out using a drum dryer that bring the product to the desired moisture content of 2%.

The residue solid is sent to a four-stage counter current washer to remove the ethanol remained in its soaking liquid. After washing, the solid is lightly squeezed and sent through a belt conveyor to the following extraction section.

6.1.8. Protein and Arabinoxylans extraction section

The last extraction stage of the process is a multistage extraction performed according to the patent WO 2012/069889 A1 (58). The purpose is to obtain a protein rich and an arabinoxylans rich powder extracts using an integrated process of simultaneous extraction of proteins and arabinoxylans using alkaline solutions with increasing concentration.

At this point of the process the BSG's composition is mainly lignocellulosic containing a high mass concentration of proteins on a dry basis. Extracting the solutes directly from a solid matrix with these characteristics is not easy to do.

Several studies reported in the literature show how the use of alkaline solvents can improve the extraction efficiency thanks to the occurrence of the phenomenon of alkaline hydrolysis, which is able to easily dissolve lignin (50). This procedure allows to better solubilize also the arabinoxylans which are mostly not extractable with water (56).

The residue brewer's spent grain arrives with a capacity of 1,2 tons per hour completely wet (60% of water) from the previous section, therefore this time it is not necessary to pre-wet it with the extract as in the previous cases, allowing to feed it directly using the feeders installed on the extractors.

In the first three stages the BSG is extracted with three sodium hydroxide solutions with increasing concentration: 0,1 M in the first extraction, 0,5 M in the second extraction and 4 M in the third extraction. For each stage the solid is sent to the following extraction step, while the extract, after filtration, is sent to a stirred tank where citric acid is added up to reach a value of pH equal to 3. The acid condition thus created allows the separation of proteins which is completed inside a decanter where a liquid residue rich in proteins is collected from the bottom and the liquid extract rich in arabinoxylans is collected by surfacing and sent to a subsequent stirred tank.

The extract rich in arabinoxylans is further acidified with the addition of hydrochloric acid until reaching a pH of 2, to allow the precipitation of the arabinoxylans. Furthermore, to avoid the precipitation of citrates, it is necessary to add also a solution of water-ethanol (70% v_{EtOH}/v) in a quantity that must be 70% of the total volume. The solution obtained is then decanted and a stream reach in AX is collected from the bottom of the decanter, while the liquid stream containing citric acid and ethanol is sent to the solvents' recovery section. This procedure is repeated for all the three alkaline extraction stages.

The solid residue of this three-stages extraction is now mostly made of ashes, cellulose and lignin but to maximize the yield of the process a further extraction process is performed. The residue is subjected to a final extraction with water performed using the TURBEX. The residual solid is then collected and sent to the combined heat and power generator, while the extract is concentrated using a membrane nanofiltration system and a series of two co-current evaporators. The membrane system allows to remove the 80% of the water contained in the extract, while the evaporators concentrate the extract up to a solutes concentration of approximately 55% in weight.

All the protein and arabinoxylans reach stream are then sent to two different tanks and the drying process of the products is completed inside two dedicated drum dryers.

6.1.9. Ethanol and citric acid recovery section

In the last section of the plant is carried out the recovery of ethanol and citric acid used in the various sections of the plant. All streams containing ethanol and citric acid are therefore conveyed into a collection tank.

From the collection tank a stream containing ethanol, water and citric acid is sent to a first distillation column which separates the ethanol solution at the top with a concentration close to that of the solution used in the plant (70% v_{EtOH}/v). This separation allows to obtain a bottom stream containing citric acid and a low concentration of ethanol. The ethanol stream is then sent to the storage tank of the ethanol solution, while the bottom stream is sent to a decanter to precipitate the sodium chloride formed by the neutralization reaction between sodium hydroxide and hydrochloric acid.

The solution containing citric acid, before decantation need to be diluted again with a water-ethanol solution up to reach a volume concentration of 40% $v_{solution}/v_{TOT}$. This is a necessary step because sodium chloride is not soluble in ethanol and its presence facilitate the precipitation of the salt. A waste stream containing sodium chloride is then collected from the bottom of the decanter and a stream containing ethanol and citric acid is collected by weir from the top of the decanter. The aqueous stream containing ethanol and citric acid is then sent to a second distillation column which separates the ethanol solution from the top, with a concentration close to that of the solution used in the plant (70% v_{EtOH}/v), and a diluted solution of citric acid at the bottom.

The water-ethanol solution is directly sent to its own storage tank, while the citric acid solution needs to be concentrated up to the desired value of concentration required in the plant operations (33,33$\%_w$). The citric acid concentration is performed in a single stage evaporator, at the outlet of which the concentrated stream is conveyed to its storage tank to be recycled.

6.1.10. Plant Flowchart

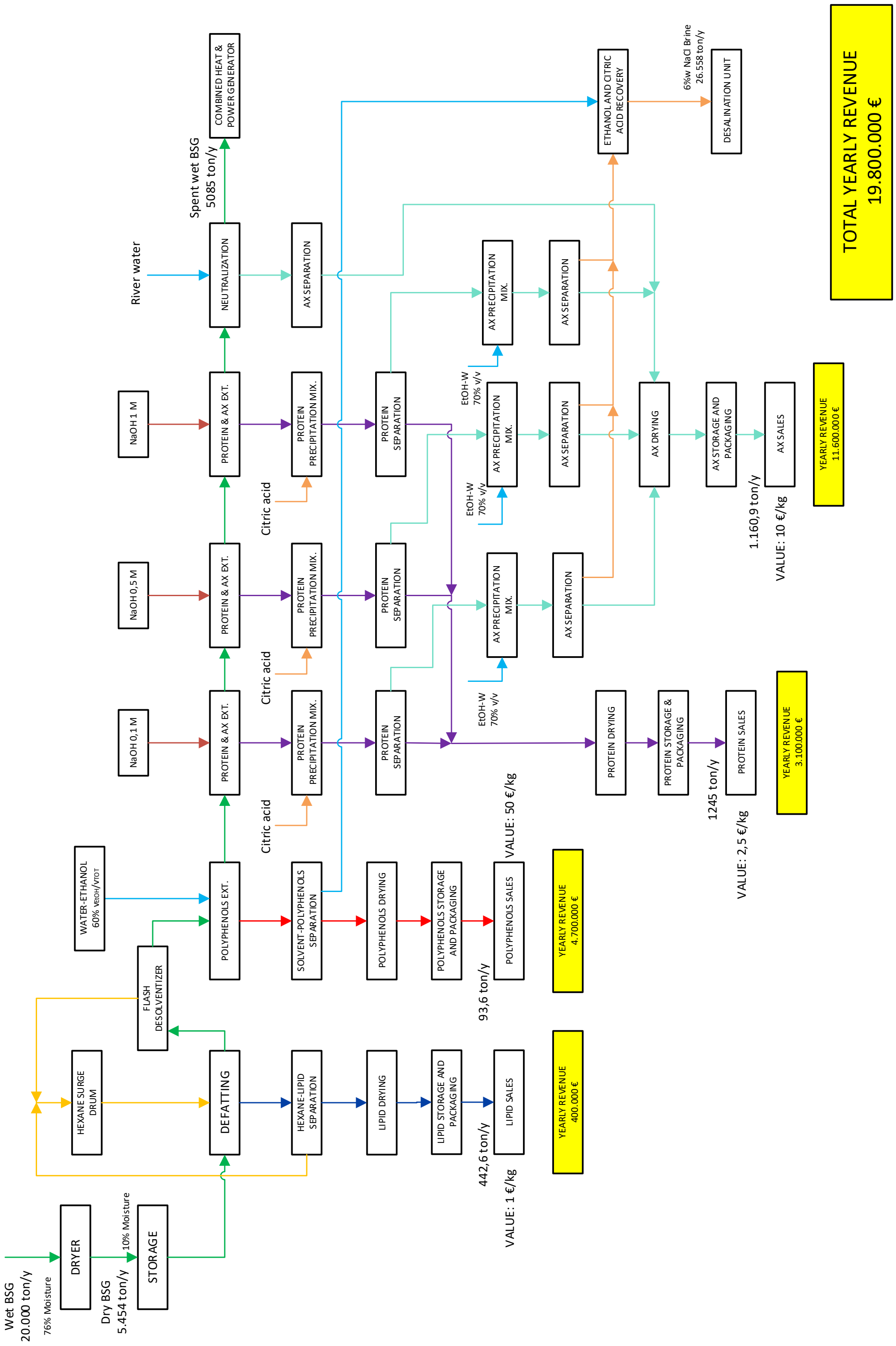


Figure 6.5 - BSG valorization plant flowchart

6.2. Design Basis

The description of the main equipment making present in the plant and the related design criteria will be described in this paragraph. In order to better understand what is reported in this section, it is recommended to consult the process flow diagram in Appendix C. It should be emphasized that the design of this plant stops at the basic conceptual design phase, with the only purpose of proposing a possible plant solution. The sizing of the individual devices was therefore not considered necessary and only the design criteria relating to their operation will be dealt in this paragraph.

6.2.1. Utilities characteristics

The utilities used in the plant are cooling water, river water, natural gas, low pressure steam, nitrogen and electric energy. Their characteristics in the production site are described in the Table 6.10.

Table 6.11 - Utilities characteristics

Utility	Characteristics		
Cooling water	Temperature IN	28	*[°C]*
	Temperature OUT	40	*[°C]*
	Pressure IN	0,4	*[Mpa g.]*
	Pressure OUT	0,25	*[Mpa g.]*
River water	Temperature	15	*[°C]*
	Pressure	1	*[bar]*
Natural gas	Pressure	0,36	*[Mpa g.]*
	LHV	32,49	*[MJ/Nm^3]*
Low pressure steam	Pressure	0,5	*[Mpa g.]*
	Temperature	158	*[°C]*
Nitrogen	Supply pression	0,6	*[Mpa g.]*
	Temperature	10	*[°C]*
Electric Energy LT	400 Volt (+/-10%)-3 Ph- 50 +/- 0,5 Hz		
Electric Energy MT	6000 Volt (+/-10%)-3 Ph- 50 +/- 0,5 Hz		

6.2.2. Utilities specific consumptions

In calculating the consumption of the various utilities, two versions are proposed: both the consumption of utilities actually required by the plant and the consumption considering the recovery of thermal and electrical energy, generated by the CHP generator, are indicated. In fact, the residual matrix from the extraction process is sent to a combined generation unit of thermal and electrical energy. According to experimental data, it is estimated that 1.25 kg/h of wood chips with 20% humidity can produce approximately 2,5 thermal kWh/h and 1 kWh/h of electricity in CHP units. Furthermore, it is assumed that the matrix in question has a calorific value equal to approximately 2/3 of that of the wood chips.

Table 6.12 - Utilities hourly consumptions

Utility	Hourly consumption (withot CHP)	Unit	Hourly consumption (with CHP)	Unit
Cooling water	948,03	$[m^3/h]$	948,03	$[m^3/h]$
River water	3,24	$[m^3/h]$	3,24	$[m^3/h]$
Natural gas	151,98	$[m^3/h]$	151,98	$[m^3/h]$
Low pressure steam	28859,32	*[kg/h]*	28566,13	*[kg/h]*
Nitrogen	0,75	$[m^3/h]$	0,75	$[m^3/h]$
Electric Energy	344,62	*[kWh]*	200,80	*[kWh]*

Table 6.13 - Utilities yearly consumptions

Utility	Yearly consumption (withot CHP)	Unit	Yearly consumption (with CHP)	Unit
Cooling water	7821224	$[m^3/y]$	7821224	$[m^3/y]$
River water	26765	$[m^3/y]$	26765	$[m^3/y]$
Natural gas	1253798	$[m^3/y]$	1253798	$[m^3/y]$
Low pressure steam	238089	*[ton/y]*	235671	*[ton/y]*
Nitrogen	6187	$[m^3/y]$	6187	$[m^3/y]$
Electric Energy	2843	*[MWh/y]*	1657	*[MWh/y]*

Table 6.14 - Utilities specific consumptions

Utility	Specific consumption (withot CHP)	Unit	Specific consumption (withot CHP)	Unit
Cooling water	2,66	$[m3/kg_{PROD}]$	2,66	$[m3/kg_{PROD}]$
River water	0,01	$[m3/kg_{PROD}]$	0,01	$[m3/kg_{PROD}]$
Natural gas	0,43	$[m3/kg_{PROD}]$	0,43	$[m3/kg_{PROD}]$
Low pressure steam	80,92	$[kg/kg_{PROD}]$	80,10	$[kg/kg_{PROD}]$
Nitrogen	0,002	$[m3/kg_{PROD}]$	0,002	$[m3/kg_{PROD}]$
Electric Energy	0,97	$[kW/kg_{PROD}]$	0,56	$[kW/kg_{PROD}]$

The total estimated electrical consumption of the equipment amounts to 344,2 kWh, from which it is subtracted the amount of electric energy obtained by the combustion of the residue BSG in a combined heat and power unit, equal to 143,8 kWh.

Since the sizing of the equipment was not carried out, nor their spatial arrangement assumed, it was not possible to evaluate the electrical consumption relating to pumps. Since the latter is not very relevant to the overall electrical consumption of the system, it was decided to neglect it in this first analysis stage. The consumption of electricity shown in the previous tables is therefore to be understood as decreased by the contribution of the pumps.

6.2.3. Pre-treatment and storage section

6.2.3.1. Underground storage tank S-101

Brewer's spent grain arriving from the local breweries is unloaded from vans and temporarily stored in the underground collection tank **S-101**. Thanks to a bucket elevator, BSG is collected from the underground tank and sent, with a capacity of 2,42 tons per hour, to the press **P-101** to reduce its water content.

6.2.3.2. Press P-101

The solid loaded through the bucket elevator is fed to the **BP-101** belt press. Here the solid is squeezed, reducing its water content from 70% to 60%. Belt press remove approximately 762 kg/h of water, reducing the capacity of the BSG stream from 2,4 tons per hour to 1,6 tons per hour.

6.2.3.3. Hoppers H-101 and H-102

The squeezed BSG is then sent by gravity to a receiving hopper **H-101**, equipped with a lump breaker to avoid the formation of agglomerates and facilitate the unloading. The receiving hopper must have at least one or two hours of hold-up and it is connected to the dosing hopper **H-102** with a vertical pipe. The discharge from the receiving hopper to the dosing hopper takes place by gravity thanks to the opening and closing of a servo-assisted gate valve, controlled by the high- and low-level switch of the dosing hopper. The dosing hopper rests on load cells and is connected to the rotary feed valve **FE-101** that assure a continuous feed of BSG to the Rotary Drum Dryer **DD-101**.

6.2.3.4. Rotary valve feeder RVF-101

The rotary feed valve **FE-101** is regulated by an inverter motor whose rotational speed is controlled by the weight control loop, deriving from the signal of the weight taken from the load cells on which the dosing hopper rests. The rotary feeding valve assure the correct feeding of BSG to the drum dryer **DD-101**, avoiding the rising of the hot fumes coming from the dryer.

6.2.3.5. Rotary Drum Dryer DD-101

BSG dried inside a drum dryer using a hot air stream. The flow of air required for drying is evaluated considering the water content of the wet BSG, the temperature and relative humidity of the external air and the operating temperatures of the process, following a dryer standard design procedure (37). The evaluated air stream, resulting from the sum of the air leaving the solid cooler **FBE-101** and the air pre-heater **E-101**, is heated up to 200°C using an air vein burner powered by methane and the hot gases are then sent to the Rotary Drum Dryer **DD-101** in counter current with the solid stream.

The dryer is a single tube type and the drying process takes place by direct contact between the hot gas stream and the wet solid stream and. This operational choice ensures lower investment and operating costs, without compromising the quality of the raw material: no problems have been recorded relating to direct contact between the hot gas stream and the BSG.

6.2.3.6. Hot gas purification system CY-101, PF-101/102

The gases leaving the Drum Dryer **DD-101** need to be purified before their discharge to the atmosphere. The first step is the passage inside the cyclone for powders **CY-101**, in order to eliminate traces of any small particles dragged during the drying process. The fumes coming out of the cyclone are finally sent to a bag filter system constituted by two filters **PF-101** and **PF-102** working alternately, in order to eliminate traces of particulate still present in the stream.

The solids removed are collected and disposed of in landfills in accordance with local regulations.

6.2.3.7. Open-back air draught burner B-101

The open-back air draught burner **B-101** has the purpose of supply the necessary heat to the air current used for drying to allow it to reach the temperature of 200 °C. It is fed with methane and allows to heat air currents quickly, effectively and with low emissions. Open-back air draught burners have also the advantage of being very easy to install, start and use.

6.2.3.8. Blowers BL-101 and BL-102

The total air flow to be sent to the dryer is obtained by mixing two distinct streams: the first stream is constituted by the hot air conveyed by the blower **BL-102** and coming out from the fluidized bed solid cooler **FBE-101**, while the remaining part is conveyed by the air blower **BL-101**. Both blowers are centrifugal, and their choice was made by consulting the Stiavelli-Irio centrifugal fans catalogue (59).

The fan **BL-101** conveys a volumetric flow of air of about 23160 m^3/h. For this purpose, it was chosen a centrifugal fan capable of feeding up to 18 m^3 per minute with an installed power of 22 kW.

The fan **BL-101** conveys a volumetric flow of air of about 680 m^3/h. It was chosen a centrifugal fan capable of feeding up to 400 m^3 per minute with an installed power of 0,75 kW.

6.2.3.9. Fluidized bed solids cooler FBE-101

The dried BSG reach a temperature of about 80 °C after the drying process and need to be cooled before being subjected to grinding and being stored. It is collected by gravity from the Drum Dryer **DD-101** and, by means of a conveyor belt **BE-101**, it is fed to the **FBE-101** heat exchanger. It consists of a fluid bed heat exchanger where the solids are suspended and mixed by a stream of cold air. Maximum contact occurs between the solid particles and the air stream, resulting in high heat transfer efficiency and uniform cooling of the solids.

The outlet solids temperature is fixed to 40°C. The outlet air stream pass through a purification section constituted by a cyclone integrated in the heat exchangers and a battery of filter bags **FB-103** and **FB-104** working alternatively. Finally, the hot air stream it is recycled to be used as part of the hot air stream needed to perform the drying process.

The cooled solid is collected by weir inside the fluidized bed and, by means of a rotary valve that prevents the gas from escaping the heat exchanger, is conveyed onto the belt conveyor **BE-102**. The belt conveyor conveys the BSG to a receiving hopper **H-103**, controlled by weight cells, which unloads directly into the dried BSG storage silo **TK-101**.

6.2.3.10. Hammer mill M-101

The dry solid extracted from the storage silo, before being sent to the first extraction section, is subjected to a grinding. Grinding is done using the grain hammer mill **M-101**, which reduces the size of the BSG down to an average diameter of 0.5mm.

A hammer mill consists of a steel drum containing a horizontal rotating shaft on which a series of hammers are mounted. Hammers are fixed to the central rotor, which is spun at high speed inside the drum while the solid matrix to be grinded is fed from the top. The grinded solids are expelled trough screens in the drum of the selected size.

6.2.4. Lipids and Polyphenols extraction section

6.2.4.1. Dry solid feeding systems

The feeding of the solid to the TURBEX, during various tests carried out in the commissioning phase, proved to be a critical part of the entire extraction process. Especially in the case of non-powdered solids, the lack of wetting of the solid can lead to the formation of agglomerates that tend to plug and block the screw feeder, interrupting the continuity of the process. For this purpose, it is necessary to carry out a complete wetting of the solid before feeding it, but in order not to change the counter-current operation of the machinery, this is done using the extract obtained rather than fresh liquid.

Adding fresh water could alter the flow-dynamic operating principles of the extractor, also diluting the extract obtained: since the extracting liquid is fed in counter current with the solid, the outlet of the extract is located near the solid entrance. Therefore, if fresh water is fed together with the solids, it risks escaping from the extractor without taking part, if not to a limited extent, in the extraction process, leading to a decrease in the total efficiency of the TURBEX. To avoid this, it was decided to recirculate the extract and mix it with the solids, maintaining the flow-dynamic conditions and the efficiency of the extractor almost unaltered.

The feeding system consist of four separate devices: a stirred tank, a decanter centrifuge and a progressive cavity feeder. The same technology is adopted both to feed the solid to the battery of extractors that takes care of the extraction of the lipids fraction and to feed it to the battery that takes care of extracting the polyphenols. To avoid redundancies, the system will only be described once.

Dry solids are fed to a stirred through a dosing hopper plus screw conveyor system, like the one described in the previous section. The mixing between the solids and the extract is carried out inside a stirred tank equipped with a paddle stirrer that guarantees continuous mixing between the two phases. The output of the obtained slurry, with a high concentration of solvent, is then sent to a decanter centrifuge.

The operating principle of the machinery is based on the separation of substances with different densities by exploiting the centrifugal force generated by high rotational speeds, increasing the settling speed of the solid particles contained in the slurry. The slurry stream is pumped inside the decanter centrifuge through an inlet placed in the middle of the machinery. The feed goes into a horizontal rotating bowl composed of a cylindrical part and a conical part. The separation take place inside the cylindrical part: since the solids has a higher density, they tend to collect on the wall of the drum. A screw conveyor collects the solids and pushes them towards the conical part of the machinery where they are also subjected to an increasing compression, facilitating the release of additional liquid. The solid therefore comes out of the exit of the conical section, while the liquid extract is collected on the opposite side.

In the cases of this extraction plant, it is not necessary to separate the solid with a very low moisture content, but rather the separation efficiency of the centrifugal decanter must be set in such a way as to ensure that the solid is just perfectly soaked. The outlet liquid content is taken slightly higher than the imbibition value of the solid. The solid is therefore fed by gravity perfectly soaked inside the dosing hoppers of the progressive cavity feeders connected to the extractors.

The collection hopper must be sized to have a hold-up of 2-3 hours. For safety reasons, the collection hopper must be equipped with an upper closure able to avoid the accidental fall of solid bodies which, getting stuck between the stator and rotor of the TURBEX, could cause serious damage to the machine. A metal detector is also added in order to preventively identify the presence of metal bodies, such as screws or bolts accidentally dragged into the feed.

The feeder consists of a food grade progressive cavity pump. This type of pumps is a kind of positive displacement pump, mainly constituted by a helical stainless-steel rotor which turns inside a helical stator made of a resilient elastomer. The rotor tightly seals against the stator while it rotates, creating a series of small, fixed shape, discrete cavities. The pumping of the fluid takes place tanks to the formation of this cavities, leading to a volumetric flow rate proportional to the rotation rate of the motor.

It was chosen a Nova Rotors DX Series, which as the following characteristics: the drive is coupled directly to the pump via a flange. This solution is extremely cost effective and compact, considerably reducing installation costs and simplifying maintenance. The forces generated by the hydraulic part are supported by the drive itself. Each drive used is adequately selected based on their specific technical parameters and are subject to numerous duration tests with heavy loads (39).

As previously mentioned, this solution must be adopted during to feed the solid to the first two batteries of extractors. The first feeding system is that of the lipids extraction, where hexane is used as the extracting solvent. The calculated liquid/solid wetting ratio between hexane and BSG is 0,27. The extract obtained is recycled inside the stirred tank **MX-201** which allows to obtain a slurry with a hexane concentration of approximately 40%by weight. By means of the volumetric sludge pump **P-201**, the slurry is fed to the decanter centrifuge **DC-201**, which separate a slurry of solid with a hexane concentration of 20% by weight and the liquid extract stream. The wet solids are collected by gravity inside the hoppers of the progressive cavity feeders **PCF-201** and **PCF-202**. The progressive cavity feeders are directly connected to the lipids' extractors **EX-201** and **EX-202** operating in parallel. The liquid extract stream leaving the centrifugal decanter, before being sent to the subsequent purification operations, is subjected to safety filtration with the filter bags system **FB-201/202** to avoid the entrainment of solid particles.

The second feeding system is that of the polyphenols extraction, where a water-ethanol solution (70% v_{EtOH}/V_{tot}) is used as the extracting solvent. The calculated liquid/solid wetting ratio between hexane and BSG is 2,67. The extract obtained is recycled inside the stirred tank **MX-202** which allows to obtain a slurry with a solvent concentration of approximately 93% by weight. By means of the volumetric sludge pump **P-206**, the slurry is fed to the decanter centrifuge **DC-202**, which separate a slurry of solid with a solvent concentration of 72% by weight and the liquid extract stream. The wet solids are collected by gravity inside the hoppers of the progressive cavity feeders **PCF-203** and **PCF-204**. The progressive cavity feeders are directly connected to the polyphenols' extractors **EX-203** and **EX-204** operating in parallel. The liquid extract stream leaving the centrifugal decanter, before being sent to the subsequent purification operations, is subjected to safety filtration with the filter bags system **FB-203/204** to avoid the entrainment of solid particles.

6.2.4.2. Lipids extractors EX-201/202/203

For the lipid extraction section, it was decided to use n-hexane, the same solvent used in traditional extraction plants, but using the TURBEX EX30 as the extractor instead of the traditional single stage stirred tank extractors. The extraction process has been modelled considering that the TURBEX is able to perform, in a single passage inside the machinery, seven stages of counter current equilibrium. The substances that are subject to solubilization in n-hexane are mainly lipids and a part of the protein fraction of the BSG. The extraction equilibrium approach constants for these two substances were therefore calculated on the basis of the data found for a single equilibrium stage. The mass balances were then solved ignoring the possible dissolution of other compounds contained in the BSG. After having calculated the equilibrium approach constants, it was possible to carry out the mass balances for the seven stages of the machinery assuming that equilibrium approach constants don't vary from stage by stage. The used procedure is very similar to that one discussed in Chapter 3.

The flow rate of dry solid to be processed is of about 660 kilograms per hour. To keep the TURBEX efficiency high, exploiting all the process intensification mechanisms that characterize the machine, it was decided to use two extractors with a maximum capacity of 500 kg/h working in parallel. Moreover, it was deemed necessary to add one more TURBEX to be kept in stand-by, ready to use if there is the need to carry out maintenance operations, assuring so the continuity of the process. The same applies to the feeding system with progressive cavity feeder and the pressing system of the solid at the outlet with the squeezing press which are almost an integral part of the machine.

The temperature at which the extraction process is conducted is 67 °C, close to the boiling temperature of hexane. The inlet solvent temperature is therefore regulated thanks to the presence of the heat exchanger E-201.

The TURBEX allow to perform the extraction with a lipid extraction yield equal to 99,99%, also extracting 30% of the proteins present in the fed BSG. The extract stream is obtained with a flow rate of 440 kilograms per hour and a lipid concentration equal to 11.36%.

6.2.4.3. Squeezing presses SP-201/202/203

Wet BSG leaving the extractors from the solid outlet holes is collected by gravity inside hoppers connected to a food grade squeezing press system. Each TURBEX is equipped with its own squeezing press. They consist of a small residence time hopper at the bottom of which is installed a screw press that has the purpose of recovery a part of the soaking liquid trapped inside the solids, reducing their solvent concentration. The degree of squeezing of each press is manually adjusted by opening a handwheel valve located near the solid outlet.

The solid squeezed out of the squeezing press is collected by gravity from the outlet hole of the press upon a bel conveyor that bring the solids to the following devices dealing with solvent recovery.

This equipment is used with the same principle for each of the extractors present throughout the plant. From this moment on, it will no longer be described in detail, but only the details relating to the operating parameters will be mentioned in the description of the operating parameters of the extractors.

The solid, with a total flow rate of 842 kilograms per hour, leaves the extractors with a concentration of hexane equal to about 28% by weight. The presses are set in such a way as to allow the solid stream to exit with a concentration of hexane of 21.5%, equal to that of the solid completely soaked in the solvent. A flow rate of approximately 68 kilograms per hour is therefore removed and sent to the collection tank **TK-201**.

6.2.4.4. Squeezed liquid collection tank TK-201

The liquid recovered from each squeezing press is conveyed to the collection tank **TK-201**, from where it will be recirculated to the extractor and used as part of the extractant solvent. The liquid is pumped out of the tank and recycled by means of the volumetric sludge pump **P-201**.

The liquid outlet pipe of the tank must be protected by inserting a static filter that avoids the possible entrainment of solid bodies that could cause serious damage inside the extractor.

This equipment is used with the same principle for each of the extractors present throughout the plant. From this moment on, it will no longer be described in detail, but only the details relating to the operating parameters will be mentioned in the description of the operating parameters of the extractors.

6.2.4.5. Flash desolventizer FD-201

The BSG coming out of the squeezing press of the first extractor, with a capacity of 774 kilograms per hour, still contains a capacity of about 166 kilograms per hour (21% w/w) of hexane which must be recovered. A flash desolventizer is used for this purpose.

It consists of a vertical cylindrical tower equipped with a series of horizontal trays. The solid BSG coming out of the presses is collected by gravity on a belt conveyor which conveys it inside the receiving hopper **H-201**. The receiving hopper discharges into the dosing hopper of the rotary feed valve **RFV-201** which feeds the solid from the top of the flash desolventizer. The feed dosing system is like those described above.

The solid is mixed above each tray and moved down each tray by agitating sweeps anchored to a central rotating shaft. The heat necessary for increase the temperature of the solids and allow the hexane to evaporate is supplied by a nitrogen stream, placed in contact with the solid directly through the plates. The trays are designed with an upper plate, lower plate and structural members between to hold the gas stream pressurized.

The equipment was designed by solving the mass and heat balances related to the process. First the amount of hexane that can be removed is calculated, after which the maximum temperature of the nitrogen to be sent is calculated without undergoing degradation phenomena of the substances present in the BSG. The temperature at which the ferulic acid begin to undergo thermal degradation phenomena is reported to be 125 °C (60), for this reason it was chosen to use nitrogen with an inlet temperature of 115 °C. The current is heated by passing through a gas/fumes plate heat exchanger fed by a current of burnt gas coming from the back-air draught air burner **B-201**.

It’s necessary a stream of 1114 cubic meters per hour of nitrogen to assure a recovery of 99,46% of hexane. The nitrogen is fed to the desolventizer at the delivery pressure of 0,6 MPa g.

The solid, with a flow rate of 608 kilograms per hour, is collected from the bottom of the desolventizer through a rotary valve and sent to the collection and dosing hoppers system **H-202** and **SC-201** to be then fed through a screw conveyor to the next extraction section. The nitrogen stream containing the hexane removed from the solid is purified passing through the cyclone **CY-201** and the filter **F-203** and finally sent to the subsequent equipment for the recovery of the hexane.

6.2.4.6. Roto-concentrator RC-201

The first step of the hexane recovery from the nitrogen stream is a passage through the rotoconcentratore **RC-201**. The roto-concentrator is a variable flow adsorption system for the continuous elimination of volatile organic compounds (VOCs) from a low concentration gas flow, by adsorption of VOCs with adsorber material in water-repellent zeolite and subsequent release of the VOCs themselves in a reduced heated air flow, but more concentrated, for subsequent treatment (61).

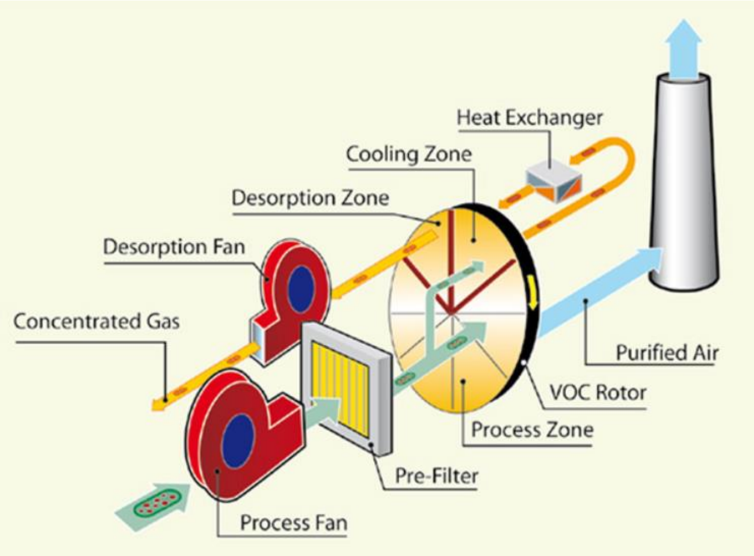


Figure 6.6 - Operation diagram of a rotary concentrator for the recovery of VOC from a generic air stream (61).

Air with a low hexane concentration enters the inlet chamber through a filter that removes dust and excess particles. From the inlet chamber, the air passes through the rotor where hexane is adsorbed, then the purified nitrogen arrives in the outlet chamber and is expelled to be recycled. The adsorbed hexane is removed from the rotor by passing a reduced flow of hot nitrogen through a part of the rotor in the opposite direction to the main gas flow to be treated, with the result of obtaining a reduced gas flow but with a higher hexane concentration. The nitrogen flow used for this operation is taken upstream of the rotor, and it is the same that guarantees the cooling phase of the rotor after regeneration.

The equipment is design by solving the mass balances, using as a design parameter a ratio of pure nitrogen stream obtained to a supplied nitrogen stream equal to 0,9. From the 1,6 ton per hour stream of nitrogen containing hexane fed, 1,25 tons per hour of pure nitrogen and approximately 342 kilograms per hour of a gas stream are obtained. The residue gas stream, composed of 59% by weight of hexane and 41% of nitrogen, it is sent to the second separation step.

6.2.4.7. Direct condensation tower for hexane T-201

The gaseous stream containing hexane coming out of the roto-concentrator is fed to the bottom of a direct condensation tower, where liquid hexane is rained from above at a temperature of 0 °C. The separation between hexane and nitrogen occurs by condensation of hexane thanks to the removal of heat deriving from contact with cold liquid hexane.

A liquid stream of almost pure hexane (99,86% w/w), with a capacity of 681 kilograms per hour and a temperature of 18,8 °C, is then separated from the bottom of the column and sent to the hexane ward tank TK-202. A stream of nitrogen saturated with hexane, with a capacity of 175 kilograms per hour and a temperature of 5 °C, is collected from the top of the column. Although the nitrogen stream contains a small part of hexane, it can safely be recirculated, after recompression in **C-201**, to the flash desolventizer **FD-201** because it is still able to provide enough thrust force for the separation process. The Aspen Plus software was used to size the column and to fine-tune the mass and thermal balances.

The fed stream is previously cooled by first passing inside the economizer **E-302** which preheats the nitrogen gas stream leaving the condensation column, and then a chilled water-fed heat exchanger **E-204** which complete the cooling bringing it to a temperature of 20 °C.

6.2.4.8. Hexane distillation tower T-202

The liquid extract containing the lipid fraction, after filtration the filter bags system **FB-201/202**, is sent to a distillation column, where the hexane is separated at the top and the lipid fraction with a hexane residue equal to about 7% by weight. Before entering the column, the extract is collected in the buffer tank **TK-203** and from there it is re-launched into the column by means of the volumetric pump **P-206**. The buffer tank must be sized to have a hold-up of two hours, and it is used to guarantee the continuous supply of liquid to the column, in order to prevent its operation from being compromised due to oscillations in the previous sections.

The distillation column is simulated using the Aspen Plus software, to obtain the partition coefficients between distillate and bottom stream, the condenser and reboiler duties and the fundamental construction characteristics of the same. The optimal distillate to feed ratio, the number of stages and the power stage are first obtained using the DSTWU block, then the column is simulated using the RadFrac block. Simulations was made using n-Hexadecanoic acid as representative molecule for the lipids fraction and Ferulic acid as representative molecule for the polyphenols fraction.

The column guarantees an overhead recovery of the hexane equal to 98,96% and is made up of four real stages, with feeding to the third stage. The reflux ratio is set equal to 0,2 and the distillate to feed ratio equal to 0.945. The temperature of the top stream is of about 67 °C, while the bottom stream has a temperature of 136 °C and is sent to the subsequent steam stripping column **T-203** to eliminate the residual hexane.

6.2.4.9. Hexane stripper T-203

To eliminate the residual hexane in the bottom stream of the hexane distillation column, it is decided to operate a stripping with steam, which is carried out in the **T-203** column. This column was also designed with the help of the Aspen Plus software, with which a sensitivity analysis was carried out to determine the optimal quantity of steam to be sent.

A stream with a capacity of about 57 kilograms per hour is fed to the top of the columns and it's put in contact with a rising stream of low-pressure steam fed to the bottom of the column with a capacity of 7 kilograms per hour. The steam provides the heat necessary to allow the evaporation of the hexane and the thrust to allow its entrainment to the top of the column.

Following stripping, the lipid stream is obtained at with a capacity of 53,6 kilograms per hour and a temperature of 136 °C. It represents the first product of the process and after the column it is sent to the product storage tanks. The temperature is not lowered, on the contrary it is advisable to let the product flow inside pipes traced with steam, so as to avoid cooling and consequent solidification on the walls of the pipes. The vapor stream containing the hexane, with a flow rate of 10 kilograms per hour and a temperature of 136 °C, is instead cooled inside the **E-207** heat exchanger and sent to the decanter D-201 for subsequent decantation.

6.2.4.10. Hexane decanter D-201

The **DC-201** equipment consists of a static decanter inside which is sent the liquid stream of water and hexane, collected at the head of the **T-203** column and cooled to a temperature of 25 °C inside the **E-209** heat exchanger.

The decanter has the purpose of separating by gravity the hexane that is collected by surfacing from above and the water that is collected from the bottom of the decanter. The clarified stream is first collected inside the well **TK-209** and subsequently sent to the hexane ward tank by means of the centrifugal pump **P-209**.

6.2.4.11. Polyphenols extractors EX-204/205/206

For the polyphenols extraction, it was decided to use a water ethanol solution 60% v_{EtOH}/v_{tot}, the same solvent used in traditional extraction plants, but even in this case using the TURBEX EX30 as the extractor instead of the traditional single stage stirred tank extractors. The water ethanol solution with a concentration of 70% v_{EtOH}/v_{tot} is used in other sections within the plant and is stored in the **TK-406** ward tank. Before obtaining the 60% solution, a dilution with water must therefore be provided in a stirred tank with a hold-up time of a couple of hours.

The extraction process has been modelled considering that the TURBEX is able to perform, in a single passage inside the machinery, seven stages of counter current equilibrium. The substances that are subject to solubilization in the water ethanol solution are mainly polyphenols, sugars and proteins. The extraction equilibrium approach constants for these three substances were therefore calculated based on the data found for a single equilibrium stage. The mass balances were then solved ignoring the possible dissolution of other compounds contained in the BSG. After having calculated the equilibrium approach constants, it was possible to carry out the mass balances for the seven stages of the machinery assuming that equilibrium approach constants don’t vary from stage by stage.

The flow rate of dry solid to be processed is of about 610 kilograms per hour. As in the previous case, to keep the TURBEX efficiency high, exploiting all the process intensification mechanisms that characterize the machine, it was decided to use two extractors with a maximum capacity of 500 kg/h working in parallel. Moreover, it was deemed necessary to add one more TURBEX to be kept in stand-by, ready to use if there is the need to carry out maintenance operations, assuring so the continuity of the process. The same applies to the feeding system with progressive cavity feeder and the pressing system of the solid at the outlet with the squeezing press.

In the absence of experimental data on the real extraction efficiency of TURBEX with hydroalcoholic solutions, the extraction is carried out with very high extracting capacities, like those used in traditional extraction processes. To evaluate the optimal liquid over solid ratio, two distinct evaluations were carried out: the first related to the extraction yield of polyphenols, while the second is an approximate estimate of the costs related to the increase in the liquid over solid ratio. In the second analysis, the costs related to all the upstream processes that must undergo the streams of extract and liquid recovered from the solid are calculated, in terms of water and steam used in subsequent equipment for the recovery of ethanol.

In both cases, the elbow of the curve stands around a value of liquid over solid ratio equal to 15, which is therefore taken as the value to be used for the extraction process.

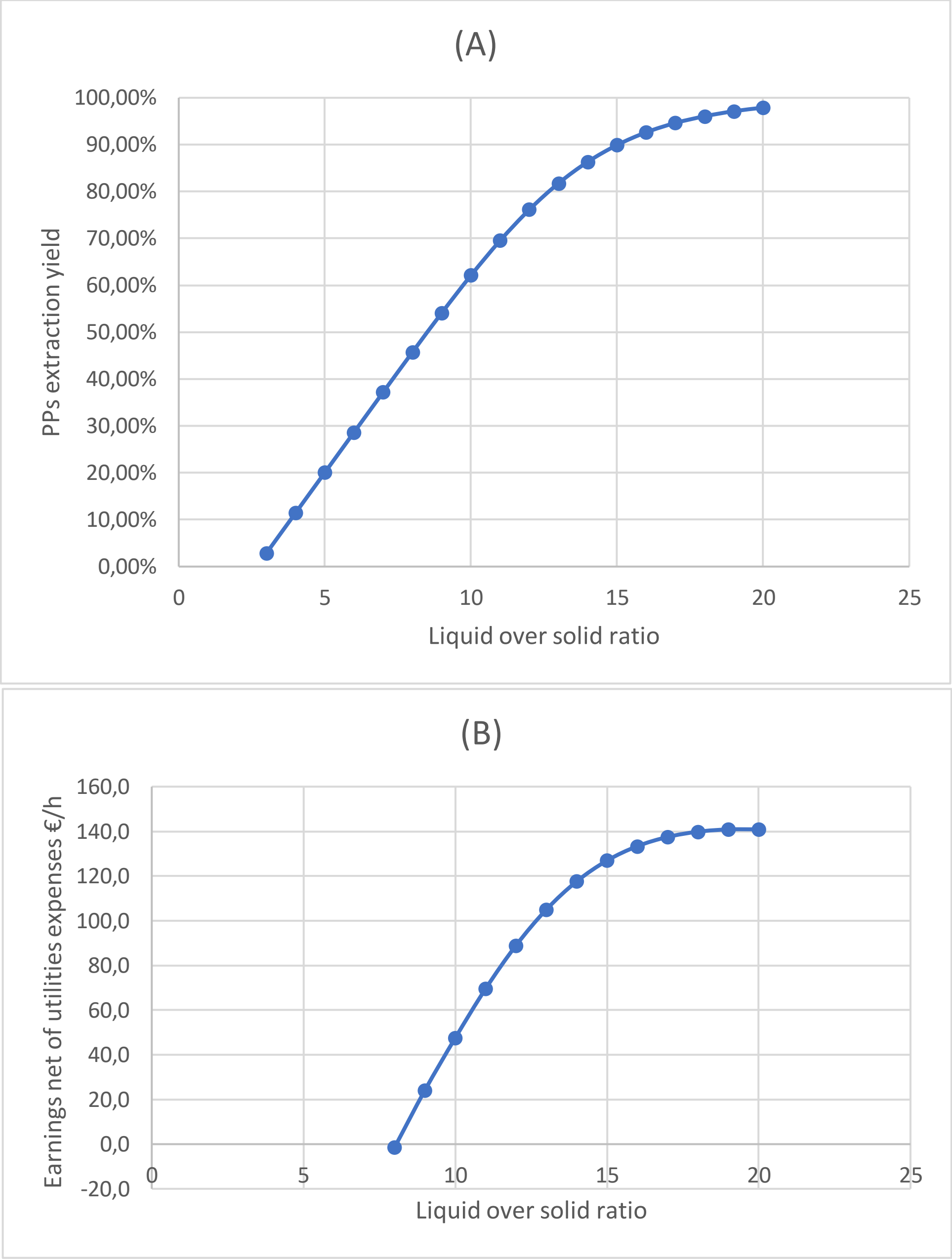


Figure 6.7 - - (A) Variation of the PPs extraction yield as the L / S ratio varies ; (B) Gain variation as the L / S ratio varies

At the exit of the flash desolventizer the solid appears again as a dry solid, therefore before being fed to the battery of extractors it needs to be wet using a system similar to that seen previously: the extract obtained is recirculated inside the stirred tank **MX-202** and mixed with the dry solid, then the slurry obtained is fed to the centrifugal decanter **DC-202** which separates the extract from the soaked solid slurry to be fed to the progressive cavity feeder **PCF-204/205** of the extractors.

The TURBEX allow to perform the extraction with a lipid extraction yield equal to 89,92%, also extracting 10,98% of sugar and 3,42% of proteins present in the fed

BSG. The extract stream is obtained with a flow rate of 7,5 tons per hour and a very low concentration of polyphenols, equal to 0,09%.

The solid at the exit from the extractors is squeezed inside the squeezing press system **SP-204/305** at the outlet of which a liquid stream of solvent is obtained which is recirculated and the solid soaked in ethanol which is sent to the multistage washer **W-201**.

6.2.4.12. Multistage washer for solids W-201

The solid soaked in hydroalcoholic solution must be washed to recover the ethanol present before being sent to the following extraction steps. For this purpose, the W-201 counter-current four-stage belt washer is used, where the hydroalcoholic solution is removed and replaced by water.

The washer is designed in such a way as to guarantee the removal of more than 99% of the ethanol present in the solid. Finally, a slight squeezing is carried out downstream of the washer with the squeezing press **SP-207** to eliminate the excess water and ensure the feeding, by means of the screw conveyor, of wet solid to the next extraction stage. All the liquid leaving the machinery is finally collected and conveyed to the ethanol recovery section.

6.2.4.13. Membrane filtration system UFM-201/202 and NFM-201/202

The extract from the centrifugal decanter **D-202**, after filtration in the bag filter system **FB-203/204**, is collected in the tank **TK-205**. From there, by means of the **P-212** centrifugal pump, it is sent at a pressure of 5 bar to a two-stage membrane filtration system. In fact, the extract contains a concentration of polyphenols lower than that of the sugar fraction extracted. To increase the commercial value of the final product, it is therefore necessary to increase the concentration of polyphenols making them the prevailing fraction.

The first step is a membrane ultrafiltration stage carried out with a Desal Gk (WRF5) membrane, which have a permeability of 79.8% to sugars, 11% to polyphenols and 1% to proteins. In this way, at the cost of a small loss of polyphenols, it is possible to obtain a retentate current with a much lower concentration of sugars. The retentate current is then conveyed inside the intermediate tank **TK-206** from where, through the centrifugal pump P-213, it is pumped at a pressure of 15 bar at the second filtration step.

The second filtration step consists of a nanofiltration using a membrane of the MPF-36 (1000 Da) type, which guarantees a rejection of 11% to sugars, 97.5% to polyphenols and 99% to proteins. At the end of the process, a liquid extract with a capacity of 7.49 tons per hour and a very low concentration of polyphenols, is reduced to an extract of 132 kilograms per hour with a concentration of polyphenols of 4.54% by weight compared to 3.59% of proteins and 0.23% of sugars.

The liquid stream eliminated by the membrane filtration process is finally sent to a filtering system capable of eliminating the solute residues and is finally sent to the ethanol recovery section. Instead, the concentrated extract stream is sent to the next concentration phase.

6.2.4.14. Double effect evaporators EV-201/202

To concentrate the extract before the final drying and recover the ethanol present, the extract is sent to a double effect evaporation system. The liquid extract stream recovered by the membrane filtration system is collected inside the **TK-207** tank and then pumped through the **P-214** centrifugal pump to the firs evaporator **EV-201**.

The first evaporator works at atmospheric pressure and manages to concentrate the extract up to a concentration of solutes equal to about 11%. The vapor stream evaporated by the first effect is sent to the second effect as a condensing current which provides the heat necessary to complete the evaporation of the ethanol. The second evaporator works in depression at 0.8 bar and completes the evaporation of the ethanol bringing the extract to a total concentration of solutes of 22%. To carry out the balances, the liquid-vapor equilibrium data of the ethanol and water solution at the evaporator operating pressures are used.

From the liquid stream fed, having a flow rate of 133 kilograms per hour, 82,4 kilograms per hour of solvent are removed, bringing the final extract to a flow rate of approximately 50,6 kilograms per hour and a concentration of polyphenols equal to 11%. The extract is then sent to the TK-208 tank before final drying.

6.2.4.15. Drum dryer for polyphenols DD-201

At the outlet of the **EV-202** evaporator, the extract stream needs to be further concentrated until it reaches a mass water content of 2% and the appearance of a powder. To carry out this operation, it was decided to use a low-pressure steam powered drum dryer, in which the contact between the utility and the process current occurs indirectly.

The chosen drum dryer configuration is equipped with two rotating cylinders inside which the steam is sent. The material in the form of a paste is distributed evenly on the surface of the rotating drums, heated by the contact with the steam and subsequently collected by means of special blades that detach it from the cylinders and make it fall on the launch shoe of the finished product.

To prevent the temperature of the extract from rising rise up to values that could thermally deteriorate the active ingredients present in the extract, it was decided to send saturated steam to the drum with a pressure of 2 bar instead of 6 bar, guaranteeing also a time of limited contact.

6.2.5. Proteins and Arabinoxylans extraction section

6.2.5.1. Extractor for alkaline extractions EX-301 to EX-309

The residue wet BSG, coming from previous extraction section has a composition that is mainly lignocellulosic containing a high mass concentration of proteins on a dry basis. The last extraction stage of the process is a multistage alkaline extraction performed according to the patent WO 2012/069889 A1 (58), but using the TURBEX EX30 as the extractor. This is carried out in three successive stages in which the extracting solution is a sodium hydroxide solution with a gradually increasing concentration. The purpose is to obtain a protein rich and an arabinoxylans rich powder extracts using an integrated process of simultaneous extraction of proteins and arabinoxylans. Considering that the extraction takes place in a similar way for all three alkaline extractions, in order to avoid redundancies, the process will be described in general in this single paragraph.

Table 6.15 - Composition on a dry basis of BSG entering the third section of the plant

Water	0,00%
Cellulose	22,36%
Hemicellulose	34,26%
Insoluble lignin	11,11%
Proteins	27,49%
Polyphenols	0,16%
Lipids	0,00%
Ash	4,63%

The use of alkaline solvents improve the extraction efficiency tanks to the occurrence of the phenomenon of alkaline hydrolysis, which is able to easily dissolve lignin (50) allowing to better solubilize also the arabinoxylans which are mostly not extractable with water (56).

The BSG coming from the W-201 washer with a capacity of 1,2 tons per hour is completely wet (60% of water), for this reason it’s not necessary to use stirred tanks and decanter centrifuges to prepare a slurry to fed to the extractors. Furthermore, the solid coming out of the squeezing press at the exit of each extractor also comes out completely soaked. In light of this, each extraction section is equipped with a receiving hopper system where the wet solid is collected, and then discharged by gravity to the dosing hopper of the progressive cavity feeders of the extractors.

In the first three stages the BSG is extracted with three sodium hydroxide solutions with increasing concentration: 0,1 M in the first extraction, 0,5 M in the second extraction and 4 M in the third extraction. For each stage the solid is sent to the following extraction step, while the extract, after filtration, is sent to the following purification steps.

As in the previous cases, for each extraction reference was made to data relating to the single equilibrium extraction stage. From there, the equilibrium approach constants were calculated for each of the substances that tend to dissolve. Given the nature of the extractant used, it has been assumed that practically all substances can be partially solubilized within the liquid. Once the constants were determined, the mass balances for each extractor were set and resolved, assuming that these are able to perform as seven counter current equilibrium stages and that the equilibrium constants of each component do not vary from one stage of balance to the other.

A cross investigation was carried out to find the optimal ratio between extracting liquid and solid fed. It has been evaluated that the extraction of proteins does not gain a clear improvement by increasing the extraction rate, while an increase in the extraction rate at the third stage of extraction involves a more significant increase in the extraction yield of arabinoxylans. Considering the results obtained, it was used a liquid over solid ratio equal to 0,65 for the firs alkaline extraction, a value of 0,4 for the second and a value of 1 for the third.

Table 6.16 - Some of the results of the cross-investigation on the best L/S ratio combination between the three alkaline extraction stages.

L/S stages	FP recovery	FP purity	AX recovery	AX purity	FP rec. Var	AX rec. Var.
0,8-0,8-0,8	95,85%	89,60%	62,28%	83,31%	*+10,72%*	*+38,84%*
0,65-0,8-0,8	95,83%	90,13%	62,26%	83,57%	*+10,69%*	*+38,78%*
0,65-0,4-0,8	94,59%	92,60%	60,45%	85,12%	*+9,27%*	*+34,75%*
0,65-0,4-0,6	91,93%	92,69%	52,70%	85,52%	*+6,19%*	*+17,47%*
0,65-0,8-0,4	94,84%	90,52%	48,43%	84,41%	*+9,56%*	*+7,96%*
0,65-0,4-1	95,53%	92,41%	67,90%	84,72%	*+10,35%*	*+51,36%*

Compared to the initial content of proteins and arabinoxylans of the BSG in input to this section, the three stages extraction efficiencies are expressed below.

- The first extraction step with 0,1 M sodium hydroxide solution is able to extract about the 35% of total proteins and 8% of total arabinoxylans present in the BSG fed in this third section. It is constituted by the receiving hopper H-301, the progressive cavity feeders PCF-301/302/303, the TURBEX EX30 extractors EX-301/302/302 and the filter bag system FB-301/302;
- The second extraction step with 0,5 M sodium hydroxide solution has a protein extraction yield of 62,86% and an arabinoxylans extraction yield of 10,13%. It extracts about the 40,87% of proteins and 9,31% of arabinoxylans. It is constituted by the receiving hopper H-302, the progressive cavity feeders PCF-304/304/305, the TURBEX EX30 extractors EX-304/304/305and the filter bag system FB-303/304;

- The third extraction step with 1 M sodium hydroxide solution has a protein extraction yield 97,97% of and an arabinoxylans extraction yield of 60,5%. It extracts about the 23,65% of proteins and 50,01% of arabinoxylans. It is constituted by the receiving hopper H-303, the progressive cavity feeders PCF-306/307/308, the TURBEX EX30 extractors EX-306/307/308 and the filter bag system FB-305/306.

6.2.5.2. Protein sedimentation

The extracts stream coming from the alkaline extractions, after filtration, is sent to a high-shear homogenizer with low residence time where citric acid is added up to reach a value of pH equal to 3. The acid condition thus created allows the separation of proteins which is completed inside a decanter where a liquid residue rich in proteins is collected from the bottom and the liquid extract rich in arabinoxylans is collected by surfacing inside a pot tank and sent to the following operations.

The equipment used for the protein precipitation processes are:

- For the first alkaline extract it’s used the stirred tank **MX-301**, the decanter **D-301**, the pot tank **TK-304**, the volumetric progressive cavity type pump **P-303** and the centrifugal pumps **P-302** and **P-304**;
- For the second alkaline extract it’s used the stirred tank **MX-304**, the decanter **D-303**, the pot tank **TK-306**, the volumetric progressive cavity type pump **P-311** and the centrifugal pumps **P-310** and **P-312**;
- For the first alkaline extract it’s used the stirred tank **MX-307**, the decanter **D-305**, the pot tank **TK-308**, the volumetric progressive cavity type pump **P-3020** and the centrifugal pumps **P-319** and **P-321**;

All the three streams containing the protein fractions, collected from the bottom of the decanters, are conveyed to the **TK-312** tank before being subjected to the final drying process.

6.2.5.3. Arabinoxylans sedimentation

The extracts rich in arabinoxylans, collected by surfacing from the protein’s decanters **D-301**, **D-303** and **D-305** are further acidified with the addition of hydrochloric acid until reaching a pH of 2, to allow the precipitation of the arabinoxylans. Furthermore, to avoid the precipitation of citrates, it is necessary to add also a solution of water-ethanol (70% v_{EtOH}/v) in a quantity that must be 70% of the total volume. These two operations are carried out in series inside two high-shear homogenizers with low residence time.

The solutions obtained are then decanted and streams reach in arabinoxylans are collected from the bottom of the decanter, while the liquid stream containing citric acid and ethanol are collected by surfacing inside the tank pots of the decanters and then sent to the solvents' recovery section. This procedure is the same repeated for all the three alkaline extraction stages.

The equipment used for the protein precipitation processes are:

- For the first alkaline extract it's used the stirred tanks **MX-302** and **MX-303**, the decanter **D-302**, the pot tank **TK-305**, the volumetric progressive cavity type pump **P-307** and the centrifugal pumps **P-304**, **P-305**, **P-306** and **P-308**;
- For the second alkaline extract it's used the stirred tanks **MX-305** and **MX-306**, the decanter **D-304**, the pot tank **TK-307**, the volumetric progressive cavity type pump **P-315** and the centrifugal pumps **P-312**, **P-313**, **P-314** and **P-316**;
- For the third alkaline extract it's used the stirred tanks **MX-308, MX-309**, the decanter **D-306**, the pot tank **TK-308**, the volumetric progressive cavity type pump **P-322** and the centrifugal pumps **P-319**, **P-320**, **P-321** and **P-323**;

All the three streams containing the arabinoxylans fractions, collected from the bottom of the decanters, are conveyed to the **TK-313** tank before being subjected to the final drying process.

6.2.5.4. Final arabinoxylans extractors EX-310/311/312

The solid residue of this three-stages extraction is now mostly made of ashes, cellulose and lignin but to maximize the yield of the process a further extraction process is performed. The residue is subjected to a final extraction with water performed using the TURBEX EX30. This stage is also called neutralization, as it has the double purpose of eliminating the residual basicity of the solid following alkaline extractions.

As in the previous cases, the extraction equilibrium constants are determined from data relating to the single equilibrium stage. The balances are then set and resolved assuming that the TURBEX can perform 7 stages of equilibrium in counter current and that the equilibrium approach constants do not vary stage by stage. Even for this last stage, the optimal ratio of extracting liquid to solid fed is investigated. Considering that the extracted arabinoxylans fraction represents the product with the lowest economic value, it is decided to maintain an L/S value of 0.8, high enough to neutralize the solid matrix and obtain an extraction efficiency 20,5% thus reducing the downstream purification costs.

The residual solid is then collected and sent to the combined heat and power generator, while the extract is sent to the following devices to be concentrated before the last drying process.

6.2.5.5. Membrane filtration system FM-301/302

The liquid extract obtained from the neutralization operation is collected inside the **TK-310** buffer tank which must be sized to ensure a two-hour hold-up. From there, by means of the **P-325** pump, the diluted extract is pumped at a pressure of 20 bar and sent to a filtration system with polyamide membranes **FM-301/302** that have a rejection to xylose of 99.13% and a recovery in the water permeate equal to 80%. Xylose is taken as the representative molecule of the arabinoxylan fraction.

The membranes are mounted in two batteries operating alternately to ensure the continuity of the process even during the restoration of the membrane. The liquid stream of permeate containing traces of arabinoxylans is disposed of as wastewater while the retentate is sent to the subsequent concentration operation.

6.2.5.6. Double effect evaporators EV-301/302

To concentrate the extract before the final drying the extract is sent to a double effect evaporation system. The liquid extract stream recovered by the membrane filtration system is collected inside the **TK-311** buffer tank and then pumped through the **P-326** volumetric pump to the firs evaporator **EV-301**.

The first evaporator works at atmospheric pressure and manages to concentrate the extract up to a concentration of solutes equal to about 17,25%. The vapor stream evaporated by the first effect is sent to the second effect operating in depression at 0,8 bar. The vapour extracted from the first evaporator works as the condensing current which provides the heat necessary to complete the evaporation of the water contained in the extract up to a target solute concentration of 55%. The two evaporators are sized by setting the operating pressures and ensuring that the heat duty of each evaporator is the same.

The concentrated extract stream, collected from the bottom of the second evaporator, is sent by means of the volumetric pump **P-327** to the storage tank **TK-313** for the liquid streams containing arabinoxylans.

6.2.5.7. Drum Drier for proteins and arabinoxylans DD-301/302

The extract streams containing proteins and arabinoxylans needs to be further concentrated until they reach the desired final product characteristics: a mass water content of 2% and the appearance of a powder. To carry out this operation, it was decided to use two distinct low-pressure steam powered drum dryer, in which the contact between the utility and the process current occurs indirectly.

The chosen drum dryer configuration is equipped with two rotating cylinders inside which the steam is sent. The material in the form of a paste is distributed evenly on the surface of the rotating drums, heated by the contact with the steam and subsequently collected by means of special blades that detach it from the cylinders and make it fall on the launch shoe of the finished product.

To prevent the temperature of the extract from rising rise up to values that could thermally deteriorate the active ingredients present in the extract, it was decided to send saturated steam to the drum with a pressure of 2 bar instead of 6 bar, guaranteeing also a time of limited contact.

The **DD-301** drum dryer deals with the drying of the proteins fraction, while the **DD-302** drum dryer deals with the drying of the arabinoxylans fraction.

6.2.6. Ethanol and citric acid recovery section

6.2.6.1. Ethanol distillation column T-401

Ethanol is used in various operations within the plant and in large quantities. Having a considerable cost and being subject to state taxation, it must be recovered with high efficiency, minimizing losses. Citric acid is also used in considerable quantities and is collected mixed with ethanol from the clarified streams of the decanters for the precipitation of arabinoxylans. The recovery of ethanol and citric acid is carried out in the last section of the plant.

The first step consists in the distillation of the ethanol inside the **T-401** distillation column, carried out in such a way as to recover a top stream with the greatest possible quantity of a hydroalcoholic solution having a volume concentration of about 70% of ethanol.

All streams containing ethanol and citric acid are therefore conveyed into the **TK-401** collection tank, which must be sized to have a hold-up time of a couple of hours. From the collection tank the stream containing ethanol, water and citric acid is pumped through the centrifugal pump **P-401** to the **T-101** distillation column. To calculate the partition coefficients of the column and the fundamental constructive and operational parameters, the column is simulated on Aspen using first a DSTWU block and finally a RadFrac block.

To carry out the operation as required, it is found that the column must have a number of stages equal to six, and feed sent to the second stage. The column works with a reflux ratio equal to 1.2 and distilled to feed ratio equal to 0,55. A total flow of about 19,5 tons per hour is fed to the column. The obtained distillate stream, with a flow rate of 12,6 tons per hour, has an ethanol concentration of 33.7%, while the bottom stream, with a flow rate of 6.9 tons per hour, has a citric acid concentration of 5%. The recovery of ethanol is practically unitary.

The distillate stream is then sent to the water ethanol solution storage tank **TK-407** while the bottom stream is sent to the stirred tank **MX-401**.

6.2.6.2. Sodium chloride decanter D-41

The solution containing citric acid must now be treated in such a way as to favour the sedimentation of the sodium chloride which forms from the acid-base neutralization reaction between hydrochloric acid and sodium hydroxide.

The solution containing citric acid, before decantation need to be diluted again with a water-ethanol solution up to reach a volume concentration of 40% $v_{solution}/v_{TOT}$. This is a necessary step because sodium chloride is not soluble in ethanol and its presence facilitate the precipitation of the salt. The mixing operation with the hydroalcoholic solution is carried out inside the high-shear homogenizer **MX-401**, which is sized in order to have a low residence time. The P-406 pump draws the liquid from the bottom of the stirred tank MX-401 and feeds it to the D-401 decanter.

The **D-401** decanter consists of a static decanter where a waste stream containing sodium chloride is then collected from the bottom through the volumetric progressive cavity like **P-407**, and a stream containing ethanol and citric acid is collected by surfacing from the top of the decanter inside the **TK-403** pot tank. The precipitate consists of a brine with a flow rate of 3.2 tons per hour and a concentration of sodium chloride of 6.83%, while the clarified one consists of a liquid stream containing water, 31% ethanol and 5% citric acid, which is sent to the buffer tank **TK-404** which must be sized with a hold-up time of two hours.

6.2.6.3. Ethanol recovery column T-402

The aqueous stream containing ethanol and citric acid is then sent to the second distillation column **T-402**, which separates the ethanol solution from the top, with a concentration close to that of the solution used in the plant (70% $v_{EtOH/}v$), and a diluted solution of citric acid at the bottom.

As for the previous case, to calculate the partition coefficients of the column and the fundamental constructive and operational parameters, the column is simulated on Aspen using first a DSTWU block and finally a RadFrac block.

The column is designed having a number of stages equal to seven, and feed is sent to the third stage. The column works with a reflux ratio equal to 1,2 and distilled to feed ratio equal to 0,81. A total flow of about 7 tons per hour is fed to the column. The obtained distillate stream, with a flow rate of 3,3 tons per hour, has an ethanol concentration of 34,16%, while the bottom stream, with a flow rate of 3,7 tons per hour, has a citric acid concentration of 9,58%. The recovery of ethanol is practically unitary.

The water-ethanol solution is directly sent to the water ethanol solution storage tank **TK-407**, while the citric acid solution needs to be concentrated.

6.2.6.4. Citric Acid concentrator EV-401

The citric acid solution obtained as the bottom stream of the distillation column **T-402** has a citric acid concentration of 9,58% and needs to be concentrated up to the desired value of concentration required in the plant operations (33,33%$_w$). To carry out the operation, it is decided to use an evaporator which uses low pressure steam as the thermal vector.

The output current from the **T-402** column is sent to the **TK-406** buffer tank which must be sized with a two-hour hold-up. By means of the centrifugal pump **P-410** the solution is sent to the evaporator **EV-401**. The evaporator works at a pressure of 1 bar. and allows to obtain a stream of citric acid solution at the desired concentration with a flow rate of about 1 ton per hour.

6.3. Process control logic

Pre-treatment and storage section:

- High- and low-level control of the **H-102** hopper manipulated by opening or closing the servo-assisted valve located in the vertical drop line of the solid from the **H-101** dosing hopper;
- Weight control in the **H-102** dosing hopper by means of the load cells on which it rests by acting on the rotation speed of the rotary feed valve **RFV-101**;
- Control of the temperature of the output gas current from the **DD-101** Rotary Drum Dryer by manipulating the methane flow rate sent to the open-back air draught burner **B-101**;
- Control of the ratio between the flow rate of methane and the flow rate of air fed to the open-back air draught burner **B-101** by manipulating the power delivered by the blower **BL-101**;
- Control of the cooled solid temperature coming from the fluidized bed cooler **FBE-102** by manipulating the air flow draught from the outside through the blower **BL-102**;
- Control of the temperature of the hot air stream outgoing from the economizer **E-301** by manipulating the bypass valve of the stream;
- Weight control in the **H-103** dosing hopper by means of the load cells on which it rests by acting on the rotation speed of the rotary valve which doses the quantity of leaves to be inserted in each big bag.
- High- and low-level control of the **H-105** hopper manipulated by opening or closing the servo-assisted valve located in the vertical drop line of the solid from the **H-104** dosing hopper;
- Weight control in the **H-102** dosing hopper by means of the load cells on which it rests by acting on the rotation speed of the rotary feed valve **RFV-101**.

Lipids and polyphenols extraction section:

- Level control of stirred tanks **MX-201** and **MX-202** by manipulating the delivery of volumetric pumps **P-201** and **P-212**;
- Weight control through load cells of the dosing hoppers connected to the progressive cavity feeders of the extractors by manipulating the rotation speed of the feeder motors;
- Rotation speed control of TURBEX with an SC loop;
- Control of the ratio between the flow rate of solid fed and the flow rate of liquid fed to the extractors **EX-201/202** by manipulating the inlet flow of fresh n-hexane;
- Control of the temperature of the liquid fed to the extractors **EX-201/202** by manipulating the flow of low-pressure steam fed to the **E-201** heat exchanger;
- Control of the level of the squeezed liquid collection tanks **TK-201** and **TK-204** by manipulating the delivery of the volumetric pumps **P-202** and **P-213**;
- High- and low-level control of the hopper of the rotary feed valve **RFV-201** manipulated by opening or closing the servo-assisted valve located in the vertical drop line of the solid from the **H-201** dosing hopper;
- Weight control in the hopper of the rotary feed valve **RVF-201** by means of the load cells on which it rests by acting on the rotation speed of the rotary feed valve motor;
- Temperature control of the nitrogen fed to the flash desolventizer **FD-201** by manipulating the methane flow rate fed to the open-back draught air burner **B-201**;
- Control of the ratio between the flow rate of methane and the flow rate of air fed to the open-back air draught burner **B-201** by manipulating the power delivered by the blower **BL-201**;
- Control of the flow of nitrogen fed to the flash desolventizer **FD-201** by manipulating the flow of fresh nitrogen fed;
- Control of the temperature of the supply to the direct condensation column of the hexane **T-201** by manipulating the flow rate of chilled water fed to the heat exchanger **E-204;**
- **T-201** column pressure control by manipulating the opening of the head stream outlet valve;
- Temperature control of the liquid hexane fed to **T-201** by manipulating the flow rate of refrigerant sent to the **E-205** exchanger;
- Control of the liquid level at the bottom of the **T-201** column by manipulating the delivery of the **P-203** pump;
- Control of the flow rate of hexane fed to the **T-201** column by manipulating the delivery of the **P-204** pump;
- Control of the temperature of the hydroalcoholic solution supplied to the **EX-204/205** extractors by manipulating the flow of low-pressure steam fed to the **E-210** heat exchanger;

- Control of the ratio between the flow rate of solid fed and the flow rate of liquid fed to the extractors **EX-204/205** by manipulating the inlet flow of fresh water-ethanol solution;
- Control of the flow rate fed to the **T-202** column by manipulating the delivery of the volumetric pump **P-206**;
- Pressure control of the **T-202** column by manipulating the chilled water flow rate sent to the **E-207** condenser;
- Level control of the **TK-205** pot tank by manipulating the delivery of the P-**207** pump;
- Control of the recirculated liquid at the head of the column by manipulating the delivery of the **P-207** pump;
- Temperature control of the **T-202** column by manipulating the flow of low-pressure steam fed to the kettle reboiler **E-208**;
- Level control in the kettle reboiler tank by manipulating the delivery of the **P-208** pump;
- Control of the pressure at the head of the **T-203** column by manipulating the opening of the valve of the top stream outlet line;
- Control of the liquid level at the bottom of the **T-203** column by manipulating the delivery of the volumetric pump **P-209;**
- Temperature control of the condensate fed to the **D-201** decanter by manipulating the cooling water flow fed to the **E-209** heat exchanger;
- Control of the level of the decanter **D-201** by manipulating the delivery of the volumetric pump **P-210**;
- Level control of the **TK-209** pot tank by manipulating the delivery of the **P-211** pump;
- Control of the flow rate fed to the membrane ultrafiltration system by manipulating the delivery of the **P-214** pump;
- Control of the flow rate fed to the membrane nanofiltration system by manipulating the delivery of the **P-215** pump;
- Differential pressure control downstream and upstream of the membranes to switch from one membrane to another and start regeneration;
- Control of the flow rate of liquid fed to the **EV-201/202** evaporators by manipulating the delivery of the **P-216** pump;
- Pressure control of the evaporators **EV-201/202** by manipulating the overhead gas stream outlet valve;
- Control of the flow rate fed to the drum dryer **DD-201** by manipulating the delivery of the **P-217** volumetric pump;
- Control of the temperature of the solids leaving the **DD-201** drum dryer by manipulating the flow of steam supplied;

Proteins and arabinoxylans extraction section:

- High- and low-level control of the hoppers connected to the progressive cavity feeders of the extractors manipulated by opening or closing the servo-assisted valve located in the vertical drop line of the solid from the **H-301 H-302 H-303** and **H-304** receiving hoppers;

- Weight control in the hoppers connected to the progressive cavity feeders of the extractors by means of the load cells on which they rest by acting on the rotation speed of the feeder's motors;
- Rotation speed control of all the TURBEX with an SC loop;
- Control of the ratio between the flow rate of solid fed and the flow rate of liquid extractant fed to the extractors by manipulating the inlet flow of fresh extractant;
- Control of the level of the squeezed liquid collection tanks **TK-301** and **TK-302 TK-303** and **TK-309** by manipulating the delivery of the volumetric pumps **P-301 P-309 P-317** and **P-325**;
- Control of the ratio between the flow rate of citric acid solution and the flow rate of liquid extract fed to the stirred tanks **MX-301 MX-304** and **MX-307** by manipulating the inlet flow of the citric acid solution;
- Level control of the decanters by manipulating the delivery of the volumetric pumps that collect the residue;
- Level control of the decanter pot tanks by manipulating the delivery of the pumps installed downstream;
- Control of the ratio between the flow rate of water ethanol solution and the flow rate of liquid extract fed to the stirred tanks **MX-303 MX-306** and **MX-309** by manipulating the inlet flow of the water ethanol solution;
- Control of the flow rate fed to the membrane filtration system by manipulating the delivery of the **P-326** pump;
- Differential pressure control downstream and upstream of the membranes to switch from one membrane to another and start regeneration;
- Control of the flow rate fed to the evaporators **EV-301** and **EV-302** by manipulating the delivery of the **P-327** volumetric pump;
- Pressure control of the evaporators **EV-301** and **EV-302** by manipulating the overhead gas stream outlet valve;
- Level control in the **EV-302** evaporator by manipulating the delivery of the volumetric pump **P-328**;
- Flow control of extracted delivery to **DD-301** and **DD-302** drum dryers by manipulating the delivery of **P-330** and **P-329** volumetric pumps;
- Control of the operating temperature of the drum dryers by manipulating the incoming steam flow.

Ethanol and citric acid recovery section:

- Control of the flow rate fed to the **T-401** column by manipulating the delivery of the **P-401** centrifugal pump;
- Pressure control of the **T-401** column by manipulating the flow of cooling water sent to the **E-401** condenser;
- Level control of the **TK-402** pot tank by manipulating the delivery of the **P-402** pump;

- Control of the recirculated liquid at the head of the column **T-401** by manipulating the delivery of the **P-402** pump;
- Temperature control of the **T-401** column by manipulating the flow of low-pressure steam fed to the kettle reboiler **E-402**;
- Level control in the kettle reboiler **E-402** tank by manipulating the delivery of the **P-403** pump;
- Control of the ratio between the flow rate of liquid and water-ethanol solution fed to the stirred tank **MX-401** by manipulating the flow rate of the water-ethanol solution fed;
- Checking the level of the stirred tank **MX-401** by manipulating the delivery of the **P-406** pump;
- Level control of the **D-401** decanter by manipulating the delivery of the **P-407** volumetric pump;
- Level control of the **TK-403** decanter pot tank by manipulating the delivery of the **P-408** pump installed downstream;
- Control of the flow rate fed to the **T-402** column by manipulating the delivery of the **P-409** centrifugal pump;
- Pressure control of the **T-402** column by manipulating the flow of cooling water sent to the **E-403** condenser;
- Level control of the **TK-405** pot tank by manipulating the delivery of the **P-404** pump;
- Control of the recirculated liquid at the head of the column **T-402** by manipulating the delivery of the **P-404** pump;
- Temperature control of the **T-402** column by manipulating the flow of low-pressure steam fed to the kettle reboiler **E-404**;
- Level control in the kettle reboiler **E-404** tank by manipulating the delivery of the **P-405** pump;
- Control of the flow rate fed to the evaporator **EV-401** by manipulating the delivery of the **P-410** pump;
- Pressure control of the evaporator **EV-401** by manipulating the overhead gas stream outlet valve;
- Level control in the **EV-401** evaporator by manipulating the bottom liquid outlet valve;
- **TK-407** tank level control by manipulating the flow of ethanol sent;
- Control of concentration of ethanol in the **TK-407** tank by manipulating the flow of water sent;
- **TK-408** tank level control by manipulating the flow of acid sent;
- Control of concentration of citric acid in the **TK-408** tank by manipulating the flow of water sent.

Chapter 7

Conclusion

The development of this thesis project, carried out in collaboration with the Andritz group, made it possible to study the prototype of the TURBEX EX30 multistage countercurrent cavitational extractor and to obtain an initial idea on the extraction performance achievable by the machinery, confirming that it is a disruptive technology capable of achieving performance that no other machine in the world can achieve.

The device can be classified as a counter-current multistage extractor that intensifies the extraction process of bioactive compounds from plant matrices by exploiting the phenomena related to hydrodynamic cavitation generated by an appropriately designed rotor-stator system, to micro-mixing carried out in a regime of high turbulence and to generation of high shear stress. It represents the first existing extractor model in the world capable of exploiting all these phenomena in a combined way by feeding the solid in countercurrent with the liquid, presenting itself as a destructive technology in the field of solid-liquid extraction.

The analyses conducted in parallel between the laboratories of the DSTF (Department of Pharma Science and Technology) of the University of Turin and the DISAT (Department of Applied Science and Technology) laboratories of the Polytechnic of Turin, allowed to analyse the extract samples, taken during the functional tests to which the machine was subjected, and the morphological variation undergone by the solid during a single passage inside the extractor.

The results obtained show that the machinery can achieve, in a single passage, performances equal to several theoretical equilibrium stages, ranging between 7 and 15, with an extraction efficiency greater than 97,5%. Following the extraction, the matrix has been reduced in size due to the cavitation phenomena and the high shear stress to which it is subjected inside the TURBEX extraction chamber, presenting also a reduced moisture content. All the signs are registered in accordance with the theory underlying the process intensification phenomena desired by the machinery.

To show the potential applicability of the machinery in the industrial field, with the results obtained it was possible to design two plants for the valorisation of wastes from the agro-food chain. In particular, a plant for the recovery of polyphenols from olive leaves and a plant for the recovery of lipids, polyphenols, proteins and dietary fibers from the brewer's spent grain were designed.

The first plant located in in Turkey, in the city of Içel, is designed to process 25000 tons/y of olive leaves collected as waste from tree pruning and olive oil production. The plant works for a total of 20 weeks and is designed to operate with a turndown ratio between 60% and 110% of nominal capacity. The resulting product consists of a 2% moisture polyphenol powder, containing oleuropein for about 32% by weight, with an overall recovery of 93.21% of the oleuropein initially present in the matrix. Estimating a commercial value of the bulk product of 20 €/kg, the plant allows to potentially obtain an estimated yearly revenue of 49.700.000 €.

The second plant located in in Siberia, in the city of Novosibirsk, is designed to process 20000 tons/y of brewer’s spent grain collected as a residue from the breweries in the neighbouring areas. The plant works for a total of 49 weeks and is designed to operate with a turndown ratio between 60% and 110% of nominal capacity. The plant allows to obtain four distinct products, which find different uses ranging from the nutraceutical, food, cosmetic sector to the production of fine chemicals.

- The first product is obtained from the extraction of the lipid fraction of the BSG and consists of a viscous liquid stream composed of 93% lipids, produced with a capacity of 442 tons per year and a commercial value of 1 €/kg. The overall yield of lipids extraction is of about 99,8%.
- The second product consists of a 53% in weight polyphenol powder with a high concentration of ferulic acid, obtained with a capacity of 93.6 tons per year and a commercial bulk value of 50 €/kg. The overall yield of polyphenols extraction, influenced by the previous extraction process, is around 54.6%.
- The third product consists of a 90% in weight protein powder, obtained with a capacity of 1245 tons per year and a commercial bulk value of 2,5 €/kg. The overall yield of proteins extraction is around 95.5%.
- The last product consists of a dietary fiber powder rich in arabinoxylans, obtained with a capacity of 1160,9 tons per year and a commercial bulk value of 10 €/kg. The overall yield of proteins extraction is around 67,9%.

Based on the value of the obtained products, the plant has an overall estimated yearly revenue of 19.800.100 €.

The two projects have again highlighted that the high extraction performance of TURBEX can be used for industrial applications, bringing as benefits the reduction of operating costs related to the use of large volumes of solvents, long extraction times and maximum exploitation of the raw material. All these advantages are achievable using a machine that has a slimmer silhouette than conventional percolation or stirred vessel extractors.

This thesis can be considered as the first study carried out on the TURBEX EX30 industrial prototype, with the aim of having a first ballpark idea about the potential of the machinery and confirming the expectations related to it. New studies will be carried out on the machinery, which in the coming months will be tested using different plant matrices with the aim of studying and fine-tuning the optimal operating parameters. From what was found with this study, it is confirmed that the TURBEX EX30 has all the prerogatives to gain a foothold in the world market, opening the doors to more efficient, profitable and sustainable extraction processes.

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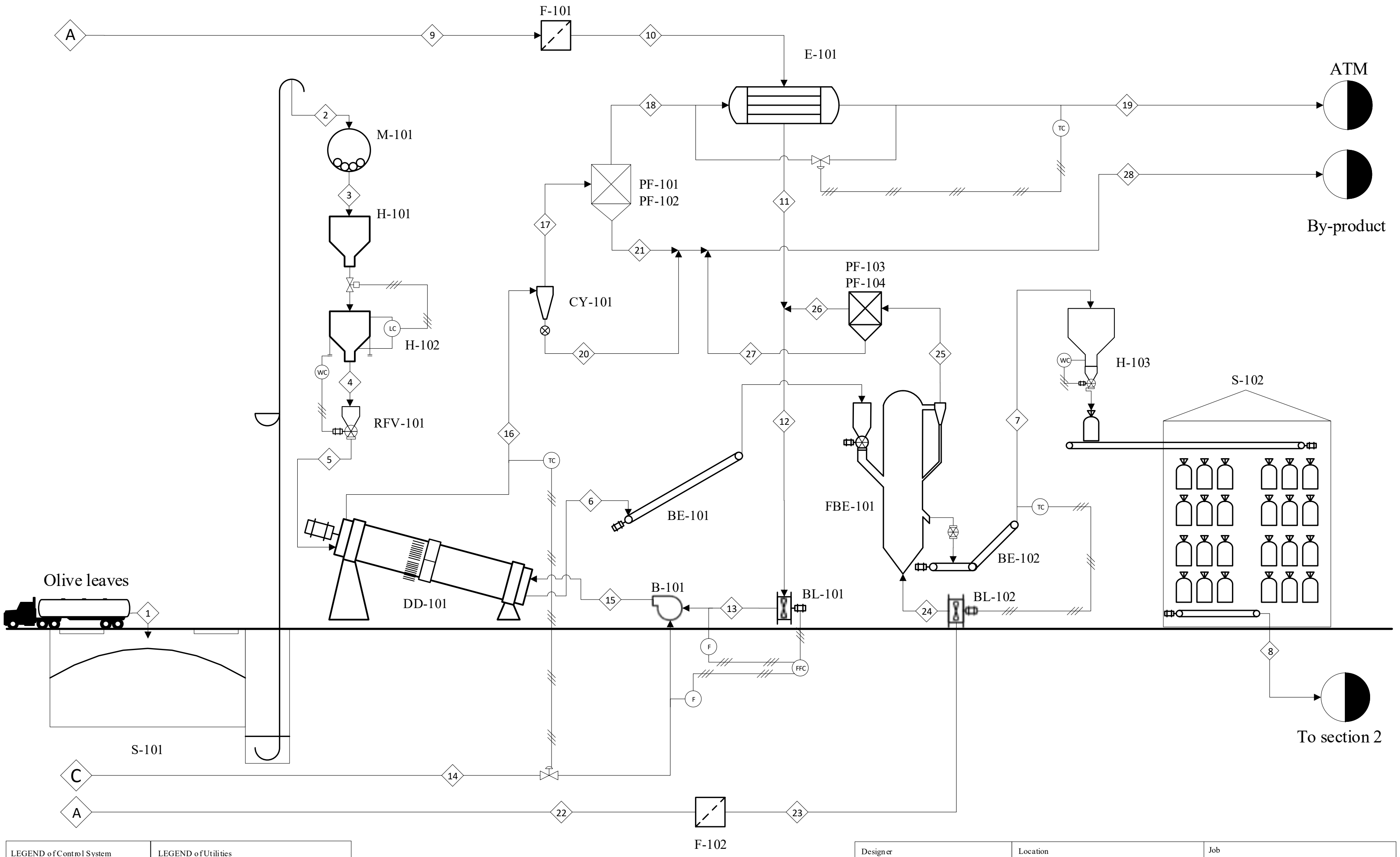
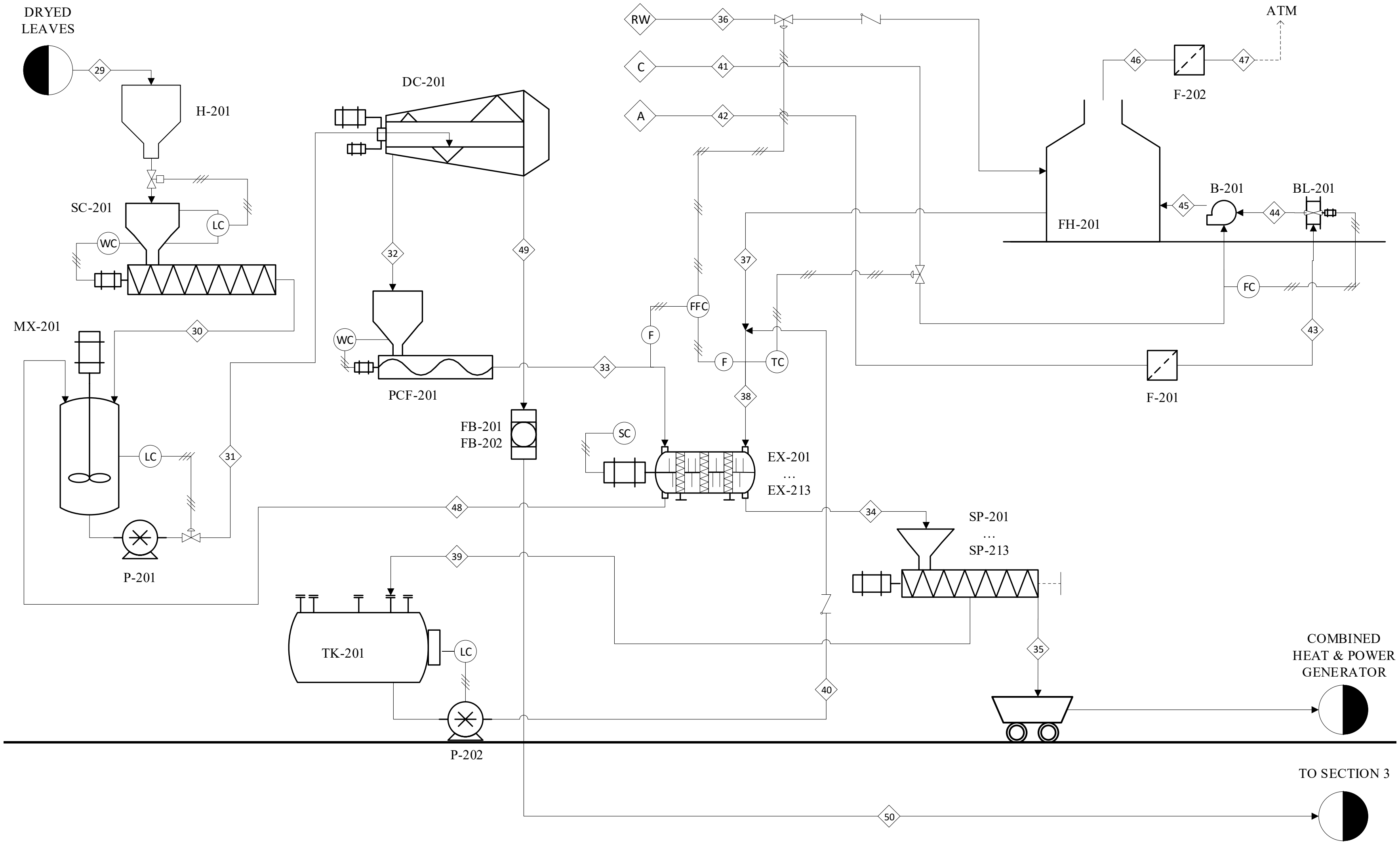
Appendix A.

Olive leaves plant Process Flow Diagram (PFD)



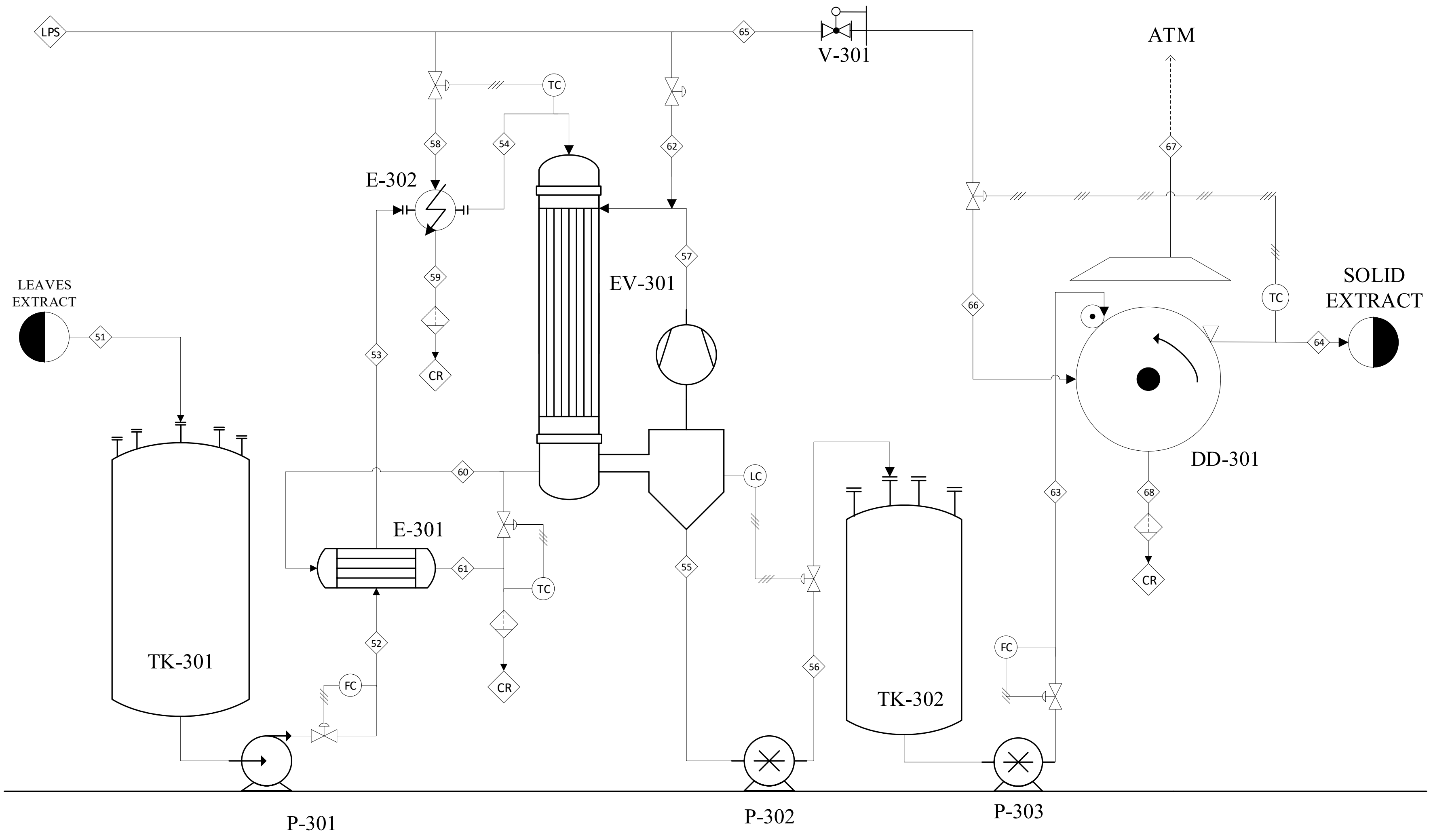
LEGEND of Control System	LEGEND of Utilities	
LC – Level Controller PC – Pressure Controller TC – Temperature Controller FC – Flow Controller WC – Weight Controller	A – Air C - Methane RW – River Water DW – Desalinated Water MW – Main Water	LPS – Low Pressure Steam CS – Condensing Steam ATM – To Atmosphere

Designer Alessio Mannalà	Location Içel – Mercin (Turkey)	Job Oleuropein Extract
	Unit Olive Leaves Processing	Doc n° OLP-FS-001
Date 27/08/2020	Plant OL – 25000 ton/year	Sheet 1 of 3
	Description Flow scheme	Revision 1



LEGEND of Control System	LEGEND of Utilities	
LC – Level Controller PC – Pressure Controller TC – Temperature Controller FC – Flow Controller WC – Weight Controller	A – Air C - Methane RW – River Water DW – Desalinated Water MW – Main Water	LPS – Low Pressure Steam CS – Condensing Steam ATM – To Atmosphere

Designer Alessio Mannalà	Location Içel – Mercin (Turkey)	Job Oleuropein Extract
	Unit Olive Leaves Processing	Doc n° OLP-FS-002
Date 27/08/2020	Plant OL – 25000 ton/year	Sheet 2 of 3
	Description Flow scheme	Revision 1



LEGEND of Control System	LEGEND of Utilities	
LC – Level Controller PC – Pressure Controller TC – Temperature Controller FC – Flow Controller WC – Weight Controller	A – Air C - Methane RW – River Water DW – Desalinated Water MW – Main Water	LPS – Low Pressure Steam CS – Condensing Steam ATM – To Atmosphere

Designer Alessio Mannalà	Location Içel – Mercin (Turkey)	Job Oleuropein Extract
	Unit Olive Leaves Processing	Doc n° OLP-FS-003
Date 27/08/2020	Plant OL – 25000 ton/year	Sheet 3 of 3
	Description Flow scheme	Revision 1

Appendix B.

Olive leaves plant Stream Table

Table 8.1 - Olive leaves Valorization plant: Stream Table Section 1

Stream n°	**Flowrate *[ton/h]***	**Temperature *[°C]***	**Pressure *[bar]***	**Vapor Fraction**	**Liquid Fraction**	**Solid Fraction**	**w%** $_{\text{Oleuropein}}$	**w%** $_{\text{Extractable}}$	**w%** $_{H_2O}$	**w%** $_{\text{Unextratable}}$	**w%** $_{O_2}$	**w%** $_{N_2}$	**w%** $_{CO_2}$	**w%** $_{CH_4}$
1	12,40	17,0	1,00	0,00	0,00	1,00	3,51%	32,24%	49,83%	14,42%	0,00%	0,00%	0,00%	0,00%
2	12,40	17,0	1,00	0,00	0,00	1,00	3,51%	32,24%	49,83%	14,42%	0,00%	0,00%	0,00%	0,00%
3	12,40	20,0	1,00	0,00	0,00	1,00	3,51%	32,24%	49,83%	14,42%	0,00%	0,00%	0,00%	0,00%
4	12,40	20,0	1,00	0,00	0,00	1,00	3,51%	32,24%	49,83%	14,42%	0,00%	0,00%	0,00%	0,00%
5	12,40	20,0	1,00	0,00	0,00	1,00	3,51%	32,24%	49,83%	14,42%	0,00%	0,00%	0,00%	0,00%
6	6,91	80,0	1,00	0,00	0,00	1,00	6,30%	57,84%	10,00%	25,86%	0,00%	0,00%	0,00%	0,00%
7	6,91	40,0	1,00	0,00	0,00	1,00	6,30%	57,84%	10,00%	25,86%	0,00%	0,00%	0,00%	0,00%
8	6,91	40,0	1,00	0,00	0,00	1,00	6,30%	57,84%	10,00%	25,86%	0,00%	0,00%	0,00%	0,00%
9	141,90	10,0	1,00	1,00	0,00	0,00	0,00%	0,00%	4,66%	0,00%	28,40%	71,00%	0,00%	0,00%
10	141,90	10,0	1,00	1,00	0,00	0,00	0,00%	0,00%	4,66%	0,00%	28,40%	71,00%	0,00%	0,00%
11	141,90	44,6	1,00	1,00	0,00	0,00	0,00%	0,00%	0,60%	0,00%	28,40%	71,00%	0,00%	0,00%
12	157,49	44,1	1,00	1,00	0,00	0,00	0,00%	0,00%	0,60%	0,00%	28,40%	71,00%	0,00%	0,00%
13	157,49	44,1	1,00	1,00	0,00	0,00	0,00%	0,00%	0,60%	0,00%	28,40%	71,00%	0,00%	0,00%
14	0,27	10,0	1,00	1,00	0,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	100,00%
15	157,76	200,0	1,00	1,00	0,00	0,00	0,00%	0,00%	0,98%	0,00%	27,67%	70,88%	0,47%	0,00%
16	163,25	110,0	1,00	1,00	0,00	0,00	0,00%	0,00%	4,31%	0,00%	26,74%	68,50%	0,45%	0,00%
17	163,25	110,0	1,00	1,00	0,00	0,00	0,00%	0,00%	4,31%	0,00%	26,74%	68,50%	0,45%	0,00%
18	163,25	110,0	1,00	1,00	0,00	0,00	0,00%	0,00%	4,31%	0,00%	26,74%	68,50%	0,45%	0,00%
19	163,25	80,0	1,00	1,00	0,00	0,00	0,00%	0,00%	4,31%	0,00%	26,74%	68,50%	0,45%	0,00%
20	-	110,0	1,00	0,00	0,00	1,00	-	-	-	-	-	-	-	-
21	-	110,0	1,00	0,00	0,00	1,00	-	-	-	-	-	-	-	-
22	15,59	10,0	1,00	1,00	0,00	0,00	0,00%	0,00%	4,66%	0,00%	28,40%	71,00%	0,00%	0,00%
23	15,59	10,00	1,00	1,00	0,00	0,00	0,00%	0,00%	4,66%	0,00%	28,40%	71,00%	0,00%	0,00%
24	15,59	10,00	1,00	1,00	0,00	0,00	0,00%	0,00%	4,66%	0,00%	28,40%	71,00%	0,00%	0,00%
25	15,59	40,0	1,00	1,00	0,00	0,00	0,00%	0,00%	4,66%	0,00%	28,40%	71,00%	0,00%	0,00%
26	15,59	40,00	1,00	1,00	0,00	0,00	0,00%	0,00%	4,66%	0,00%	28,40%	71,00%	0,00%	0,00%
27	-	40,00	1,00	0,00	0,00	1,00	-	-	-	-	-	-	-	-
28	-	86,67	1,00	0,00	0,00	1,00	-	-	-	-	-	-	-	-

Table 8.2 - Olive leaves Valorization plant: Stream Table Section 2

Stream n°	**Flowrate** ***[ton/h]***	**Temperature** ***[°C]***	**Pressure** ***[bar]***	**Vapor Fraction**	**Liquid Fraction**	**Solid Fraction**	**w% Oleuropein**	**w% Extractable**	**w% H_2O**	**w% Unextratable**	**w% O_2**	**w% N_2**	**w% CO_2**	**w% CH_4**
29	5,81	15,0	1,0	0,00	0,00	1,00	6,30%	57,84%	10,00%	25,86%	0,00%	0,00%	0,00%	0,00%
30	5,81	15,0	1,0	0,00	0,00	1,00	6,30%	57,84%	10,00%	25,86%	0,00%	0,00%	0,00%	0,00%
31	23,44	46,3	1,0	0,00	0,39	0,61	3,50%	18,16%	71,93%	6,41%	0,00%	0,00%	0,00%	0,00%
32	10,20	46,3	1,0	0,00	0,00	1,00	4,70%	35,13%	45,44%	14,73%	0,00%	0,00%	0,00%	0,00%
33	10,20	46,3	1,0	0,00	0,00	1,00	4,70%	35,13%	45,44%	14,73%	0,00%	0,00%	0,00%	0,00%
34	8,42	50,0	1,0	0,00	0,00	1,00	0,30%	31,87%	50,00%	17,83%	0,00%	0,00%	0,00%	0,00%
35	8,42	50,0	1,0	0,00	0,00	1,00	0,30%	31,87%	50,00%	17,83%	0,00%	0,00%	0,00%	0,00%
36	17,42	15,0	1,0	0,00	1,00	0,00	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%
37	17,42	50,0	1,0	0,00	1,00	0,00	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%
38	18,99	50,0	1,0	0,00	1,00	0,00	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%
39	1,57	50,0	1,0	0,00	1,00	0,00	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%
40	1,57	50,0	1,0	0,00	1,00	0,00	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%
41	0,05	10,0	1,0	1,00	0,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	100,00%
42	1,05	10,0	1,0	1,00	0,00	0,00	0,00%	0,00%	0,60%	0,00%	28,40%	71,00%	0,00%	0,00%
43	1,05	10,0	1,0	1,00	0,00	0,00	0,00%	0,00%	0,60%	0,00%	28,40%	71,00%	0,00%	0,00%
44	1,05	10,0	1,0	1,00	0,00	0,00	0,00%	0,00%	0,60%	0,00%	28,40%	71,00%	0,00%	0,00%
45	1,10	2286,0	1,0	1,00	0,00	0,00	0,00%	0,00%	11,48%	0,00%	7,65%	67,55%	13,32%	0,00%
46	1,10	180,0	1,0	1,00	0,00	0,00	0,00%	0,00%	11,48%	0,00%	7,65%	67,55%	13,32%	0,00%
47	1,10	180,0	1,0	1,00	0,00	0,00	0,00%	0,00%	11,48%	0,00%	7,65%	67,55%	13,32%	0,00%
48	13,24	50,0	1,0	0,00	1,00	0,00	2,58%	5,10%	92,33%	0,00%	0,00%	0,00%	0,00%	0,00%
49	13,24	46,3	1,0	0,00	1,00	0,00	2,58%	5,10%	92,33%	0,00%	0,00%	0,00%	0,00%	0,00%
50	13,24	46,3	1,0	0,00	1,00	0,00	2,58%	5,10%	92,33%	0,00%	0,00%	0,00%	0,00%	0,00%

Table 8.3 - Olive Leaves Valorization Plant: Stream Table Section3

Stream n°	**Flowrate** ***[ton/h]***	**Temperature** ***[°C]***	**Pressure** ***[bar]***	**Vapor Fraction**	**Liquid Fraction**	**Solid Fraction**	**w%** $_{\text{Oleuropein}}$	**w%** $_{\text{Extractable}}$	**w%** $_{\text{H2O}}$	**w%** $_{\text{Unextratable}}$	**w%** $_{\text{O2}}$	**w%** $_{\text{N2}}$	**w%** $_{\text{CO2}}$	**w%** $_{\text{CH4}}$
51	9,46	50,0	1,0	0,00	1,00	0,00	2,58%	5,10%	92,33%	0,00%	0,00%	0,00%	0,00%	0,00%
52	9,46	50,0	1,0	0,00	1,00	0,00	2,58%	5,10%	92,33%	0,00%	0,00%	0,00%	0,00%	0,00%
53	9,46	65,0	1,0	0,00	1,00	0,00	2,58%	5,10%	92,33%	0,00%	0,00%	0,00%	0,00%	0,00%
54	9,46	85,1	1,0	0,00	1,00	0,00	2,58%	5,10%	92,33%	0,00%	0,00%	0,00%	0,00%	0,00%
55	2,42	100,0	1,0	0,00	1,00	0,00	10,07%	19,93%	70,00%	0,00%	0,00%	0,00%	0,00%	0,00%
56	2,42	100,0	1,0	0,00	1,00	0,00	10,07%	19,93%	70,00%	0,00%	0,00%	0,00%	0,00%	0,00%
57	7,04	194,0	2,0	1,00	0,00	0,00	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%
58	0,35	158,8	6,0	1,00	0,00	0,00	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%
59	0,35	158,8	6,0	1,00	0,00	0,00	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%
60	7,04	120,2	2,0	0,00	1,00	0,00	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%
61	7,04	100,0	2,0	0,00	1,00	0,00	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%
62	-	158,8	6,0	1,00	0,00	0,00	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%
63	2,42	95,0	1,0	0,00	1,00	0,00	10,07%	19,93%	70,00%	0,00%	0,00%	0,00%	0,00%	0,00%
64	0,74	95,0	1,0	0,00	0,00	1,00	32,90%	65,10%	2,00%	0,00%	0,00%	0,00%	0,00%	0,00%
65	1,69	158,0	6,0	1,00	0,00	0,00	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%
66	1,69	153,5	2,0	1,00	0,00	0,00	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%
67	1,68	100,0	1,0	1,00	0,00	0,00	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%
68	1,69	120,0	2,0	0,00	1,00	0,00	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%

Appendix C.

Olive leaves equipment list

Pre-treatment and storage section:

The first section of the plant has a total of 22 devices. The list is shown in full in the Table 8.4.

Table 8.4 - Pre-treatment and storage section equipment list

NAME TAG	DESCRIPTION
B-101	Open back-air draught burner
BE-101	Belt elevator
BE-102	Belt elevator
BL-101	Air Blower
BL-102	Air Blower
CY-101	Cyclone for dust
DD-101	Rotary Drum Dryier
E-101	Gas-gas heat exchanger
F-101	Air Filter
F-102	Air filter
FBE-101	Fludized beed cooler for solids
H-101	Receiving hopper
H-102	Dosing hopper
H-103	Dosing hopper
M-101	Mill for olive leaves grinding
PF-101	Bag filter for dust
PF-102	Bag filter for dust
PF-103	Bag filter for dust
PF-104	Bag filter for dust
RFV-101	Rotrary feeding valve
S-101	Underground collecting tank for green olive leaves
S-102	Storage warehous for dried olive leaves

Extraction section:

The second section of the plant has a total of 53 devices, 2 of which are pumps. The list is shown in full in the Table 8.5.

Table 8.5 - Extraction section equipment list.

NAME TAG	DESCRIPTION
B-201	Burner for FH-201
BL-201	Air Blower for B-201
DC-201	Decanter Centrifuge
EX-201	TURBEX EX30 Extractor
EX-202	TURBEX EX30 Extractor
EX-203	TURBEX EX30 Extractor
EX-204	TURBEX EX30 Extractor
EX-205	TURBEX EX30 Extractor
EX-206	TURBEX EX30 Extractor
EX-207	TURBEX EX30 Extractor

EX-208	TURBEX EX30 Extractor
EX-209	TURBEX EX30 Extractor
EX-210	TURBEX EX30 Extractor
EX-211	TURBEX EX30 Extractor
EX-212	TURBEX EX30 Extractor
EX-213	TURBEX EX30 Side Extractor
F-201	Air Filter
F-202	Air filter
FB-201	Filter bag for liquids
FB-202	Filter bag for liquids
FH-201	Fired heater
H-201	Receiving hopper
MX-201	Stirred tank
P-201	Volumetric pump for sludges
P-202	Volumetric pump for sludges
PCF-201	Progressive cavity feeder
PCF-202	Progressive cavity feeder
PCF-203	Progressive cavity feeder
PCF-204	Progressive cavity feeder
PCF-205	Progressive cavity feeder
PCF-206	Progressive cavity feeder
PCF-207	Progressive cavity feeder
PCF-208	Progressive cavity feeder
PCF-209	Progressive cavity feeder
PCF-210	Progressive cavity feeder
PCF-211	Progressive cavity feeder
PCF-212	Progressive cavity feeder
PCF-213	Side Progressive cavity feeder
SC-201	Screw conveyor
SP-201	Squeezing press
SP-202	Squeezing press
SP-203	Squeezing press
SP-204	Squeezing press
SP-205	Squeezing press
SP-206	Squeezing press
SP-207	Squeezing press
SP-208	Squeezing press
SP-209	Squeezing press
SP-210	Squeezing press
SP-211	Squeezing press
SP-212	Squeezing press
SP-213	Side Squeezing press
TK-201	Squeezed liquid collection tank

Concentration section:

The third section of the plant has a total of 10 devices, 3 of which are pumps. The list is shown in full in the Table 8.6.

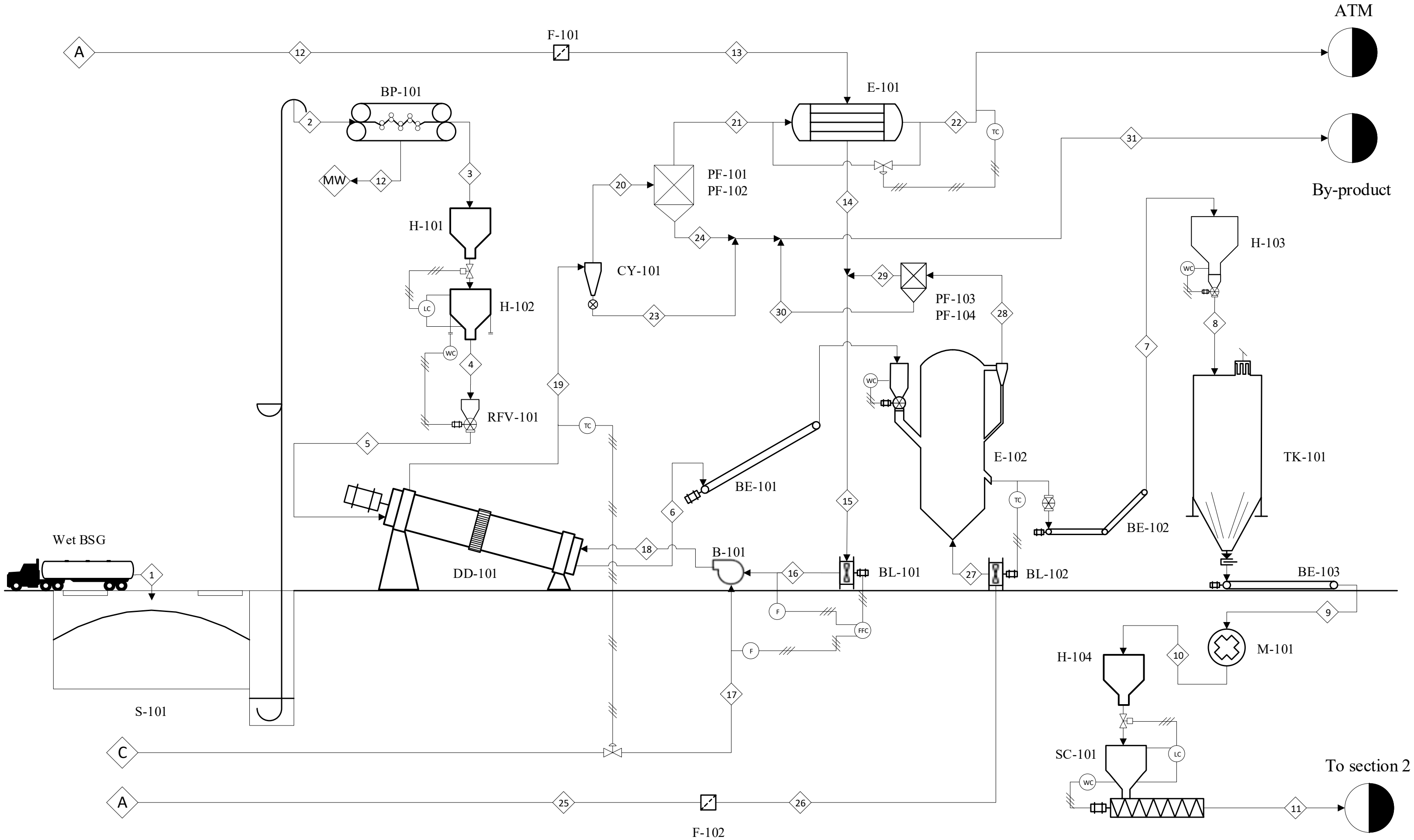
Table 8.6 - Concentration section steam table

NAME TAG	DESCRIPTION
DD-301	Rotary Drum Dryier
E-301	Economizer heat exchanger
E-302	Heater
EV-301	Evaporator
P-301	Centrifugal pump
P-302	Volumetric pump for sludges
P-303	Volumetric pump for sludges
TK-301	Buffer tank
TK-302	Buffer tank
V-301	Lamination valve for steam

The plant has a total of 85 devices, 5 of which are pumps.

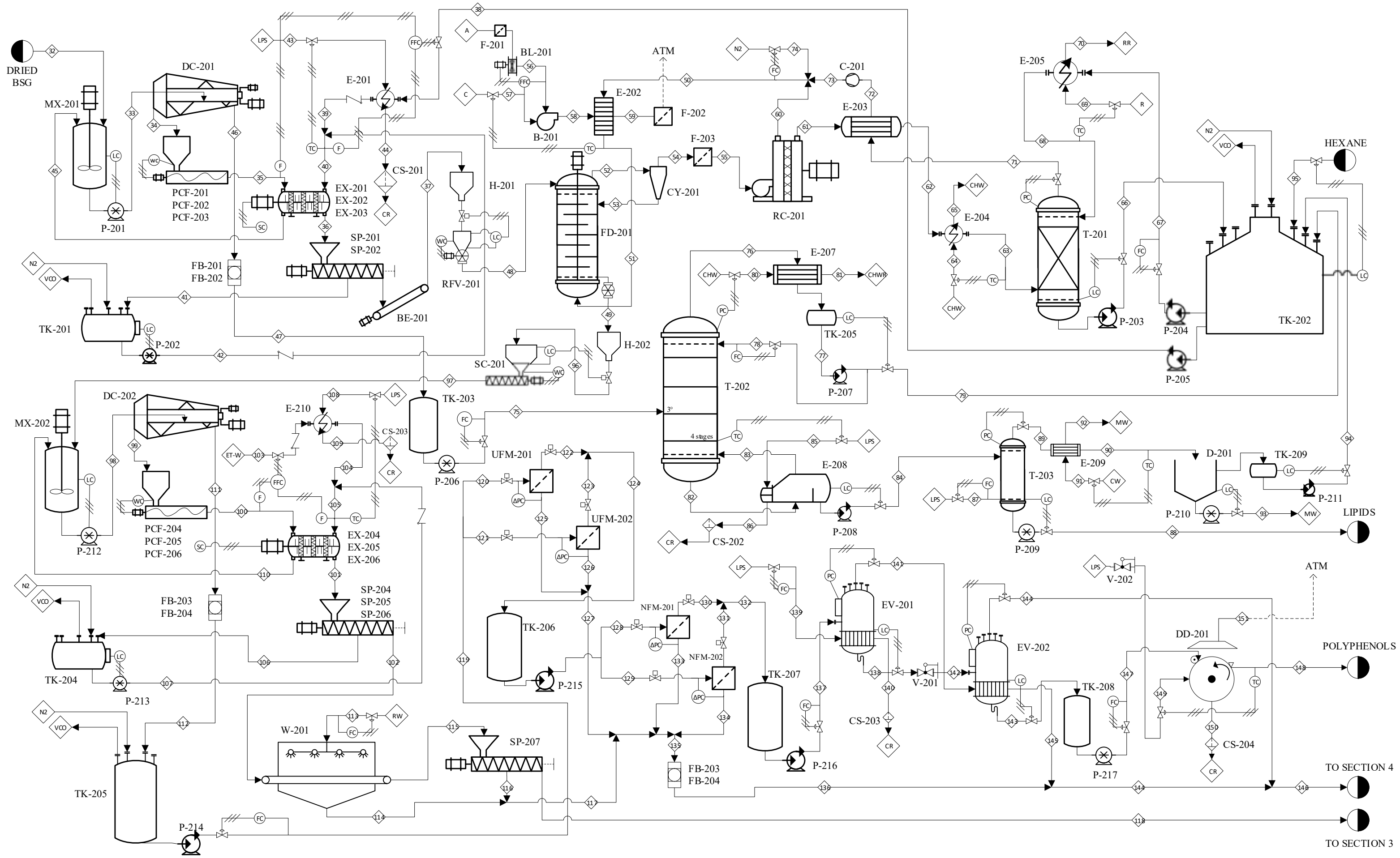
Appendix D.

BSG plant Process Flow Diagram (PFD)



LEGEND of Control System		LEGEND of Utilities		
LC – Level Controller	F – Flow Sensor/Transmitter	LPS – Low Pressure Steam	A – Air	NaOH 1M – 1M Sodium Hydroxide Solution
PC – Pressure Controller	SC – Switch Controller	CR – Condense Return	C - Methane	NaOH 0.5M – 0.5M Sodium Hydroxide Solution
TC – Temperature Controller		CW – Cooling water	R – Refrigerant (R134a)	NaOH 0.1M – 0.1M Sodium Hydroxide Solution
FC – Flow Controller		RW – River Warter	CA – Citric acid solution	
WC – Weight Controller		MW – Main water	HCL – Cloridric acid solution	
FFC – Flow Fraction Ratio Controller		HW – Hot Water	ET-W – Water Ethanol solution (70%$_{mol}$)	
		CHW – Chilled Water		

Designer Alessio Mannalà	Location Novosibirsk (Siberia – CSI)	Job Oils, Polyphenols, Proteins and Arabinoxylans Extracts
	Unit Brewer's Spent Grain Processing	Doc n° BSG-FS-001
Date 10/09/2020	Plant BSG – 20000 ton/y	Sheet 1 of 4
	Description Flow scheme	Revision 1



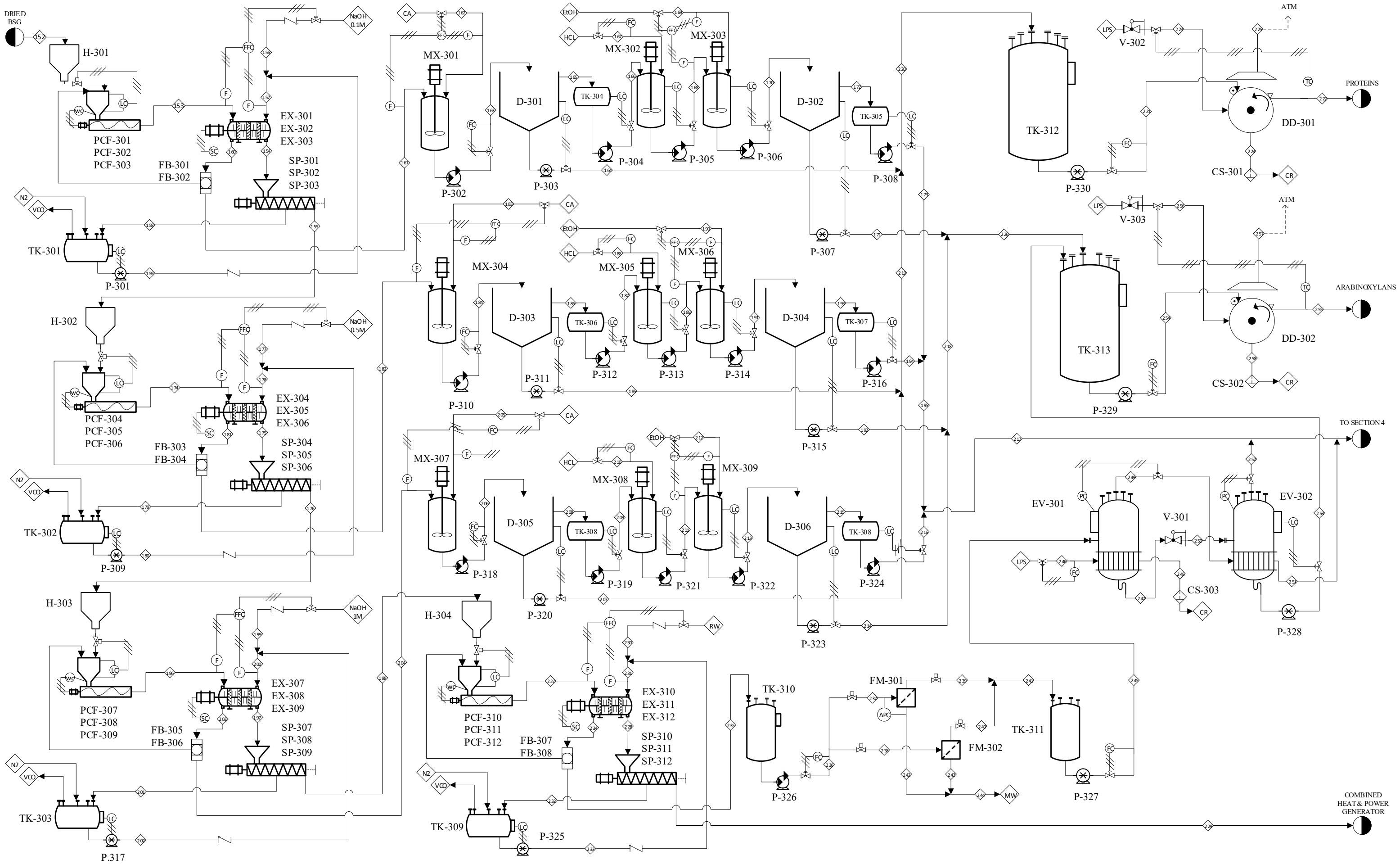
LEGEND of Control System

LC – Level Controller
PC – Pressure Controller
TC – Temperature Controller
FC – Flow Controller
WC – Weight Controller
FFC – Flow Fraction Ratio Controller
F – Flow Sensor/Transmitter
SC – Switch Controller

LEGEND of Utilities

LPS – Low Pressure Steam
CR – Condense Return
CW – Cooling water
RW – River Warter
MW – Main water
HW – Hot Water
CHW – Chilled Water
A – Air
C - Methane
R – Refrigerant (R134a)
CA – Citric acid solution
HCL – Cloridric acid solution
ET-W – Water Ethanol solution (70%$_{mol}$)
NaOH 1M – 1M Sodium Hydroxide Solution
NaOH 0.5M – 0.5M Sodium Hydroxide Solution
NaOH 0.1M – 0.1M Sodium Hydroxide Solution

Designer Alessio Mannalà	Location Novosibirsk (Siberia – CSI)	Job Oils, Polyphenols, Proteins and Arabinoxylans Extracts
	Unit Brewer's Spent Grain Processing	Doc n° BSG-FS-002
Date 10/09/2020	Plant BSG – 20000 ton/y	Sheet 2 of 4
	Description Flow scheme	Revision 1



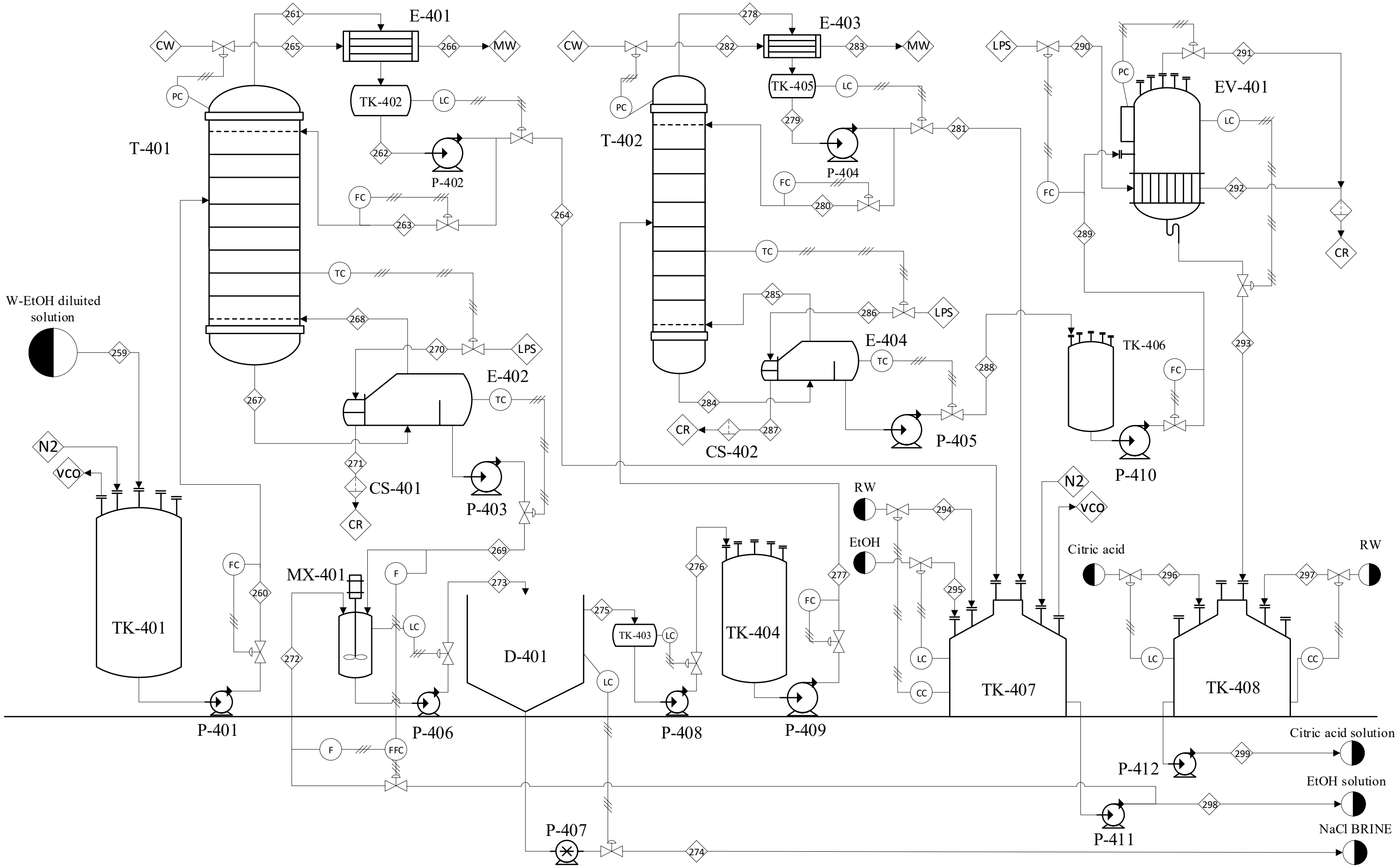
LEGEND of Control System

LC – Level Controller
PC – Pressure Controller
TC – Temperature Controller
FC – Flow Controller
WC – Weight Controller
FFC – Flow Fraction Ratio Controller
F – Flow Sensor/Transmitter
SC – Switch Controller

LEGEND of Utilities

LPS – Low Pressure Steam
CR – Condense Return
CW – Cooling water
RW – River Warter
MW – Main water
HW – Hot Water
CHW – Chilled Water
A – Air
C - Methane
R – Refrigerant (R134a)
CA – Citric acid solution
HCL – Cloridric acid solution
ET-W – Water Ethanol solution (70%$_{\text{mol}}$)
NaOH 1M – 1M Sodium Hydroxide Solution
NaOH 0.5M – 0.5M Sodium Hydroxide Solution
NaOH 0.1M – 0.1M Sodium Hydroxide Solution

Designer Alessio Mannalà	Location Novosibirsk (Siberia – CSI)	Job Oils, Polyphenols, Proteins and Arabinoxylans Extracts
	Unit Brewer's Spent Grain Processing	Doc n° BSG-FS-003
Date 10/09/2020	Plant BSG – 20000 ton/y	Sheet 3 of 4
	Description Flow scheme	Revision 1



LEGEND of Control System		LEGEND of Utilities		
LC – Level Controller PC – Pressure Controller TC – Temperature Controller FC – Flow Controller WC – Weight Controller FFC – Flow Fraction Ratio Controller	F – Flow Sensor/Transmitter SC – Switch Controller	LPS – Low Pressure Steam CR – Condense Return CW – Cooling water RW – River Warter MW – Main water HW – Hot Water CHW – Chilled Water	A – Air C - Methane R – Refrigerant (R134a) CA – Citric acid solution HCL – Cloridric acid solution ET-W – Water Ethanol solution (70%$_{mol}$)	NaOH 1M – 1M Sodium Hydroxide Solution NaOH 0.5M – 0.5M Sodium Hydroxide Solution NaOH 0.1M – 0.1M Sodium Hydroxide Solution

Designer Alessio Mannalà	Location Novosibirsk (Siberia – CSI)	Job Oils, Polyphenols, Proteins and Arabinoxylans Extracts
	Unit Brewer's Spent Grain Processing	Doc n° BSG-FS-004
Date 10/09/2020	Plant BSG – 20000 ton/y	Sheet 4 of 4
	Description Flow scheme	Revision 1

Appendix E.

BSG plant Stream Table

Table 8.7 – BSG Valoriation Plant: Stream, Table Section 1

n°	Flowrate [kg/h]	Temp. [°C]	Pressure [bar]	V_{FRAC}	L_{FRAC}	S_{FRAC}	WEIGHT COMPOSITION																	
							H_2O	Cell.	Hemicell.	Lignin	Protein	PPs	Lipids	Ash	C_6H_{14}	Ethanol	NaOH	C.A.	HCl	NaCl	N_2	O_2	CO_2	CH_4
1	2424,2	10,0	1,0	0,00	0,00	1,00	76,00%	5,21%	7,10%	2,30%	5,90%	0,46%	2,06%	0,96%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
2	2424,2	10,0	1,0	0,00	0,00	1,00	76,00%	5,21%	7,10%	2,30%	5,90%	0,46%	2,06%	0,96%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
3	1662,3	10,0	1,0	0,00	0,00	1,00	65,00%	7,60%	10,36%	3,36%	8,61%	0,67%	3,01%	1,40%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
4	1662,3	10,0	1,0	0,00	0,00	1,00	65,00%	7,60%	10,36%	3,36%	8,61%	0,67%	3,01%	1,40%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
5	1662,3	10,0	1,0	0,00	0,00	1,00	65,00%	7,60%	10,36%	3,36%	8,61%	0,67%	3,01%	1,40%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
6	661,2	80,0	1,0	0,00	0,00	1,00	12,00%	19,10%	26,05%	8,45%	21,65%	1,67%	7,57%	3,52%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
7	661,2	25,0	1,0	0,00	0,00	1,00	12,00%	19,10%	26,05%	8,45%	21,65%	1,67%	7,57%	3,52%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
8	661,2	25,0	1,0	0,00	0,00	1,00	12,00%	19,10%	26,05%	8,45%	21,65%	1,67%	7,57%	3,52%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
9	661,2	25,0	1,0	0,00	0,00	1,00	12,00%	19,10%	26,05%	8,45%	21,65%	1,67%	7,57%	3,52%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
10	661,2	25,0	1,0	0,00	0,00	1,00	12,00%	19,10%	26,05%	8,45%	21,65%	1,67%	7,57%	3,52%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
11	661,2	25,0	1,0	0,00	0,00	1,00	12,00%	19,10%	26,05%	8,45%	21,65%	1,67%	7,57%	3,52%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
12	28375,8	10,0	1,0	1,00	0,00	0,00	0,60%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	71,00%	28,40%	0,00%	0,00%
13	28375,8	10,0	1,0	1,00	0,00	0,00	0,60%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	71,00%	28,40%	0,00%	0,00%
14	28375,8	42,0	1,0	1,00	0,00	0,00	0,60%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	71,00%	28,40%	0,00%	0,00%
15	29209,3	42,5	1,0	1,00	0,00	0,00	0,60%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	71,00%	28,40%	0,00%	0,00%
16	29209,3	42,5	1,0	1,00	0,00	0,00	0,60%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	71,00%	28,40%	0,00%	0,00%
17	85,3	10,0	1,0	1,00	0,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	100,00%
18	29294,6	200,0	1,0	1,00	0,00	0,00	1,25%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	70,79%	27,16%	0,80%	0,00%
19	30295,8	110,0	1,0	1,00	0,00	0,00	4,52%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	68,45%	26,26%	0,77%	0,00%
20	30295,8	110,0	1,0	1,00	0,00	0,00	4,52%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	68,45%	26,26%	0,77%	0,00%
21	30295,8	110,0	1,0	1,00	0,00	0,00	4,52%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	68,45%	26,26%	0,77%	0,00%
22	30295,8	80,0	1,0	1,00	0,00	0,00	4,52%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	68,45%	26,26%	0,77%	0,00%
23	-	110,0	1,0	0,00	0,00	1,00	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
24	-	110,0	1,0	0,00	0,00	1,00	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
25	833,5	10,0	1,0	1,00	0,00	0,00	0,60%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	71,00%	28,40%	0,00%	0,00%
26	833,5	10,0	1,0	1,00	0,00	0,00	0,60%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	71,00%	28,40%	0,00%	0,00%
27	833,5	10,0	1,0	1,00	0,00	0,00	0,60%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	71,00%	28,40%	0,00%	0,00%
28	833,5	60,0	1,0	1,00	0,00	0,00	0,60%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	71,00%	28,40%	0,00%	0,00%
29	833,5	60,0	1,0	1,00	0,00	0,00	0,60%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	71,00%	28,40%	0,00%	0,00%
30	-	60,0	1,0	0,00	0,00	1,00	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
31	-	93,3	1,0	0,00	0,00	1,00	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-

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Table 8.8 - BSG Valorization Plant: Stream Table, Section 2

n°	Flowrate [kg/h]	T [°C]	P [bar]	V_{FRAC}	L_{FRAC}	S_{FRAC}	WEIGHT COMPOSITION																	
							H_2O	Cell.	Hemic.	Lignin	Protein	PPs	Lipids	Ash	C_6H_{14}	Ethanol	NaOH	C.A.	HCl	NaCl	N_2	O_2	CO_2	CH_4
32	661,2	25,0	1,00	0,00	0,00	1,00	12,00%	19,10%	26,05%	8,45%	21,65%	1,67%	7,57%	3,52%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
33	1291,7	46,0	1,00	0,00	0,31	0,69	6,14%	9,77%	13,33%	4,32%	11,08%	1,22%	9,42%	1,80%	42,90%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
34	851,2	46,0	1,00	0,00	0,00	1,00	9,32%	14,83%	20,23%	6,56%	16,81%	1,47%	8,41%	2,73%	19,62%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
35	851,2	46,0	1,00	0,00	0,00	1,00	9,32%	14,83%	20,23%	6,56%	16,81%	1,47%	8,41%	2,73%	19,62%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
36	842,2	61,0	1,00	0,00	0,00	1,00	9,42%	14,99%	20,45%	6,63%	17,00%	0,92%	0,00%	2,76%	27,83%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
37	774,2	61,0	1,00	0,00	0,00	1,00	10,25%	16,31%	22,24%	7,21%	18,49%	1,00%	0,00%	3,01%	21,49%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
38	593,2	25,0	1,00	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
39	593,2	67,0	1,00	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
40	621,5	67,0	1,00	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
41	28,3	61,0	1,00	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
42	28,3	61,0	1,00	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
43	28,8	158,0	6,00	1,00	0,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
44	28,8	158,0	6,00	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
45	630,5	61,0	1,00	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,75%	11,36%	0,00%	87,89%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
46	440,5	61,0	1,00	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,75%	11,36%	0,00%	87,89%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
47	440,5	61,0	1,00	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,75%	11,36%	0,00%	87,89%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
48	774,2	61,0	1,00	0,00	0,00	1,00	10,25%	16,31%	22,24%	7,21%	18,49%	1,00%	0,00%	3,01%	21,49%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
49	607,8	68,7	1,00	0,00	0,00	1,00	13,05%	20,77%	28,33%	9,19%	23,55%	1,27%	0,00%	3,83%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
50	1429,2	82,3	7,00	1,00	0,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	2,58%	0,00%	0,00%	0,00%	0,00%	0,00%	97,42%	0,00%	0,00%	0,00%
51	1429,2	115,0	7,00	1,00	0,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	2,58%	0,00%	0,00%	0,00%	0,00%	0,00%	97,42%	0,00%	0,00%	0,00%
52	1595,7	68,7	1,00	1,00	0,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	12,68%	0,00%	0,00%	0,00%	0,00%	0,00%	87,32%	0,00%	0,00%	0,00%
53	-	68,7	1,00	0,00	0,00	1,00	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
54	1595,7	68,7	1,00	1,00	0,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	12,68%	0,00%	0,00%	0,00%	0,00%	0,00%	87,32%	0,00%	0,00%	0,00%
55	1595,7	68,7	1,00	1,00	0,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	12,68%	0,00%	0,00%	0,00%	0,00%	0,00%	87,32%	0,00%	0,00%	0,00%
56	20,1	15,0	1,00	1,00	0,00	0,00	0,60%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	71,00%	28,40%	0,00%	0,00%
57	1,0	15,0	1,00	1,00	0,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	100,00%
58	21,1	2291,0	1,00	1,00	0,00	0,00	11,50%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	67,54%	7,60%	13,35%	0,00%
59	21,1	180,0	1,00	1,00	0,00	0,00	11,50%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	67,54%	7,60%	13,35%	0,00%
60	1254,0	68,7	7,00	1,00	0,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%
61	341,7	68,7	1,00	1,00	0,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	59,22%	0,00%	0,00%	0,00%	0,00%	0,00%	40,78%	0,00%	0,00%	0,00%
62	341,7	49,0	1,00	1,00	0,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	59,22%	0,00%	0,00%	0,00%	0,00%	0,00%	40,78%	0,00%	0,00%	0,00%
63	341,7	20,0	1,00	1,00	0,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	59,22%	0,00%	0,00%	0,00%	0,00%	0,00%	40,78%	0,00%	0,00%	0,00%
64	406,8	5,0	1,00	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
65	406,8	15,0	1,00	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
66	681,4	18,8	1,00	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
67	515,0	25,0	1,00	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
68	515,0	0,0	1,00	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
69	74,3	-20,0	10,00	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
70	74,3	-20,0	10,00	1,00	0,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
71	175,3	5,0	3,00	1,00	0,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	21,03%	0,00%	0,00%	0,00%	0,00%	0,00%	78,97%	0,00%	0,00%	0,00%
72	175,3	53,6	3,00	1,00	0,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	21,03%	0,00%	0,00%	0,00%	0,00%	0,00%	78,97%	0,00%	0,00%	0,00%

n°	Flowrate [kg/h]	T [°C]	P [bar]	VFRAC	LFRAC	SFRAC	WEIGHT COMPOSITION																	
							H_2O	Cell.	Hemic.	Lignin	Protein	PPs	Lipids	Ash	C_6H_{14}	Ethanol	NaOH	C.A.	HCl	NaCl	N_2	O_2	CO_2	CH_4
73	175,3	53,6	7,00	1,00	0,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	21,03%	0,00%	0,00%	0,00%	0,00%	0,00%	78,97%	0,00%	0,00%	0,00%
74	0,9	25,0	7,00	1,00	0,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%
75	440,5	61,0	1,00	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,75%	11,36%	0,00%	87,89%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
76	-	68,0	1,00	1,00	0,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,01%	0,00%	99,99%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
77	-	68,0	1,00	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,01%	0,00%	99,99%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
78	-	68,0	1,00	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,01%	0,00%	99,99%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
79	383,1	68,0	1,00	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,01%	0,00%	99,99%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
80	3953,1	5,0	1,00	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
81	3953,1	15,0	1,00	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
82	-	136,0	1,00	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	5,78%	87,19%	0,00%	7,02%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
83	-	136,0	1,00	1,00	0,00	0,00	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
84	57,3	136,0	1,00	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	5,78%	87,19%	0,00%	7,02%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
85	68,8	158,0	6,00	1,00	0,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
86	68,8	158,0	6,00	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
87	7,0	158,0	6,00	1,00	0,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
88	53,6	136,0	1,00	0,00	1,00	0,00	0,73%	0,00%	0,00%	0,00%	0,00%	6,18%	93,09%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
89	10,7	134,0	1,00	1,00	0,00	0,00	61,90%	0,00%	0,00%	0,00%	0,00%	0,00%	0,43%	0,00%	37,68%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
90	10,7	25,0	1,00	1,00	0,00	0,00	61,90%	0,00%	0,00%	0,00%	0,00%	0,00%	0,43%	0,00%	37,68%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
91	406,3	28,0	1,00	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
92	406,3	40,0	1,00	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
93	6,7	25,0	1,00	0,00	1,00	0,00	99,32%	0,00%	0,00%	0,00%	0,00%	0,00%	0,68%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
94	4,0	25,0	1,00	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
95	-	25,0	1,00	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
96	607,8	68,7	1,00	0,00	0,00	1,00	13,05%	20,77%	28,33%	9,19%	23,55%	1,27%	0,00%	3,83%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
97	607,8	68,7	1,00	0,00	0,00	1,00	13,05%	20,77%	28,33%	9,19%	23,55%	1,27%	0,00%	3,83%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
98	9724,9	60,2	1,00	0,00	0,77	0,23	43,60%	1,47%	1,77%	0,57%	1,53%	0,17%	0,00%	0,24%	0,00%	50,64%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
99	851,2	60,2	1,00	0,00	0,00	1,00	36,75%	5,80%	7,73%	2,51%	6,47%	0,41%	0,00%	1,04%	0,00%	39,28%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
100	851,2	60,2	1,00	0,00	0,00	1,00	36,75%	5,80%	7,73%	2,51%	6,47%	0,41%	0,00%	1,04%	0,00%	39,28%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
101	2228,6	60,2	1,00	0,00	0,00	1,00	37,40%	5,04%	7,73%	2,51%	6,20%	0,03%	0,00%	1,04%	0,00%	40,05%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
102	1256,9	60,2	1,00	0,00	0,00	1,00	30,90%	8,94%	13,70%	4,44%	11,00%	0,06%	0,00%	1,85%	0,00%	29,10%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
103	8145,3	25,0	1,00	0,00	1,00	0,00	45,80%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	54,20%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
104	8145,3	60,0	1,00	0,00	1,00	0,00	45,80%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	54,20%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
105	9117,1	60,0	1,00	0,00	1,00	0,00	45,80%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	54,20%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
106	971,7	60,0	1,00	0,00	1,00	0,00	45,80%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	54,20%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
107	971,7	60,0	1,00	0,00	1,00	0,00	45,80%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	54,20%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
108	481,5	158,0	6,00	1,00	0,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
109	481,5	158,0	6,00	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
110	630,5	60,0	1,00	0,00	1,00	0,00	45,64%	0,18%	0,00%	0,00%	0,07%	0,09%	0,00%	0,00%	0,00%	54,02%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
111	7496,3	60,0	1,00	0,00	1,00	0,00	45,64%	0,18%	0,00%	0,00%	0,07%	0,09%	0,00%	0,00%	0,00%	54,02%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
112	7496,3	60,0	1,00	0,00	1,00	0,00	45,64%	0,18%	0,00%	0,00%	0,07%	0,09%	0,00%	0,00%	0,00%	54,02%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
113	2513,7	25,0	1,00	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%

n°	**Flowrate [kg/h]**	**T [°C]**	**P [bar]**	**VFRAC**	**LFRAC**	**SFRAC**	**WEIGHT COMPOSITION**																	
							H_2O	**Cell.**	**Hemic.**	**Lignin**	**Protein**	**PPs**	**Lipids**	**Ash**	**C_6H_{14}**	**Ethanol**	**NaOH**	**C.A.**	**HCl**	**NaCl**	**N_2**	**O_2**	**CO_2**	**CH_4**
114	2513,7	25,0	1,00	0,00	1,00	0,00	85,57%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	14,43%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
115	1256,9	25,0	1,00	0,00	0,00	1,00	59,76%	8,94%	13,70%	4,44%	11,00%	0,06%	0,00%	1,85%	0,00%	0,24%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
116	-	-	1,00	0,00	1,00	0,00	85,57%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	14,43%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
117	2513,7	25,0	1,00	0,00	1,00	0,00	85,57%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	14,43%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
118	1256,9	25,0	1,00	0,00	0,00	1,00	59,76%	8,94%	13,70%	4,44%	11,00%	0,06%	0,00%	1,85%	0,00%	0,24%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
119	7496,3	60,0	5,00	0,00	1,00	0,00	45,64%	0,18%	0,00%	0,00%	0,07%	0,09%	0,00%	0,00%	0,00%	54,02%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
120	7496,3	60,0	5,00	0,00	1,00	0,00	45,64%	0,18%	0,00%	0,00%	0,07%	0,09%	0,00%	0,00%	0,00%	54,02%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
121	-	60,0	5,00	0,00	1,00	0,00	45,64%	0,18%	0,00%	0,00%	0,07%	0,09%	0,00%	0,00%	0,00%	54,02%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
122	1476,8	60,0	5,00	0,00	1,00	0,00	45,37%	0,19%	0,00%	0,00%	0,33%	0,42%	0,00%	0,00%	0,00%	53,69%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
123	-	60,0	5,00	0,00	1,00	0,00	45,37%	0,19%	0,00%	0,00%	0,33%	0,42%	0,00%	0,00%	0,00%	53,69%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
124	1476,8	60,0	5,00	0,00	1,00	0,00	45,37%	0,19%	0,00%	0,00%	0,33%	0,42%	0,00%	0,00%	0,00%	53,69%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
125	6019,5	60,0	5,00	0,00	1,00	0,00	45,71%	0,18%	0,00%	0,00%	0,00%	0,01%	0,00%	0,00%	0,00%	54,10%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
126	-	60,0	5,00	0,00	1,00	0,00	45,71%	0,18%	0,00%	0,00%	0,00%	0,01%	0,00%	0,00%	0,00%	54,10%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
127	6019,5	60,0	5,00	0,00	1,00	0,00	45,71%	0,18%	0,00%	0,00%	0,00%	0,01%	0,00%	0,00%	0,00%	54,10%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
128	1476,8	60,0	15,00	0,00	1,00	0,00	45,37%	0,19%	0,00%	0,00%	0,33%	0,42%	0,00%	0,00%	0,00%	53,69%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
129	-	60,0	15,00	0,00	1,00	0,00	45,37%	0,19%	0,00%	0,00%	0,33%	0,42%	0,00%	0,00%	0,00%	53,69%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
130	132,9	60,0	15,00	0,00	1,00	0,00	41,97%	0,23%	0,00%	0,00%	3,59%	4,54%	0,00%	0,00%	0,00%	49,67%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
131	-	60,0	15,00	0,00	1,00	0,00	41,97%	0,23%	0,00%	0,00%	3,59%	4,54%	0,00%	0,00%	0,00%	49,67%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
132	132,9	60,0	15,00	0,00	1,00	0,00	41,97%	0,23%	0,00%	0,00%	3,59%	4,54%	0,00%	0,00%	0,00%	49,67%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
133	1343,9	60,0	15,00	0,00	1,00	0,00	45,71%	0,19%	0,00%	0,00%	0,01%	0,01%	0,00%	0,00%	0,00%	54,09%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
134	-	60,0	15,00	0,00	1,00	0,00	45,71%	0,19%	0,00%	0,00%	0,01%	0,01%	0,00%	0,00%	0,00%	54,09%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
135	9877,1	51,1	1,00	0,00	1,00	0,00	55,85%	0,14%	0,00%	0,00%	0,00%	0,01%	0,00%	0,00%	0,00%	44,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
136	9877,1	51,1	1,00	0,00	1,00	0,00	55,85%	0,14%	0,00%	0,00%	0,00%	0,01%	0,00%	0,00%	0,00%	44,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
137	132,9	60,0	15,00	0,00	1,00	0,00	41,97%	0,23%	0,00%	0,00%	3,59%	4,54%	0,00%	0,00%	0,00%	49,67%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
138	92,9	81,0	1,00	0,00	1,00	0,00	51,01%	0,33%	0,00%	0,00%	5,14%	6,50%	0,00%	0,00%	0,00%	37,02%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
139	26,9	158,0	6,00	1,00	0,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
140	26,9	158,0	6,00	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
141	40,0	81,0	1,00	1,00	0,00	0,00	20,97%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	79,03%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
142	92,9	81,0	0,80	0,00	1,00	0,00	51,01%	0,33%	0,00%	0,00%	5,14%	6,50%	0,00%	0,00%	0,00%	37,02%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
143	50,5	75,0	1,00	0,00	1,00	0,00	77,54%	0,61%	0,00%	0,00%	9,44%	11,95%	0,00%	0,00%	0,00%	0,46%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
144	42,3	75,0	0,80	1,00	0,00	0,00	19,36%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	80,64%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
145	40,0	81,0	1,00	0,00	1,00	0,00	20,97%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	79,03%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
146	9917,1	51,2	1,00	0,00	1,00	0,00	55,71%	0,14%	0,00%	0,00%	0,00%	0,01%	0,00%	0,00%	0,00%	44,14%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
147	50,5	75,0	1,00	0,00	1,00	0,00	77,54%	0,61%	0,00%	0,00%	9,44%	11,95%	0,00%	0,00%	0,00%	0,46%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
148	11,3	75,0	1,00	0,00	0,00	1,00	2,00%	2,71%	0,00%	0,00%	42,06%	53,22%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
149	41,0	153,5	2,00	1,00	0,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
150	41,0	153,5	2,00	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
151	39,2	100,0	1,00	1,00	0,00	0,00	99,40%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,60%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%

Table 8.9 - BSG Valorization Plant: Stream Table, Section 3

n°	Flowrate [kg/h]	T [°C]	P [bar]	V_{FRAC}	L_{FRAC}	S_{FRAC}	WEIGHT COMPOSITION																	
							H_2O	Cell.	Hemic.	Lignin	Protein	PPs	Lipids	Ash	C_6H_{14}	Ethanol	NaOH	C.A.	HCl	NaCl	N_2	O_2	CO_2	CH_4
152	1256,9	25,0	1,0	0,00	0,00	1,00	59,76%	8,94%	13,70%	4,44%	11,00%	0,06%	0,00%	1,85%	0,00%	0,24%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
153	1256,9	25,0	1,0	0,00	0,00	1,00	59,76%	8,94%	13,70%	4,44%	11,00%	0,06%	0,00%	1,85%	0,00%	0,24%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
154	1256,9	25,0	1,0	0,00	0,00	1,00	65,14%	8,76%	12,60%	4,35%	7,15%	0,06%	0,00%	1,84%	0,00%	0,09%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
155	1252,0	25,0	1,0	0,00	0,00	1,00	65,00%	8,80%	12,65%	4,37%	7,18%	0,06%	0,00%	1,85%	0,00%	0,09%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
156	812,0	25,0	1,0	0,00	1,00	0,00	99,60%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,40%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
157	817,0	25,0	1,0	0,00	1,00	0,00	99,60%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,40%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
158	4,9	25,0	1,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
159	4,9	25,0	1,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
160	817,0	25,0	1,0	0,00	1,00	0,00	91,33%	0,28%	1,69%	0,14%	5,92%	0,00%	0,00%	0,02%	0,00%	0,23%	0,40%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
161	817,0	25,0	1,0	0,00	1,00	0,00	91,33%	0,28%	1,69%	0,14%	5,92%	0,00%	0,00%	0,02%	0,00%	0,23%	0,40%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
162	22,1	25,0	1,0	0,00	1,00	0,00	66,67%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	33,33%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
163	839,0	25,0	1,0	0,00	1,00	0,00	90,68%	0,27%	1,65%	0,13%	5,76%	0,00%	0,00%	0,02%	0,00%	0,22%	0,39%	0,88%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
164	73,9	25,0	1,0	0,00	1,00	0,00	28,57%	0,68%	5,11%	0,34%	65,09%	0,00%	0,00%	0,21%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
165	765,2	25,0	1,0	0,00	1,00	0,00	96,67%	0,23%	1,31%	0,11%	0,04%	0,00%	0,00%	0,00%	0,00%	0,24%	0,43%	0,96%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
166	765,2	25,0	1,0	0,00	1,00	0,00	96,67%	0,23%	1,31%	0,11%	0,04%	0,00%	0,00%	0,00%	0,00%	0,24%	0,43%	0,96%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
167	8,0	25,0	1,0	0,00	1,00	0,00	63,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	37,00%	0,00%	0,00%	0,00%	0,00%	0,00%
168	773,2	25,0	1,0	0,00	1,00	0,00	96,32%	0,23%	1,30%	0,11%	0,04%	0,00%	0,00%	0,00%	0,00%	0,24%	0,42%	0,95%	0,00%	0,38%	0,00%	0,00%	0,00%	0,00%
169	1497,6	25,0	1,0	0,00	1,00	0,00	35,20%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	64,80%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
170	2270,8	25,0	1,0	0,00	1,00	0,00	56,01%	0,08%	0,44%	0,04%	0,01%	0,00%	0,00%	0,00%	0,00%	42,82%	0,14%	0,32%	0,00%	0,13%	0,00%	0,00%	0,00%	0,00%
171	15,8	25,0	1,0	0,00	1,00	0,00	28,57%	9,13%	55,86%	4,54%	1,83%	0,06%	0,00%	0,01%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
172	2255,1	25,0	1,0	0,00	1,00	0,00	56,20%	0,01%	0,06%	0,01%	0,00%	0,00%	0,00%	0,00%	0,00%	43,12%	0,14%	0,33%	0,00%	0,13%	0,00%	0,00%	0,00%	0,00%
173	2255,1	25,0	1,0	0,00	1,00	0,00	56,20%	0,01%	0,06%	0,01%	0,00%	0,00%	0,00%	0,00%	0,00%	43,12%	0,14%	0,33%	0,00%	0,13%	0,00%	0,00%	0,00%	0,00%
174	1252,0	25,0	1,0	0,00	0,00	1,00	65,00%	8,80%	12,65%	4,37%	7,18%	0,06%	0,00%	1,85%	0,00%	0,09%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
175	1252,0	25,0	1,0	0,00	0,00	1,00	71,13%	8,61%	11,37%	4,23%	2,67%	0,06%	0,00%	1,84%	0,00%	0,09%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
176	1032,6	25,0	1,0	0,00	0,00	1,00	65,00%	10,43%	13,78%	5,13%	3,23%	0,07%	0,00%	2,23%	0,00%	0,11%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
177	281,5	25,0	1,0	0,00	1,00	0,00	98,04%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	1,96%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
178	500,8	25,0	1,0	0,00	1,00	0,00	98,04%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	1,96%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
179	219,3	25,0	1,0	0,00	1,00	0,00	98,04%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	1,96%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
180	219,3	25,0	1,0	0,00	1,00	0,00	98,04%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	1,96%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
181	500,8	25,0	1,0	0,00	1,00	0,00	82,71%	0,48%	3,20%	0,34%	11,28%	0,00%	0,00%	0,02%	0,00%	0,00%	1,96%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
182	500,8	25,0	1,0	0,00	1,00	0,00	82,71%	0,48%	3,20%	0,34%	11,28%	0,00%	0,00%	0,02%	0,00%	0,00%	1,96%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
183	66,5	25,0	1,0	0,00	1,00	0,00	63,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	37,00%	0,00%	0,00%	0,00%	0,00%	0,00%
184	567,2	25,0	1,0	0,00	1,00	0,00	80,83%	0,42%	2,83%	0,30%	9,96%	0,00%	0,00%	0,02%	0,00%	0,00%	1,73%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
185	84,7	25,0	1,0	0,00	1,00	0,00	28,57%	0,49%	4,20%	0,35%	66,27%	0,00%	0,00%	0,11%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
186	482,5	25,0	1,0	0,00	1,00	0,00	90,01%	0,41%	2,59%	0,30%	0,07%	0,00%	0,00%	0,00%	0,00%	0,00%	2,04%	4,59%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
187	482,5	25,0	1,0	0,00	1,00	0,00	90,01%	0,41%	2,59%	0,30%	0,07%	0,00%	0,00%	0,00%	0,00%	0,00%	2,04%	4,59%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
188	24,2	25,0	1,0	0,00	1,00	0,00	63,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	37,00%	0,00%	0,00%	0,00%	0,00%	0,00%
189	506,7	25,0	1,0	0,00	1,00	0,00	88,72%	0,39%	2,46%	0,28%	0,07%	0,00%	0,00%	0,00%	0,00%	0,00%	1,94%	4,37%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
190	929,7	25,0	1,0	0,00	1,00	0,00	35,20%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	64,80%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
191	1436,4	25,0	1,0	0,00	1,00	0,00	54,08%	0,14%	0,87%	0,10%	0,02%	0,00%	0,00%	0,00%	0,00%	41,94%	0,68%	1,54%	0,00%	0,62%	0,00%	0,00%	0,00%	0,00%
192	22,4	25,0	1,0	0,00	1,00	0,00	28,57%	8,34%	55,49%	6,01%	1,50%	0,08%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%

n°	Flowrate [kg/h]	T [°C]	P [bar]	V_{FRAC}	L_{FRAC}	S_{FRAC}	WEIGHT COMPOSITION																	
							H_2O	Cell.	Hemic.	Lignin	Protein	PPs	Lipids	Ash	C_6H_{14}	Ethanol	NaOH	C.A.	HCl	NaCl	N_2	O_2	CO_2	CH_4
193	1414,0	25,0	1,0	0,00	1,00	0,00	54,48%	0,01%	0,00%	0,01%	0,00%	0,00%	0,00%	0,00%	0,00%	42,61%	0,69%	1,57%	0,00%	0,63%	0,00%	0,00%	0,00%	0,00%
194	1414,0	25,0	1,0	0,00	1,00	0,00	54,48%	0,01%	0,00%	0,01%	0,00%	0,00%	0,00%	0,00%	0,00%	42,61%	0,69%	1,57%	0,00%	0,63%	0,00%	0,00%	0,00%	0,00%
195	3669,1	25,0	1,0	0,00	1,00	0,00	55,54%	0,01%	0,03%	0,01%	0,00%	0,00%	0,00%	0,00%	0,00%	42,92%	0,36%	0,80%	0,00%	0,33%	0,00%	0,00%	0,00%	0,00%
196	1032,6	25,0	1,0	0,00	0,00	1,00	65,00%	10,43%	13,78%	5,13%	3,23%	0,07%	0,00%	2,23%	0,00%	0,11%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
197	1032,6	25,0	1,0	0,00	0,00	1,00	77,97%	9,58%	5,44%	4,56%	0,07%	0,06%	0,00%	2,21%	0,00%	0,11%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
198	649,9	25,0	1,0	0,00	0,00	1,00	65,00%	15,21%	8,65%	7,24%	0,10%	0,10%	0,00%	3,51%	0,00%	0,18%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
199	649,9	25,0	1,0	0,00	1,00	0,00	86,09%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	13,91%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
200	1032,6	25,0	1,0	0,00	1,00	0,00	86,09%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	13,91%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
201	382,7	25,0	1,0	0,00	1,00	0,00	86,09%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	13,91%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
202	382,7	25,0	1,0	0,00	1,00	0,00	86,09%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	13,91%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
203	1032,6	25,0	1,0	0,00	1,00	0,00	73,12%	0,86%	8,34%	0,58%	3,17%	0,01%	0,00%	0,02%	0,00%	0,00%	13,91%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
204	1032,6	25,0	1,0	0,00	1,00	0,00	73,12%	0,86%	8,34%	0,58%	3,17%	0,01%	0,00%	0,02%	0,00%	0,00%	13,91%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
205	971,5	25,0	1,0	0,00	1,00	0,00	66,67%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	33,33%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
206	2004,1	25,0	1,0	0,00	1,00	0,00	69,99%	0,44%	4,30%	0,30%	1,63%	0,00%	0,00%	0,01%	0,00%	0,00%	7,16%	16,16%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
207	84,7	25,0	1,0	0,00	1,00	0,00	28,57%	0,22%	3,55%	0,15%	66,94%	0,00%	0,00%	0,48%	0,00%	0,00%	0,03%	0,07%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
208	1955,5	25,0	1,0	0,00	1,00	0,00	71,02%	0,45%	4,32%	0,30%	0,01%	0,00%	0,00%	0,00%	0,00%	0,00%	7,34%	16,56%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
209	1955,5	25,0	1,0	0,00	1,00	0,00	71,02%	0,45%	4,32%	0,30%	0,01%	0,00%	0,00%	0,00%	0,00%	0,00%	7,34%	16,56%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
210	353,7	25,0	1,0	0,00	1,00	0,00	63,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	37,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
211	2309,3	25,0	1,0	0,00	1,00	0,00	69,79%	0,38%	3,65%	0,25%	0,01%	0,00%	0,00%	0,00%	0,00%	0,00%	6,22%	14,02%	0,00%	5,67%	0,00%	0,00%	0,00%	0,00%
212	3727,2	25,0	1,0	0,00	1,00	0,00	35,20%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	64,80%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
213	6036,4	25,0	1,0	0,00	1,00	0,00	48,43%	0,15%	1,40%	0,10%	0,00%	0,00%	0,00%	0,00%	0,00%	40,01%	2,38%	5,36%	0,00%	2,17%	0,00%	0,00%	0,00%	0,00%
214	138,7	25,0	1,0	0,00	1,00	0,00	28,57%	6,28%	60,73%	4,22%	0,14%	0,06%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
215	1414,0	25,0	1,0	0,00	1,00	0,00	48,90%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	40,95%	2,43%	5,49%	0,00%	2,22%	0,00%	0,00%	0,00%	0,00%
216	1414,0	25,0	1,0	0,00	1,00	0,00	48,90%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	40,95%	2,43%	5,49%	0,00%	2,22%	0,00%	0,00%	0,00%	0,00%
217	5083,0	25,0	1,0	0,00	1,00	0,00	53,69%	0,01%	0,03%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	42,37%	0,93%	2,11%	0,00%	0,85%	0,00%	0,00%	0,00%	0,00%
218	161,2	25,0	1,0	0,00	1,00	0,00	28,57%	6,57%	60,00%	4,47%	0,33%	0,06%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
219	169,5	25,0	1,0	0,00	1,00	0,00	28,57%	0,35%	3,87%	0,25%	66,61%	0,00%	0,00%	0,29%	0,00%	0,00%	0,01%	0,03%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
220	243,3	25,0	1,0	0,00	1,00	0,00	28,57%	0,45%	4,25%	0,28%	66,15%	0,00%	0,00%	0,27%	0,00%	0,00%	0,01%	0,02%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
221	243,3	25,0	1,0	0,00	1,00	0,00	28,57%	0,45%	4,25%	0,28%	66,15%	0,00%	0,00%	0,27%	0,00%	0,00%	0,01%	0,02%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
222	150,9	60,0	1,0	0,00	0,00	1,00	1,96%	0,68%	6,00%	0,41%	90,60%	0,01%	0,00%	0,32%	0,00%	0,00%	0,01%	0,02%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
223	67,4	153,5	2,0	1,00	0,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
224	67,4	153,5	2,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
225	56,2	100,0	1,0	1,00	0,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
226	176,9	25,0	1,0	0,00	1,00	0,00	28,57%	6,80%	59,63%	4,48%	0,46%	0,06%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
227	649,9	25,0	1,0	0,00	0,00	1,00	65,00%	15,21%	8,65%	7,24%	0,10%	0,10%	0,00%	3,51%	0,00%	0,18%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
228	649,9	25,0	1,0	0,00	0,00	1,00	66,81%	15,21%	6,88%	7,24%	0,10%	0,10%	0,00%	3,48%	0,00%	0,18%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
229	616,4	25,0	1,0	0,00	0,00	1,00	65,00%	16,04%	7,25%	7,63%	0,11%	0,11%	0,00%	3,67%	0,00%	0,19%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
230	486,4	25,0	1,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
231	519,9	25,0	1,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
232	33,5	25,0	1,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
233	33,5	25,0	1,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%

n°	**Flowrate [kg/h]**	**T [°C]**	**P [bar]**	**V_{FRAC}**	**L_{FRAC}**	**S_{FRAC}**	**WEIGHT COMPOSITION**																	
							H_2O	**Cell.**	**Hemic.**	**Lignin**	**Protein**	**PPs**	**Lipids**	**Ash**	**C_6H_{14}**	**Ethanol**	**NaOH**	**C.A.**	**HCl**	**NaCl**	**N_2**	**O_2**	**CO_2**	**CH_4**
224	67,4	153,5	2,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
225	56,2	100,0	1,0	1,00	0,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
226	176,9	25,0	1,0	0,00	1,00	0,00	28,57%	6,80%	59,63%	4,48%	0,46%	0,06%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
227	649,9	25,0	1,0	0,00	0,00	1,00	65,00%	15,21%	8,65%	7,24%	0,10%	0,10%	0,00%	3,51%	0,00%	0,18%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
228	649,9	25,0	1,0	0,00	0,00	1,00	66,81%	15,21%	6,88%	7,24%	0,10%	0,10%	0,00%	3,48%	0,00%	0,18%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
229	616,4	25,0	1,0	0,00	0,00	1,00	65,00%	16,04%	7,25%	7,63%	0,11%	0,11%	0,00%	3,67%	0,00%	0,19%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
230	486,4	25,0	1,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
231	519,9	25,0	1,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
232	33,5	25,0	1,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
233	33,5	25,0	1,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
234	519,9	25,0	1,0	0,00	1,00	0,00	97,74%	0,00%	2,22%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
235	519,9	25,0	1,0	0,00	1,00	0,00	97,74%	0,00%	2,22%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
236	519,9	25,0	20,0	0,00	1,00	0,00	97,74%	0,00%	2,22%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
237	519,9	25,0	20,0	0,00	1,00	0,00	97,74%	0,00%	2,22%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
238	-	25,0	20,0	0,00	1,00	0,00	97,74%	0,00%	2,22%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
239	113,3	25,0	20,0	0,00	1,00	0,00	89,71%	0,00%	10,11%	0,00%	0,02%	0,00%	0,00%	0,16%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
240	-	25,0	20,0	0,00	1,00	0,00	89,71%	0,00%	10,11%	0,00%	0,02%	0,00%	0,00%	0,16%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
241	113,3	25,0	20,0	0,00	1,00	0,00	89,71%	0,00%	10,11%	0,00%	0,02%	0,00%	0,00%	0,16%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
242	406,6	25,0	20,0	0,00	1,00	0,00	99,98%	0,00%	0,02%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
243	-	25,0	20,0	0,00	1,00	0,00	99,98%	0,00%	0,02%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
244	406,6	25,0	20,0	0,00	1,00	0,00	99,98%	0,00%	0,02%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
245	113,3	25,0	1,0	0,00	1,00	0,00	89,71%	0,00%	10,11%	0,00%	0,02%	0,00%	0,00%	0,16%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
246	66,5	158,0	6,0	1,00	0,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
247	67,6	100,0	1,0	0,00	1,00	0,00	82,75%	0,00%	16,94%	0,00%	0,03%	0,00%	0,00%	0,27%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
248	66,5	158,0	6,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
249	45,7	100,0	1,0	1,00	0,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
250	67,6	100,0	0,8	0,00	1,00	0,00	82,75%	0,00%	16,94%	0,00%	0,03%	0,00%	0,00%	0,27%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
251	45,7	100,0	1,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
252	46,4	94,0	0,8	1,00	0,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
253	21,2	94,0	1,0	0,00	1,00	0,00	45,00%	0,00%	54,02%	0,00%	0,11%	0,00%	0,00%	0,87%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
254	198,1	32,4	1,0	0,00	1,00	0,00	30,33%	6,07%	59,03%	4,00%	0,43%	0,06%	0,00%	0,09%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
255	140,7	60,0	1,0	0,00	0,00	1,00	1,92%	8,54%	83,10%	5,63%	0,60%	0,08%	0,00%	0,13%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
256	67,1	153,5	2,0	1,00	0,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
257	57,4	100,0	1,0	1,00	0,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
258	67,1	153,5	2,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%

Table 8.10 - BSG Valorization Plant: Stream Table, Section 4

n°	Flowrate [kg/h]	T [°C]	P [bar]	V_{FRAC}	L_{FRAC}	S_{FRAC}	WEIGHT COMPOSITION																	
							H_2O	Cell.	Hemic.	Lignin	Protein	PPs	Lipids	Ash	C_6H_{14}	Ethanol	NaOH	C.A.	HCl	NaCl	N_2	O_2	CO_2	CH_4
259	19526,2	25,0	1,0	0,00	1,00	0,00	53,54%	0,07%	0,01%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	43,03%	0,80%	1,81%	0,00%	0,73%	0,00%	0,00%	0,00%	0,00%
260	19526,2	25,0	1,0	0,00	1,00	0,00	53,54%	0,07%	0,01%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	43,03%	0,80%	1,81%	0,00%	0,73%	0,00%	0,00%	0,00%	0,00%
261	-	80,4	1,0	1,00	0,00	0,00	33,71%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	66,29%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
262	-	80,4	1,0	0,00	1,00	0,00	33,71%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	66,29%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
263	-	80,4	1,0	0,00	1,00	0,00	33,71%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	66,29%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
264	12673,4	80,4	1,0	0,00	1,00	0,00	33,71%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	66,29%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
265	751053,1	28,0	1,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
266	751053,1	40,0	1,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
267	-	100,0	1,0	0,00	1,00	0,00	90,23%	0,20%	0,02%	0,00%	0,00%	0,01%	0,00%	0,00%	0,00%	0,00%	2,29%	5,16%	2,08%	0,00%	0,00%	0,00%	0,00%	0,00%
268	-	100,0	1,0	1,00	0,00	0,00	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
269	6852,8	100,0	1,0	0,00	1,00	0,00	90,23%	0,20%	0,02%	0,00%	0,00%	0,01%	0,00%	0,00%	0,00%	0,00%	2,29%	5,16%	2,08%	0,00%	0,00%	0,00%	0,00%	0,00%
270	20116,2	158,0	6,0	1,00	0,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
271	20116,2	158,0	6,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
272	3352,9	25,0	1,0	0,00	1,00	0,00	35,20%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	64,80%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
273	10205,8	64,4	1,0	0,00	1,00	0,00	72,15%	0,14%	0,01%	0,00%	0,00%	0,01%	0,00%	0,00%	0,00%	21,29%	1,53%	3,46%	0,00%	1,40%	0,00%	0,00%	0,00%	0,00%
274	3219,2	64,4	1,0	0,00	1,00	0,00	92,37%	0,43%	0,04%	0,01%	0,00%	0,03%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	6,83%	0,00%	0,00%	0,00%	0,00%
275	6986,6	64,4	1,0	0,00	1,00	0,00	63,84%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	31,10%	0,00%	5,06%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
276	6986,6	64,4	1,0	0,00	1,00	0,00	63,84%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	31,10%	0,00%	5,06%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
277	6986,6	64,4	1,0	0,00	1,00	0,00	63,84%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	31,10%	0,00%	5,06%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
278	-	80,4	1,0	1,00	0,00	0,00	34,16%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	65,84%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
279	-	80,4	1,0	0,00	1,00	0,00	34,16%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	65,84%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
280	-	80,4	1,0	0,00	1,00	0,00	34,16%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	65,84%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
281	3300,0	80,4	1,0	0,00	1,00	0,00	34,16%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	65,84%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
282	196567,7	28,0	1,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
283	196567,7	40,0	1,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
284	-	100,0	1,0	0,00	1,00	0,00	90,41%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,01%	0,00%	9,58%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
285	-	100,0	1,0	1,00	0,00	0,00	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-	-
286	5068,0	158,0	6,0	1,00	0,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
287	5068,0	158,0	6,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
288	3686,6	100,0	1,0	0,00	1,00	0,00	90,41%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,01%	0,00%	9,58%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
289	3686,6	100,0	1,0	0,00	1,00	0,00	90,41%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,01%	0,00%	9,58%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
290	2820,1	158,0	6,0	1,00	0,00	1,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
291	2625,7	100,0	1,0	1,00	0,00	0,00	1,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
292	2820,1	158,0	6,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
293	1060,8	100,0	1,0	0,00	1,00	0,00	66,67%	0,01%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,02%	0,00%	33,30%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
294	-	25,0	1,0	0,00	1,00	0,00	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
295	-	25,0	1,0	0,00	1,00	0,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
296	-	25,0	1,0	0,00	0,00	1,00	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
297	-	25,0	1,0	0,00	1,00	0,00	1,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
298	-	25,0	1,0	0,00	1,00	0,00	35,20%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	64,80%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
299	-	25,0	1,0	0,00	1,00	0,00	66,67%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	33,33%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%

Appendix F.

BSG plant Equipment list

Pre-treatment and storage section:

The first section of the plant has a total of 27 devices. The list is shown in full in the Table 8.7.

Table 8.11 - Pre-treatment and storage section equipment list

NAME TAG	DESCRIPTION
B-101	Open-back air draught burner
BE-101	Belt elevator
BE-102	Belt elevator
BE-103	Belt elevator
BL-101	Air Blower
BL-102	Air Blower
BP-101	Belt Press for solids
CY-101	Cyclone for dust
DD-101	Rotary drum dryer for solids
E-101	Economizer plate heat exchanger
E-102	Fluidized bed cooler for solids
F-101	Air filter
F-102	Air filter
H-101	Receiving hopper
H-102	Dosing hopper
H-103	Dosing hopper
H-104	Receiving hopper
H-105	Dosing hopper
M-101	Hammer mill for cereals
PF-101	Filter bag for gas
PF-102	Filter bag for gas
PF-103	Filter bag for gas
PF-104	Filter bag for gas
RFV-101	Rotary feeding valve
S-101	Underground storage for BSG
SC-101	Screw conveyor
TK-101	Silo for dry BSG storage

Lipids and polyphenols extraction section:

The second section of the plant has a total of 95 devices, 17 of which are pumps and 1 is a compressor. The list is shown in full in the Table 8.8.

Table 8.12 - Lipids and polyphenols extraction section equipment list

NAME TAG	DESCRIPTION
B-201	Open-back air draught burner
BE-201	Belt elevator
BL-201	Air Blower

C-201	Nitrogen compressor
CS-201	Condense separator
CS-202	Condense separator
CS-203	Condense separator
CS-203	Condense separator
CS-204	Condense separator
CY-201	Cyclone for dust
D-201	Static decanter
DC-201	Decanter centrifuge
DC-202	Decanter centrifuge
DD-201	Drum dryier for Polyphenols
E-201	Heater for fres n-hexane extractant feed
E-202	Plate heater gas/nitrogen
E-203	Exonomizer plate heat exchanger
E-204	Cooler heat exchanger
E-205	Cooler heat exchanger
E-207	Distillation column condenser
E-208	Kettle Reboiler
E-209	Condeser
E-210	Eather for the fresh ethanol extractanct solution
EV-201	Evaporator
EV-202	Evaporator
EX-201	TURBEX EX30 for defatting
EX-202	TURBEX EX30 for defatting
EX-203	Side TURBEX EX30 for defatting
EX-204	TURBEX EX30 for polyphenol extraction
EX-205	TURBEX EX30 for polyphenol extraction
EX-206	Side TURBEX EX30 for polyphenol extraction
F-201	Air filter
F-202	Gas filter
F-203	Nitrogen filter
FB-201	Filter bag for liquids
FB-202	Filter bag for liquids
FB-203	Filter bag for liquids
FB-203	Filter bag for liquids
FB-204	Filter bag for liquids
FB-204	Filter bag for liquids
FD-201	Flash desolventizer for solids
H-201	Receiving hopper
H-202	Receiving hopper
MX-201	Stirred tank
MX-202	Stirred tank
NFM-201	Nanofiltration membrane
NFM-202	Nanofiltration membrane
PCF-201	Progressive cavity feeder

PCF-202	Progressive cavity feeder
PCF-203	Progressive cavity feeder
PCF-204	Progressive cavity feeder
PCF-205	Progressive cavity feeder
PCF-206	Progressive cavity feeder
RC-201	Roto-concentrator
RFV-201	Rotrary feeding valve
SP-201	Squeezing press for solids
SP-202	Squeezing press for solids
SP-204	Squeezing press for solids
SP-205	Squeezing press for solids
SP-206	Squeezing press for solids
SP-207	Squeezing press for solids
T-201	Hexane direct condensation tower
T-202	Distillation column for n-hexane
T-203	Stripping column for n-hexane
TK-201	Squeezed liquid collection tank
TK-202	N-hexane storage tank
TK-203	Buffer tank for the n-hexane distillation column
TK-204	Squeezed liquid collection tank
TK-205	Pot tank for n-hexane distillation column condenser
TK-205	Buffer tank for the liquid polyphenols extract
TK-206	Buffer tank for nanofiltration membrane system
TK-207	Buffer tank for evaporators
TK-208	Buffer tank for the polyphenols drum dryer
UFM-201	Ultrafiltration membrane
UFM-202	Ultrafiltration membrane
V-201	Lamination valve
V-202	Lamination valve
W-201	Counter current washer for solids
P-201	Volumetric pump for sludges
P-202	Volumetric pump for sludges
P-203	Centrifugal pump
P-204	Centrifugal pump
P-205	Centrifugal pump
P-206	Volumetric pump
P-207	Centrifugal pump
P-208	Centrifugal pump
P-209	Volumetric pump
P-210	Volumetric pump for sludges
P-211	Centrifugal pump
P-212	Volumetric pump for sludges
P-213	Centrifugal pump
P-214	Centrifugal pump
P-215	Centrifugal pump

P-216	Centrifugal pump
P-217	Volumetric pump for sludges

Proteins and arabinoxylans extraction section:

The third section of the plant has a total of 118 devices, 30 of which are pumps. The list is shown in full in the Table 8.9.

Table 8.13 - Proteins and arabinoxylans extraction section equipment list

NAME TAG	DESCRIPTION
CS-301	Condense separator
CS-302	Condense separator
CS-303	Condense separator
D-301	Static decanter for proteins precipitation
D-302	Static decanter for arabinoxylans precipitation
D-303	Static decanter for proteins precipitation
D-304	Static decanter for arabinoxylans precipitation
D-305	Static decanter for proteins precipitation
D-306	Static decanter for arabinoxylans precipitation
DD-301	Drum dryer for proteins
DD-302	Drum dryer for arabinoxylans
EV-301	Evaporator
EV-302	Evaporator
EX-301	TURBEX EX30 for the first alkaline extraction
EX-302	TURBEX EX30 for the first alkaline extraction
EX-303	Side TURBEX EX30 for the first alkaline extraction
EX-304	TURBEX EX30 for the second alkaline extraction
EX-305	TURBEX EX30 for the second alkaline extraction
EX-306	Side TURBEX EX30 for the second alkaline extraction
EX-307	TURBEX EX30 for the third alkaline extraction
EX-308	TURBEX EX30 for the third alkaline extraction
EX-309	Side TURBEX EX30 for the third alkaline extraction
EX-310	TURBEX EX30 for arabunoxylans extraction with water
EX-311	TURBEX EX30 for arabunoxylans extraction with water
EX-312	Side TURBEX EX30 for arabunoxylans extraction with water
FB-301	Filter bag for liquids
FB-302	Filter bag for liquids
FB-303	Filter bag for liquids
FB-304	Filter bag for liquids
FB-305	Filter bag for liquids
FB-306	Filter bag for liquids
FB-307	Filter bag for liquids
FB-308	Filter bag for liquids
FM-301	Filtration membrabe
FM-302	Filtration membrabe

H-301	Receiving hopper
H-302	Receiving hopper
H-303	Receiving hopper
H-304	Receiving hopper
MX-301	Hig shear flow homogenizer
MX-302	Hig shear flow homogenizer
MX-303	Hig shear flow homogenizer
MX-304	Hig shear flow homogenizer
MX-305	Hig shear flow homogenizer
MX-306	Hig shear flow homogenizer
MX-307	Hig shear flow homogenizer
MX-308	Hig shear flow homogenizer
MX-309	Hig shear flow homogenizer
PCF-301	Progressive cavity feeder
PCF-302	Progressive cavity feeder
PCF-303	Side Progressive cavity feeder
PCF-304	Progressive cavity feeder
PCF-305	Progressive cavity feeder
PCF-306	Side Progressive cavity feeder
PCF-307	Progressive cavity feeder
PCF-308	Progressive cavity feeder
PCF-309	Side Progressive cavity feeder
PCF-310	Progressive cavity feeder
PCF-311	Progressive cavity feeder
PCF-312	Side Progressive cavity feeder
SP-301	Squeezing press for solids
SP-302	Squeezing press for solids
SP-303	Side Squeezing press for solids
SP-304	Squeezing press for solids
SP-305	Squeezing press for solids
SP-306	Side Squeezing press for solids
SP-307	Squeezing press for solids
SP-308	Squeezing press for solids
SP-309	Side Squeezing press for solids
SP-310	Squeezing press for solids
SP-311	Squeezing press for solids
SP-312	Side Squeezing press for solids
TK-301	Squeezed liquid collection tank
TK-302	Squeezed liquid collection tank
TK-303	Squeezed liquid collection tank
TK-304	Pot tank for the static decanter
TK-305	Pot tank for the static decanter
TK-306	Pot tank for the static decanter
TK-307	Pot tank for the static decanter
TK-308	Pot tank for the static decanter

TK-309	Squeezed liquid collection tank
TK-310	Buffer tank for the membrane filtration system
TK-311	Buffer tank for the evaporators
TK-312	Buffer tank for protein drum dryer
TK-313	Buffer tank for arabinoxylans drum dryer
V-301	Lamination valve
V-302	Lamination valve
V-303	Lamination valve
P-301	Volumetric pump for sludges
P-302	Centrifugal pump
P-303	Volumetric pump for sludges
P-304	Centrifugal pump
P-305	Centrifugal pump
P-306	Centrifugal pump
P-307	Volumetric pump for sludges
P-308	Centrifugal pump
P-309	Volumetric pump for sludges
P-310	Centrifugal pump
P-311	Volumetric pump for sludges
P-312	Centrifugal pump
P-313	Centrifugal pump
P-314	Centrifugal pump
P-315	Volumetric pump for sludges
P-316	Centrifugal pump
P-317	Volumetric pump for sludges
P-318	Centrifugal pump
P-319	Centrifugal pump
P-320	Volumetric pump for sludges
P-321	Centrifugal pump
P-322	Centrifugal pump
P-323	Volumetric pump for sludges
P-324	Centrifugal pump
P-325	Centrifugal pump
P-326	Centrifugal pump
P-327	Volumetric pump
P-328	Centrifugal pump
P-329	Volumetric pump
P-330	Volumetric pump

Ethanol and citric acid recovery section:

The third section of the plant has a total of 31 devices, 12 of which are pumps. The list is shown in full in the Table 8.10.

Table 8.14 - Ethanol and citric acid recovery section equipment list

NAME TAG	DESCRIPTION
CS-401	Condense separator
CS-402	Condense separator
D-401	Static decanter for sodium chloride
E-401	Condenser of the first ethanol distillation column
E-402	Kettle reboiler of the first ethanol distillation column
E-403	Condenser of the second ethanol distillation column
E-404	Kettle reboiler of the second ethanol distillation column
EV-401	Evaporator
MX-401	Hig shear flow homogenizer
T-401	First ethanol distillation tower
T-402	Second ethanol distillation tower
TK-401	Buffer thank for the feed of the first distillation tower
TK-402	Pot tank for the first distillation column condenser
TK-403	Pot tank for the static decanter
TK-404	Buffer tank for the feed of the second distillation column
TK-405	Pot tank for the second distillation column condenser
TK-406	Buffer tank for the feed of the evaporator
TK-407	Storage tank for the wather ethanol solution
TK-408	Storage tank for the citric acid solution
P-401	Centrifugal pump
P-402	Centrifugal pump
P-403	Centrifugal pump
P-404	Centrifugal pump
P-405	Centrifugal pump
P-406	Centrifugal pump
P-407	Centrifugal pump
P-408	Centrifugal pump
P-409	Centrifugal pump
P-410	Centrifugal pump
P-411	Centrifugal pump
P-412	Centrifugal pump

The plant has a total of 271 devices, 59 of which are pumps.