Politecnico di Torino

Ingegneria Chimica e dei Processi Sostenibili
A.a. 2021/2022
Sessione Marzo 2022

Experimental development of an integrated BSG valorisation plant and design of a 2.000 tpy demo-unit

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“Ξυνὸν δὲ μοί ἐστιν,

ὁππόθεν ἄρξωμαι·

τόθι γὰρ πάλιν ἵξομαι αὖθις.”

1 Sommario

Lo scopo di questo lavoro di tesi è stato lo sviluppo di un processo in scala pilota per la valorizzazione del brewer's spent grain, attraverso l'estrazione di prodotti ricchi in polifenoli, proteine ed arabino-xylani. Il processo è basato su quello, in scala di laboratorio, ideato e brevettato da un gruppo di ricerca dell'Universidade de Aveiro (Coelho, et al., 2014). L'obiettivo del lavoro è stato dimostrare la fattibilità di una versione in scala pilota del processo che utilizza l'estrattore cavitazione mutistadio contro-corrente denominato Turbex. Il Turbex è un estrattore innovativo brevettato da G. Cavaglià che, grazie alla sua capacità di generare e sfruttare alti valori di turbolenza, sforzi di taglio e cavitazione controllata, è in grado di realizzare un alto numero di stadi ideali di equilibrio, che grazie alla sua configurazione in contro-corrente rende possibile il raggiungimento di elevate rese di estrazione con un ridotto utilizzo di estraente, rendendo tale apparecchiatura estremamente competitiva nel ruolo di estrattore, specialmente, ma non solo, con matrici di partenza di origine biologica, come è il caso con il brewer's spent grain. La campagna sperimentale condotta ha permesso di validare risultati precedentemente ottenuti, di ottenerne altri e di rendere possibile un confronto fra le estrazioni batch, le estrazioni convenzionali e le estrazioni col Turbex. I dati ottenuti sono poi stati utilizzati nello sviluppo concettuale dello scale-up a scala di impianto pilota del processo.

Il brewer's spent grain rappresenta uno dei più importanti scarti agro-alimentari in termini di produzione nel mondo. Stante la produzione di 20 g di questo scarto per ogni ettolitro di birra prodotto, e che nel 2020 la produzione di birra mondiale si è assestata a circa 1.8 miliardi di ettolitri, la quantità di BSG prodotta annualmente rende tale prodotto un materiale di enorme disponibilità; al giorno d'oggi, il brewers' spent grain (BSG) non conosce un utilizzo di alto valore: per lo più viene ceduto a costo zero dai birrifici ad allevatori nelle immediate vicinanze, per essere utilizzato come materiale di foraggio; quando questo non è possibile, il BSG può persino rappresentare un costo, dovuto allo smaltimento, per il birrificio. Ciò nonostante, questo materiale contiene un certo numero di prodotti dall'alto potenziale valore aggiunto, che rendono degni di studio processi volti alla loro estrazione. Nello specifico, il BSG contiene un importante contenuto di proteine, arabino-xylani e polifenoli (la cui estrazione è obiettivo del processo sviluppato in questa tesi), oltre ad altri composti edibili come altri monosaccaridi. L'utilizzo di polifenoli, proteine e arabino-xylani come integratori dietetici nell'uomo è stato oggetto di diversi studi.

I polifenoli predominanti nel BSG sono acidi idrossicinnamici, prevalentemente acido p-cumarico, acido ferulico e acido sinapico.
L'acido p-cumarico è uno dei tre isomeri dell'acido cumarico. Esibisce funzioni biologiche tra le quali proprietà anti-ossidanti, anti-infiammatorie, anti-diabetiche e anti-cancerogene. L'acido ferulico è un composto reperibile nelle pareti cellulari vegetali, legato covalentemente ad altre molecole. Varie le sue applicazioni

nell'industria alimentare, farmaceutica e cosmetica. Diversi studi ne dimostrano le proprietà anti-ossidanti, anti-infiammatorie e anti-cancerogene.

Le proteine extrattte dal BSG sono state suggerite come potenziali integratori: uno dei loro possibili utilizzi è quello di essere aggiunto alla farina di grano per biscotti, avendo dimostrato un effetto posivito in termini di sapore, valori nutrizionali e qualità del prodotto. Anch'esse sono state collegate con proprietà anti-ossidanti e anti-infiammatorie sugli umani. Gli aranino-xylani, infine, trovano ampio utilizzo come addensanti alimentari e agenti stabilizzatori. Esibiscono inoltre interessanti proprietà anti-ossidanti, prebiotiche, favoriscono l'abbassamento del colesterolo, e aumentano la funzionalità del sistema immunitario.

Il processo sviluppato dal team di ricerca dell'Università di Aveiro consiste in una sequenza di estrazioni con soluzione alcaline di NaOH o KOH, la cui concentrazione nella fase estraente aumenta ad ogni successivo step estrattivo.

Nello studio pubblicato, le concentrazioni di KOH nella miscela estrattiva erano, rispettivamente, 0.1, 0.5 e 4 M, con l'aggiunta di metabisolfito di sodio nel ruolo di agente anti-ossidante. Tutte le estrazioni sono state condotte in batch, con un rapporto liquido/solido pari a 10. Alla conclusione di ogni processo estrattivo, il residuo solido è stato separato per filtrazione dalla fase liquida. Alla fase liquida così recuperata è stata quindi addizionata un quantità di soluzione satura di acido citrico sufficiente a causare un abbbassamento del pH ad un valore pari a 3, in modo da causare la precipitazione di una fase solida ricca di proteine. La miscela è stata fatta sedimentare per 24 ore; si è proceduto quindi alla rimozione della fase liquida. Il solido recuperato è stato sottoposto a centrifugazione, causando una ulteriore separazione della fase liquia e solida; la prima viene successivamente trattata per causare la precipitazione degli arabino-xylani, mentre la seconda rappresenta il primo prodotto proteino. La fase liquida recuperata è stata ulteriormente acidificata sino ad un pH uguale a 2 mediamente aggiunta di soluzione al 36% di HCl. Il pH fortemente acido causa la totale protonazione dell'acido citrico fa sì che questo rimanga solubile nella soluzione. Si è proceduto quindi all'aggiunta di una soluzione acquosa di etanolo, che provoca la precipitazione degli arabino-xylani. Ancora una volta, la soluzione viene seprarata dalla fase solida per mezzo di decantazione e centrifugazione, permettendo di ottenere un secondo prodotto, ricco di arabino-xylani. L'etanolo è quindi recuperato dalla fase liquida mediante distillazione, permettendone il riutilizzo. Dopo la rimozione dell'etanolo, la miscela risultante contiene acido citrico e NaCl, la cui rimozione avviene successivamente alla sua precipitazione, permettendo di ottenere una soluzione satura di acido citrico che può quindi, anch'essa, essere riutilizzata nel processo. Il residuo solido che è stato separato dall'estratto a seguito della prima estrazione viene trattato nuovamente con un secondo step estrattivo, utilizzando la miscela 0.5 M. Gli step successivi sono identici a quelli seguenti la prima estrazione alcalina, e permettono anch'essi l'ottenimento di due prodotti (proteine e arabino-xylani) ed il recupero di etanolo e acido citrico; infine, ancora una volta, il residuo solido della seconda estrazione alcalina viene sottoposto ad una terza ed ultima estrazione alcalina (4M), a cui ancora una volta seguono i passaggi sopra descritti e l'ottenimento di altre due frazioni. Come ultimo passaggio, il residuo solido della terza estrazione alcalina è stato sottoposto ad un'estrazione con acqua distillata nel ruolo di liquido estraente. La precipitazione dell'estratto e la sua centrifugazione permettono quindi di ottenere un quarto prodotto ricco di arabino-xylani. Il processo ideato dal team di ricerca portoghese è quindi in grado di ottenere prodotti di valore dal BSG con una perdita irrisoria di solvente ed acido citrico, rendendo interessante l'applicazione di questo processo ad una scala più ampia. Nel lavoro svolto dall'autore di questo lavoro presso I laboratori del dipartimento di chimica dell'Università di Aveiro, l'esperienza oggetto dello studio pubblicato è stata replicata, con una modifica: precedentemente agli step di estrazione sopra descritti, sono stati effettuati due step step di estrazione di polifenoli. La prima estrazione è avvenuta con una miscela 60/40 di etanolo ed acqua. L'estrazione è stata eseguita con un rapporto liquido/solido pari a 10, in un becher a temperatura ambiente, usando un miscelatore magnetico a 400 RPM per un'ora. La miscela è stata quindi fatta riposare per 1 ora alla temperatura di 4°C. Ultimato questo step, il solido è stato seprato dall'estratto mediante filtrazione. L'estratto è stato quindi centrifugato una prima volta, per separare il prodotto solido dal liquido residuo. Dopo la prima separazione, è stata quindi esguita una seconda centrifugazione, da cui è stato ottenuto il primo prodotto polifenolicoche è stato quindi liofilizzato. Il solido estratto, residuo della prima estrazione con etanolo/acqua, è stato sottoposto ad un secondo step di estrazione per l'ottenimento di una seconda frazione ricca di polfenoli. Tutte le condizioni di estrazione sono state le stesse della prima estrazione, con l'unica differenza che la soluzione estraente, in questo caso, era composta di sola acqua. Successivamente alla seprazione, condotta come prima spiegata, il residuo solido è stato sottoposto agli step del processo originale descritti precedentemente. I prodotti ottenuti, insieme alla matrice di BSG iniziale, sono stati sottoposti ad una serie di test di caratterizzazione. Nello specifico si è proceduto ai seguenti test:

- Analisi della composizione monosaccaridica:

Le frazioni sottoposte a questo test sono state: il BSG di partenza, il residuo solido risultante dall'ultima estrazione, i quattro prodotti ricchi di arabino-xylani (che verranno nel seguito indicati con AX1, AX2, AX3 e AX4, dove il numero rappresenta l'ordinale dell'estrazione alcalina che ne ha permesso l'ottenimento), i tre prodotti a base di proteine (indicati con PP1, PP2 e PP3, con lo stesso razionale per la nomenclatura) e i due prodotti ricchi di polifenoli. Questo test, tipico, è concettualmente suddiviso in due macro-fasi. Nella prima, si romposono I legami glicosidici che legano i vari monosaccaridi attraverso una reazione di idrolisi acida con soluzione 72% di acido solforico ed in presenza di calore; così si cause la rottura dei legami glicosidici. Successivamente, si procede all'aggiunta di uno standard interno e quindi ad una riduzione con $NaBH_4$; prima che questa venga svolta è però indispensabile neutralizzare la soluzione per evitare la decomposizione dell'agente riducente. La reazione di riduzione provoca la conversione dei gruppi aldeidici dei moonosaccaridi, che diventano quindi alditoli. L'aggiunta di acido acetico glaciale permette quindi di degradare il $NaBH_4$ residuo. Gli alditoli ottenuti vengono quindi acetilizzati con anidride acetica e metilimidazole nel ruolo di catalizzatore. Una serie di successive estrazioni con dicloro-metano (in grado di solubilizzare i composti organici) permette il trasferimento dei monosaccardi acetilizzati in questa fase. La fase acquosa di origine viene quindi rimossa con aspirazione, ed il solvente residuo viene evaporato, lasciando solo i monosaccardi acetilati e lo standard interno. Conclusa questa prima macrofase, si procede alla seconda, che consiste nella volatizzazione di questi in un solvente appropriato che ne permette quindi l'analisi con gas-cromatografia. Il gas cromatografo produce dei segnali a tempi di eluizione corrispondenti a ben precisi monomeri. Conoscendo la concentrazione iniziale dello standard interno e dal confronto tra l'area del suo segnale e quella degli altri monosaccaridi, è possibile calcolare la concentrazione di questi ultimi.

- Analisi elementale. Sono stati sottoposti a questo test il BSG, il residuo finale, le frazioni PP1, PP2 e PP3, le frazioni polifenoliche ottenute in laboratorio e le frazioni polifenoliche ottenute mediante i test di estrazione condotti col turbex (estrazione idroalcolica e con sola acqua).

Questo test è svolto con speciali macchinari, ed è in grado di restituire le percentuali con cui determinati elementi (C, H, N, S) sono presenti nei composti analizzati. In maniera indiretta, questo test permette di stimare la presenza di proteine utilizzando il cosiddetto medoto di Kjendahl, attraverso una semplice moltipliacazione della percentuale di N per un appropriato fattore (in questo caso pari a 6.25).

- Analisi del contenuto fenolico totale; test svolto secondo il metodo di Ciolin-Focalteu. Sono stati testati in laboratorio in questa maniera le due frazioni polifenoliche ottenute. Il contenuto fenolico degli estratti ottenuti col Turbex sono stati analizzati in separata sede.

Questo test permette di dedurre il contenuto fenolico totale di un composto, espresso come mgGAE/g (mg equivalenti di acido gallico per grammo). La dissoluzione dei composti insieme al reagente di Folin-Ciocalteu permette di dedurre l'attività fenolice sulla base di una calibrazione ottenuta con acqua distillata contenente concentrazioni note di acido gallico. Uno specifico macchinario è in grado di misurarne l'assorbanza, che è correlata all'attività fenolica. Dal confronto (calibrazione) con i punti a concentrazione nota di acido gallico, è possibile stimare l'attività fenolica dei composti analizzati

- Analisi di metilazione: permette di stimare il grado di polimerizzazione ed il grado di ramificazione di un polisaccaride. Si provoca la rottura dei legami glicosidici e la loro metilazione. I gruppi metili sostituiscono i gruppi OH che si formano nel monosaccaridi a seguito della loro de-polemirazzazione, così che essi fungano effettivamente da "segnaposto" per individuare a quali atomi di carbonio erano interessati dal legame glicosidico, permettendo di capire se il legame fosse, per esempio, terminale etc. Uno stesso monomero metilizzato in differenti punti ha tempi di eluizione differenti, e pertanto una gas-cromatografia è capaci di discriminarli e dalla relativa concentrazione dei vari monosaccardi metilati è possibile dedurre il grado di polimerizzazione ed il grado di ramificazione

- Analisi granulometrica del BSG.

I risultati ottenuti per i vari esperimenti sono riportati di seguito.

	PESO		G/G OF DRY BSG
AX1	0,24097	g	2,10%
AX2	0,89828	g	7,82%
AX3	1,01381	g	8,83%
AX4	0,40192	g	3,50%
PP1	1,68753	g	14,69%
PP2	1,77684	g	15,47%
PP3	0,67782	g	5,90%
ETOH POLYPHENOLS	0,38432	g	3,35%
WATER POLYPHENOLS	0,2073	g	1,80%
RESIDUE	2,23566	g	19,47%
LOSS	1,96063	g	17,07%

Estrazione idroalcolica		
TPC	12,33729	mgGAE/g
Deviazione standard	2,101876	
Coefficiente di deviazione	0,170368	
TPC in BSG	4,765	mgGAE/g
Resa estrazione polifenoli	8,66%	

Estrazione con acqua		
TPC	24,92115	mgGAE/g
Deviazione standard	1,904152	
Coefficiente di deviazione	0,076407	
TPC in BSG	4,765	mgGAE/g
Resa estrazione polifenoli	9,44%	

Sample a	**Sample B**	**media**	**deviazione standard**	**coefficiente variazione**
% AX in AX1				
90,64%	86,96%	88,80%	0,018399929	0,020720973
% AX in AX2				
77,82%	73,09%	75,45%	0,023654895	0,031350699
% AX in AX3				
54,15%	48,83%	51,49%	0,026616797	0,051693873
% AX in AX4				
58,00%	85,32%	71,66%	0,136607257	0,19063168
% AX in PP1				
2,29%	1,48%	1,88%	0,004015551	0,213155315
% AX in PP2				
10,99%	10,35%	10,67%	0,003184119	0,029849228
% AX in PP3				
15,51%	21,23%	18,37%	0,02859185	0,155646401
% AX in Residue				
32,07%	21,32%	26,69%	0,053764197	0,201418557
% AX in estratto polifenolice (estr idroalcolica)				
1,25%				
% AX in estratto polifenolice (estr. Con acqua)				
10,25%				

RESA AX SU AX NEL BSG	
AX1	7,21%
AX2	22,83%
AX3	17,58%
AX4	9,70%
TOTAL AX YIELD IN AX PRODUCTS	57,32%

Specie	relative content
t-XYL	8,50
4-XYL	55,09
3,4-XYL	0,69
2,4-XYL	1,06
TOT XYL	65,34

$$DP = \frac{8.50}{65.34} = 7.687$$

$$DB = \frac{65.34 - 8.50}{65.34} = \frac{56.84}{65.34} = 0.8699$$

Dimensione setaccio (micrometri)	fraction
-	1,6%
63	7,4%
90	11,4%
150	24,8%
300	54,6%

I risultati ottenuti hanno permesso di caratterizzare la matrice di partenza (che per sua natura ha una composizione variabile in dipendenza di una moleplicità di fattori, dal periodo di raccolta del cereale al trattamento durante la birrificazione, etc.), e di valutare le differenze nei prodotti per il processo modificato con l'aggiunta di polifenoli. Per quanto riguarda la sola estrazione di polifenoli, è stato possibile confrontare i risultati dell'estrazione batch con quelli del Turbex, permettendo un confronto fra i due e permettendo altresì di ipotizzare le rese raggiungibili dall'apparecchiatura per le successive estrazioni. I dati ottenuti hanno permesso il design di un processo su scala pilota. Il processo, per come sviluppato, differisce in alcuni punti dal processo di laboratorio: nella fattispecie, date le alte rese raggiungibili dal Turbex, si è reputato di procedere con un solo stadio di estrazione alcalino in luogo di 3, in quanto tali stadi sarebbero risultati ridondanti. Altresì, si è proceduto con una sola estrazione idroalcolica di polifenoli, rinunciando a quella con sola acqua, data la resa oltre 90% di polfenoli ottenuta. Il processo è suddiviso in quattro sezioni. Nella prima sezione, relativa allo stoccaggio del BSG ed al suo pretrattamento, si procede ad un dewatering con roll-filter press e quindi ad un essiccamento con tamburo rotante per far sì che il contenuto finale di umidità resti al di sotto del 10%: questo è fatto per diminuire la volumetria occupata dal BSG (che arriva dai birrifici con un contenuto di umidità oltre il 70%) e per prevenirne il deterioramente, così che possa essere stoccato per periodi più lunghi. La seconda sezione è quella relativa all'estrazione di polifenoli, realizzaat con una stazione Turbex che utilizza una miscela 60/40 di etanolo ed acqua come solvente. Dall'estrazione si ottengono quindi un solido, che viene inviato alla sezione successiva, ed un liquido contenente polifenoli. Tale liquido presenta un alto livello di impurezze. Per aumentarne il valore commericale, è necessario procedere ad una purificazione, ottenuta mediante un passaggio attraverso una sezione di ultrafiltrazione e quindi di nanofiltrazione. Il liquido così ottenuto, ricco di polifenoli, viene quindi sottoposto ad una evaporazione in un evaportore a tubi verticali ed ad un ultimo essiccamento con asciugatore a cilindri, che permette di ottenere un prodotto finale sotto forma di polvere.

Il solido proveniente dalla prima estrazione, viene quindi sottoposto preliminarmente ad un lavaggio con acqua, e quindi ad una nuova estrazione con una soluzione alcalina di NaOH 4M, sempre con Turbex. In questo caso, il liquido ottenuto viene prima acidificato con acido citrico in un apposito tank, e la soluzione inviata ad un decanter centrifugo per ottenere la separazione della fase solida da quella liquida. La fase solida è ricca di proteine, e viene inviata ad uno stadio di centrifugazione e di successivo essicamento un essiccatore a cilindri. La fase liquida invece viene ulteriormente acidificata con una soluzione 36% di HCl in un tank apposito, e quindi, in un tank successivo, viene miscelata con una soluzione 60/40 di etanolo. La soluzione risultante viene inviata ad un decanter centrifugo, e la fase solida risultante viene sottoposta ad una centrifugazione e ad un essiccamento con essiccatore a cilindri per ottenere una polvere ricca di arabino-xylani. Il solvente utilizzato, proveniente dai liquidi separati, contiene acqua, etanolo, acido citrico, NaCl ed HCl. La miscela, proveniente dalle diverse parti dell'impianto, è inviata alla quarta sezione, dove si procede ad una prima distillazione per il recupero di etanolo. La miscela etanolica recuperata viene inviata allo stoccaggio e da qui al resto dell'impianto. Il prodotto di fondo contiene NaCl, er la cui rimozione si procede con una miscelazione con una miscela idroalcolica che ne causa la precipitazione, seguita da decantazione centrifuga. L'NaCl ottenuto è quindi un sottoprodotto dell'impianto che può essere venduto. La fase liquida separata viene quindi inviata ad una colonna di distillazione per il recupero della miscela etanolica. Il prodotto di fondo contiene acido citrico e acqua. Per riottenere una soluzione satura in acido citrico, questa corrente viene processata con un evaporatore a triplice effetto, che causa l'allontanamento dell'acqua; la soluzione di acido citrico può quindi essere ricircolata nell'impianto.
L'impianto, per come sviluppato, è in grado di processare 2000 tonnellate annue di BSG.

Utility	Consumo annuo	
Elettricità	3292	kWh/year
LPS	23556	ton/year
Acqua di raffreddamento	632661	m3/year

Prodotto	Produzione annua	Ricavi annui
Polifenoli	5.56 t	362.300,00 €
Proteine	110 t	330.300,00 €
AX	143 t	1.713.000,00 €
NaCl	1880 t	56.000,00 €
Tot. ricavi annui		2.461.300 €

La documentazione di processo redatta si compone di Process Description, Design Basis, PFD, P&ID, Plot Plan, Equipment list, Instrument List, Interlock description.

Una valutazione economica dell'economicità del processo è stata redatta, composta di CAPEX, OPEX, e alcuni indicatori economici di rilievo. Dall'analisi, si è stimato che l'impianto sia in grado di generare un utile annuo pari a 867.000 euro.
Con un investimento iniziale di 9.660.000 euro, ed un ROI annuo di 8.98%, si stima che l'impianto abbia un PBP di 12 anni. L'indice IRR calcolato per l'impianto è pari a 6.36%.

1. INTRODUCTION

1.1 Definition of thesis target and of the enviroment in which this thesis program has been developed

The main object of this work is to illustrate the design of a demonstration-scale process for the valorisation of Brewer's spent grain (from now on referred to as "BSG") by extraction of a polyphenol rich extract, a protein-rich extract and an arabino-xylans (from now on referred to as "AX")-rich extract. To accomplish this result, it has been necessary to conduct analysis of the raw materials and of the products to precisely asses their carachterisation and behaviour. The process is based on the methodology developed in (Vieira, et al., 2014) and subsequently patented (Coimbra Rodrigues da Silva, et al., 2012), and features two main modifications: the addition of the Turbex cavitational extractor (Cavaglià, 2018) in the role of solid-liquid extractor, and the addition of a preliminaryhydroalcoholic extraction of a polyphenols-rich material. The aim of this work is to demonstrate that a continuous-regime version of the process, able to process 2000 ton/year of BSG, is feasible and economically viable. This work has been carried out in the context of a internship at the Universidade de Aveiro; during this work, experiments for the characterization of raw materials and products samples have been carried out, as a first step, to validate previously obtained results and, secondly, to make a comparison of the extraction yields obtained with the process as descripted by (Vieira, et al., 2014) and the ones obtained with TURBEX. Secondly, in the same university, under the supervision of my thesis supervisors at the Universidade de Aveiro and my thesis supervisors at the Politecnico di Torino, the actual design of the process took place: with this, we mean the creation of the process flowsheeting (containing, among the other things, all the energy and mass balances for every equipment of the process, their sizing and the choice of suitable equipment among the ones available in the market, and also chemicals, utilities and energy consumptions of the process), and the redaction of all the necessary documentation. In the *design basis*, the theoretical grounds, the results employed (both experimentally obtained and found on the scientific literature), the assumptions and hypothesis used during the design of all equipment are explained in detail. In the *process description*, we present a detailed explanation of the operations of the process for how they have been designed; furthermore, in this document the designed control systems' logic is explained. Also, the *equipment list*, the *line list*, and the *instrument list* are presented as ancillary documentation. Finally, we present an economical evaluation of the process by means of preliminary CAPEX and OPEX estimations, as to assess the economical viability of the process.

The process is based on the extraction of several high added value products. First, the BSG matrix undergoes ad solid-liquid extraction with an ethanol-water solution as extracting medium, to obtain a product rich in polyphenols. The solid fraction leaving this first extraction process is subsequently sent to the next step of the process: the combined recovery of protein-rich and AX-rich materials. The protein material extraction is also carried out with TURBEX as extracting equipment, and with an alkali solution as extracting medium. After the extraction, the extract is acidified to cause the precipitation of proteins, which are then separated from the liquid; in turn, the liquid is further acidified and treated with an ethanolic solution to cause the precipitaion of AX. The citric acid, the hydrochloric acid and ethanol used as chemicals in the process are for the most part recovered and recycled. The process as developed originally is interesting in the context of green economy because of the fact that:

- Valorises and abundant industry by-product by extracting high added-value compounds
- Its full integration allows for a high reagents recovery and minimizes chemical consumption

In the aforementioned paper, the extraction step is carried out both in 24 hours and 2 hours, in a batch fashion. It's easy to see, that on an industrial scale, this would translate to big volume equipment even for the shortest extraction conditions. The Turbex realises a continuous version of the extraction steps and reduces the volumetric encumbrance of the plant; moreover, as will be explained, this type of equipment is able to realise higher inter-phase mass exchange rates than traditional solid-liquid extraction equipment; thus the design respects the conditions of "significantly enhancing transport rates" and "is that it is smaller – perhaps by orders of magnitude – than that it supersedes", thus placing it in the context of process intensification (Reay, et al., 2013).

The performance of the cavitational solid-liquid extractor Turbex will be assessed and compared to that of other extraction equipment; to carry out such comparison, and preliminarely to the process design phase, it has been necessary to carry out proper analysis on the samples as treated by the Turbex extractor; the methods and materials used to analyse the samples will be presented in this work, along with their results and the results' interpretation.

1.2 Introduction on brewer's spent grain: sources, charateristics, applications, market

Brewer's spent grain (BSG) is a lignocellulosic material, and the major by-product of the production of beer, representing 85% of the total by-products generated. Around 20 kg of BSG are produced for 100 litres of beer (Mussatto, et al., 2006). BSG is the solid residue left after separation of the wort during the brewing process, specifically in the mashing phase. (Santos, et al., 2003). We refer to mashing as the phase of the production of beer, following the malting phase, during which the malt is slowly heated, together with water and adjuncts, to cause the enzymatic conversion of starch and proteins in simpler units; the sugary liquid extract, called the wort, will then go on to the fermentation step, after being separated from the solid fraction (BSG) by means of filtration (Ensminger, 1994).

In some breweries, barley is the only cereal used: in this case the BSG is then composed by its milled grains, which in turn are composed by three main parts: the germ (embryo), the endosperm (comprising the aleurone and starchy endosperm) and the grain coverings , with the last divided into three fractions: the seed coat (the innermost layers surrounding the aleurone); overlying the seed coat are the pericarp layers, which, in turn, are covered by the husk (Kunze, 1996). However, other types of cereal beside barley are often added to the mash: among them, the most common are wheat, rye and maze; in this case, BSG represents the total solid fraction comprising these other cereals too (Fix, 1999). After the separation, the resulting BSG contains around 70-80% moisture (Vieira, et al., 2014).

BSG's composition is dependent on barley variety, time of harvest, characteristics of hops and other adjuncts added, and brewery technology (Santos, et al., 2003). In Table 1-1, compositions of BSG as found by different studies are reported. (Jackowski, et al., 2020).

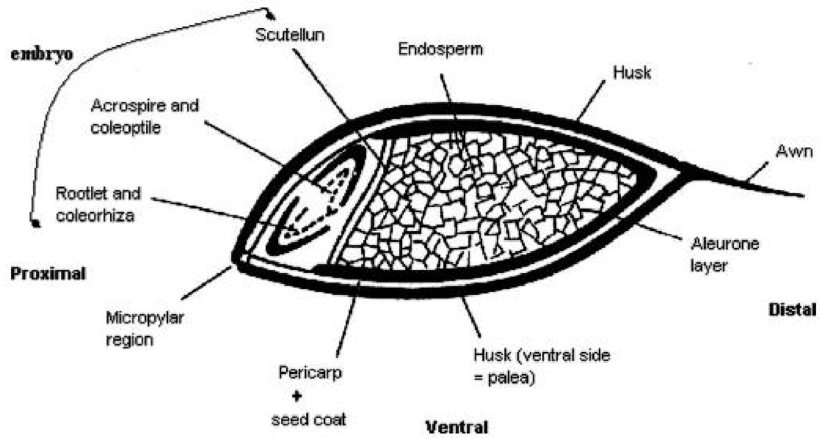
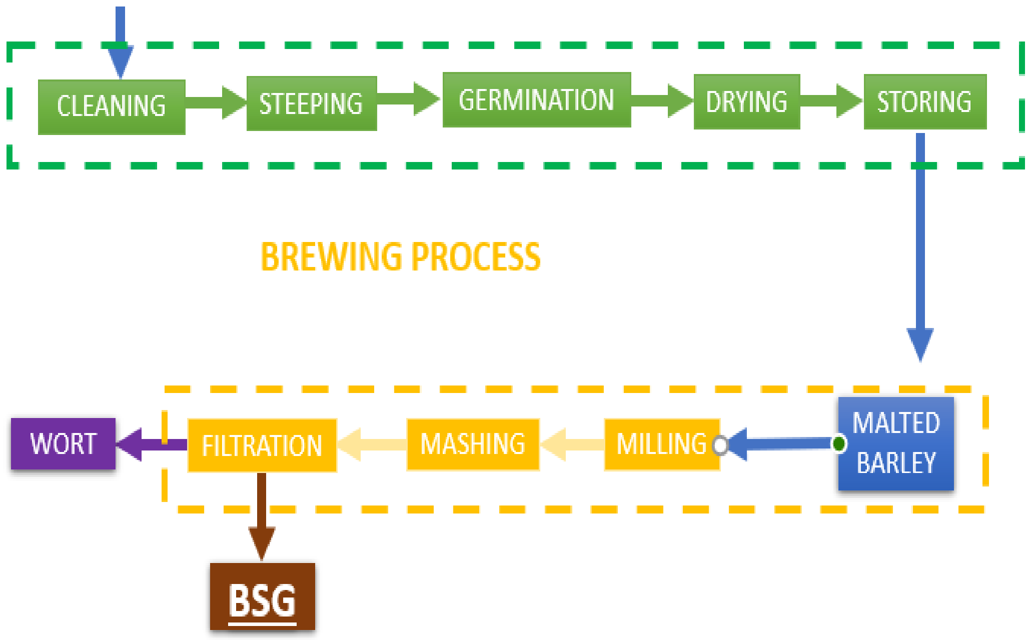


Figure 1-2 Schematic of barley grain (Mussatto, et al., 2006

As we can see, the compositions found in literature are indeed very different from

Figure 1-1 Production of beer process and origin of BSG. Adapted from (Mussatto, et al., 2006)



each other. Nonetheless, we can give a general description of BSG as a material rich in sugars, minerals and proteins, with variable composition but mainly composed of fibres (cellulose, hemicellulose and lignin) and protein (Mussatto, 2014). Regarding the categories which are the main interest of this work (namely, the proteins and the arabino-xylans, the latters belonging to the hemicellulose category), we have that the main proteins are A, B, and C hordenins, (accounting for around 50% of the proteic fraction), followed by glutenins and lastly albumines, which are the less abundant one, accounting for around 2% of the protein fraction; AX are the most relevant among the hemicellulosic compounds; less abundant, but still important, are the phenolic compounds (Vieira, et al., 2014).

The predominant phenols in BSG are hydroxycinnamic acids (Bartolomé & Gomez-Cordovés, 1999), mainly p-coumaric acid, ferulic acid and sinapic acid, contained in amounts that depend, among other things, from the type of treatment (kilning) that the BSG has undergone. Higher kilning temperature are correlated with lesser total phenolic content in the BSG (Moreira, et al., 2013).

Ferulic acid is a phytochemical found in plant cell walls, covalently bonded as side chains of other molecules. Ferulic acid has many applications in food, health, cosmetic and pharmaceutical industries, and is known to possess several physiological functions, ranging from anti-oxidant, to anti-inflammatory and anti-cancer activities, among the others (Mussatto, et al., 2007). Its application as anti-oxidant is due to the chemical structure of the compund: the presence of a phenolic nucleus and extendend side chain, allows the ferulic acid to form resonance-stabilized phenoxy-radical which accounts for its radical-scavenging effect (Zhao & Moghadasian, 2008).

p-coumaric acid is one of the three isomers of the coumaric acid. Several applications are described in literature. Biological functions exhibited by p-coumaric acid include anti-oxidant, anti-inflammatory, antidiabetic, anti-ulcer and anti-cancer activities (Ilavenil, et al., 2016).

The high protein content of BSG is one of the most interesting charateristic of this material. Proteins extracted from it exhibit interesting physiochemical properties, suggesting its uses as an addition to what flour for bakery, where it has been shown to have positive effetcs on flavor, nutricional value and quality of cookies. Protein co-precipitates are also an interesting valuation route, because of their increasing demand and for the growing interest in sustainability and waste recycling. The main interest for proteins from BSG is caused by its interesting properties as anti-oxidant and anti-inflammatory. (Wen, et al., 2019).

AX are hemicellulosic carbohydrates polymers.. They occur in various tissues of cereal grains and are the predominant matrix polysaccharides in cereal grain cell walls. Structurally, they are composed by a back-bone of $\beta(1\rightarrow4)$ likend D--xylopyranosil residues with α-L-arabinofuranosil groups as side chains. Cereal xylans are mainly substituted with monomeric arabinofuranosyl groups at C-2, C-3 or both in the xylose backbone. Arabinofuranoxyl groups are irregularly distributed along the xylan chainAX constitute a dietary fiber, influencing the nutritional quality of cereal-based foods. Hydroxycinnamic acids esterified at the

C-5 position are present the arabinoxe groups linked to C-3 in the xylose chain. The dimerization of ferulic acid forming a covalent link between AX chains.

They have applications as a food thickeners and stabilizing agents. They exhibit physiological activities as antioxidant, prebiotics, cholesterol-lowering agents, blood sugar modifiers, and immunity enhancers (Bastos, et al., s.d.).

COMPOSITION OF BSG (% ON DRY WEIGHT)

Authors	Lignin	Cellulose	Hemicellulose	Ash	Protein	Lipids	Phenolics	Starch
Kanauchi et al. (2001)	*11,90*	*25,40*	*21,80*	*2,40*	*24,00*	*10,60*	*ND*	*ND*
Carvalheiro et al. (2004)	*21,70*	*21,90*	*29,60*	*1,20*	*24,60*	*ND*	*ND*	*ND*
Silva et al. (2004)	*16,90*	*25,30*	*41,90*	*4,60*	*ND*	*ND*	*ND*	*ND*
Mussatto and Roberto (2006)	*27,80*	*16,80*	*28,40*	*4,60*	*15,20*	*ND*	*ND*	*ND*
Celus et al. (2006)	*ND*	*0,30*	*22,50*	*3,30*	*26,70*	*ND*	*ND*	*1,00*
Xiros et al. (2008)	*11,50*	*12,00*	*40,00*	*3,30*	*14,20*	*13,00*	*2,00*	*2,70*
Jay et al. (2008)	*20-22*	*31-33*	*ND*	*ND*	*15-17*	*7,00*	*1,0-1,5*	*11,00*
Treimo et al. (2009)	*12,6 ±0,1*	*45,9 (tot. Carbohydrates)*			*23,4 ±1,4*	*ND*	*ND*	*7,8± 0,2*
Roberston et al. (2010)	*13-17*	*ND*	*22-29*	*ND*	*20-24*	*ND*	*ND*	*2,0-8,0*
Khidzir et al. (2010)	*56,74 ± 9,38*	*40,20± 17,71*	*ND*	*2,27 ±0,76*	*6,41 ±0,31*	*2,50 ±0,11*	*ND*	*0,28 ±0,06*
Waters et al. (2012)	*ND*	*26,00*	*22,20*	*1,10*	*22,10*	*ND*	*ND*	*ND*
Nuno et al. (2013)	*19,40 ± 0,34*	*21,73± 1,36*	*19,27± 1,18*	*4,18 ±0,03*	*24,69 ±1,04*	*ND*	*ND*	*ND*
Sobukola et al. (2012)	*9,19± 0,011*	*60,64±0,26 (tot. Carbohydrates)*		*2,48 ±0,02*	*24,39 ±0,46*	*6,18 ±0,13*	*ND*	*ND*
Kemppai-nen et al. (2016)	*19,60*	*45 (tot. Carbohydrates)*		*4,10*	*20,30*	*ND*	*ND*	*ND*
Yu et al. (2020)	*ND*	*51,0±0,7 (tot. Carbohydrates)*		*4,1± 0,1*	*23,4 ±0,1*	*9,4± 0,1*	*ND*	*ND*

Table 1-1 BSG composition in different studies

In 2019, the global beer production amounted to 1.91 billion hectolitres (Conway, 2021), allowing us to infer a BSG production of around 38 million tons per year. The huge production and availability of this material, combined with its low price and the properties so far presented, make BSG an extremely interesting raw material in a Chemical Eningeer's perspective.

2. STATE OF THE ART

One of the promising routes for the valorisation of BSG is the extraction of valued products for human consumption. Some extractive processes have been developed on a laboratory scale, some of which have been patented.

1.3 Survey of industrial scale technologies available on the market

To the best of our knowledge, there is no estabilished industrial route for the extraction of AX and proteins from BSG.

Historically, BSG represented a by-product requiring disposal, which poses environmental problems. Currently the main use of BSG is as animal feed: most often for dairy cattle, where BSG has proved to be beneficial under several aspects; also, its use as feed for poultry, pigs and fish has been investigated and found to be beneficial for the animals (Mussatto, et al., 2006). BSG is usually sold by breweries to nearby fams; but because of the increasing centralization of the farming structures and of the high production of BSG per hectoliter of beer, its disposal as animal feed has become increasingly difficult (Steiner, et al., 2015). For this reason, breweries sell BSG to farmers at low prices, and when this is not possible, its disposal becomes an expense for the brewery. Moreover, BSG must be dried quickly to avoid microbial spoilage, and due to its high moisture content, this proves to be economically burdening (Yu, et al., 2019).

One viable industrial application of BSG is presented in (Weger, et al., 2016); the authors performed a mechanical dewatering of the BSG by means of a screw press, obtaining a solid cake and a press water; the first one can be burned along woodchips for heat generation: a certain ratio between cake and woodchips must be mantained to comply with NOx emissions. The press water is then subjected to anaoribic digestion for the production of biogas. The process, according to the authors, ic capable of genereting around 40 000 euros per year of revenue, with a payback period of 11 years.

Other uses have been studied in the recent years: namely as a source for the production of biodiesel, the production of phenolic compounds, lactic acid and for the growth, and cultivation of microorganisms, fungi and enzymes (Karlovic, et al., 2020) (Panjicko, et al., 2017).

Another promising route for the valorisation of BSG is represented by its possiblity to be used for the production of activated and non-activated carbon materials, to be used for decolourising, solvent recovery, water treatment, deodorizing, gas impurities removal, metals recovery and catalysis (Jackowski, et al., 2020).

For what concerns its use for human consumption, the addition of BSG to humand diet has been proven to have several beneficial effects, as accelerated transit time, increased faecal weight, fat excretion, decreased gallstone incidence and reduced plasma cholesterol and postprandial serum glucose levels. In this case, BSG is powdered and made into a flour used for bakery products (Mussatto, 2014). Some of the food preparation of BSG have been also patented: for example a BSG protein powder in (MacKay, et al., 2018) and a beverage (Gil-Martinez & Arendt, 2018). AX display immunomodulatory, antioxidant and anticarcinogenic effect (Vieira, et al., 2014).

1.4 Survey of patent literature

To the best of the author's knowledge, the earliest patented method for the recovery of protein fractions from BSG is presented in Japanese Unexamined Patent publication No. 51-129776, and it involves the usa of an alkaline aqueour solution of pH 11-12 at 104°C-121°C, followed by isoelectric precipitation. This process was found to be un-satisfactory because of the protein degradation caused by the severe extraction conditions. In the patent by (Kishi, et al., 1989), these operations are preceded by pressing of BSG in wet state, followed by the separation by sieving of the low-protein husks from the protein-richer crushed gems and other particles, thus allowing for the extraction to be conducted in less severe conditions (namely, temperatures between 60°C and 100°C). This process still suffers from partial degradation of the protein fraction and high energy consumption due to the high temperatures required (Vieira, et al., 2014). Other inventors tried to obtain protein fractions by means different thant extractions: namely, in the patent by (McEwan & White, 2019), BSG is cell milled after hygienic extraction and drying, and the resulting powder is fractionanated in a finer protein-rich powder and a coarser one which may be recycled to the cell mill for further refining. In the patent by (Munch, et al., s.d.), the fractionation is obtained by means of an air classifier.

To the best of our knowledge, there is no estabilished industrial route for the extraction of AX and proteins from BSG.

So far, the already mentioned process firstly described in (Vieira, et al., 2014) and patented in (Coimbra Rodrigues da Silva, et al., 2012) is the only patented process able to realise a simultaneous recovery of both proteincs and AX in different fractions. The process as presented in the aforementioned works consists on a series of alkaline extractions of increasing strength, carried out first on the

BSG and then on the solid residue of the preceding extraction. The agent used to achieve the extraction is KOH, present in 0.1 M, 0.5 M and 4 M, respectively, in the three extraction steps, with the addition of sodium meta-bisulphite as anti-oxydant. Beside the solid residue, every extraction step originates an extract in a liquid phase. Every extract is then treated with a saturated aqueous solution of citric acid, as to obtain, from each extract, a fraction containing precipitated arabino-xylans; the solution deprived of AX is then further acidified to a pH 2 with a 37% aqueous solution of HCl, and then treated with an aqueous solution of ethanol (70% v/v). This step causes the proteins to precipitate, allowing them to be separated from the ethanolic solution by means of decantation and filtration. The citric acid present, due to its full protonation at a pH below 2, remains soluble in this solution. The ethanol was recovered by distillation of the ethanolic solution resultant from the precipitation of polysaccharides, leaving an aqueous solution containing citric acid and NaCl dissolved. The NaCl is partially removed as a precipitate upon concentration of the solution, allowing to obtain an aqueous solution saturated in citric acid. An extraction with ethanol is also performed to the precipitated NaCl to recover co-precipitated citric acid.

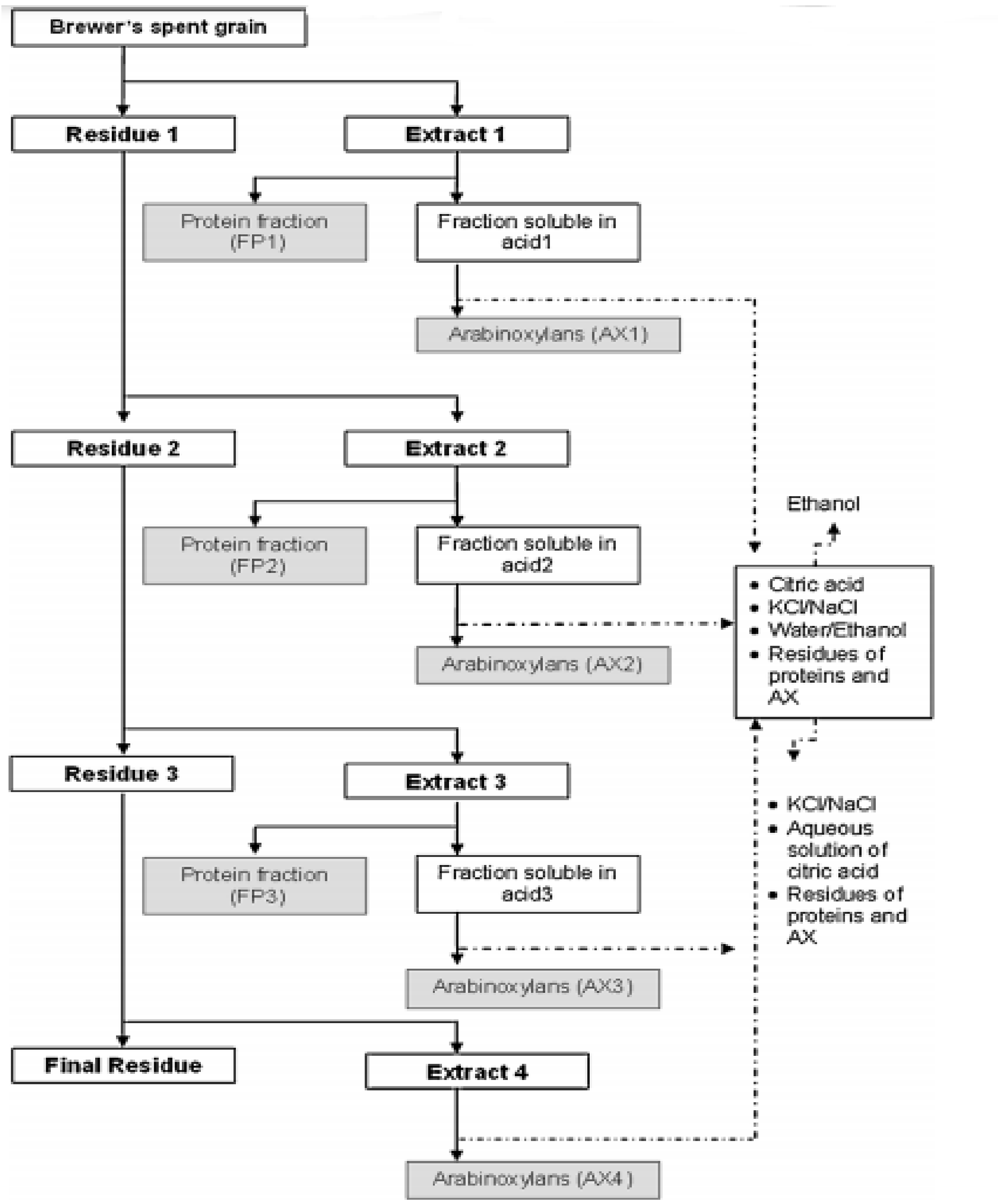


Figure 2-1 Patented process for the extraction of AX and proteins from BSG

The ethanol is recovered by evaporation and the two aqueous solutions containing the citric acid are combined and reused as a saturated citric acid solution. According to the authors, the processi s able to recover around 82% of the total protein fraction contained.

As already mentioned, the possiblity of spoiling is a very worrying one when dealing with BSG. Its high moisture content promotes detrimental microbial activity. Thus, an industrial process must be concerned with a way to stabilize the stored raw-materials waiting to be processed. Several drying methods specific for BSG are available in the patent literature.

- BSG dehydratation with fluidized bed or rotary drum with hot air in countercurrent. Its drawback is the high energy demand, and it only achieves residual moisture content of 10%-15%.
- BSG dehydratation by screw conical press with sieve or travelling screen press for mechanical dewatering, followed by thermal dehydratation in convection dryer or by solar energy.
- Dilution of BSG up to 95% moisture followed by separation with vibrating sieving to recover the fibrous fraction, which is then dehydrated in a centrifugal settler down to 30% moisture levels, and finally a last drying by either paste dryer or extrusion dryer.

BSG dehydratation by means of a filter press, with integrated compression by means of a thermal fluid and vacuum drying, with a specifically designed control chain for the control of the pressure in the filter, as described in the patent (Carvalho, et al., 2010).

	Process a)	Process b)	Process c)	Present Invention
BSG dilution	-	-	95%	-
Mechanical dehydration type	-	Worm extruder or travelling screen press	Vibrating sieve followed by a centrifugal settler for the protein fraction and screw-press for the fibrous fraction	Membrane filter press.
Thermal dehydration type	Fluidized bed	Thermal dryer or solar energy	Thermal dryer for the protein fraction	Vacuum thermal drying, in the filter press.
Advantages	Maintenance of the BSG nutritional characteristics	Thermal drying with energy costs reduced by mechanical dewatering	Protein concentrate with commercial value for animal food	- Exudate with polyphenols for the pharmaceutical and cosmetic industry - Reduced production costs (50€/ton) - Maintenance of the BSG nutritional characteristics - Reduction of the ruminant digestive problems during the weaning
Disadvantages	High energy expenditure	- High equipment and operating costs - Reduced BSG nutritional value.	- Great water consumption - High equipment and operating costs	-
Final product	Dry BSG	Dry BSG with low nutritional value	- Protein concentrate - Fibrous fraction	- Dry BSG with high nutritional value - Exudate with polyphenols.

Figura 2-2: Comparison between several BSG dehydratation patented technologies, from the patent by (Carvalho, et al., 2010)

1.5 Survey of scientific literature

Concerning the extraction of carbohydrates from the BSG, several techniques have been investigated in the scientific literature. The AX fraction in BSG can be divided into two categories: water-unextractable (WUAX) and water-extractable (WEAX). The difference in behavior is dictated by their molecular weight, the ratio between xylose and arabinose, and the degree of cross-linking with other polymers (Severino, et al., 2015). Some of the technologies for the recovery of AX are then based on pretreatment with an ethanolic solution followed by a number of simple water extractions.

The use of alkaline aqueous solutions for the extraction of AX from various vegetable matrixes and specifically cereal brans has been extensively studied (Kale, et al., 2013). In (Hoije , et al., 2005) we found an exaple of arabino-xylans extraction from barley husks.with the highest yield of AX being over 80%. The alkaline extractions descripted in the literature require require some form of pretreatment of the raw-materials.

More recenlty, a variety of improved techiniques for protein recovery from BSG have been developed (Bonifacio-Lopes, et al., 2019); among them we can cite ultrafiltration membranes (Tang, et al., 2009) , the use of microwaved-heated water (Coelho, et al., 2014), and ultrasonic-assisted extraction in water (Tang, et al., 2010) and in alkaline solutions (Reis, et al., 2015); regarding the microwave-assisted extraction, the effect of the microwaves it thst of rapidly heating the solvent, reducing the amount of solvent needed and the time required for the operation. In this case, higher temperature correspond to higher extraction yield on one hand, but on the other causes depolymerization, debranching and deesterification of the polysaccharides and the formation of brown products due to Maillard reactions. The ultrasounds are able to induce pressure variations, causing the forming and istantaneous collapse of bubbles, causing the breakage of cell wals and improved extraction performances, with a pronounced reduction of the time needed for the extraction; one drawback of this methodology is the possibility if polymers break-down by the ultrasounds (Bonifacio-Lopes, et al., 2019).

3. FUNDAMENTALS

1.6 Fundamentals of solid-liquid extraction with respect to vegetable matrixes

Extraction is a separation process based on the differences in solubility of different materials in a given solvent. In the solid-liquid extraction, we extract one or more components from a solid mixture to a liquid solvent. This operation is also referred to as "leaching". We distinguish an extraction from a washing, although the physical phenomena is the same: we call a process "extraction" if the useful product is the extractable solute, whereas we call a process washing if its purpose is the removal of one or more components from the desired, insoluble ones (Berk, 2009).

An extraction operation can be conducted in both a batch or a continuous regime. For both regimes, the key concept in an extraction (like in many other separation operations) is the concept of equilibrium stage. An equilibrium stage is a theoretical concept. It can be imagined as a "black-box", receiving as input a solid stream and a liquid solvent stream. Frox the box two streams leave as output: a solid and a liquid one, in mutual thermodynamical equilibrium.

When dealing with vegetable matter, we often work with material with high porosities. By that, we mean the presence of many highly tortuous channels in the bulk of the solid. The transport mechanism of the solute from the solid to the bulk of the solvent is thus composed of a series of elementary mechanisms:

1. Transport of the solvent from the bulk liquid to the solid surface
2. Diffusion of the solvent into the porosities of the solid
3. Interphase transport fo the solute from the solid to the solvent imbibed in the pores
4. Diffusion of the solute in the solvent present in the porosities
5. Transport of the solute from the solid surface to the bulk of the solvent.

Each mechanism proceeds at its own rate. If the contact time between matrix and solvent is not long enough, the composition of the fluid imbibed in the solid may differ considerabily from the composition of the bulk fluid. Let's suppose a stage where the solvent if sufficient to dissolve all the extractable soluteIf the quantity of solvent and the solid matrix is truly inert: then the stage is at equilibrium if concentration of the solute in the extract is equal to that of the imbibed solution. In practice, equilibrium conditions are not always realised in a single stage. In cases where the solute is ditributed through a more or less permeable solid, the rate of leaching is largely governed by the rat of diffusion through the solid. Thus, the number of real stages to achieve a certain separation is generally different from the number of ideal stages. In (McCabe, et al., 1993) we find a definition for the so called Murphree efficiency η_M:

$$\eta_M = \frac{X_0 - X}{X_0 - X^*} \quad (3.1)$$

Where

- X_0 is the concentration of the extract entering the stage
- X is the concentration of the extract leaving the stage
- X^* is the concentration of the extract if equilibrium was attained

Having so defined the stage efficiency, the number of real stages can be calculated from the number of ideal stages using the following relation:

$$\eta_M = \frac{Number\ of\ ideal\ stages}{Number\ of\ real\ stages} \quad (3.2)$$

The mechanism by which the mass transpher occur is diffusion. In leaching, the diffusion of solute through the solid phase is followed by diffusion into the liquid. Among the models used when dealing with this type of phenomena, the most used ones are the film theory and the compenetration theory; according to (McCabe, et al., 1993) those two models are actually just two limit cases of a more general model.

Diffusion arises in presence of a gradient of chemical potential, or in other words, when a gradient of activity is present. In chemical engineering, the gradients of activity in leaching usually coincide with gradients of concentration. In this case, the molar flux of a component A in a solvent B is described by Fick's first law of diffusion:

$$\vec{J}_A = -D_{AB} \nabla C_A \quad (3.3)$$

Where:

- $\vec{J}_A$ is the molar flux of component A in the solvent (mol/m2/s) due to diffusion
- D_{AB} is the diffusion coefficient of component A in solution with B (m2/s)
- C_A is the molar concentration of component A in the mixture.

parlare del film

In most cases, the flow caused by diffusion is accompanied by the flow caused by convection. When this occurs, the total molar flow of A N_A is the sum of the two contributions.

$$N_A = y_A N - \vec{J}_A \quad (3.4)$$

Where:

- N is the total molar flow
- N_A is the molar flow of A

- y_A is the molar fraction of A in the solvent

In the case that only the component A diffuses, the total molar flow coincides with the molar flow of A. In that case, assuming: the validity of he first Fick's law, the unidirectionality of the flow it's possible to write:

$$N_A = y_A N_A - D_{AB}\frac{dc_A}{dx} \quad (3.5)$$

$$N_A = y_A N_A - D_{AB}\rho_M\frac{dy_A}{dx} \quad (3.6)$$

$$N_A(1 - y_A) = D_{AB}\rho_M\frac{dy_A}{dx} \quad (3.7)$$

Integrating across a film of thickness δ, we find the profile reported in fig.3.1

$$\int_0^\delta \frac{N_A}{D_{AB}\rho_M}dx = \int_{y_{A,i}}^{y_A} \frac{dy_A}{(1 - y_A)} \quad (3.8)$$

$$\frac{N_A}{D_{AB}\rho_M}\delta = \frac{\ln(1 - y_A)}{\ln(1 - y_{A,i})} \quad (3.9)$$

This treatment is valid for stagnant fluids. However in industry, the most common regime is the turbolent one, because of its ability to increase mass transfer rates and interphasic surface. Nonetheless, it's still possible to use some these results in turbulent regimes.

The variation of component A in the solid is only due to the flux leaving it. It's common to write the mass transport with the massic form of the general equation for the transport of properties:

$$\frac{\partial M_A}{\partial t} = kA(C_A - C_{A,i}) \quad (3.10)$$

This equation may be solved with different approaches, the most common being the film theory and the penetration theory. The first one assumes the presence of a stagnant layer of fluid bewteen the solid surface and the liquid bulk, where only diffusion occurs. In this case, it's possible to show that the molar flux of A leaving the solid can also be rewritten as:

$$A\frac{D_{AB}}{\delta}(C_A - C_{A,i}) = kA(C_A - C_{A,i}) \quad (3.11)$$

$$\frac{D_{AB}}{\delta} = k \quad (3.12)$$

So in turbulent regime, the resistance to mass transfer is the same as the one ina stagnant layer of thickness δ. In this case the mass exchange coefficient varies with the first power of the diffusion coefficient. For most cases this has been show not to be exact. Still, this model finds large use in the practice. For the one-way diffusion flow, we can substitute in (nr.eq)

The two film theory is among the most used models employed for the description of mass transfer through an interface. The theory posits the existance of two adjacent layers, each on one of the two sides of the interface. In this case, there exist two equilibrium concentration at the interfase, one for each side. The two equilbrium concentration are related to one another by the repartition coefficient, that depends on the system. Since in each layer a gradient of concentration is present, a diffusive flow will arise as consequence in each film. If we assume steady state, it's easy to show that the two massic flows ought to be equal, otherwise there would be accumulation on the interface. For each film we can write an expression for the flow like previously shown:

$$k_S a(x_{bulk} - x_i) = k_L a(y_i - y_{bulk}) \quad (3.13)$$

Finally, it's easy to show that if write the flowrate as the product of a exchange coefficient K_y, using as driving force the difference between the bulk concentration and the equilibrium concentration at the interface, then we have that:

$$\frac{1}{K_y} = \frac{m}{k_L} + \frac{1}{k_S} \quad (3.14)$$

Another theory widely empolyed to model the interface mass trasnfer is the penetration theory. Penetration theory is actually more of a collection of various forms of model, all of them being classified as surface-renewal models; that is to say, models implying either formation of new surface at frequent intervals of replacement of fluid elements at the surface with fresh fluid from the bulk. Mathematically, the theory supposes a non-stationary balance on the film. The balance then is represented by the following differential equation with boundary conditions:

$$\frac{\partial C_A}{\partial t} = D_A \frac{\partial^2 C_a}{\partial x^2} \quad (3.15)$$

$$\begin{cases} C_A = C_{A,0} \quad at\ t = 0 \\ C_A = C_{A,i} \quad at\ x = 0, t > 0 \end{cases}$$

From this balance, we obtain an expression for the flow, which in turn can be averaged on time untila certain time t_T-finally, the flow obtained is equal to the classic expression of the flow as product of exchange coefficient and driving force, allowing us to obtain an expression that correlates mass transfer coefficient and diffusivity:

$$k = 2\sqrt{\frac{D_A}{\pi t_T}} \quad (3.16)$$

Danckwerts gives a similar expression for k:

$$k = \sqrt{D_A s} \quad (3.17)$$

Where s is the surface renewal rate.

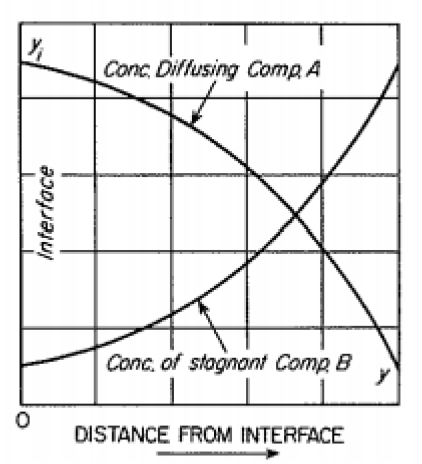


Figure 3-1 Concentration profiles across a film

In practice, to achieve the desired separation, more than one equilibrium stage may be needed. In this case the extraction is carried out in a series of stages, that may be arranged in a co-current or in a counter-current fashion; moreover, both co- and counter-current extraction may be either in continuous, semi-continuous or batch regime. The latter finds applications, for example, in the extraction of pigments from plants, isolation of protein from oilseeds, and the production of meat and yeast extractions; more often though, continuous and semi-continuous configuration are the ones used in industry (Berk, 2009). In this case, the movement of the solid with respect to the solvent may be realized in several different ways. Counter-current set ups are usually favoured; the difference bewteen the concentration of solute in the solvent and its equilibrium concentration in the same phase are the driving force that causes the mass transfer to arise. Counter-current set up, by virtue of their nature, allow to keep the driving force more constant along the equipment. This translates in a more performing and a more efficient operation. From now on, the same nomenclature as in (McCabe, et al., 1993) will be followed. We will consider a counter-current multistage extraction, composed of N stages. The liquid stream leaving the n-th stage will be denoted by the symbol V_n and the molar fraction of the desired component in said stream will be denoted with the symbol y_n;similarly, the solid stream leaving the n-th stadium will be denoted by the symbol L_n and the molar fraction of the desidred component in said stream will be denoted by the symbol x_n. The stages are numbered so that the stage number 1 is the one where the solid is fed. The numeration then follows the direction of the solid stream. Conversely, the liquid stream will be fed at stage N. To denote the fresh feed of solvent and of solid, we will use the subscript a (L_a, x_a, V_a, y_a) to denote the entering solid at stage 1 and the solvent solution leaving stage 1; similarly, we use the subscript b $((L_b, x_b, V_b, y_b)$ to denote the solid residue leaving stage N and the fresh solvent feed entering the same stage. Let's suppose for the moment that the solvent present is enough to absorb all of the solute, that there is no adsorption and that the concentrion in the imbibed liquid and in the solvent is the same, i.e., we consider the stages to be equilibrium stages. Also, we assume the solute-free solid to be insoluble in the solvent; this means that the flow of this solid is constant along the operation. The solid retains a quantity of imbibed liquid which may or may not be constant along the operation. Then, we use L to refer to this retained liquid instead. We can write the mass balances of the control volume of fig.3.2; one for the total mass, and one for the desired component

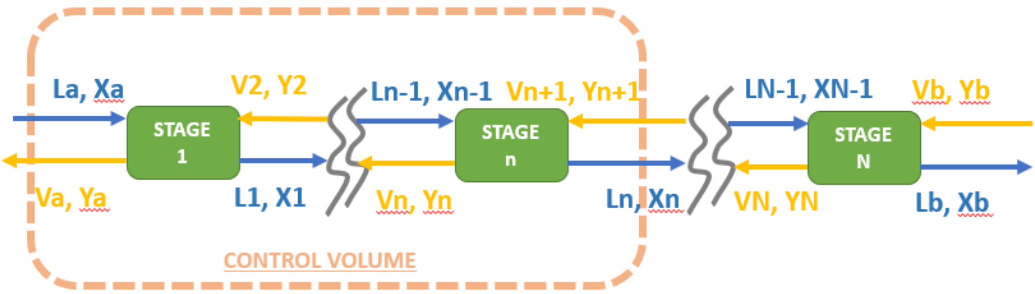


Figure 3-2 Schematic of counter-current extraction

$$V_a + L_n = V_{n+1} + L_a \quad (3.18)$$

$$V_a y_a + L_n x_n = V_{n+1} y_{n+1} + L_a x_a \quad (3.19)$$

Rearranging the terms, we obtain the operating-line equation:

$$y_{n+1} = \left(\frac{L_n}{V_{n+1}}\right) x_n + \frac{V_a y_a - L_a x_a}{V_{n+1}} \quad (3.20)$$

Passing through the points (x_a, y_a) and (x_b, y_b). Generally, the slope can change for each stage, although in certain case they may be assumed constant, giving a constant slope $L/_V$; this is the case of constant underflow. This implies that the solid always retains the same amount of liquid. This hypothesis is true if the density and viscosity of the solution doesn't change considerably with solute concentration. The considtions in every stage can then be easily determined. The number of stages may be calculated analitically of by means of a graphical method like McCabe-Thiele.

Extraction equipment, as mentioned, can be in first instance classified as either batch or continuous regime equipment. Another possible distinction is between equipment where the leaching is accomplished by percolation, and the equipment in which particulate solids are dispersed in the liquid and subsequently separated (Dahlstrom, et al., 1997)

In the following paragraphs, several types of equipment are described. If not differently specified, the source for these descriptions is Perry's Chemical Engineering Handbook, section 18-55.

Among the batch solid-liquid extractor, we cite: batch tanks, batch stirred tanks, Pachuca tanks. Bacth tanks belong to the percolation leaching equipment category. In this case, the quipment is simply a large tank filled with solid until a certain height. Solvent is then sprayed until deemed economically convenient. After that, the solid is removed from the tank. Is not uncommon to have a diffusion battery: that is to say, a serie of of batch tanks, where the solvent is fed in a “counter-current” fashion, starting from the most exhaust solid. Batch tanks may be agitated with an impeller. After the extraction step, it is necessary to separate solid and liquid phases. This may be accomplished with filters, centrifuges or thickeners, dpending on the nature of the system to be treated. Pachuca extractors are composed by a cylindrical tank with a conical bottom. Air is admitted from the bottom, agitating the solid along its way to the outlet. Pachuca extractor are a relatively big equipment, and they are used mainly in the leaching of metal ores.

Semi-continuous extraction are a typical industrial design, to improve the efficiency of purely batch systems. A typical configuration is the one in figure 3.3; ont of the tanks is in stand-by; the solvent is fed to the first extractor, and after percolation, is sent to the next tank and so on until the solvent leaves the last operating tank. When the first tank (extracted with pure solvent) is exhausted, it gets disconnected from the battery, and the solid is substituted with a fresh one, while the tank in stand-by is now attached to the tail end of the battery, and the fresh solvent is fed to the secondo extractor; the circle thene goes, disconnecting the second tank when the solid is exhaust etc (Berk, 2009).

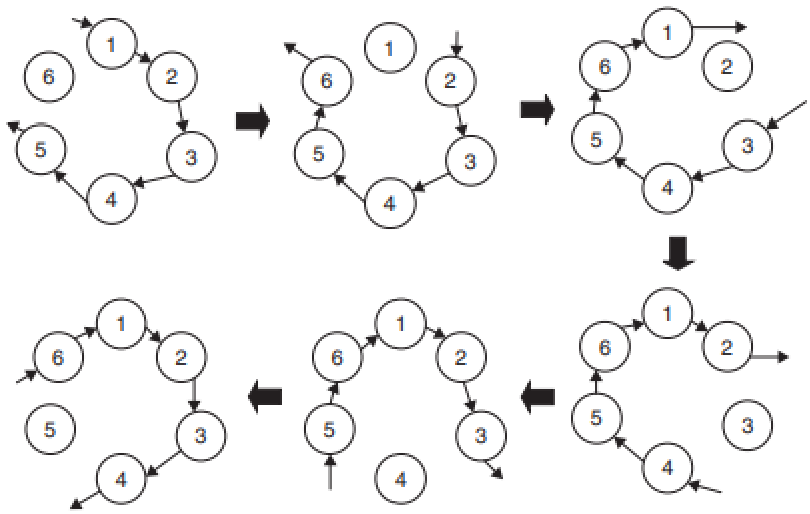


Figure 3-3 Batch extractors "merry go-round" set-up

Continuous extracting equipment is a more diverse family. Many types of equipment are available, and the choice of the right equipment depends on the nature of the material to be handled. Bollman extractor is one commond device of extraction. This apparatus consists in an endless-moving belt carrying perforated baskets, moving along the vertical direction. Solid is fed at the top of the descending section, and here is put in contact with enriched solvent pumped up from the bottom of the apparatus ("half-micella"). Fresh solvent is instead fed to the most exhausted solid, at the top of the rising section. The perforated baskets allow for the solvent to fall to the lower buckets, realising a semi counter-current regime. The exhaust solid is then discharged at the top of the apparatus on a paddle conveyor, while the extract is collected from the bottom. Horizonalt-basket extractor are also quite common. They are comprised of a big circular tank, divided in walled, anular compartments, revolving around a central axis. Each compartment, during its rotation, encounters a solid feed point, a solvent feed point (usually sprayed), a drainage section and finally a solid discharge section. The solvent is fed to the most exhaust solid; after the drainage, it is fed to the next solid compartment, thus realising a counter-current contact between solid and liquid. Similar in principle are the de Smelt and the Lurgi extractors, differing in that the disposition of the equipment is linear rather than circular.

Kennedy extractor is also one of the most common continuous extractor, with a very simple concept. The extractor is a simple long channel, devided into sections, each one provided with a paddle. The channel is usually inclined. The solid is fed to one end (usually the one at the lower height), and the solvent is fed to the other. Every section is provided by a littler basing in which solid and solvent come into contact. The solvent flows down because of gravity, while the paddles move the solid from one section to the next one, in the opposite direction of the liquid flow. The mechanical action of the extractor may make it unsuitable for fragile materials.

Among the continuous dispersed-solids extractor, the most famous is probabily the Bonotto extract, a type of vertical plate extractor. This type of equipment is comprised of a vertical column containing a series of trays, each one containing an opening opposite to the one of the plate directly above. Along the axis of the column, a mechanical rotating tree revolves around the vertical axis of the equipment. The mechanical tree is equipped with a blade for each tray, whose aim is to move the solid towards the tray opening, soo that it may fall onto the lower tray. The solid is fed from above and moves by gravity, while the solvent is fed from the bottom of the extractor, filling it to the top, where it is recovered. The solid is recovered by a screw converyor place on the bottom ot the apparatus.

Finally, screw conveyor extractor, exemplified by the Hildebrandt extractor, are often classified as percolating extractor although this is not, strictly speaking, the case.

1.7 Fundamentals of liquid extract separation and purification techniques

The purification of the extracts in the plant is achieved by means of two separate membrane steps. This allows for the recovery of liquid streams richer in the desired compound or, inversely, of a stream poorer in un-desired components content. Membranes can be applied to separation of liquid and gaseous stream. By passing through a porous membrane, the feed liquid is separated in two streams: the permeate and the retentate. Based on their size, some particles have the ability to pass thorugh the porosities, whereas molecules or particles with size bigger than the porosities don't. The stream that is filtrated bu the porosities is called permeate, whereas the other resulting stream is claeed the retentate. Although certain particles have the ability to pass through the membranes porosities, that does not mean that every particle able to do it will. With respect to each membrane in a certain set of conditions (feed pressure, operational time, configuration), it's possible to define a retention coefficent R for each component, defined as the ratio of its concentration in the permeate and in the feed. Another important desing factor in the membranes is the Molecula Weight Cut-Off (MWCO). This is conventionally defined as the moleculaar weight to which 90% of the solute is retained (McCabe, et al., 1993). The transport is accomplished by different mechanisms: diffusion and viscous transport both play a role. The driving force for membrane separation is the transmembrane pressure, i.e. the difference of pressure between two sides of a membrane; secondary driving forces may also be related to the electrical potential between feed and product. For pressure-driven separation the transport relationship is given by (Singh, 2012):

$$J = K\frac{\Delta P}{t} \quad (3.21)$$

Where:

- J is the flux of trhough the membrane
- K is the permeability constant
- ΔP is pressure differential, i.e. the driving force
- t is the membrane thickness

Based on the size of the particles to be separated, different types of membrane exist: microfiltration, ultrafiltration, nanofiltration and reverse osmosis. Microfiltration is defined as a membrane separation process that uses a MWCO of over 400 kDa. The pore size is usually bewteen 0.5 and 100 nm (Ray, et al., 2020). In the food industry is used in applications such as wine, juice and beer clarification, as well as wastewater treatment and plmas separation from blood (Charcosset, 2012). It is also frequently used for the separation of microbial cells from medium (Hori & Unno, 2011). Ultrafiltration differs from mircofiltration basically by pore size, whose dimension range from 0.01 and 0.5 nm; they tend to have a more asymmetric structure than the MF. Typical values of MWCO for the

UF membranes range betewwen 1 and 400 kDa. Like the MF, the osmotic pressure of microsolutes is practically negligible, allowing this type of membane to operate at relatively low pressure of 2-5 bar. The range of applications for this technology is indeed quite vast, from clarification in the food industry, to water tratment (often as a preliminary step before more intense separations) (Singh & Hankins, 2016). Nanofiltration (NF) exhibits typical MWCO values between 200 and 1000 Da; they have low rejection of monovalent ions and high rejection of divalent ions, as well as higher flux compared to RO membranes, charateristics that allowed NF thecnology to in part be in competition with RO in certain cases, given its lesser energetic toll. Whereas the first three work with a similar principle, reverse osmosis is quite different, and its used mainly for water purification: since it is a special case that is not employed in this work, further discussion about the reverse osmosis will not be presented. Membranes come in different types and materials. Polymeric membranes are among the most common: for the MF, crystalline polymers are usually preferred because of their chemical and thermal stability; whereas for UF amorphous glassy polymers are preferred for control of the small pore size (Singh, 2012). Beside the polymeric membranes, inorganic material are also available: ceramics, glass, zeloites and sintered metals among them. Ceramic membranes can withstand higher fluxes and tempertaures than their polymeric counterparts, as well as a longer life: but they present a higher cost.

4. TURBEX SOLID-LIQUID EXTRACTOR

1.8 Introduction of TURBEX liquid extractor

In the context of process intensification, an important addition to the process as developed by the portoguese research team is the use of TURBEX, i.e. a device

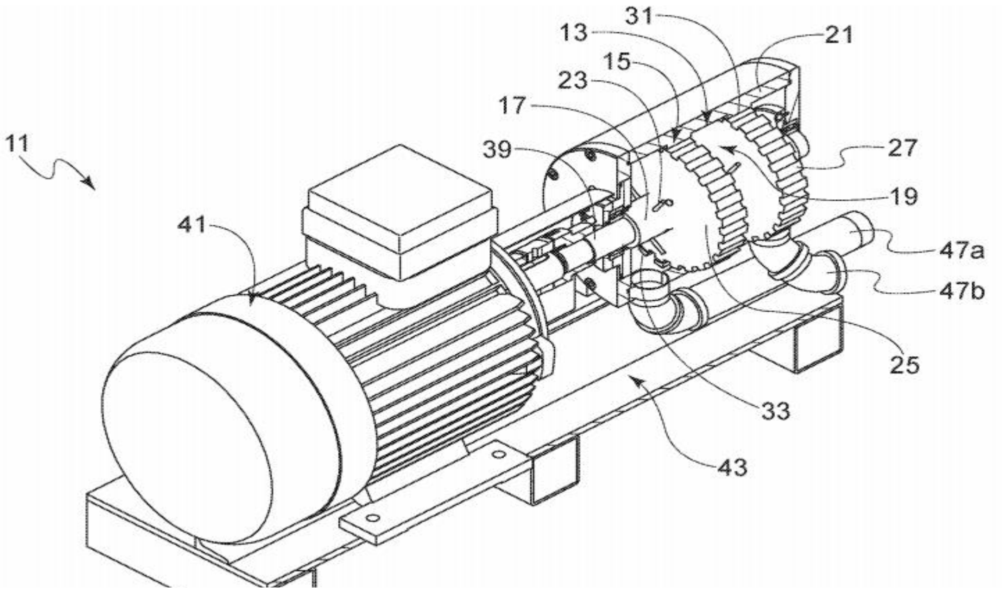


Figure 4-1 Schematic of a Turbex cavitational extractor

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able to increase phase contact surface (and hence matter and heat transfer) by taking advantage of turbulence and cavitation (Cavaglià, 2018). This causes enhanced extraction yields when compared to other most common extractors.

The functional principle of the Turbex is the presence of stages of high turbulence and high shear stress. TURBEX is always composed by (at least) one high turbulence mixing stage and (at least) one high shear stress stage. Stages are composed by a couple of rotor and stator. The high turbulence mixing chamber is composed by at least one radial pin intergal with the rotor, on a plance perpendicualr to the rotor's axis of rotation. The pins cross section is circular. The high shear stress instead are equipped with radial teeth on their stator, likewise provided with radial teeth: those radial teeth have a specific parabolic profile. The rotational speed of the equipment can ranghe from 500 do 5000 rpm.

The solid matrix is fed to the equipment with an hopper, whereas the liquid phase is withdrawn from a port which is equipped with a grid which has the function of preventing the solid phase to be withdrawn together with the liquid.

Mass transfer phenomena are governed by three main quantities, upon which depends the magnitude of the transfer: mass transfer coefficient, exchange area and driving force; the TURBEX extractor is able to influence all of these parametres:

- The phenomena increasing the exchage area are the high shear stress and the controlled cavitation. The high shear stress contributes to the generation of interfacial area, whereas the cavitation, by creating bubbles that by imploding generate pressures in the order of 1000/2000 bars. This results in a stress imposed on the solid matrix, which results in its rupture, with increase the number and the size of the porosities which are in contact with the extracting phase
- The phenomena influencing the exchange coefficient are the high turbulence and, again, the controlled cavitation. It is well known the turbulence has a positive influence on the mass transfer coefficients, and the Turbex is able to realise values of the Reynolds number of over 500.000. The beneficial role of the cavitation is clarified if one thinks about the Arrhenius equation: the exchange coefficient is a function which increases the temperature. The small bubble created inside the apparatus (0.05 to 5 microns) implode with times in the order or the millisecond or less: this causes the local increase of the temperature up to 1000-2000 K.
- The counter-current modality, which is the preferred configuration of the equipment, ensures high values for the driving force (i.e. the difference of concentration of the compunds in the solid phase and the extractant)

The extraction yield that TURBEX has been shown to be able to reach make this equipment extremely interesting for an industrial process; even more so when we take into account the low residence times required, which is in the order of seconds/minutes. Several experiments have been conducted to evaluate its extraction ability, particularly with vegetable matrixes; the yields in all cases were higher than those obtainable with traditional extraction equipment. The L/S ratio required to achieve the desired yields was also smaller than in conventional extractors, which results in a better economicity for the plant, by lowering the quantity of solvents that must be treated for recycling purposes.

1.9 Solid-liquid extraction Turbex performance

In this chapter is presented the result of the test of the Turbex in the extraction of polyphenols. The yield is presented alongside the one obtained during the laboratory experiments (single stage batch extraction) and with traditional equipment. Two tests have been conducted on the extracting ability of the Turbex with regard to polyphenols. In both cases, the rotational speed off the Turbex was set at 900 RPM, and the L/S nominal ratio was kept at 10. The first extractant was a ethanolic solution (60/40 ethanol water), whereas for the second test pure water was used. In each case, the test was conducted a room temperature.

EtOH-Water 60/40 extraction		
Fresh BSG Total Phenolic content	4,765	mgGAE/g
Batch extraction	3,35%	
Total Phenolis-rich product mass yield (Turbex)	16,90%	
Total Phenolis-rich product mass yield (Conventional)	15,80%	
TPC (Batch extraction)	12,33	mgGAE/g
TPC (Turbex)	25,60	mgGAE/g
TPC (Conventional	17,59	mgGAE/g
Yield on BSG polyphenols (Batch extraction)	8,7%	
Yield on BSG polyphenols (Turbex)	90,8%	
Yield on BSG polyphenols (Conventional)	58,3%	

Water extraction		
Fresh BSG Total Phenolic content	4,765	mgGAE/g
Batch extraction	1,80%	
Total Phenolis-rich product mass yield (Turbex)	18,30%	
Total Phenolis-rich product mass yield (Conventional)	16,90%	
TPC (Batch extraction)	24,92	mgGAE/g
TPC (Turbex)	11,74	mgGAE/g
TPC (Conventional	12,63	mgGAE/g
Yield on BSG polyphenols (Batch extraction)	9,4%	
Yield on BSG polyphenols (Turbex)	45,1%	

Yield on BSG polyphenols (Conventional)	44,8%

From the evidence presented, it has been shown the higher potential for extraction of the equipment. With respect to the batch extraction we see an overall higher yield on polyphenols with respect to traditional means of extraction. The ethanol-water extraction appears to yield less product, but with a higher concentration and with a higher yield if considering only the polyphenols. This seems to indicate that the ethanolic solution is the most viable one, as it is more selective towards the polyphenols and gives a purer product than the extraction with pure water.

5. EVALUATION OF AVAILABLE EXPERIMENTAL RESULTS

1.10 Description of experiments done and of experimental results obtained

In this chapter are presented the experiments conducted to investigate the composition and granulometry of the initial matrix and the composition of the extracted fractions, and their relative results. After the presentation of the matherial and methods relative to each experiment,

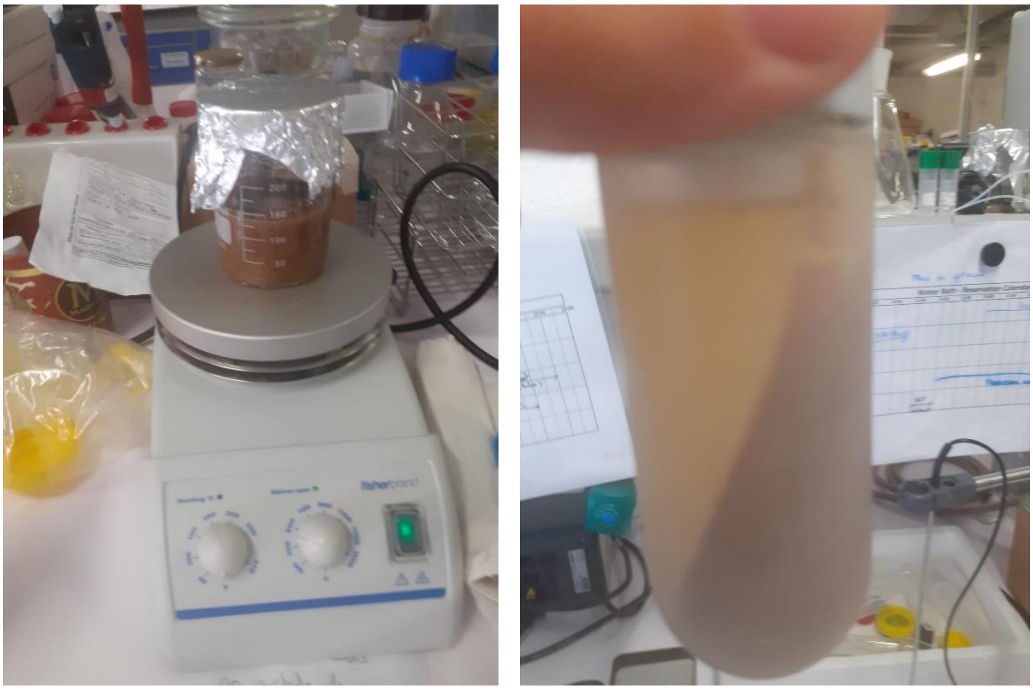
1.10.1 Polyphenols extraction

For the extraction of polyphenols, a sample of dry BSG (10% humidity) of 12.761 grams has been weighted on an electronical balance. The extracting medium chosen was an ethanolic aqueous solution at 60% v/v ethanol concentration. The L/S ration bewteen solvent and solid was 9.79 mL/mg, correspoinding to 125 mL of ethanolic solution. The solvent was prepared additioning 31.25 mL of distilled water to 93.75 mL of Ethanolic aqueous solution at 96% v/v concentration. The BSG and the ethanolic solution were put together in a beaker. The extraction was carried in 1 hour at room temperature, with moderate mixing provided by a magnetic stirrer. At the end of the extraction, the extract was filtrated with a funnel, through a piece of filtration fabric, and collected in a volumetri balloon. The filtrate has been centrifugated at 4°C and 15000 RPM x g in a Heraeus Biofuge Stratos centrifuge, to cause the sedimentation of the polyphenolic fraction, which was then separated from the liquid fraction by means of a filtration. The BSG recovered from the first extraction was then extracted again with pure water. 125 mL of distilled water were added to the BSG and the extraction was carried out in a beaker for 1 hour, at room temperature and with moderate agitation provided by a magnetic stirrer. Once again, the extract was separated from the residue by means of a filtration, and centrifugated at 4°C and 15000 RPM x g, followed by a filtration. The residue from the extraction was stored and underwent the polyphenols and AX estraction steps as explained in the next paragraph. The polyphenolic extracts obtained were freeze-dried and stored in a container to prevent the absorption of atmospheric humidity.

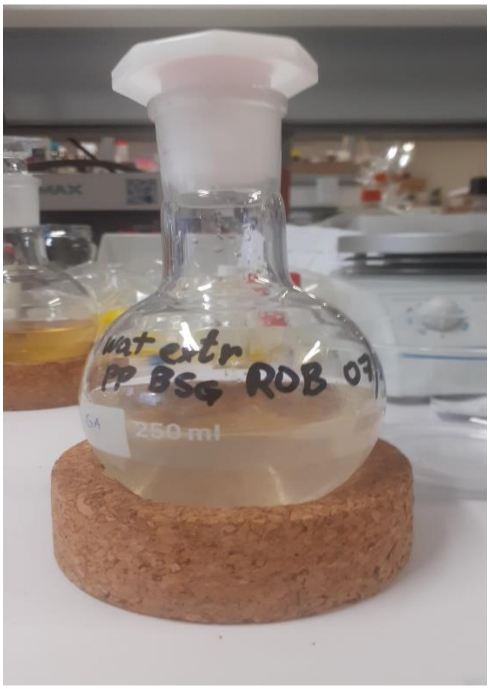
Material	Weight (mg)	Yield on BSG d.w.
BSG sample	12761	-
BSG d.w. (90%)	11485	-
Polyphenolic fraction from ethanolic extracion	384.32	3.35%
Polyphenolic fraction from water extracion	207.3	1.80%

Table 5-1

Figure 5-1 a) Hydro-alcoholic extraction set-up. b) Extract sample after centrifugation. c) Hydro-alcoholic extraction filtrate. d) Water extraction filtrate



a) b)



c)

d)

1.10.2 Protein and arabino-xylane extractions

The BSG recovered from the two stages of polyphenols extraction were submitted of the proteins and AX extraction steps. The BSG was mixed with 100 mL of alkaline solution. This solution was prepared mixing 100 mL of distillate water with 400 mg of NaOH, resulting in a solution of 0.1 M concentration. 95,65 mg of sodium meta-bisulphite $Na_2S_2O_5$ as anti-oxydant were also added. The extraction step was carried out in 24 hours at room temperature, with moderate mixing provided by a magnetic stirrer. When the extraction was completed, a first acidification with a saturated citric acid aqueous solution was carried out to cause the protein precipitation, which happens when the isoelectric point of the proteins is reached. The precipitation manifets itself visually with a sharp change in colour. The acid was added until a reading with a pH-meter confirmed a pH lower than 3. After the precipitation, the solution was filtrated to separate the BSG residue from the extract. The extract was centrifugated at 4°C and 15000 RPM x g to cause the sedimentation of the protein fraction. The protein fraction was separated from the solvent by means of filtration, freeze-dried and stored. The liquid extract, after separation from the protein fraction, was measured to be 110 mL, to which 295 mL of an aqueous solution of ethanol at 96% v/v concentration was added, resulting in a solution with about a 70% v/v ethanol concentration. This step is carried out in 2 hours at room temperature, with moderate mixing. After completion, an aqueous solution of HCl 36% v/v was gradually added until a pH below 2 was reached, which was measured with a pH-meter. This step induces the precipitation of the AX fraction, which is then separated from the supernatant and washed with distilled water. Upon completion of the washing, the AX were recovered by means of a centrifugation at 4°C and 15000 RPM x g and a filtration. The AX fraction obtained was collected, freeze-dried, and stored under vacuum to prevent hydration. The BSG residue from the first alkaline extraction was submitted to a second alkaline extraction step, using 100 mL of 0.5 M NaOH aqueous solution, together with 95.74 mg of sodium meta-bisulphite. The precitiptation and the recovery of the protein fraction followed the same steps as in the first alkaline extraction. The liquid extract was measured to be 92 mL, to which 250 mL of 96% v/v ethanolic solution was added to obtain a concentration of ethanol of around 70% v/v. The addition of the 36% v/v HCl solution until the reaching of a pHlower than 2 caused the precipitation of the secondo AX fraction, which was recovered, stored and treated the same way as the first AX fraction. The BSG residue recovered from the second filtration was submitted to a third alkaline extraction, with same conditions as for the previous two extraction, except that the molarity of the alkaline solution this time was 4 M. In this case the sodium bi-sulphite added was 94.97 mg. The liquid recovered after the protein fraction precipitation amounted to 214 mL, to which 575 mL of 96% v/v ethanolic solution was added, resulting in a final ethanol concentration of around 70% v/v. The steps in this extraction are qualitatively the same as in the first two extractions. The process so far thus yielded 3 protein-rich fraction and 3 AX-rich fractions. The BSG residue from the 4 M alkaline extraction was submitted to a final extraction with 75 mL of distilled water. This extraction was conducted for 2 hours at room temperature. After the completion of the extraction, 250 mL of ethanolic solution 96%v/v were added to cause the precipitation of a fourth and final AX fraction, which again was recovered with the same modality as for the previous AX fractions.

Material		mass yields on dry BSG %
BSG		-
0,1 M		
	PP1	14,69
	AX1	2,10
0,5 M		
	PP2	15,47
	AX2	7,82
4 M		
	PP3	5,90
	AX3	8,83
	AX4	3,50
Final residue		19,47

Table 5-2

Material	AX in extractsa as % of AX in BSG

BSG		-
0,1 M		
	PP1	1%
	AX1	18%
0,5 M		
	PP2	6%
	AX2	24%
4 M		
	PP3	0%
	AX3	17%
	AX4	15%
Final r		15%

Table 5-3

Material		monosaccharide molar % on total monosaccharide									
		ARA		XYL		MAN		GAL		GLC	
		S1	S2	S1	S2	S1	S2	S1	S2	S1	S2
BSG		26%	29%	47%	39%	2%	1%	2%	2%	23%	29%
0,1 M											
	PP1	34%	36%	21%	18%	0%	0%	23%	21%	22%	26%
	AX1	43%	43%	50%	51%	0%	0%	2%	2%	5%	4%
0,5 M											
	PP2	21%	18%	47%	50%	0%	0%	2%	1%	30%	31%
	AX2	30%	27%	54%	53%	0%	2%	0%	3%	16%	15%
4 M											
	PP3	10%	9%	54%	52%	1%	1%	1%	1%	34%	36%
	AX3	30%	27%	54%	53%	0%	2%	0%	3%	16%	15%
	AX4	26%	34%	55%	55%	0%	0%	3%	0%	16%	10%
Final residue		15%	15%	21%	21%	1%	1%	1%	1%	62%	62%

Table 5-4

1.10.3 Sugar analysis: description of the steps and theoretical background

The sugar analysis, based on acidic hydrolysis, is a technique used to assess the relative massic proportions in which the various monosaccharides are present in the analysed carbohydrates. It is one of the most common analysis techniques when dealing with carbohydrates. However, it fails to give information about the polysaccharides' structures and bonds.

This analysis was carried out in the laboratories of Builidng 29, at the Universidade de Aveiro. The analysis was carried out following the protocol in force in said department and here descripted.

Polysaccharides are polymers consisting of more than two monosaccharides linked covalently by glycosidic linkages. (Muhamad, et al., 2017)

The analysis is conceptually subdivided in two phases: in the first the polysaccharides are "broken" into the elementary sugars by which they are composed; said sugars are then converted in alditol acetates that can be volatized in an appropriate eluent; the second phase is the analysis of the alditol acetates by means of a gas-chromatography.

The first step is the acid hydrolysis reaction, conducted in presence of strong acid and heat; this causes the glycosidic linkage between two adjacent linked monosaccharides to be cleaved (Cui, 2005), "freeing" the different monosaccharides.

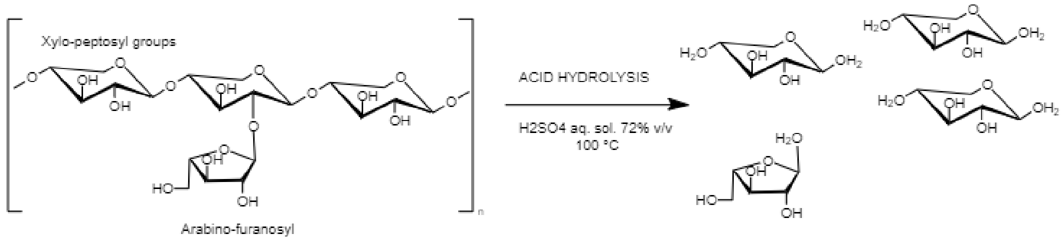


Figura 5-2: Glicosidic linkage hydrolysis of AX

A sample between 1-2 mg is weighted on an electronic scale. The hydrolysis agent in our case was sulphuric acid (72% aqueous solution). 200 µL of sulphuric acid are added to the sample and mixed: then, 2,2 mL of distilled water are added. The heat is provided by a heating bloc, which heats the tubes at 100°C for 2,5 hours.

This time has been estabilshed considering that, during hydrolysis, the monosaccharides can undergo degradation by the acid; however, not all glycosidic linkages are broken at the same rate, and so the time must be sufficient for all the linkages to be hydrolyzed. By doing analysis on the same sample subjected to different times of heating, we would that the amounts of monosaccharides measured for different heating times would reac. a maximum; by these considerations, we infer that the time must be a compromise between the two factors: not too short (so we can "free" al the monosaccharides) but not too long (as to avoid degradation) (Cui, 2005).

After that, the tubes are cooled in an ice bath. The next step is to add 200 µL of internal standard; in this case, a solution of water and 2-deoxyglucose in concentration 1 mg/mL.

The internal standard is necessary as a reference to integrate the gas-chromatography results. Knowing the area of the peak relative to the internal standard, the areas of the peaks relative to the monosaccharides, and the quantity of internal standard, we obtain the quantities of the various monosaccharides.

The next step consists in transferring of 1 mL of the sample in a culture tube, and neutralizing it with 200 µL of ammonia 25% aqueous solution; it's important to confirm that the pH is above 7. That's because the next step is the reduction, where the reducing agent is $NaBH_4$ (sodium borohydride). But sodium borohydride in an acid solution would decompose (Banfi, et al., 2001), so we neutralize it. Sodium borohydride reduces the aldehydic group of the monosaccharides, converting them in alditols (Cui, 2005). $NaBH_4$ is put into a solution of NH_3 3M, in concentration 150 mg/mL. 100 µL of this solution is added to the sample in the culture tube. The tubes are then put in an ice bath, where 2 x 50 µL of glacial acetic acid are added, to decompose the excess sodium borohydride left from the reduction step.

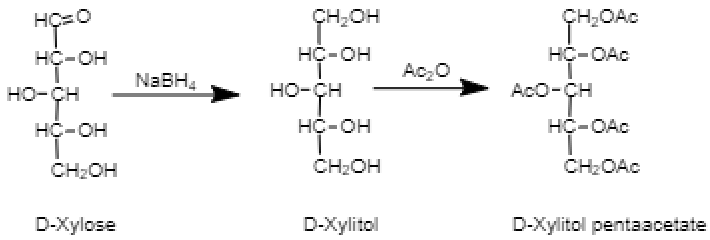


Figure 5-3

At this point the aldehydes have been converted to alditols, so now we proceed to acetylate them. We transfer 300 µL of sample in SOVIREL tubes with inert teflon caps. The tubes are now put into an ice bath, and 450 µL of 1-methylimidazole are added, along with 1 mL of acetic anhydride. The acetic anhydride is the reagent, whereas 1-methylimidazole is the catalyst. The ice bath is needed because of the exothermic nature of the acetylation reaction (Wachowiak & Connors, 1979). The prepared sample is then shaked and put onto a heating bloc at 30°C for 30 minutes to allow the acetylation reaction to take place.

After that, 3 mL of distilled water and 2.5 mL of dichloromethane are added to the sample. Dichloromethane is used for the liquid extraction of the monosaccharides from the aqueous phase; that's because dichloromethane is a good solvent for organic compunds and is hydrophobic (Rossberg, et al., 2006).

The samples are then centrifugated for 30 seconds at 3000 rpm, to cause the separation of the organic phase from the aqueous one. When this is done, the aqueous phase is removed by means of a pasteur pipette. To remove the un-desired components, another step of addition of distilled water and dichloromethane, followed by centrifugation and water removal is repeated as before. Finally a last washing similar to the last two, but conducted with only distilled water, is performed. The dichloromethane is evaporated in a *speedvac* (vacuum centrifuge). Two separate additions of 1mL anhydrous acetone, each one followed by evaporation in the speedvac, are performed.

The reactions phase are now concluded. The dry sample obtained is ready for analysis in a gas-chromatographe. The injection is performed using 2 µL of anhydrous acetone as solvent. The samples obtained are analysed in a Flame Ionisation DetectionGas Chromatograph (GC-FID); this tipe of equipment is able to provide a signal which can be correlated to the concentration of internal standard to infer the concentration of the various compunds in the sample. The compunds can be distinguished by their characteristic elution time. The fraction upon which this analysis was carried out were: the 3 protein fractions, the 4 AX fraction , the two polyphenolic fractions, the starting BSG and the finale residue from the final AX extraction with water. The analysis as descripted provided unsatisfactory results for the AX, especially the first fraction, because of their high purity; for this reason, for the AX fraction the analysis was repeated with a doubled concentration of internal standard with respect to what previously desccripted in this paragraph.

1.10.4 Methylation analysis

Methylation analysis is one of the most common methods of carbohydrate structural analysis. It is structured in two steps: chemical derivatization, followed by gas-liquid chromatograph-mass spectroscopy (Cui, 2005). In the derivatization step, we induce a methylation reaction of the carbohydrates with a methyl-group donor. The methyl group will react with the free hydroxyl-groups of the carbohydrates to form methyloxide groups. In a second time, a acid hydrolisis is performed, with the aim of breaking the glycosidic linkages between the monosaccharides. The hydrolysis leaves the methyloxide groups intact. Monomers are then reduced and acetylated to give volatile products. These volatile products can be then identified, along with their relative proportions, by means of gas-liquid chromatograph-mass spectroscopy (GC-MS). The substitution patterns on the monomers proviide information about which positions, in the monomers structures, presents a free hydroxyl group; from it, we can infer the structure of the carbohydrates and give average value for Branching Degree (BD) and Degree of polymerization (DP). To put it simply, the methoxyl groups function as “placeholders”, occupying the hydroxyl groups which are not “employed” by the molecule to form a linkage, and to preserve this information that would otherwise be lost during the acid hydrolisis. Linking the results obrained by the GC-MS with previous knowledge of the general structure of AX, it’s possible to have a good idea of their average structure.

The analysis starts with the weighting of 1-2 mg of sample, which is placed in a sovirel tube. 1 ml of anhydrous di-methyl sulphoxide is added as solvent. The tubes are then stirred overnight to provide an adequate solubilization of the sample. The solubilisation must be as complete as possbile, to avoid under-methylation that would hinder the quality of the analysis. The methylation reaction require an alkaline environment. This is realised with the addition of 40 mg of powdered NaOH, followed by a 30 minutes stirring. The methyl-group donor employed is CH3I. 80 microlitres are added, and the solution is vigorously stirred for 20 minutes. The solution is then neutralised with 1M HCl. Water is added to destroy the residual CH3I. After that, a liquid-liquid extraction of the methylated carbohydrates is performed, using 3 mL of dicholoromethane as solvent. The sample is then vigorously stirred to favor the contact between the aqueous and the organic phase, to cause the solvent to migrate towards the extract phase. The emulsion is then centrifuges for 30 s at 3000 rpm, to cause the separation of the two phases. The aqueous phase is then removed with a vacuum pipette. The organic phase is then washed with distillate water, and then the steps of stirring, centrifugating, and vacuum-separation of the two phases are repeated.

At this point, the methylation reaction is complete, and we can proceed with the hydrolysis. This is performed with the addition of 500 micro-litres of 2M TFA. The reaction takes place at 121°C and lasts for 1 hour, during which the solution is stirred 2 or 3 times. TFA is then evaporated in a speedvac.

The next steps are reduction and acetylation of the hydrolysates. The reduction of the monosaccharides to alditols is conducted in an alkaline environment; for this reasion, 300 micro-litres of 2M NH3 are added. The reductive agent employed is 20 mg of NaBD4. This compound introduces a deuterium atom onto che C1 position and facilitates the distinction between C1 and C6 carbons. The reaction is conducted at 30°C (provided by a heating block) for 1 hour. 100 micro-litres of acetic acid are then added to cause the conversione of excess NaBD4 in boric acid. This step must be carried out in an ice bath because of the exothermictiy of the reaction. 450 mL of 1-methylimidazole are added, as catalyst for the acetylation reaction.; 3mL of acetic andhydride are used as reagent. The solution is mixed and incubated at 30°C for 30 minutes.

What we obtained are called partially methylated alditol acetates (PMAA), that can be analysed with GC-MS. To do so, we perform another liquid-liquid extraction using distilled water and dichloromethane, not unlike previously described. After the same steps of washing and vacuum separation, we are left with an organic phase containing the PMAA compounds. The solvent is then evaporated in a speedvac; after the evaporation, 1 ml of anhydrous acetone is added and then evaporated in the speedvac. This step is repeated. We are left with a solid residue that can be analysed in a GC-MS apparatus.

1.10.5 BSG granulometry

The granulometric distribution of the brewers' spent grain was investigated. The mechanical apparatus used for this analysis consisted in a column of 4 forated sieves with different mesh sizes. A certain amount of material is firstly measured as well as the weight of each single sieve. The plates are stacked onto each other in a pile, with the finer mesh at the bottom, and a mesh size wich increased along the vertical axis for each plate. The measured amount of BSG is then placed in the top sieve, and the whole stack is inserted in a vibrating machine for a certain amount of time (in this case, 10 minutes). The vibration causes the particles of size less than that of the mesh to fall down, until they reach a rieve with a mesh finer that the particle's size. In this way, the original sample gets distributed along the sieve stack. Once the separation is complete, the amount from each sieve is weighted. By knowing the mass fraction that stopeed in each sieve, we able to determine the fraction of particles that have a size comprised between that of the mesh of the sieve from which the amount is collected, and the size of the mesh of the higher up sieve. The mesh employed during this analysis were 63, 90, 150 and 300 µm.

1.11Experimental results

1.11.1 BSG sugar analysis

The sugar analysis of the BSG has been done in two different ways: with and without hydrlosis. This was done as to highlight the content of cellulose. For the non-cellulosic analysis, the samples and internal standard quantity are reported in the following table:

Sample 1 mass	1.32	mg
Sample 2 mass	1.25	Mg
Internal standard mass	0.2122	mg
Internal standard volume	0.2	mL
Internal standard concentration	1.061	mg/mL

Table 5-5

The gas-chromatography of both samples led to the following areas, redeable in the chromatograph reported in Appendix A.

Sample 1	Areas, µV*s	Area monosaccharide/area internal standard
Arabinose area	144629.79	0.589244014
Xylose area	265830.09	1.083032681
Mannose area	13187.02	0.053725948
Galactose area	16386.78	0.066762263
Glucose area	152941.16	0.623105814
Internal standard area	245449.74	1
Sample 2	Areas, µV*s	Area monosaccharide/area internal standard
Arabinose area	1560971.1	0.622210325
Xylose area	210673.5	0.83975297
Mannose area	4077.87	0.01625481
Galactose area	13592.69	0.0541811
Glucose area	183517.5	0.731509167
Internal standard area	250875.1	1

Table 5-6

The results relative to the “cellulosic” BSG are as follows:

Sample 1 mass	2.49	Mg
Sample 2 mass	2.8	Mg
Internal standard mass	0.2122	Mg
Internal standard volume	0.2	mL
Internal standard concentration	1.061	mg/mL

Table 5-7

Sample 1	Areas, µV*s	Area monosaccharide/area internal standard
Arabinose area	235491.04	1.003451066
Xylose area	483173.97	2.058853004
Mannose area	12192.81	0.051954793
Galactose area	22224.03	0.094698833
Glucose area	394637.91	1.681591925
Internal standard area	234681.14	1
Sample 2	Areas, µV*s	Area monosaccharide/area internal standard
Arabinose area	285212.3	1.274150969
Xylose area	596764.6	2.66597321
Mannose area	14816.57	0.066191219
Galactose area	30367.45	0.135662877
Glucose area	536105	2.394983715
Internal standard area	223845	1

Table 5-8

1.11.2 Sugar analysis of polyphenols-rich products

Two different polyphenols extraction route have been tested: one using a 60/40 v/v ethanol-water solution as extracant, and one using pure water as extractant.

Water-ethanol extraced sample mass	1.18	Mg
Water extracted sample mass	1.05	Mg
Internal standard mass	0.2122	Mg
Internal standard volume	0.2	mL
Internal standard concentration	1.061	mg/mL

Table 5-9

Hydro-alcoholic extraction	Areas, µV*s	Area monosaccharide/area internal standard
Arabinose area	13155.68	0.051916039
Xylose area	4412.71	0.017413803
Mannose area	0	0
Galactose area	0	0

Glucose area	219567.82	0.866476798
Internal standard area	253403	1
Pure water extraction	Areas, µV*s	Area monosaccharide/area internal standard
Arabinose area	61547.32	0.262197318
Xylose area	57470.27	0.244828705
Mannose area	0	0
Galactose area	15073.72	0.064215452
Glucose area	189201.7	0.806016785
Internal standard area	234736.7	1

Table 5-10

1.11.3 Sugar analysis of arabinoxylanes-rich products

The various products are labelled as follows: AX1 refers to the product obtained in the first alkaline extraction, AX2 to the product obrained in the second alkaline extraction, AX3 to the prouct obtained in the third alkaline extraction, and AX4 to the product obtained in the last extraction, performed with pure water.

AX1		
Sample 1 mass	1.09	mg
Sample 2 mass	1.47	Mg
Internal standard mass	0.2122	mg
Internal standard volume	0.2	Ml
Internal standard concentration	1.061	mg/mL

Table 5-11

AX1		
Sample 1	Areas, µV*s	Area monosaccharide/area internal standard
Arabinose area	442427.55	2.136261196
Xylose area	521805.05	2.519536358
Mannose area	0	0
Galactose area	30464.02	0.14709556
Glucose area	59057.54	0.285159408
Internal standard area	207103.6	1
Sample 2	Areas, µV*s	Area monosaccharide/area internal standard
Arabinose area	526334	2.773371176
Xylose area	616908	2.519536358
Mannose area	0	0
Galactose area	34265	0.14709556
Glucose area	57590	0.285159408
Internal standard area	189781	1

Table 5-12

AX2

Sample 1 mass	1.78	mg
Sample 2 mass	2.07	Mg
Internal standard mass	0.4214	mg
Internal standard volume	0.2	Ml
Internal standard concentration	2.107	mg/mL

Table 5-13

AX2		
Sample 1	Areas, µV*s	Area monosaccharide/area internal standard
Arabinose area	682019.52	1.245239096
Xylose area	1118300.37	2.041805698
Mannose area	0	0
Galactose area	51352.21	0.093759457
Glucose area	279410.66	0.510151202
Internal standard area	547701.66	1
Sample 2	Areas, µV*s	Area monosaccharide/area internal standard
Arabinose area	855446.64	1.379943523
Xylose area	1370156.8	2.210236016
Mannose area	0	0
Galactose area	63641.38	0.102661588
Glucose area	368456.04	0.59436615
Internal standard area	619914.24	1

Table 5-14

AX3		
Sample 1 mass	0.9	mg
Sample 2 mass	2.08	Mg
Internal standard mass 1	0.2122	mg
Internal standard mass 2	0.4214	
Internal standard volume	0.2	Ml
Internal standard concentration 1	1.061	mg/mL
Internal standard concentration 1	2.107	mg/mL

Table 5-15

AX3		
Sample 1	Areas, µV*s	Area monosaccharide/area internal standard 1
Arabinose area	229543.74	0.824320676
Xylose area	410003.25	1.472373659
Mannose area	0	0
Galactose area	4517.24	0.016221982
Glucose area	142823.33	0.512896688
Internal standard area	278464.13	1
Sample 2	Areas, µV*s	Area monosaccharide/area internal standard 2
Arabinose area	434223.37	0.81913334

Xylose area	843370.08	1.590961239
Mannose area	30531	0.057594689
Galactose area	55868.4	0.105391999
Glucose area	294038.2	0.554683395
Internal standard area	530100.96	1

Table 5-16

AX4		
Sample 1 mass	1.25	mg
Sample 2 mass	2.28	Mg
Internal standard mass	0.4212	mg
Internal standard volume	0.2	Ml
Internal standard concentration	2.106	mg/mL

Table 5-17

AX4		
Sample 1	Areas, µV*s	Area monosaccharide/area internal standard
Arabinose area	247804.47	0.556119477
Xylose area	519181.78	1.165140807
Mannose area	0	0
Galactose area	28554.51	0.064081649
Glucose area	174636.24	0.391916314
Internal standard area	445595.74	1
Sample 2	Areas, µV*s	Area monosaccharide/area internal standard
Arabinose area	318102.2	1.780001648
Xylose area	507268.2	2.838516072
Mannose area	0	0
Galactose area	4574.13	0.025595419
Glucose area	116604.3	0.652481832
Internal standard area	178708.9	1

Table 5-18

1.11.4 Sugar analysis of protein-rich products

Three protein-rich products are obtained in three sequential alkaline extractions: the products are thus labelled as PP1, PP2 and PP3, where the number indicates the extraction from which the product has been obtained.

PP1		
Sample 1 mass	1.78	mg
Sample 2 mass	1.17	Mg
Internal standard mass	0.2122	mg
Internal standard volume	0.2	Ml

Internal standard concentration	1.061	mg/mL

Table 5-19

PP1		
Sample 1	Areas, µV*s	Area monosaccharide/area internal standard
Arabinose area	26522.99	0.118347581
Xylose area	16440.85	0.073360312
Mannose area	0	0
Galactose area	22167.35	0.098923387
Glucose area	21249.9	0.094818656
Internal standard area	224110.96	1
Sample 2	Areas, µV*s	Area monosaccharide/area internal standard
Arabinose area	13898.65	0.054767387
Xylose area	6842.32	0.026962042
Mannose area	0	0
Galactose area	9958.8	0.039242477
Glucose area	11990.54	0.047248513
Internal standard area	253776.03	1

Table 5-20

PP2		
Sample 1 mass	0.9	mg
Sample 2 mass	1.79	Mg
Internal standard mass	0.2122	mg
Internal standard volume	0.2	Ml
Internal standard concentration	1.061	mg/mL

Table 5-21

PP2		
Sample 1	Areas, µV*s	Area monosaccharide/area internal standard
Arabinose area	37235.97	0.142049892
Xylose area	84901.43	0.323886794
Mannose area	0	0
Galactose area	45177.25	0.017232662
Glucose area	64370.22	0.245563169
Internal standard area	262133.04	1
Sample 2	Areas, µV*s	Area monosaccharide/area internal standard
Arabinose area	65397.1	0.229343984
Xylose area	183531.1	0.643633443
Mannose area	0	0
Galactose area	2707.68	0.009495683
Glucose area	135606.2	0.47556349
Internal standard area	285148.5	1

Table 5-22

PP3		
Sample 1 mass	1.52	mg
Sample 2 mass	2.12	Mg
Internal standard mass	0.2122	mg
Internal standard volume	0.2	Ml
Internal standard concentration	1.061	mg/mL

Table 5-23

PP3		
Sample 1	Areas, µV*s	Area monosaccharide/area internal standard
Arabinose area	55712.43	0.175084652
Xylose area	297820.42	0.935945257
Mannose area	8708	0.027366194
Galactose area	4602.03	0.014452568
Glucose area	230217.96	0.723494405
Internal standard area	318202.82	1
Sample 2	Areas, µV*s	Area monosaccharide/area internal standard
Arabinose area	78902.38	0.326127052
Xylose area	434221.13	1.794765353
Mannose area	12290.39	0.050799845
Galactose area	6991.65	0.028898573
Glucose area	363657.02	1.503102846
Internal standard area	241937.55	1

Table 5-24

1.11.5 Sugar analysis of the final residue

The final residue is the leftover solid from the extraction process, i.e. the leftover solid from the last, water-based extraction of the AX4 product.

Final residue		
Sample 1 mass	1.59	mg
Sample 2 mass	2.38	Mg
Internal standard mass	0.2122	mg
Internal standard volume	0.2	Ml
Internal standard concentration	1.061	mg/mL

Table 5-25

Final residue		
Sample 1	Areas, µV*s	Area monosaccharide/area internal standard
Arabinose area	200090.3	0.994746974
Xylose area	283250.18	1.408175506
Mannose area	13971.09	0.069457138
Galactose area	13573.28	0.067479429
Glucose area	974304.38	4.843744719

Internal standard area	201146.93	1
Sample 2	Areas, µV*s	Area monosaccharide/area internal standard
Arabinose area	219816.3	1.008331002
Xylose area	301380	1.382475739
Mannose area	218000.2	0.063538892
Galactose area	13851.49	0.073377508
Glucose area	15996.31	4.887336515
Internal standard area	1065440	1

Table 5-26

1.11.6 Elementary analysis

An elemental composition anaylis has been performed on the BSG and the various products: the nitrogen content is representative of the protein content, as will be explained in the section 5.3, relative to the interpreation of the experimental results. Also Carbon, Hydrogen and Sulphur content was measured. For each analysed material, two samples were tested. The average content of each element was averaged in the two samples and the standard deviation was calculated.

	Sample Mass mg	%C	%H	%N	%S
BSG sample A	1,919	46,7780	5,779	3,572	0
BSG sample B	1,174	46,6890	6,129	3,879	0
Final residue sample A	2,106	42,0610	5,329	0,569	0
Final residue B	1,816	42,0620	5,49	0,355	0
PP1 sample A	1,803	54,2110	6,666	9,85	1,298
PP1 sample B	1,152	54,5200	6,933	10,109	1,204
PP2 sample A	1,534	35,6990	4,274	1,686	0
PP2 sample B	1,372	35,7350	4,355	2,047	0
PP3 sample A	0,969	40,2390	40,239	0,878	0
PP3 sample B	0,752	39,6830	39,683	0,874	0
Water extracted polyphenolic product sample A (batch)	0,959	33,016	5,756	2,012	0
Water extracted polyphenolic product sample B (batch)	1,027	33,319	5,531	2,029	0
EtOH-water extracted polyphenolic product sample A (batch)	1,546	40,965	6,441	3,583	0
EtOH-water extracted polyphenolic product sample B (batch)	2,229	40,917	6,71	3,67	0
Water extracted polyphenolic product sample A (with Turbex)	1,66	44,952	6,317	4,095	0,448
Water extracted polyphenolic product sample B (with Turbex)	1,925	44,859	6,078	4,054	0,288

EtOH-water extracted polyphenolic product sample A (with Turbex)	1,492	47,649	6,596	5,374	0,643
EtOH-water extracted polyphenolic product sample B (with Turbex)	1,76	47,531	6,416	5,333	0,51

Table 5-27

	%C	st. dev.	%H	st.dev.	%N	st.dev.	%S	st. dev.
BSG (average)	46,7335	0,0445	5,9540	0,1750	3,7255	0,1535	0,0000	-
Final residue (average)	42,0615	0,0005	5,4095	0,0805	0,4620	0,1070	0,0000	-
pp1 (average)	54,3655	0,1545	6,7995	0,1335	9,9795	0,1295	1,2510	0,0470
pp2 (average)	35,7170	0,0180	4,3145	0,0405	1,8665	0,1805	0,0000	-
pp3 (average)	39,9610	0,2780	39,9610	0,2780	0,8760	0,0020	0,0000	-
Water polyphenolic extract	33,1675	0,1515	5,6435	0,1125	2,0205	0,0085	0	-
Ethanolic polyphenolic extract	40,941	0,0240	6,5755	0,1345	3,6265	0,0435	0	-
Turbex water polyphenolic extract	44,9055	0,0465	6,1975	0,1195	4,0745	0,0205	0,368	0,0800
Turbex ethanolic polyphenolic extract	47,59	0,0590	6,506	0,0900	5,3535	0,0205	0,5765	0,0665

Table 5-28

1.11.7 Folin-Ciocalteu analysis of the polyphenols-rich products

The phenolic content of the two polyphenols-rich products obtained (water extraction and ethanol-water extraction) was also carried out. For each extraction, two samples from the product were analysed. Each sample was tested three times. Seven concentration of the Folin-Ciocalteu reagent were tested, ranging from 0 to 100 µg/mL, to obtain the calibration data.

Calibration concentrations	
5	ug/ml
10	ug/ml
25	ug/ml
50	ug/ml
75	ug/ml
100	ug/ml

Table 5-29

Samples masses		
EtOH-water extraction sample A	1,68	mg
EtOH-water extraction sample B	3,77	mg
Pure water extraction sample A	1,11	mg
Pure water extraction sample B	1,313	mg

Table 5-30

For the ethanol-water extraction, the following values of absorbance were measured for the calibarion solutions and the samples.

RESULTS (absorbance) for EtOH-water extraction								
0 ug/ml	5 ug/ml	10 ug/ml	25 ug/ml	50 ug/ml	75 ug/ml	100 ug/ml	A	B
0,059	0,059	0,089	0,145	0,25	0,346	0,82	0,126	0,292
0,045	0,062	0,087	0,147	0,246	0,38	0,518	0,126	0,186
0,047	0,068	0,083	0,131	0,231	0,386	0,503	0,135	0,286

Table 5-31

For the pure water extraction, the following values of absorbance were measured for the calibarion solutions and the samples.

RESULTS (absorbance) for pure water extraction								
0 ug/ml	5 ug/ml	10 ug/ml	25 ug/ml	50 ug/ml	75 ug/ml	100 ug/ml	A	B
0,044	0,063	0,123	0,119	0,153	0,325	0,204	0,104	0,108
0,0337	0,061	0,075	0,118	0,223	0,307	0,412	0,059	0,133
0,039	0,049	0,075	0,119	0,216	0,328	0,408	0,108	0,133

Table 5-32

1.12Interpretation of experimental results

1.12.1 Sugar analysis

As explained in section 5.3, the data relative to the areas in µV*s for the samples, and their ratio with respect to the area relative to the Interal standard, allow for the calculation of the concentration of the various monosaccharide species in the samples, using the folowing relation:

$$Monosaccharides\ mass = \frac{Area\ of\ monosaccharide}{Area\ of\ internal\ standard} mass\ of\ interal\ standard$$

For each analysed material, the total mass of five monosaccharide species (Arabinose, Xylose, Manose, Galactose and Glucose) has been measured. From the masse values, by simple moltiplication by the monosaccharide's molecular mass, the number of moles has been calculated, as well as the molar percentage of each monosaccharide on the total sugars content. Almost every material was tested twice, allowing for the calculation of a mean content value and a standard deviation.

“Cellulosic” BSG				
SAMPLE 1				
	area monosaccharide/ area int. St.	monosaccharides mass micrograms	monosaccharide s micromoles	monosacchari des molar %
ARA	1,003451066	212,9323161	1,418319564	22%
XYL	2,058853004	436,8886074	2,910068656	45%
MAN	0,051954793	11,02480703	0,061195892	1%
GAL	0,094698833	20,09509229	0,111540255	2%
GLC	1,681591925	356,8338065	1,980649459	31%
TOTAL SUGARS		1037,774629	6,481773825	
SAMPLE 2				
	area monosaccharide/ area int. St.	monosaccharides mass micrograms	monosaccharide s micromoles	monosacchari des molar %
ARA	1,274150969	270,3748357	1,800938091	21%
XYL	2,66597321	565,7195153	3,768197664	44%
MAN	0,066191219	14,04577657	0,077964523	1%
GAL	0,135662877	28,78766258	0,159789424	2%
GLC	2,394983715	508,2155444	2,820912214	33%
TOTAL SUGARS		1387,143334	8,627801915	

Table 5-33

“Non cellulosic” BSG				
SAMPLE 1				
	area monosaccharide/ area int. St.	monosaccharides mass micrograms	monosaccharide s micromoles	monosacchari des molar %
ARA	0,589244014	125,0375797	0,832862051	26%
XYL	1,083032681	229,8195349	1,530803537	47%
MAN	0,053725948	11,40064619	0,063282079	2%
GAL	0,066762263	14,16695213	0,078635391	2%
GLC	0,623105814	132,2230537	0,733920147	23%
TOTAL SUGARS		512,6477667	3,239503205	100%
SAMPLE 2				
	area monosaccharide/ area int. St.	monosaccharides mass micrograms	monosaccharide s micromoles	monosacchari des molar %
ARA	0,622210325	132,033031	0,87945801	29%

XYL	0,839754297	178,1958618	1,186943727	39%
MAN	0,016254581	3,449222123	0,019145752	1%
GAL	0,0541811	11,49722945	0,063816771	2%
GLC	0,731509167	155,2262452	0,86160216	29%
TOTAL SUGARS		480,4015896	3,010966421	100%

Table 5-34

The diffence between the “cellulosic” BSG results and the “non cellulosic” BSG lies in the fact that in the latter the pre-hydrolisis step has been skipped. This explains the difference in the total sugar content in the two cases. By skipping the pre-hydrolisis step, the cellulose didn’t solubilize in the analyzed solvent. By averaging the toal sugar content in the two cases, we can surmise how much of the various monosaccharide species were present as monomers in the cellulose, by simply taking the difference between the two average sugar content.

Cellulosic BSG sample A mass	2.49	mg
Cellulosic BSG sample B mass	2.8	mg
Cellulosic BSG sample A total sugars	1.038	mg
Cellulosic BSG sample B total sugars	1.397	mg
Cellulosic BSG sample A specific sugar content	0.417	mg sugars/mg BSG
Cellulosic BSG sample B specific sugar content	0.499	mg sugars/mg BSG
Average sugar specific content in cellulosic BSG	0.458	mg sugars/mg BSG
Non-Cellulosic BSG sample A mass	1.32	mg
Non-Cellulosic BSG sample B mass	1.25	mg
Non-Cellulosic BSG sample A total sugars	0.513	mg
Non-Cellulosic BSG sample B total sugars	0.480	mg
Non-Cellulosic BSG sample A specific sugar content	0.387	mg sugars/mg BSG
Non-Cellulosic BSG sample B specific sugar content	0.384	mg sugars/mg BSG
Average sugar specific content in non-cellulosic BSG	0.385	mg sugars/mg BSG
Cellulose specific content in BSG	0.073	mg sugars/mg BSG

Table 5-35

In the following tables are presented the sugar analysis results relative to the four AX and the three protein products, as well as the final residue. Each product was analysed in duplicates.

AX1
SAMPLE 1

	area monosaccharide/ area int. St.	monosaccharides mass micrograms	monosaccharides micromoles	monosaccharides molar %
ARA	2,136261996	453,3147956	3,019481753	43%
XYL	2,519536358	534,6456151	3,561217712	50%
MAN	0	0	0	0%
GAL	0,14709556	31,21367781	0,173255316	2%
GLC	0,285159408	60,51082641	0,335872704	5%
TOTAL SUGARS		1079,684915	7,089827486	100%
SAMPLE 2				

	area monosaccharide/ area int. St.	monosaccharides mass micrograms	monosaccharides micromoles	monosaccharides molar %
ARA	2,773371176	588,5093636	3,919998425	43%
XYL	3,250621215	689,7818218	4,594563523	51%
MAN	0	0	0	0%
GAL	0,180548707	38,31243563	0,212657835	2%
GLC	0,305352126	64,79572106	0,359656533	4%
Total sugars		1381,399342	9,086876317	100%

Table 5-36

Sugars on dry weight of sample, AX1		
	SAMPLE 1	SAMPLE 2
ARA	42%	40%
XYL	49%	47%
MAN	0%	0%
GAL	3%	3%
GLC	6%	4%
TOT	99%	94%

Table 5-37

AX2
SAMPLE 1

	area monosaccharide/ area int. St.	monosaccharides mass micrograms	monosaccharides micrmoles	monosaccharides molar %
ARA	1,245239096	524,7437551	3,495262473	33%
XYL	2,041805698	860,416921	5,731145814	54%

MAN	0	0	0	0%
GAL	0,093759457	39,510235	0,219306367	2%
GLC	0,510151202	214,9777164	1,193259971	11%
TOTAL SUGARS		1639,648627	10,63897462	100%
SAMPLE 2				

	area monosaccharide/ area int. St.	monosaccharides mass	monosaccharides moles	monosaccharides molar %
ARA	1,379943523	581,5082004	3,87336442	33%
XYL	2,210236016	931,3934571	6,203912989	53%
MAN	0	0	0	0%
GAL	0,102661588	43,26159298	0,240128735	2%
GLC	0,59436615	250,4658955	1,390241427	12%
TOTAL SUGARS		1806,629146	11,70764757	100%

Table 5-38

Sugars on dry weight of sample, AX2		
	SAMPLE 1	SAMPLE 2
ARA	29%	28%
XYL	48%	45%
MAN	0%	0%
GAL	2%	2%
GLC	12%	12%
TOT	92%	87%

Table 5-39

AX3
SAMPLE 1

	area monosaccharide/ area int. St.	monosaccharides mass micrograms	monosaccharides micromoles	monosaccharides molar %
ARA	0,824320676	174,9208475	1,165129205	30%
XYL	1,472373659	312,4376904	2,081114304	54%
MAN	0	0	0	0%
GAL	0,016221982	3,442304501	0,01910693	0%
GLC	0,512896688	108,8366772	0,604111219	16%
TOTAL SUGARS		599,6375197	3,869461658	100%
SAMPLE 2				

	area monosaccharide/ area int. St.	monosaccharides mass	monosaccharides moles	monosaccharides molar %
ARA	0,81913334	345,1827896	2,299225935	27%
XYL	1,590961239	670,431066	4,465670193	53%
MAN	0,057594689	24,270402	0,13471881	2%
GAL	0,105391999	44,4121885	0,246515256	3%
GLC	0,554683395	233,7435825	1,297422194	15%
TOTAL SUGARS		1318,040029	8,443552388	100%

Table 5-40

Sugars on dry weight of sample, AX3		
	SAMPLE 1	SAMPLE 2
ARA	19%	17%
XYL	35%	32%
MAN	0%	1%
GAL	0%	2%
GLC	12%	11%
TOT	67%	63%

Table 5-41

AX4				
SAMPLE 1				
	area monosaccharide/ area int. St.	monosaccharides mass micrograms	monosaccharides micromoles	monosaccharides molar %
ARA	0,556119477	234,2375238	1,560231292	26%
XYL	1,165140807	490,7573078	3,268882354	55%
MAN	0	0	0	0%
GAL	0,064081649	26,99119074	0,149817888	3%
GLC	0,391916314	165,0751515	0,916269713	16%
TOTAL SUGARS		917,0611739	5,895201247	100%
SAMPLE 2				
	area monosaccharide/ area int. St.	monosaccharides mass micrograms	monosaccharides micromoles	monosaccharides molar %

ARA	1,780001648	749,7366943	4,993916568	34%
XYL	2,838516072	1195,582969	7,963651299	55%
MAN	0	0	0	0%
GAL	0,025595419	10,78079062	0,05984009	0%
GLC	0,652481832	274,8253475	1,525451529	10%
TOTAL SUGARS		2230,925802	14,54285949	

Table 5-42

Sugars on dry weight of sample, AX4		
	SAMPLE 1	SAMPLE 2
ARA	19%	32,9%
XYL	39%	52,4%
MAN	0%	0,0%
GAL	2%	0,5%
GLC	13%	12,1%
TOT	73%	98%

Table 5-43

PP1				
SAMPLE 1				
	area monosaccharide/ area int. St.	monosaccharides mass micrograms	monosaccharides micromoles	monosaccharides molar %
ARA	0,118347581	25,1133567	0,167277404	34%
XYL	0,073360312	15,56705826	0,103690523	21%
MAN	0	0	0	0%
GAL	0,098912387	20,98920852	0,116503156	23%
GLC	0,094818656	20,12051878	0,111681388	22%
TOTAL SUGARS		81,79014225	0,499152471	
SAMPLE 2				
	area monosaccharide/ area int. St.	monosaccharides mass	monosaccharides moles	monosaccharides molar %
ARA	0,054767387	11,62163948	0,077410507	36%
XYL	0,026962042	5,72134533	0,038109274	18%
MAN	0	0	0	0%
GAL	0,039242477	8,327253602	0,046221434	21%
GLC	0,047248513	10,02613441	0,055651279	26%

TOTAL SUGARS		35,69637283	0,217392495	

Table 5-44

Sugars on dry weight of sample, PP1		
	SAMPLE 1	SAMPLE 2
ARA	1,41%	0,99%
XYL	0,87%	0,49%
MAN	0,00%	0,00%
GAL	1,18%	0,71%
GLC	1,13%	0,86%
TOT	5%	3%

Table 5-45

PP2

SAMPLE 1

	area monosaccharide/ area int. St.	monosaccharides mass micrograms	monosaccharide s micromoles	monosacchari des molar %
ARA	0,142049892	30,14298706	0,200779238	21%
XYL	0,323886794	68,72877775	0,457795096	47%
MAN	0	0	0	0%
GAL	0,017232662	3,656770814	0,020297351	2%
GLC	0,245563169	52,10850446	0,289234594	30%
TOTAL SUGARS		154,6370401	0,96810628	100%

SAMPLE 2

	area monosaccharide/ area int. St.	monosaccharides mass micrograms	monosaccharide s micromoles	monosacchari des molar %
ARA	0,229343984	48,66679348	0,324164347	18%
XYL	0,643633443	136,5790165	0,909738337	50%
MAN	0	0	0	0%
GAL	0,009495683	2,014983896	0,011184413	1%
GLC	0,47556349	100,9145725	0,560138613	31%
TOTAL SUGARS		288,1753664	1,80522571	100%

Table 5-46

Sugars on dry weight of sample, PP2		
	SAMPLE 1	SAMPLE 2
ARA	3,35%	2,72%
XYL	7,64%	7,63%

MAN	0,00%	0,00%
GAL	0,41%	0,11%
GLC	5,79%	5,64%
TOT	17%	16%

Table 5-47

PP3				
SAMPLE 1				
	area monosaccharide/ area int. St.	monosaccharides mass micrograms	monosaccharide s micromoles	monosacchari des molar %
ARA	0,175084652	37,15296315	0,247471945	10%
XYL	0,935945257	198,6075834	1,322904039	54%
MAN	0,027366194	5,807106298	0,032233766	1%
GAL	0,014462568	3,068956982	0,017034619	1%
GLC	0,723494405	153,5255128	0,852162038	34%
TOTAL SUGARS		398,1621227	2,471806408	
SAMPLE 2				
	area monosaccharide/ area int. St.	monosaccharides mass micrograms	monosaccharide s micromoles	monosacchari des molar %
ARA	0,326127052	69,20416048	0,46096157	9%
XYL	1,794765343	380,8492059	2,536796149	52%
MAN	0,050799845	10,77972707	0,059835515	1%
GAL	0,028898573	6,132277234	0,034037951	1%
GLC	1,503102846	318,958424	1,77041754	36%
TOTAL SUGARS		785,9237946	4,862048725	

Table 5-48

Sugars on dry weight of sample, PP3		
	SAMPLE 1	SAMPLE 2
ARA	2%	3%
XYL	13%	18%
MAN	0%	1%
GAL	0%	0%
GLC	10%	15%
TOT	26%	37%

Table 5-49

Final residue				
SAMPLE 1				
	area monosaccharide/ area int. St.	monosaccharides mass micrograms	monosaccharide s micromoles	monosacchari des molar %
ARA	0,994746974	211,0853079	1,406016838	15%
XYL	1,408175506	298,8148424	1,990373959	21%
MAN	0,069457138	14,7388046	0,081811345	1%
GAL	0,067479429	14,31913485	0,0794801	1%
GLC	4,843744719	1027,842629	5,705165572	62%
TOTAL SUGARS		1566,800719	9,262847814	100%
SAMPLE 2				
	area monosaccharide/ area int. St.	monosaccharides mass	monosaccharide s moles	monosacchari des molar %
ARA	1,008331002	213,9678387	1,42521707	15%
XYL	1,382475739	293,3613519	1,954048837	21%
MAN	0,063538892	13,48295299	0,074840433	1%
GAL	0,073377508	15,57070724	0,086427105	1%
GLC	4,887336515	1037,092808	5,756509816	62%
TOTAL SUGARS		1573,475659	9,297043261	100%

Table 5-50

Ethanol-water extracted polyphenols				
	area monosaccharide/ area int. St.	monosaccharides mass microgram	monosaccharide s micromoles	monosacchari des molar %
ARA	0,051916039	11,01658345	0,073380293	7%
XYL	0,017413803	3,695209062	0,024613395	2%
MAN	0	0	0	0%
GAL	0	0	0	0%
GLC	0,866476798	183,8663765	1,020572694	91%
TOTAL SUGARS		198,578169	1,118566383	100%

Table 5-51

Pure water extracted polyphenols

	area monosaccharide/ area int. St.	monosaccharides mass micrograms	monosaccharides micromoles	monosaccharides molar %
ARA	0,262197318	55,63827082	0,370600618	21%
XYL	0,244828705	51,95265117	0,346051097	20%
MAN	0	0	0	0%
GAL	0,064215452	13,62651884	0,075635651	4%
GLC	0,806016785	171,0367618	0,949360356	55%
TOTAL SUGARS		292,2542026	1,741647722	100%

Table 5-52

Sugars on dry weight of sample		
	Hydro-alcoholic extraction	Water extraction
ARA	1%	5%
XYL	0%	5%
MAN	0%	0%
GAL	0%	1%
GLC	16%	16%
TOT	17%	28%

Table 5-53

As readable in the previous data, the purity in monosaccharides of the AX-rich products is highest for the first extraction (with an average value of 96.5%) and decreases in subsequent alkaline extractions. It grows again in the water extraction, possibly because of the lower solubility of the other compounds in water, with respect to the case of the alkaline solutions. The polyphenols-rich products also exhibit a considerable, albeit lower, sugar content, increasing with the alkalinity of the extracting solution, ranging from 4.5% up to 31.5%.

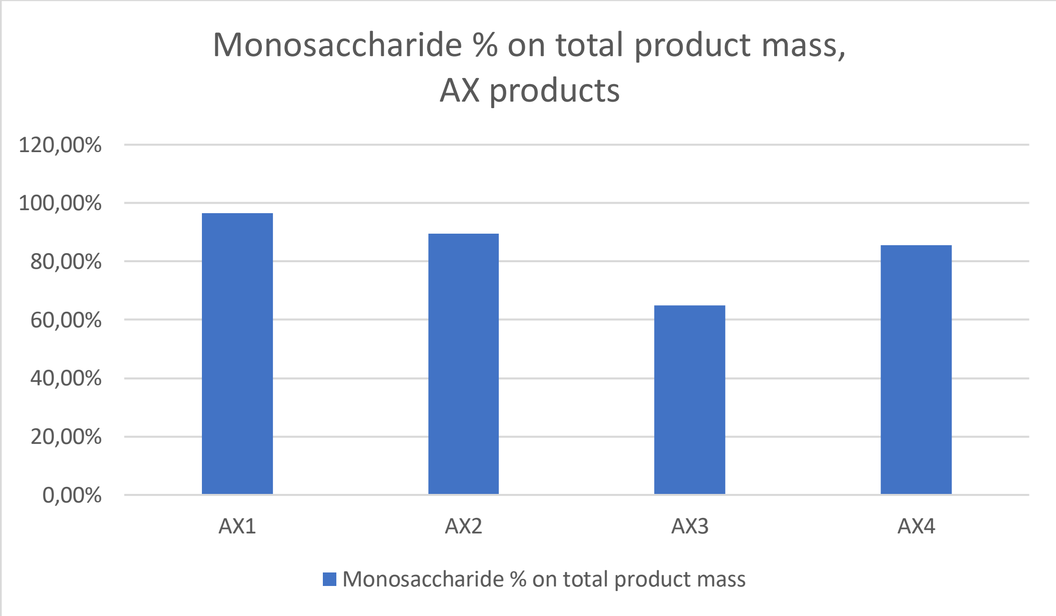


Figure 5-4

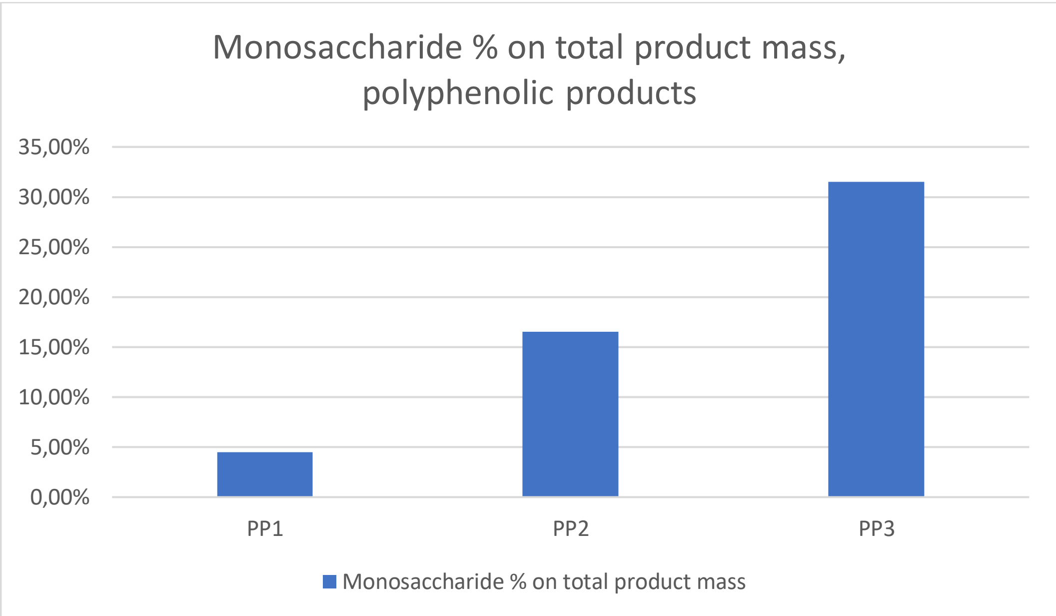


Figure 5-5

The gross product yield ona dry BSG basisì is calculated as the ratio between the total weight of the product and the initial BSG sample treated.

Using the data relative to the gross yield of the products, and the data relative to the content of arabinose and xylane, it's possible to calculate the AX yield of each product on the basis of the toal content of AX in the BSG.

Material	product weight, % on dry BSG matrix	AX in extract as % ax in BSG	Total sugars mg/g of BSG

BSG		100%	100,00%	
0,1 M				
	PP1	14,69%	1,07%	5,62
	AX1	2,10%	7,21%	20,25
0,5 M				
	PP2	15,47%	6,38%	25,74
	AX2	7,82%	22,83%	70,15
4 M				
	PP3	5,90%	4,19%	18,67
	AX3	8,83%	17,58%	57,37
Water				
	AX4	3,50%	9,70%	29,96
	Residue	19,47%	22,32%	160,26
EtOH Polyphenols			0,014%	
Water Polyphenols			0,062%	

Table 5-54

When considered together, the four AX-rich products have an overall yield of AX on the basis of the BSG's AX content equal to 58.32%. The 0.1 M alkialine extraction presents the lowest yield and the 0.5 M the highest, followed by the 4M extraction; nonetheless, one has to consider that the 4 extractions are sequential, so there is less AX to be extracted for each extraction. We can calculate the AX yield on the basis of the leftover yields as follows: for each extraction, we calculate the leftover AX by subtracting from 100% the percentages of extracted AX in the previous extraction steps. By division of the yield percentage by the leftover AX percentage, timing by 100, we obtain the corrected yield of AX on the basis of the leftover AX content.

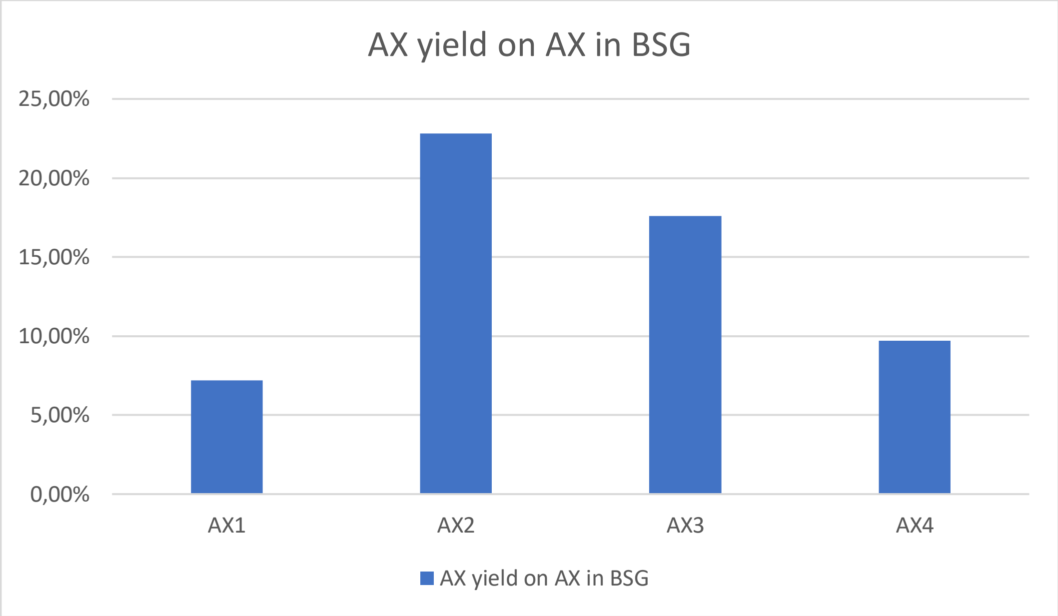


Figure 5-6

	AX in extract as % AX in BSG	Leftover AX, % AX in BSG	Corrected yield of AX on leftover AX
AX1	7.21%	100%	7.21%
AX2	22.83%	92.79%	24.60%
AX3	17.58%	69.96%	25.13%
AX4	9.70%	52.38%	18.52%

Table 5-55

One can see that when this evaluation has been carried out, the highest yield is relative to the extraction carried out with the most alkaline extractant. One fact that may be surprising is the fact that the last extraction, performed with pure water, gives yields superior to the first alkaline extraction, both in the corrected an non corrected case. This may be explained when one takes into account the fact that the water extraction is performed after all the alkaline extraction. Alkaline solutions cause swelling of the matrix, with consequent enlargement of the matrix porosities: this in turn translates with porosities more accessible to the extractant: in the case of the first alkaline extraction, there haven't been any preceding steps that could cause said swelling, and the porosities of the matrix are not yet so accessible as in the case of the last extraction.

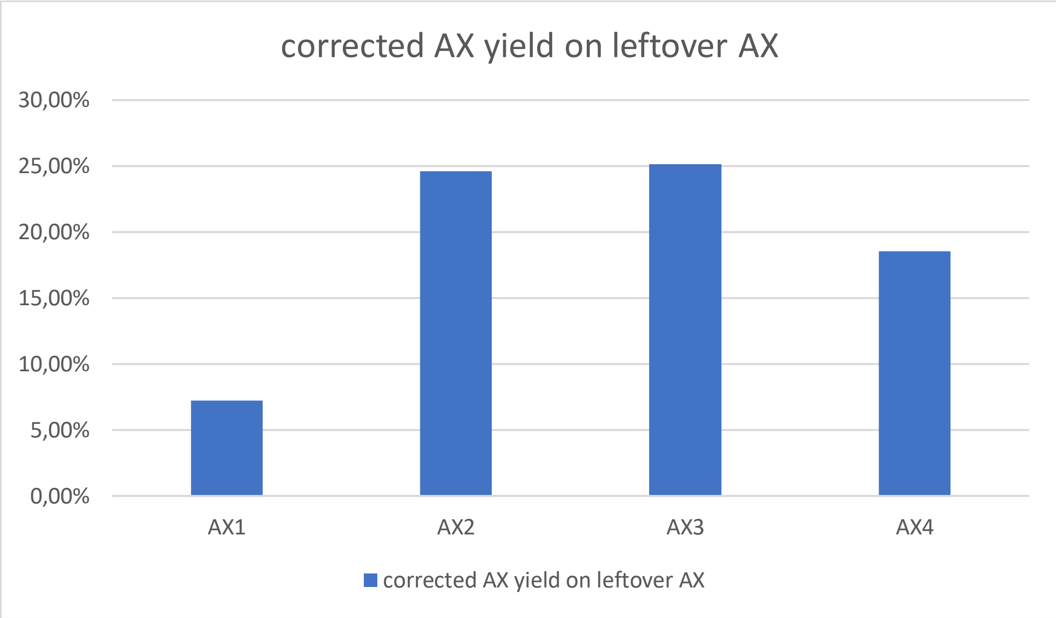


Figure 5-7

1.12.2 Total phenolic content

The absorbance presents a linear correlation to the phenolice content of the analysed sample, expressed in mGAE/g (where GAE stands for gall-acid equivalent). By using the calibration point of known concentration, it has been possible to calculate che intercept and the angula coefficient of the line which best fits the calibration points' absorbance. Once the straight line has been obtained, it's possible to use the value of absorbance relative to the samples ot obtain, in turn, their phenolic content.

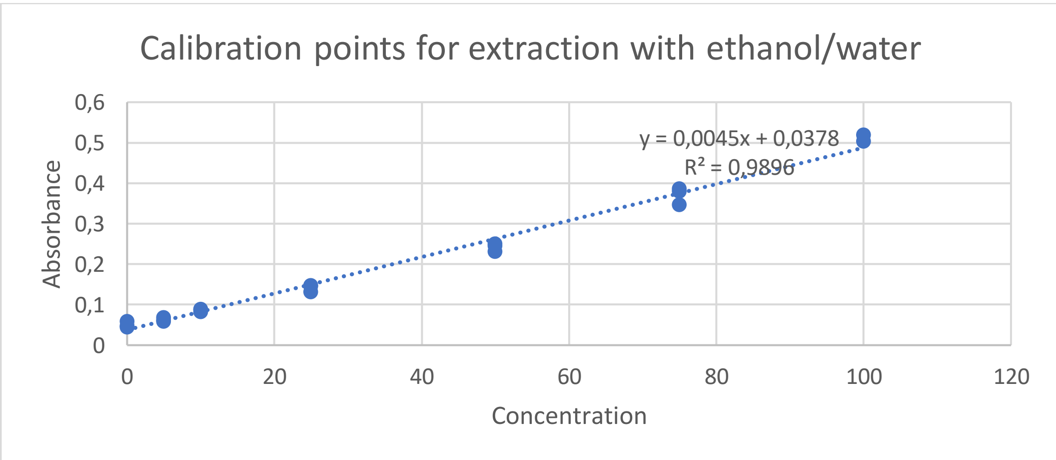


Figure 5-7

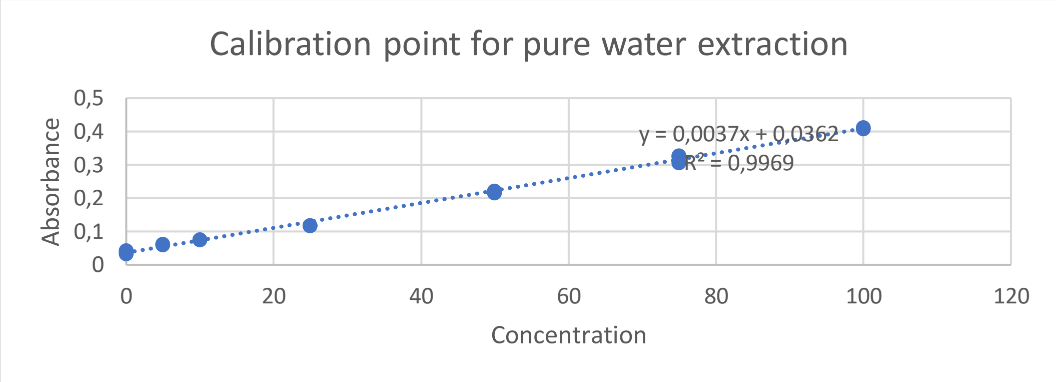


Figure 5-7

For the ethanol-water extracted material, as one can see in graph n. 5.7, we obtain a value of angulae coefficient m of 0.0045, and an intercept q of 0.0378.

$$Absorbance = m * TPC + q$$

$$TPC = (Absorbance - 0.0378)/0.0045$$

Combined with the absorbance results presented in the relative section, we obtain the following phenolic content values for the two samples:

Ethanol-water extraction		
Samples masses		
A	1,68	mg
B	3,77	mg
Tube volume (samples)	1	ml
Sample concentration	1680	ug/ml
Sample concentration	3770	ug/ml

Table 5-56

Phenolic concentration of ehtnaol-water extracted product (ug/ml)		Phenolic content of ehtnaol-water extracted product (mgGAE/g sample)	
A	B	A	B
19,4	56,3	11,5	14,93
21,4	56.0	12,7	14,6

Table 5-57

Average phenolic content	12,3	mgGAE/g
Standard deviation	2,1	
Deviation coefficient	0,1703	

Table 5-58

Concerning the extraction with pure water, the following results have been obtained:

Pure water extraction		
Samples masses		
A	1,11	mg
B	1,313	mg
Tube volume (samples)	1	ml
Sample concentration	1110	ug/ml
Sample concentration	1313	ug/ml

Table 5-59

Phenolic concentration of water-extracted product (ug/ml)		Phenolic content of water-extracted product (mgGAE/g sample)	
A	B	A	B
27,1	28,2	24,4	21,5
28,2	35.0	25,4	26,6

Table 5-60

Average phenolic content	24,9	mgGAE/g
Standard deviation	1,9	
Deviation coefficient	0,076	

Table 5-61

From the analysis, it's possible to see how the avreage phenolic content is way higher in the case of pure water extraction, with a TPC more than twice that of the product extracted with ethanol-water. For the calculation of the total yield on polyphenols in the two cases, it's necessary to calculate the mass yield of the products (i.e. the ratio between the weight of the products and the weight of the BSG sample).

	Mass, g	% Mass yield
BSG sample	12,76	100%
EtOH Polyphenols	0,38	3,35%
Water Polyphenols	0,21	1,80%

Table 5-62

To calculate the yield in polyphenols, it's sufficient to divide the product of the BSG sample mass times its TPC, by the weight of the product times its TPC

$$Yield\ on\ Polyphenols = \frac{m_{BSG} TPC_{BSG}}{m_{product} TPC_{product}}$$

Data relative to the phenolic content of the BSG has been experimentally obtained and is reported in the folowing table:

Component	Content, mg/g
Catechin	0.06
Gallic acid	0.28
P-coumaric acid	0.41
Ferulic acid	0.52
Caffeic acid	0.34

Syringic acid	1.2
Synapic acid	0.14
Other polyphenols	2.79
Total phenolic content [mgGAE/g]	4.765

Table 5-63

The values of the yield on polyphenols in the two cases are here reported:

BSG mass (g)	12,761
TPC of BSG (mgGAE/g)	4,765
Total phenols in BSG (mg)	60,807
Ethanol-water extracted product mass (g)	0,384
Ethanol-water extracted product TPC (mgGAE/g)	12,337
Ethanol-water extracted product yield	*7.8%*
Water extracted product weight (g)	0,207
Water extracted product TPC (mgGAE/g)	24,921
Water extracted product yield	*8.5%*

Table 5-64

1.12.3 Methylation analysis of BSG

The methylation anaylisis of the BSG revealed the content of the various monosaccharide groups. By distinguishing (based on the elution time) between the various types of sobstituted species, the degree of polymerization and the degree of branching in the arabino-xylanes have been calculated.

Specie	relative content
t-XYL	8,50
4-XYL	55,09
3,4-XYL	0,69
2,4-XYL	1,06
TOT XYL	65,34

Table 5-65

The degree of polymerisation (DP) is calculated as the ratio between the total xylose amount an the terminal xylose amount.

$$DP = \frac{8.50}{65.34} = 7.687$$

Whereas, the degree of branching (DB) is calculated as the ratio between non-terminal xylose content and total xylose content:

$$DB = \frac{65.34 - 8.50}{65.34} = \frac{56.84}{65.34} = 0.8699$$

1.12.4 BSG granulometry

The initial weight of BSG was 196.1 g. The weight in each sieve was weighted, and the weight of the empty sieve was subtracted to obtain the weight of BSG in each sieve. The ratio between these weight and the initial BSG was then calculated, giving the percentages of material that “stopped” in each sieve. The results are reported in the following table:

SIEVE CUT um	EMPTY SIEVE WEIGHT g	LOADED SIEVE WEIGHT (AFTER SHAKING) g	BSG WEIGHT IN SIEVE g	fraction
BOTTOM	344,54	347,69	3,15	1,6%
63	252,39	266,97	14,58	7,4%
90	243,06	265,5	22,44	11,4%
150	268,96	317,6	48,64	24,8%
300	275,04	382,15	107,11	54,6%

Table 5-66

As it readable, more than half of the material’s mass is composed by particles with a size of 300 μm. Unfortunately, sieves with bigger meshes were not available. The characterisation of the matrix for particles with size greater that 300 μm is thus in need of further investigation.

6. DEFINITION OF A TURBEX BASED BSG VALORISATION PROCESS

1.13Process flow diagram

1.14Process description

1.14.1 Targets

The aim of the process is to obtain three different products from fresh brewers’ spent grain: namely a product rich in phenolic compunds, a product rich in arabino-xylanes, and a product rich in proteins. The process basically foresees 3 in sequential extractions performed with Turbex as extracting equipment.

1.14.2 Plant Overview

The entire process scheme can be divided in 4 main sections. The first section is dedicated to raw material storage and pre-treatment. The BSG need to be dried to a humidity content of ≤ 10% whithin 24 ÷ 36 hours after its discharge from brewery, in order to avoid its spoilage. Once the BSG has been treated, it is sent to the second section, where the extraction of poliphenols—rich product is performed using a water-ethanol mixture as liquid extractant. In the same section the product is purified and sent to a packaging unit, while the extracted BSG (i.e. solid residue after polyphenol extraction) is sent to the following section. The third section is where the extraction of arabino-xylanes and protein rich product is performed. Last, a fourth section is present, where the reagents and the extracting solutions are recovered to be re-routed to the extraction sections of the plant. The BSG valorisation plant is designed with a nameplate capacity of 2.000 tons per year of fresh BSG. The availability of the raw material is variable during the year. The process is also able to work between 60% and 110% of the nominal capacity. The plant has been supposed to be operative for 350 days a year, or 8400 hours, meaning that the nameplate capacity is equivalent to 238 kg of fresh BSG per hour.

1.14.3 Utilities charateristics and consumption

The following utilities are employed during the productive process:

Utility	Charateristics	
Low pressure steam (LPS)	Temperature:	158 °C
	Pressure:	5 bar
Cooling water	Temperature IN:	20 °C
	Temperature OUT:	40 °C
	Pressure:	1 bar
Nitrogen (closed circuit)	Temperature:	25°C
	Pressure IN:	4 bar
Methane	Temperature:	25°C
	Pressure:	10 bar
Electric Energy	Voltage:	400 V
	Frequency:	50 Hz

Table 6-1

The following prices have been considered for the utilities, excluding nitrogen, which, being used in a closed loop, suffers virtually no loss.

Utility	Price
LPS	5.125 euros/ton
Cooling water	0.021 euros/ton
Methane	0.97 euro/kg
Eletric energy	0.125 euro/kWh

Table 6-2

Low pressure steam (LPS) is used as heating medium in the various equipment where an evaporation is accomplished. It is returend to the plant as hot water (HT).

Equipment	LPS consumption (kg/h)
Evaporator EV-201	34.2
Drum dryer DD-201	20.7
Drum Dryer DD-301	6.8

Drum Dryer DD-302	5.2
Distillation column C-401	1740.0
Distillation column C-402	969.0
EV-401	28.1
TOT.	2804

Table 6-3

Methane is used to heat the air employed by the rotary dryer RD-101 in the BSG pre-treatment.

Methane consumption (kg/h)	0.7

Table 6-4

The overall utilities consumptions of the plant are synthetized in the following table:

Utility	Yearly co sumption	
Electricity	3292	kWh/year
Low pressure steam	23556	ton/year
Cooling water	632661	m3/year

1.14.4 Matrix characterisation

Fresh BSG is supplied from the breweries. The matrix still reating a high level of moisture. The composition of the BSG considered during the design process is reported in table 6.7

Component	Content, g/100 g
Water	72,2
Proteins	7.2
Arabinose	3.0
Xylose	5.3
Galactose	0.3
Glucose	1.2
Cellulose	3.5
Polyphenols	0.48
Other compunds (ashes, lipids)	6.8

Table 6-5

Several phenolic compounds are present: a more detailed list of the present species and their respective content is hereby reported:

Component	Content, mg/g
Catechin	0.06
Gallic acid	0.28
P-coumaric acid	0.41

Ferulic acid	0.52
Caffeic acid	0.34
Syringic acid	1.2
Synapic acid	0.14
Other polyphenols	2.79
Total phenolic content [mgGAE/g]	4.765

Table 6-6

1.14.5 Products characterisation

Three main products and one by product are obtained in the plant. The first one is a polyphenols-rich product, the seecond is a protein-rich fraction, and the third main product is an arabino-xylanes rich product. As a result of the reaction bewteen the NaOH used in the protein extraction and the HCl used in the arabino-xylanes, a certain quantity of NaCl is also produced as a by-product, and can therefore be sold.

The polyphenols rich product presents itself as a whitish powder, with a water residual content of around 2%; the main polyphenolic compund is syringic acid, followed by Ferulic and P-coumaric acid. There is also a noteoworthy residual content in proteins; the presence of proteins could facilitate the delivery of polyphenols to lower parts of the gastrointestinal tract, due to their interaction which protects polyphenols against oxidative degradation (Shpigelman, et al., 2010).

Component	Mass, %
Phenols	31,5
Proteins	49,4
Arabinose	10,3
Xylose	2,3
Galactose	0,0
Glucose	3,0
Cellulose and other products	1,6
Water	2,0

Table 6-7

The process can obtain 570 g of product per hour, for a yearly production of 5.56 tons. Given the high content of polyphenols and proteins, both valuable biocative compounds, it has been estimated that the product could be sold at the price of 76 €/kg, for a yearly gross revenue from sales of 362300 €/year.

The protein-rich fraction also presents itself as a yellow-brown powder, with a residual content of humidity of around 2% in mass. The product is mainly composed by proteins, with cellulose and monosaccharides present in minor, but not negligible, quantities.

Component	Mass, %
Proteins	76,2
Arabinose	4,3
Xylose	8,3
Glucose	1,6
Cellulose and other products	7,4
Water	2,3

Table 6-8

For this product, the price has been estimated to be of 3.00 €/kg. With a yearly production of 110 tons, the gross revenue from its sale can be surmised to be around 330.300 €/year.

The arabino-xylanes fraction presents itself as a dry powder; it is composed mainly of arabino-xylanes and, secondarily, glucose.

Component	Mass, %
Arabinose	29,4
Xylose	57,2
Glucose	10,7
Cellulose and other products	0,5
Ethanol	0,6
Water	1,6

Table 6-9

For this product, the price has been estimated to be around 12 €/kg; with a yearly production of 143 tons, the toal estimated gross sales revenue for this product is equal to 1.713.000 €/year.

Finally, the yearly production of NaCl as a by-product is of 224 kg/hour; with a cost of 30 €/ton, the gross revenue from the sale of salt is estimated to be around 56.000 €/year.

Polyphenols-rich product	362.300,00 €/year
Protein-rich product	330.300,00 €/year
AX-rich product	1.713.000,00 €/year
NaCl	56.000,00 €/year
Tot. gross revenue from sales	2.461.300 €/year

Table 6-10

1.14.6 Fresh BSG storing and stabilisation section

Fresh BSG is fed to the valorisation plant at a nameplate capacity of 238 kg per hour. It has been supposed that the delivery of the raw material from nearby breweries happens daily, and that the BSG is delivered by means of a truck. BSG is firstly stored in the underground tank S-101, so that the truck may be able to easily drop its load into it. Given the variability of the supply, the tank has been designed to be able to accommodate a daily load of BSG in the month for which the supply is higher (June) and at a turn-down ratio of 110%. From the undersground stroage tank S-101, the BSG is then picked up by the screw conveyor SC-101 and unloaded into the hopper H-101. For the transportation of the raw material to the dewatering step, the SEEPEX SAI technology is employed, which basically consists of a volumetric conveyor which receives the fresh material from H-101, and transports is along the tubes with the aid of periodical injections of compressed air received from the air compressor C-101. The injections of air are regulated on the basis of the pressure in the tube, as read by an appropriate reader. The system allows for the transportation of the heavily humid material along longer distances. The BSG arrives to hopper H-102, where it is separated by the air used for transportation and from which it is brought to the dewatering step by feeder XF-101. The dewatering of the fresh BSG is prerformed in the roll-belt press RP-101, by applying mechanical force to the material. This step allows for a first reduction of the moisture content down to 40-60% by weight. From RP-101, we have a stream of waste water (which is re-sent to the brewery to be adequately treated and re-utilised in the beer-production process, lowering overall water foot-print of the process) and a cake which is collected in the hopper H-103. Again, the cake is transported to the next step by means of the SEEPEX SAI technology. The cake arrives to the rotary drier RD-101. The drying of the BSG is achieved using a counter-current of air at 80°C. The air is taken from the atmoshpere by the blowing fan BF-101 and is filtered and thereafter it is sent to the methane fuelled burner B-101. The flowrate of methane is regulated to ensure a temperature of 80°C at the exit of the burner. The air stream is then sent to RD-101. At the exit of the dryer, the hot air is sent to cyclone CY-101 + filter F-102 system to recover the entrained BSG particles, which are then re-routed to dryer RD-101 by means of feeder XF-103. RD-101 brings BSG moisture content down to 10%, which allows for the storing of the material without risk of spoilage. The dried BSG is sent to the storage silo SS-101 by means of a dense phase pneumatic conveying system. The system employs nitrogen as moving fluid to prevent explosion risk, as well as oxidative product degradation. The nitrogen cycle is a “closed-loop”: the BSG, conveyed by pneumatic conveying, is discharged in SS-101, from which the nitrogen is sent to cyclone CY-102 to be separated from the BSG and re-used. The nitrogen stream leaving the cyclone is firstly filtered and then re-compressed by the compressor included in the prenumatic transportation package. The stored BSG, along the BSG recoverered in CY-102, is sent, by means of a second dense phase penumatic transport, to the polyphenols extraction section

1.14.7 Polyphenols extraction section

The polyphenols extraction section receives the stabilised BSG from the previous section. BSG is collected in hopper H-201, from which screw feeded XF-201 sends the material to the first extracting step. The BSG is stored in D-201 where it is soaked with extract coming out ot the extractor TX-201. The soaking is time-operated, and when completed, the solid matrix is discarged in D-202 from which XF-202 delivers it to the Turbex extractor TX-201. D-202 is mounted on a load cell and the soaked BSG is fed to Turbex extractor with a "loss-in-weight" type weight control loop. The Turbex extractor is a Turbex EX150, able to process up to 500 kg/h. The extractors employ a 60/40 ethanol-water mixture as extractant. The extracted solids are sent to a centrifugal dencanter and after to a squeezing press, to perform solid-liquid separation. The recovered liquid is sent to drum D-203 and recyled back in the extraction step along fresh extractant. The solid is instead sent to the third section for the extraction of arabino-xylanes and proteins. The liquid extract is taken out of the turbex by means of a progressing-cavity pump. A fraction of the stream is sued to fill the aforementioned soaking drums, while the balance, after a liquid filtration, is sent to the membranes step, where it is purified from the other compounds.

Two steps of membranes are present: a ultrafiltration followed by a nano-filtration. The first step reduces the amount of proteins and cellulose which are left in the retentate stream. The second step is used to reduce the amount of sugars, which are left in the permeate stream. Each step is preceded by a buffer tank to ensure the continuity of operation and to ensure a steady, constant flow to the membranes. The configuration of both membrane section is the same, so it will be presented only once. The membranes are provided with additional modules to ensure the continuity of the process even when some of the modules undergo the cleaning phase. A time-activated system activates ON/OFF valves to divert the flow to the back-up module when the operation time for the membranes has elapsed. While the process flow is diverted, the membranes undergo their cleaning routine. The cleaning consists in a series of passages of alkali and acid solutions, with passages of cleaning water in between. The cleaning fluids pass through the membrane in the opposite sense of the process fluids, and are provided by dedicated tanks where the fluids are prepared, stored and heated before usage. The alkali and acid solutions are recycled a number of times, after which they are disposed of in the sewage. The solutions are prepared in a batch fashion in separated tanks, which, on the basis of a time-regulated system, re-fill the dedicated tanks once they have discharged their content. The permeate from the first ultrafiltration step is sent as feed of the nano-filtration step, whereas the retentate of the nano-filtration is sent to an evaporator.

The evaporator EV-201 allows for the evaporation of the hydroalcoholic solution. The evaporator employs low pressure steam as heating medium. The evaporated solution is sent to the condenser HX-201 where it is condensed by means of chilled water and then sent to the recovery section of the plant. The concentrated product leaving the evaporators is instead sent to drum-dryer DD-201, where the remaining liquid is evaporated to obtain the final polyphenolic product in powder form. DD-201 uses low pressure steam as heating medium. The evaporated solution is also sent to condenser HX-201. The solid cake obtained from DD-201 is conveyed by the screw-conveyor SC-201 and sent to an appropriate packaging unit.

1.14.8 Proteins and arabino-xylanes extraction section

The solid feed coming out of the squeezing press attached to the polyphenols extraction step of the previous section is conveyed to the mulstistage counter-current washer W-301 to remove the hydroalcoholic extracting solution. The resulting mixture of water and ethanol is collected and sent to the recovery section of the plant. After the washing step, the solid is fed to the squeezing-press SP-301 to be separated from water. After that the solid is stored in the gravity blender silo SS-301, from which a pneumtic transport line bring the solid to the first extracting step with Turbex. The pneumatic transport presents itself in the same configuration as the ones illustrated in the previous section.

The first extraction step in TX-301 uses an alkali solution of water and NaOH with 4 M concentration. The configuration of this step is identical to the one of polyphenols extraction step: the solid is soaked with liquid extract out of the turbex, the solid is processed with centrifugal decanter and squeezing press and sent to the next extraction step, and the recovered liquid sent to the surge tank D-303. The extracting solution is prepared in a separated tank by mixing of solid NaOH with water, which is then fed to the same surge tank; the liquid extract, after filtration is sent to static mixer D-307 where the extract is acidified with a saturated solution of citric acid to cause the precipitation of proteins.. The exhausted solid coming out of TX-301 and is disposed of as a waste. From D-307 the liquid mixture is pumped to the centrifugal decanter CD-302, where the the proteins deposit on the bottom of the decanter. The solid collected from the decanter is pumped by means of a progressing cavity pump to the collecting tank D-310. The liquid fraction from the decanter is pumped and sent to the static mixer D-317 where the liquid is further acidified with HCl. After the mixing, the liquid is sent to the static mixer D-308 where it is mixed with ethanol, and then sent to the centrifugal decanter CD-303. In the decanter, the supernatant is collected and pumped to the recovery section of the plant, whereas the precipitated arabinoxylanes are collected in the tank D-334. The liquid extract is sent to the static mixer D-333, where it is mixed with citric acid. The liquid coming out of the mixer is sent to the static decanter D-307, where the arabino-xylanes deposited on the bottom are collected and sent to the collecting drum D-312; the supernatant rich in citric acid is collected and sent to the recovery section of the plant.

From tank D-312, where the arabino-xylanes are collected, the material is periodicaly sent to basket cetrifuge CF-302, which operates in a batch fashion, and realises the separation from the liquid and the solid. After centrifugation, the separated solid is collected in the buffer tank D-313, from which it is continuosly fed to the drum dryer DD-301, whereas the recovered liquid is sent to the recovery section of the plant. The liquid evaporated from drum dryer D-301 is sent to HX-301 to be condensed with chilled water and also sent to the recovery section of the plant.

The protein-rich fraction collected in D-310 is likewise periodically sent to the baset centrifuge CF-301. After centrifugation, the separated solid is collected in buffer tank D-311, from which it is conveyed to the drum dryer DD-302, which evaporates the leftover liquid, leaving a finale product which is then sent to an appropriate packaging unit.

1.14.9 Recovery section

The recovery section has the aim to separate the mixtures coming from various parts of the plant and re-obtain the ethanol solution and the citric acid needed for the functioning of the process. The mixture of citric acid and ethanolic solution coming from the previous sections is collected in tank D-401. From there, the mixture is sent to the first distillation column CC-401. The head stream is rich in ethanol and is stored in

TK-401, from which it is sent to the parts of the plant utilizing the ethanolic solution. TK-401 also receives ethanol stored in TK-402 as fresh feed compensing the losses of ethanol and to adjust its concentration to the desired value. The bottom product leaving CC-401 is sent to the static mixer D-403, where it is mixed with a side stream of ethanolic solution coming from TK-401. This is done to allow the NaCl to precipitate and be remove. The liquid stream from D-403 is then sent to the centrifugal decanter CD-401- This is done to precipitate and remove the NaCl, The liquid from DC-401 is instead sent to the distillation column CC-402. This column realises a further separation of a ethanol-rich stream that omes out of the top of the column, while from the bottom a solution rich citric acid I obtained. The head product isi sent to storage tank TK-401, while the bottom product is further concentrated in the multiple effect evaporators EV-401/402/403, and after that store in TK-403. From here, the citric acid is sent to the various parts of the plant.

1.15Design basis

In this chapter the theoretical considerations used during the design of the various pieces of equipment in the process will be presented. This will include a description the physical and/or chemical phenomena of interest in the evaluation of each piece of equipment and the step-by-step procedure employed for each design. Beside the theoretical considerations, also technical issued need to be cared for. When a particular engineering choice has been performed, it will also be described in this section, along with the reasons that led to that particualr choice. Many of these choices are done on an ecomocial basis. Th procedure for the economical evaluation of the various equipment is reported in the relative section.

1.15.1 Underground storage tank S-101

The underground storage tank S-101 collects the fresh BSG from nearby breweries'. The storage tank bust be able to contain the equivalent of 24 hours worth of BSG during the periods of maximum productivity of the plant. The necessary volume has been calculated on this basis, using the density of fresh BSG to transform the mass flowrate in a volumetric one and using 24 hours as time of residence. An additional 20% of empty volume has been added to allow for access to the tank for possible maintenance operations. The tank has a volume of around 10 m^3 with 1700 mm of height, 1200 mm of width and 5100 mm of length. The tank may be provided with either a small floor slope of a system of carts to aid in the collection of the feed stock by the subsequent screw conveyor SC-101.

1.15.2 Screw conveyor SC-101

The screw conveyor SC-101 collects the BSG contained in the underground storage tank S-101 and conveys is to the hopper H-101. The conveyor is 4 metres long at an angle of 60° from the horizontal. The length has been calculated keeping in mind the height of the tank S-101, the angle, and the distance to the hopper H-101. The design and the choice of the conveyor is based on the procedure illustrated in the website of KWS (KWS contructor website, s.d.) For the choice of the screw conveyor, the volumetric flowrate of prelevated feed stock has been calculated and corrected with a coefficient dependent on the type of screw conveyor. Since in this case the conveyor is a short one, the pitch is equale to 2/3 of the conveyor diametre and the correction factor is equal to 1.5. Then, the reccomended value of though loading for the wet BSG found in (KWS constructor's website, s.d.) allowed to choose a diameter for the conveyor. Finally, the speed of the conveyor is calculated as the ratio of the corrected capacity and the capacity at maximum RPM. The screw conveyor trasnports a nameplate capacity of 248 litre per hour of BSG and is able to function with at the maximum value of productivity equal to 350 litres per hour. The conveyor has a 4 inches diametre and works at 31 RPM for nameplate capacity and 44 RPM at maximum capcity. In the same pages, value realitves to material and conveyor can be found to calculate the necessary power for the conveyor, which in this case is equal to 430 W

1.15.3 RP-101

For the sizing of the Roll press R-101, the performance of several commercially-available equipment of the same family has been collected. The investigation has revealed that roll-presses are already succesfully implemented in the industry for BSG dewatering. The final moisture content obtained ranged beteween 40% and 60% on total weight. The least efficiency was then considered to correspond to the maximum possible flowrate in the quipment, to be sure that water removal could only increase for lesser flowrates. Based on the maximum flowrate, a commercially available equipment was selected: DY750 by SHANGAI DAZHENG. The size of the equipment and the energetic power required were found on the constructor's website. The roll press processes 233 kg/hours of BSG, leaving a wastewater stream of 71 kg/hour and a dewatered cake of BSG with a flowrate of 162 kg/hour. The estimated power consumption is 1.1 kW

1.15.4 Compressor C-101 for Seepex SAI pneumatic transportation

The alternative air compressor C-101 provides periodical injection of compressed air in the solid stream coming out of the progressing cavity pump H-102. The solution, mutuated from the solution offered by Seepex (SAI), allows for a diminished power need of said pump. The compressor is activated by a pressure transmitter that allows the flow of compressed air in the solid stream pipe when the registered pressure falls below a fixed amount. To achieve a penumatic transport of the solid, the Archimedes number (Ar) of the stream has been calculated, a value of the coefficient omega (Ω) has been set to ensure the possibility of pneumatic transport (see graph below) and from that the velocity of the stream has been calculated.

$$Ar = \frac{\rho_f(\rho_p - \rho_f)D_p^3}{\mu_f^2} g \quad (6.1)$$

$$\Omega = \frac{\rho_f^2 u^3}{(\rho_p - \rho_f)\mu_f} g \quad (6.2)$$

Where (SI units):

- ρ_f is the fluid (air) density
- ρ_p is the particle density
- D_p is the particle diametre
- u is the stream velocity
- μ_f is the fluid viscosity
- g is the gravitational acceleration

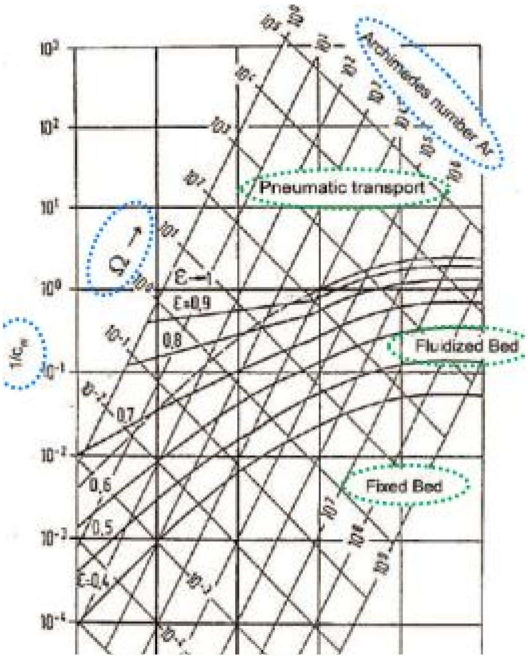


Figure 6-1 From Perry's Chemical Engineers' Handbook, 9th ed.

From the same graph, the Reynolds number has been retrieved. To estimate the necessary flowrate of air, a first hypothesi on the number of kg of air per kg of solid necessary to achieve transportation has been made, and on that hypothesis a density was calculated and then, making use of Re, the necessary diametre. The value for the diametre so obtained allows for the calculation of the flowrate of air given the calculated velocity and the air density; if the two values of air flowrate are not similar, a new hypothesis on the ratio between air and solid is made until the two flowrates are similar enough. The value obtained is 3.3 kg of air for kg of solid, for air at 4 bars. This is equivalent to a total flowrate of 1400 kg/h at maximum capacity. The power needed for the compressor has been obtained with well-known formula here reported. Surmising an efficiency of the compressor of 0.72, a value of 43 kW of power is obtained.

$$P_{is} = \frac{k}{k-1} ZRT_1 (-1 + \left(\frac{P_2}{P_1}\right)^{(k-1)/k}) * Q \quad (6.3)$$

Where:

- P_{is} is the required isoenthropic power (W)
- k is the ratio of heat capacities, equal to 1.4 for air
- Z is the compressibility factor
- R is the gas constant of air (8314/mlecoluar weith J/ kg K)
- T_1 is the aspiration temperature (K)
- Q is the mass flowrate (kg/s)

1.15.5 DD-101

For the sizing of the drum dryer DD-101, it's necesseary to estabilish a residence time for BSG in the vessel, able to grant a outlet humidity level of 10% or less. To do so, it has been necessary to obtain a drying curve for the BSG. The drying behaviour of BSG with hot air as drying medium has been investigated in (Mallen & Najdanovic-Visak, 2018); several models for the drying curve were proposed and the model parametres retro-fitted for a number of cases with different air temperatures. Among the proposed models, the Page Model was selected for its accuracy and simplicity. The authors defined the moisture ration MR as:

$$MR = \frac{X - X_E}{X_0 - X_E} \quad (6.4)$$

Where X is the moisture content at time t, whereas X_0 and X_E were the mositure contents at the start of the experiment and at the end, respectively. Using the page model, MR has been calculated as:

$$MR = \exp(-kt^n) \quad (6.5)$$

With k and n charateristic parametres of every set of conditions. The drying rate funcion $\Phi(t)$, which is the derivatve of MR has been calculated, for every point of the discretized time dominion using the approximation:

$$\frac{d}{dt} MR(t_i) \approx \frac{MR(t_{i+1}) - MR(t_{i-1})}{2\Delta t} \quad (6.6)$$

The drying curve can be then used to assess the residence time necessary to go from to moisture values X_1 and X_2 (Berk, 2009):

$$t = -\int_{X_1}^{X_2} \frac{dX}{\Phi} \quad (6.7)$$

Using the conditions of air at 80°C, we obtain a minimum residence time of 29 minutes. Since air will change temperature during the drying, this value is only used as a first trial. In the second step, it has been necessary to design a drum dryer able to ensure a residence time at least 29 minutes long. A model for the residence time in rotary drum dryers has been found in Perry's Chemical Engineering Handbook:

$$\theta = \frac{0.23L}{SN^{0.9}D} \pm 0.6\frac{BLG}{F}$$

$$B = 5(D_p)^{-0.5}$$

Where:

- Θ residence time, minutes
- L drum dryer length, ft
- D drum dryer diameter, ft
- S slope of the dryer, ft/ft
- F solid flowrate per unit surface, lb/hours/square feet
- G gas flowrate per unit surface, lb/hours/square foot
- N rotations per minute
- Dp mass average diameter of the particles

Dp has been calculated based on the granulometric distribution found in (Alonso-Riano, et al., 2020). Typical values for L/D, G, S and N have also been found in Perry's Chemical Engineering Handbook, and several values of them were tried until a satisfactory residence time was found. Using air at 80°C at the inlet, with humidity of 0,015 kg/ water/kg of dry air, an outlet temperature for the solid was surmised, equal to the wet bulb temperature of air at the inlet conditions. Calculated in this way, the sensible heat aquired by the solid has been found to be negligible with respect to the heat of vaporisation required to achieve the desired moisture content; under this hypothesys, the transformation of air in the dryer can be approximately considered adiabatic. This in turn allows us to calculate the dry bulb temperature of air at the outlet using a psychometri chart, starting from the inlet point, and proceding along the saturation line until the desired level of humidiry ratio is achieved. In the next step, the temperature inside the dryer was assumed to be the average of the temperature of air at the inlet and the temperature of air at the outlet. The drying time for air at this new temperature was calculated, as before, sugin the drying curves according to the Page model. The procedure was repeated, until the calculated average temperature inside the dryer was similar enough to the temperature used to model it. The sizing of the final iteration was taken as definitve sizing. The number and size of flights were determined upon the diameter of the dryer, with typical values also found in Perry's Chemical Engineering Handbook. The obtained values for the dryer are: 0.5 m of diametre, 4 m of length, 40 RPM of speed, 6.25 cm of fliers height, and a slope of 2 cm per m of length. The dryer treats 162 kg/h of feed stock and is able to process up to 232 kg/h. At nameplate capacity the flowrate of air needed is 590 kg/h, and 842 kg/h at maximum capacity.

1.15.6 CY-101

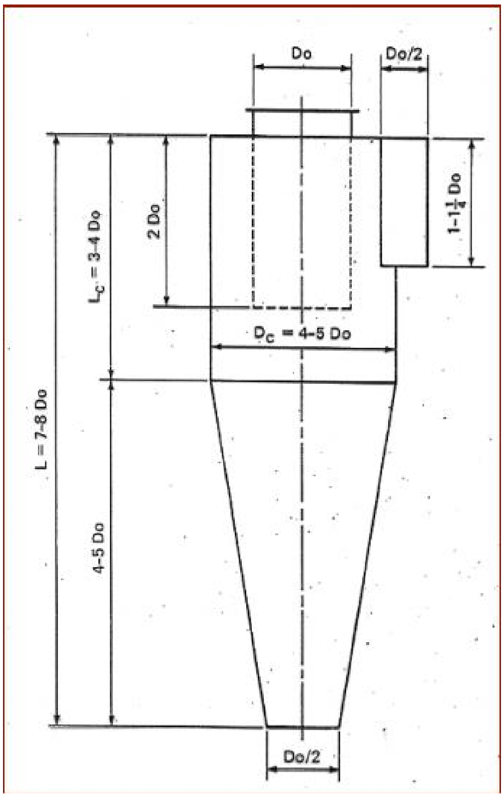


Figure 6-2 Cyclone normogram

Cyclone CY-101 has been sized according to normograms and hypothesys on the entrained solid. Different percentage of solid entrained were surmised for each size class of the granulometry found in (Alonso-Riano, et al., 2020). Removal efficiencies were surmised to be close to units, given the relatively important size of the particles. Using a typical value of 25 m/s of air at the inlet of the cyclone, and given the air flowrate to be treated, it has been possible to calculate the inlet diameter of the cyclone. The same diameter was sued to size the rest of the cyclone, based on common normographs available for this type of equipment. Based upon geometry and velocities inside the cyclone, it has been possible to surmise the pressure drops along the cyclone, making use of the model available in (Karagoz & Avci, 2007). The model consists in a tedious series of relationships based only on the inlet conditions and the geometry of the equipment. The removal efficiency of the cyclone was surmised to be virtually unitary. The cyclone has an height of 1.4 m and a diametre of 0.9 m, with a feed opening of 18 cm in diametre.

1.15.7 Pneumatic transport PL-101

The pneumatic line transports the dried BSG from DD-101 to the subsequent steps of the process. The fluid used is N2 to prevent explosion risks. Given the cost of the gas, its circuit is a closed one. The sizing of the pneumatic transport line is not unlike the one used for the Seepex SAI (cap. 6.3.4) so it will not be reported here again. The line uses 4.5 kg of N2 for each kg of solid transported, for a total N2 flowrate of 465 kg/h. The compressor power is equal to 26 kW. The flowrate from the pneumatic transport is fed to the cyclone C-102 to separate the solid from the N2, which is then recompressed and reused.

Pneumatic transportation is also used elsewhere in the plant. The criteria and the procedure for its sizing therefore will not be reported for brevity. The specifics of said equipment will be found in the equipment list.

1.15.8 CY-102

Cyclone CY-102 separates the solid from the N2 used in PL-101. The sizing of the cyclone is not unlike that of CY-101 (cap 6.3.6) so it will not be reported here again. The cyclone processes 465 kg/h of N2 and 103 kg/h of solid. The cyclone has an height of 0.8 m and a diametre of 0.5 m, with a feed opening of 10 cm in diametre.

In combination with other pneumatic transport equipment, other cyclones are present elsewhere in the plant: therefore the criteria and the procedure for its sizing will not be reported for brevity. The specifics of said equipment will be found in the equipment list.

1.15.9 BF-101

The blowing fan BF-101 is employed to move the air to the burner B-101, where it is mixed and burned with the methane coming from tank TK-101. The flowrate of external air at maximum capacity is 842 kg/h, and the installed power of the equipment is 12.5 kW if a pressure loss of 0.1 bar is surmised in the line.

1.15.10 B-101

The burner B-101 is fed with an external air flowrate from BF-101 equal to 842 kg/h. If the external air temperature is assumed to be equal to 25°C, to get an outlet at 80°C, a flowrate of around 1 kg/h of methane is required. This can be calculated with a simple enthaply balance at the burner, assuming a burning efficiency of 0.9. The burner works with a gas velocity of 2 m/s.

1.15.11 TX-201

The battery of Turbex cavitationl extractor TX-201 allows for the extraction of polyphenols from the treated BSG, using as solvent an aqueous solution of ethanol (60% v/v). The solvent to solid ratio used in the turbex is set equal to 10. The solid flowrate to be treated at nameplate capacity, coming from the previous part of the plant, is equal to 72 kg/h, but the section must be able to handle the flowrate at the maximum turnover ratio, that is 103 kg/h. The feedstock has a residual humidity of 10% by weight. Before being fed to the turbex, the solid flowrate must be soaked with a liquid current, wich is obtained by withdrawing a fraction of the extract coming out of the turbex. The solid feedstock is deposited in tank D-201, where it accumulates while being soaked by said stream; the vessel is agitated and the ratio between solid and liquid in the soaking step is set to 4. Tank D-201 operates in a batch modality, with a soaking time of around 20 minutes. At the end of the soaking step, a time-actioned valve releases the soaked solid in the tank D-202, where a weight-in-loss control based on the weight reading of a load cell, which allows for a steady feeding of the matrix to the turbex TX-201. For the calculation of the Turbex yields, there was data available both from the laboratory experiments performed and from a test on the turbex. From the laboratory experiments, is has been possible to calculate the partition constants of the various compounds of the BSG for the extraction with ethanolic solution. Comparing the data obtained from the polyphenols extraction in the case of the laboratory experiment and the turbex experiment, it has been possible to show that the Turbex is able to perform around 10 theoretical equilibium extraction stages. For the other compunds, where only data about the laboratory experiments were available, it has been surmised that the turbex is able to operate with the same number of equilibrium stages as for the polyphenols, thus allowing to calculate the extraction yields of the other components and the composition of the extract. The feedstock has a relatively low content of polyphenols (around 0.5 % d.w.), wich is increased to 2.5% in the extract. The extract presents a high percentage of cellulose, sugars and proteins, so the extract needs to undergo purification steps in the following membrane section. The nameplate capacity of the Turbex is 500 kg/h.

1.15.12 Centrifugal decanter CD-201

The centrifugal decanters collect the solid coming out of the turbex, which retains, at this stage, about 60% by weight of solvent. The decanters are an integral part of the turbex section, and one is present for every installed turbex extractor. By means of its rotation, the decanter is able to apply a centrifugal force on the fed stream, and to induce a separation of solid and liquid by virtue of their relative density difference. In the laboratory experiments, the centrifugation step was carried out in a laboratory centrifuge with a RCF of 15000. Given a diametre of around 40 cm for said centrifuge, it has been surmised that the centrifugal decanter must operate with around 1800 RPM. The solid leaving the decanter still retains a content of liquid of around 40% by weight, which will be further reduced in the following squeezing presses, also a fundamental part of the turbex section. The separated liquid, along with the liquid stream recovered in the squeezing press, is fed to the surge tank D-203, from which it will be recycled as solvent in the extraction step.

1.15.13 Squeezing press SP-201

The squeezing press realises a further sepration of liquid and solid, after the centrifugal decanters. The application of a mechanical force squeezes the liquid out the solid, reducing its liquid content. The solid comes with a concentration of liquid of around 40% by weight. The squeezing press (one for each Turbex) further reduces said amount to the amount equal to the imbibition ratio of the solid. The solid coming out of the squeezing presses ha a content of liquid of around 30% by weight. Along with the liquid coming out of the previous centrifugal decanters, the removed liquid is fed to the tank D-203, from which it is recycled back as solvent in the turbex section, along with a stream of fresh solvent coming from the surge tank D-204. The separated solid is in turn fed to the next section of the plant, where it will undergo a washing and the subsequent extraction steps.

1.15.14 Liquid collection tank D-203

The tank D-206 collects the liquid separated from the solid coming out of the Turbex, obtained from the centrifuge decanter DC-201 and the squeezing press SP-201. From the tank, the centrifugal pump P-202 withdraws a stream which is fed, along with fresh solvent from D-204. This allows for a recycle of the solvent and a better overall performance of the extraction section. The tank has been designed to hold enough liquid to be able to satisfy the liquid demand for 12 hours, in case any problem with the previous parts of the plant would arise. This allowed to calculate a volume for the tank of about 1000 litres.

1.15.15 UF-201 and NF-201

The membrane section of the process has the aim of increasing the product purity in polyphenols and to reduce the amount of solvent in the product. Choosing a membrane with a molecular cut-off lower than the average molecular weight of the cellulosic material allows to obtain a retentate with low cellulose content and richer in the products of interest. Membrane separation is a unit operation that takes advantage of the trans-membrane pressure as its driving force, which allows the solvent to move through a semi-permeable membrane, along with the solutes whose molecular weight is below the cut-ff of the membrane. Membranes are porous, and it is actually through these porosities that the solvent and the light solutes are able to permeate. It's usually considered a good practice to select a membrane whose pores have size about one tenth of the molecule to be removed

(Cheryan, 1998). The membrane filtration is structured in two steps: an ultrafiltration step (cut off 1500 Da) and a nano-filtration (cut-ff 200 Da). The permeate from the UF is fed to the NF, from which in turn a polyphenols-rich retentate is obtained, which will in turn be further concentrated with an evaporator and a drum dryer. Membranes are an interesting choice for their low encumbrance, availability on the market, low operative and chemicals cost. The choice of the working conditions of the membrane is based on technical and econimical considerations that will be explained in this paragraph. The feed pressure is instead chosen according to the manufacturer reccomendations: the pressure is set to be about 80% of the maximum allowable value of the module. To start with the sizing of the membranes, it's necessary to fix a ratio between permeate and feed flowrates, or alternatively, a Volume Concentration Ratio; in both cases, they allow us to calculate the flowrate of either permeate or retentate based on the volumetric flowrate of the feed. This parameter is a desinger's choice. It is necessary to know the rejection coefficients of the various solutes to caclulate their concentration in the product streams. The rejection coefficient is a value beteween 0 and 100%, the higher the more pronunciated is the tendency of the solute to stay in the retentate. The concentrations for solutes in the retentate are given by:

$$C_{A,retentate} = (1 - \frac{Q_{permeate}}{Q_{feed}})^{-R_A} \quad (6.8)$$

Where:

- $C_{A,retentate}$ is the concentration of solute A in the retentate stream
- $Q_{permeate}$ is the volumetric flowrate of the permeate
- Q_{feed} is the volumetric flowrate of the feed
- R_A is the rejection coefficient of solute A

With this information it's possible to determine the mass balances over the membranes. The ratio between the feed and the permeate allows to calculate the solvent flowrate in these two currents: if density variations with the solutes' concentration is neglected, this also allows to calculate the retentate solvent flowrate by difference. Knowing the solvent flowrate in the retentate and the solutes' concentration in the same current, it's possible to close the mass balances for every current.

$$Q_{feed}C_{A,feed} = Q_{permeate}C_{A,permeate} + Q_{retentate}C_{A,retentate}$$

To calculate the minimum area necessary to achieve the desired permeate and retentate flow, it is necessary to have data on the permeability of the membrane, that is to say, the permeate volumetric flowrate passing through the unit area of membrane in the unit time, per unit of driving force (trans-membrane pressure).

$$A = \frac{Q_{fEED}}{\varphi\, TMP} \quad (6.9)$$

Where:

- φ is the solvent permeability
- TMP is the trans-membrane pressure

Knowing the amount of driving force and the required volumetric flowrate of permeate, the minimum area of the membrane has been calculated. This area needs to be further increased; membranes accumulate fouling the hinder their performance, to it's necessary to proceeed with periodical cleaning of the membranes modules. For this reason, the number of real modules is higher than the ideal one. In this way, the feed can be treated without interruptions even when some of the modules require to stop. A module enters the cleaning phase when a decrease of more than 20% of the nomial permeate flowrate is registered. At this point the module is isolated from the rest of the rack my means of shut-off valves, and starts the cleaning routine. The cleaning routines are usually conducted as per reccomendation of the manufacturer, and incluedes clean water cleanings, alternated with a caustic and a acid cleaning. The solutions have been surmised to be able to remove a 0.01 kg/m2 of fooling in the case of the basic solution and 0.005 kg/m2 in the case of the acid solution, for each cleaning. After the cleaning, the solutions are returned to their storage vessel to be used again. The solution can accumulate a certain amount of fouling before becoming too dirty to be used; at this point they are discharged. Using a maximum tolerable solid concentration of 5 kg/m3, it's possible to surmise the number of cleaning cycles a solution can undergo. An operational time has also been surmised. To do so, it is necessary to obtain data on the fouling behaviour of the membrane. This data is usually presented in the literature as filtrate flowrate as a function of time. The logarithm of the ratio between initial permeate flux and permeate flux at a generic time is a linear function, whose angular coefficient is called fouling factor. This allows to estimate the time at which the permeate flux will decrease to 80% of the initial value. Regarding the best set of conditions for the membranes, the procedure followed has beeen the following. For both the UF and NF steps, several combination of values of the ratio ${Q_{permeate}}/{Q_{feed}}$ for both membrane steps have been evaluated.. For each combination, the profitability of the section has been evaluated based on profits and costs:

- The revenue of the product sales. This has been calculated as the amount of the polyphenolic-rich material obtained (considering 10% final moisture of the product) times the price of the materiale per kg. This in turn has been estimated as a linear function of the product purity in polyphenols, with a price of 50 euro/kg for a product with 20% purity and of 200 euros/kg for a product with 90% purity.
- The annualised cost of the membranes, accounting for 200 euros for square metre of memrbane

- The annual cost for the chemicals necessary for the membranes' regeneration
- The annual cost for the steam needed to evaporate the residual solvent
- The annualised cost of the multiple evaporators

This allows to surmise how the profitability of the purification section varies with the ${Q_{permeate}}/{Q_{feed}}$ ratio, and to select the most profitable one, if other technical consideration do not prevent it. In this case, low values of the ratios give higher profitability, but the lowest ones give a finale product with a concentration of cellulosic material too high to be deemed acceptable. On the other hand, too high values would cause the liquid fed to the evaporators to have a high solid content that prevents the evaporators from properly functioning. The selected membranes are from the Desal GK manufacturer. They are spiral wound-membranes, easily assembled in racks.

1.15.16 Falling film evaporator EV-201

The evaporator EV-201 concentrates the feed coming from the retentate out of the nano-filtration step. The feed stream has a solid content of 2.5% by weight, with the rest of the stream composed by the hydroalcoholic solvent. The evaporator brings this value up to 50%. The heating fluid is low pressure steam. A configuration comprising multiple effects has been investigated, but given the modest flowrate of the feed stream, it has been chosen to work with a single evaporator. The equipment has been designed as a falling film evaporator, with the inlet stream fed at the top of the evaporator. In this type of equipment, the liquid adheres to the external surface of the vertical tubes, within which flows the heating fluid. The evaporator also comprises an integrated tank for the separation of the liquid from the vapour, equipped on the top with a device useful for the disengagement of liquid droplets that may be entrained by the rising vapour. This type of equipment finds a large use in the food industry for its simplicity and high overall heat transfer coefficients. Although several relationships for the calculation of the overall heat transfer coefficient are available in the literature, they are often quite cumbersome and require a large amout of data, the majority of which needs to be experimentally assessed. For this reason, its value has been chosen thanks with the aid of a graphical correlation found on Perry's Chemical Engineers' Handbook for falling film evaporator, related to the boiling temperature of the evaporated solution. The valure has then been compared to other cases found in the literature to confirm its reasonability. . The enthaply vaporisation and the boiling temperature are fixed by the the pressure and inizial molar fraction of ethanol, and can be easily read on a Ponchon-Savarit diagram. Given the duty of the LPS and the enthalpy of vaporisation of the mixture, it is possible to calculatethe amount of solvent vaporised, making the assumption that the sensible heat transfer is negligible with respect to the latent heat. For a set amount of vaporised solvent, given the enthalpy of vaporisation of the mixture and of the LPS it is possible to calculate the amount of steam required, making the assumption that the sensible heat transfer is negligible with respect to the latent heat. The mass fractions of water and ethanol in the vapor are at the equilibrium and are the values that allow for the mass balance to close. Since the equilibrium fractions are given as a series of discrete values, it has been necessary to interpolate the equilibrium concentrations to reduce the error in the mass balance down to negligible amounts. At the end of the procedure, the mass balances obtained were double-checked with the aid of the software ASPEN Plus. Knowing the duty in the evaporator, and using the equation

$$Q = UA\Delta T \quad (6.10)$$

it's possible to determine the exchange area required. The value obtained is increased of 20% to account for the incertainty of some of the factors involved and for the froming of fouling that can decrease the heat transfer. For the design of the evaporator, it has been chosen to work with tubes of 5 inches internal diametre (12.7 cm) and of 0.75 cm in length, arranged in a triangular pitch. The required number of tubes is 11, and on this basis, given the outer diameter of the tubes and the triangular pitch, the shell diameter for the evaporator has been calculated to be of 40 cm.

1.15.17 Drum dryer DD-201

The drum dryer D-201 provides the last stage of solvent removal from the product. The solid, being fed with a solid content of 50% by weight, is processed to leave the equipment with a residual content of humidity of 2%, composed chiefly by water. The necessary flowrate of LPS has been calculated with a simple enthalpy balance, with the enthalpy necessary for the vaporisation of the solvent being provided by the condensation of the steam, an it has been calculated to be of 24 kg/h at nameplate capacity. The area necessary for the dryer has been estimated using the flowrate to be evaporated and the value of 10 kg/h of solvent evaporated per square metre of surface. Said surface has been estimated to be equal to 3.2 m^2, and a suitable dryer has been chosen from the ones available on the market: namely the equipment from Andritz, model T 5/10, with two rolls of 500 mm in diameter and 1000 mm in length.

1.15.18 HX-201

The heat exchanger HX-201 collects the evaported stream of the evaporator EV-201 and of the the drum dryer DD-201. The condensing phase (vapour solvent) flows through the shell, whereas the refrigerant liquid (water) flows through the tubes. The exchanger has been designed following the well estabilished Kern method (Kern, 1965). The enthaply balance has been redacted to calculate the necessary flowrate of water. A first overall heat transfer coefficient has been surmised, along with values for the fouling resistance. Based on this hypothesis, a first trial area has been calculated; having fixed internal diameter of the tubes and their length, the number of tubes necessary has been determined. In the cited books, several relationships allow for the calculation of the geometrical charateristics of the exchanger and, using the adimensional number Pr and Re in both tubes and shell, to calculate the heat transfer coefficients in the tube and and the shell sizes. Since the internal fluid's, external fluid's, tubes' width's and fouling's heat transfer resistances can be treated as a a seires of resistances, one can calculate a new value for the overall heat transfer coefficent U. The new coefficient can be used to repeat the same calculation in an interative way, until said value becomes aproxiately constant with each interation. The exchanger treats a current of 83 kg/h of vapour, which is totally condensed. The amount of water necessary is equal to 2250 kg/h, entering at 20°C and leaving the exchanger with a temperature of 40°C. The exchanger is composed by 200 tubes disposed in a triangual pitch, with and internal diameter of 1 inch (2.5 cm) and 1.8 metres of length, both sizes are chosen because they are easily found in commerce. The shell has an internal diameter of 0.53 m. The installation of this exchanger allows for the recovery of an importan quantity of solvent, crucial for the economicity of the process, given the high cost and taxation of ethanol.

1.15.19 Washer W-301

The washer W-301 is necessary to wash the BSG leaving the polyphenols extraction section, which carries with it imbibed liquid, composed of water and ethanol. Given the cost of ethanol, it is necessary to recover it from said stream, so that it can be recirculated in the plant. The washer is a multistage counter-current one, and the washing liquid is water. The solid is carried by a belt, and the water is sprayed from the top. The pure water is fed at the last stage. The belt is provided with holes to allow the passage of the liquid, which is collected in a tank below and pumped to the previous step, as illustrated in the figure below.

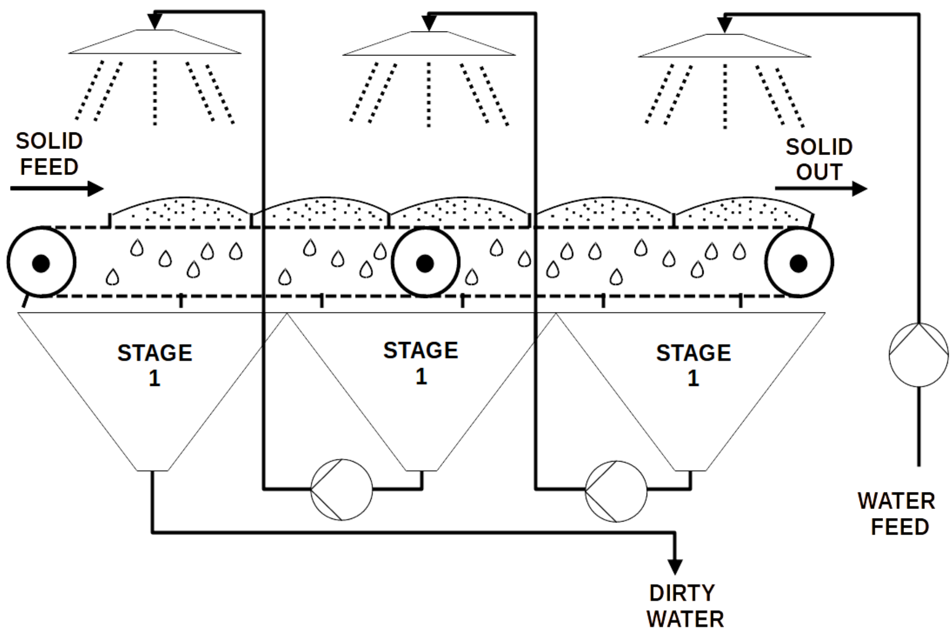


Figure 6-3 Simplified model of the counter-current multistage washer. Only three stages are represented for simplicity

It has been decided to recover 95% of the ethanol in the feed stream. With an imbition ratio of the solid of 0.4 kg liquid/kg of solid, and a L/S ratio of 3, it has been estimated that the washer must work with a number of stages equal to 6. The calculation has been carried as is customary for a solid liquid extractor, under the assumption of a repartition constant of 0.2. The water consumption is equal to around 160 kg/h when the plant operates at nameplate capacity. The recovered liquid stream has a fraction of ethanol equal to 5.3% in weight.

1.15.20 Squeezing press SP-301

The squeezing press SP-301 removes the imbibition liquid from the solid coming out of the washer W-301. The resulting liquid stream has a percentage of 1.8% of ethanol, and it is sent, along the liquid coming from the washer, to the recovery section of the plant.

1.15.21 TX-301

In this case, the yields of the Turbex section are not precisely available, since no test has been operated under the extracting conditions. Data for the equilibrium constants were obtained in the laboratory experiment for a single stage batch extraction, and the data obtained has been used to model the behavior of the turbex under the hypothesis that the equipment is, like in the case of the polyphenols extraction, able to realise 10 equilibrium stages. Data on the content of solid in the extract and in the exhausted solid has been mutuated also from the data relative to said extraction stage.The sizing of the ancillary equipment (i.e. squeezing presses SP-302, centrifugal decantners CD-301, feeder screws XF-302, and the surge tanks D-303, D-304) follows the same rationale as in the case of the turbex TX-201/202. The extractant in this case is a solution 4M of NaOH. In this case, the route of extraction differs from the one developed in (Vieira, et al., 2014), in that is composed only of one alkaline extraction step, instead of 3 with increasing alkalinity. The extractant may be also prepared with KOH, but the choice of NaOH is probabily more economical advantageous, given the lower cost and the fact that any effect on the extraction capacity should be negligible. The exhaust solid coming out of the squeezing press SP-302 is collected and sent to the next and final extraction step, whereas the liquid is sent to the next step, that is to say the acidification and protein precipitation.

1.15.22 Equipment for the preparation of the NaOH solution

The basic solution used as extractant in the TX-301 is prepared in the agitated tank D-305. This tank receives a stream of water, regulated on the basis of the liquid level. The tank D-325 contains solid NaOH, which is fed to the tank D-305 by means of the rotary valve RV-301, whose speed is regulated by a pH control system reading the pH inside D-305. It has been chosen to work with high residence times for two reasons: this allows for a better homogeneity of the solution, but more importantly allows for the NaOH to be fed slowly: the dissolution of NaOH in water is an exothermic reaction, and it is important to allow the resulting heat to be dissipiated to avoid excessive temperature increase in the tank.

1.15.23 Acidification tank D-307

In this tank, the liquid extract coming from the TX-301 extractor is acidified with a saturated solution of citric acid. The amount of citric acid solution fed to the tank must be sufficient to allow for the pH to reach a value of 3. Citric acid is a weak acid, and the theoretical calculation of the amount needed could prove to be a difficult task, especially considering the presence of different components in the solution. From the laboratory experiments, it has been necessary to add 0.9 L of saturated solution for each L of extract; this high ratio is due to the fact that NaOH is a strong base and the citric acid is a weak acid. To allow for a good homogeneity of the solution, the tank is provided with an agitator. The residence time has been set to 10 minutes. The volume of the tank, calculated on the basis of the residence time and the inlet volumetric flowrates, has been augmented of 20% as a safety measure. The tank has a volume of 350 l, with a diameter of 0.6 m and a heigth of 1.25 m.

1.15.24 Centrifugal decanters CD-302 and CD-303

The centrifugal decanter CD-302 realises the separation between the precipitated solid phase and the liquid solution, after the acidification with citric acid. The precipitated solid fraction retains a liquid content estimated in around 38% w/w, and is rich in proteins. The equipment works in a continuous fashion. Given the high tenure of solid, this fraction is withdrawn by means of a progressive cavity pump, and is conveyed to tank D-310. The liquid fraction is instead pumped by the centrifugal pump P-310 and conveyed to the D-308, where it will undergo a second acidification step. Centrifugal decanter D-303 realises instead the separation of the solid phase, rich in arabino-xylanes, from the solution after the precipitation step with ethanol in D-309. From here, the recovered solid is conveyed, by means of a progressive cavity pump to tank D-312. The recovered liquid is instead pumped by P-312 and sent to the recovery section of the plant. Based on the volumetric flowrate of the two streams, and their relative solid content, a compatible equipment has been found from vendors. The choice has been to employ the centrifugal decanter D2N as CD-302 and D2L as CD-303, both by Andritz.The chosen equipment is able to provide an acceleration of 3000 G, requires no previous thickening nor operator supervision, being fully automatised.

1.15.25 Tank D-308 for acidification with HCl

The liquid collected from the decanter CD-302 contains a high amount of sugars. To cause their precipitation, is preliminarily necessary to acidify the solution until it reaches a pH below 2. As before, given the complexity of the reactions involved in the determinatio of the pH, the need for the HCl solution has been taken as equal to the one used in the experiments, that is to say 0.2 litres of HCl 36% w/w aqueous solution for litre of solution to be acidified. To ensure the homogeneity of the pH in the solution, the tank has been provided with an impeller. The residence time has been set equal to the 10 minutes. An additional volume has been added to the volume calculated on the basis of this residence time, for safety reasons. The tank has a diameter of 0.7 m and a height of 1.5 m.

1.15.26 Tank D-312

In the tank D-312, the acidified solution withdrawn from the tank D-311 is put into contact with an ethanol solution 60% w/w, in a ratio 1/1, to cause the precipitation of the AX fraction. Except for the longer residence time (60 minutes), the procedure used for its sizing is equal to that of the other tanks, so it will not be reported again. The diameter of the tank is 1.6 m and its height 3.2 m. The outlet of the tank is directly discharged in the gravitational decanter DC-302 below.

1.15.27 Centrifuges CF-301 and CF-302

The centrifuge CF-301 treats the protein fraction coming out of the decanter DC-301. The centrifuge operates in a batch fashion, so the product is first accumulated in the buffer tank D-310. From there, is is periodically discharged in the centrifuge by means of a temporised-opening valve and actioned screw feeder. The centrifuge operates at 1200 RPM and separates the leftover liquid, leaving a solid stream containing only the imbibition liquid. Given the capacity of the centrifuge (around 80 kgs) it is necessary to accumulate 2 hours worth of material in the buffer tank, before it can be processed. The recovered liquid stream, rich in citric acid and NaOH, is sent to the recovery section of the plant, whereas the solid is fed to the buffer tank D-311 by means of the screw conveyor SC-301. The selected centrifuge has a horizontal basket of 76 cm of diameter and 30 cm of height, operating with an installed power of 3.7 kW.

The centrifuge CF-302 separates the liquid from the AX-products coming out of decanter DC-303. Also in this case, the streams coming from the decanters are first accumulated in the buffer tank D-312, from which it is discharged at regular time intervals. The same centrifuge as before is used in this case, but the accumulation time is 20 minutes, since the flowrate to be treated is bigger. The recovered liquid consists mainly in water, but it also contains ethanol, citric acid, HCl and NaOH, so it is sent to the recovery section of the plant. The solid is instead fed, by means of a screw feeder, to the buffer tank D-313.

1.15.28 Drum dryers DD-301 and DD-302

The drum dryer DD-301 receives the AX rich fraction from the buffer tank D-312, which assures a steady stream to the dryer, to which is fed by a screw conveyor. The dryer evaporates the liquid, leaving a solid product with 2% of residual liquid by weight. The calculations for the design of the dryer do not differ from the ones relative to DD-201. The contact area obtained is equal to 1.4 m2. The steam consumption is estimated to be equal to 8 kg/h.

The drum dryer DD-302 receives the protein rich fraction from the buffer tank D-311 to which is fed by a screw conveyor. The dryer evaporates the liquid, leaving a solid product with 2% of residual liquid by weight. The calculations for the design of the dryer do not differ from the ones relative to DD-201. The contact area obtained is equal to 0.8 m2. The steam consumption is estimated to be equal to 5 kg/h.

1.15.29 Distillation columns CC-401 and CC-402

Two distillation columns are present in the recovery section of the plant. The first column CC-401 receives a mixed solution containing water, ethanol, citric acid, NaCl and HCl; to this columns are fed currents originating from:

- From HX-201, i.e. the condensed solvent which was evaporated in EV-201
- Countercurrent washer W-301
- Static decanter CD-303
- Centrifuges CF-301 and CF-302
- HX-301

For this column, the fixed parametres were the ethanol recovery (99%) and the ethanol fraction in the distillate (0.6). The recovered distillate is stored in TK-401. The column treats 2656 kg/h or liquids when operating at maximum turn-over ratio, and is composed by 11 sieve trays with a diameter of 60 cm, constant along the height of the column. The bottom product is still rich in solids: the stream is thus sent to buffer tank D-403, from which in turn is sent to the centrifugal decanter CD-401 along with a current of water/ethanol to cause the precipitation of solids. The supernatant is then sent to the second distillation column. In the second column CC-402, the liquid coming from CD-401 is treated to recover a ethanol-rich distillate and a bottom product rich in citric acid. The recovery of ethanol in this column is almost unitary, and the distillate purity in ethanol has been set at 60%. This column is composed of 25 sieve trays with a constant diameter of cm. The recovered ethanolic solution is stored, whereas the bottom product is sent to the evaporator EV-401, to increase the concentration in citric acid. For both columns, the height of each stage has been assumed to be 60 cm. The procedure for the design of the column has been the same. Given the complexity of the mixtures involved, the design has been done using the ASPEN Plus software. As a first step, the DTSWU model was employed. By fixing the recovery and the purity of ethanol in the distillate, the column calculates a first version of the mass balances, as well as giving the minimum reflux ratio necessary to achieve the desired separation and the minimum number of stages. Using the same model, it is possible to tabulate the number of stages required for reflux ratios higher than the minimum one. Once that a list of reflux ratios and relative number of stages has been obtained, the simulation is repeated with the mor rigorous model RadFrac. Differently from DSTWU, this model requires as input the reflux ratio and the number of stages: one simulation is carried out for each pair obtained as described above. For each simulation, furthermore, was enabled the Tray sizing option and the utility consumption estimate. At this point, a pletora of different columns set-ups are available, each one with its number of stages, diameter, reflux ratio and utility consumption (i.e. LPS and cooling water). To choose the appropriate column, an economical evaluation has been carried out. For each possible setup, making use of the Guthrie correlations, a cost for each single plate and a cost for meter of height of the column has been calculated. The cost of each sieve tray is then multiplied by the number of stages, and the cost per metre of height is multiplied by the height of the column (which, in turn, is calculated by timing the number of real stages for the tray spacing). The cost for the trays and the cost for the column are then multiplied for the module factor and the installation factor. By summing those two costs, and actualising them with the use of the CEPCI index, we obtain the total installation cost of the column and its relative trays. After that, under the hypothesis of a depreciating period of 10 years, the cost of the installed column and trays has been devided by the depreciating period, to obtain the “economical weight” of the purchase of the equipment in a given year. The operative costs are calculated as the sum of the consumption of LPS and cooling water. By adding the capital and the oprative costs, it is possible to compare the various set ups and to individuate the reflux ratio which minimizes the sum of the costs; this reflux ratio if then chosen as the definitive one.

1.16Equipment list

Description	Tag
B-101	Burner
BF-101	Blowing fan
C-101	Air compressor
C-102	Air compressor
CY-101	Cyclone
CY-102	Cyclone
CY-103	Cyclone
F-101	Air filter
F-102	Air filter
F-103	Air filter
F-104	Air filter
H-101	Hopper
H-102	Hopper
H-103	Hopper
H-104	Hopper
H--105	Hopper
HP-10	Progressive cavity pump
HP-101	Progressive cavity pump
PT-101	Pneumatic transportation line
PT-102	Pneumatic transportation line
RD-101	Rotary dryer
RP-101	Roll press filter
S-101	Underground storage
SC-101	Screw conveyor
SS-101	Silo
XF-101	Screw feeder
XF-102	Screw feeder
XF-103	Screw feeder
CD-201	Centrifugal decanter
D-201	Soaking tank of TX-201
D-202	Feeding tank TX-201
D-203	Tank for recyled solvent of TX-201
D-204	Tank for solvent of TX-201
D-205	Buffer tank
D-206	Acid solution tank for UF section
D-207	Basic solution tank for UF section
D-208	Cleaning water tank for UF section
D-209	Acid solution tank for NF section

D-210	Basic solution tank for NF section
D-211	Cleaning water tank for NF section
D-212	Buffer tank
D-213	Buffer tank
DD-201	Drum dryer for polyphenol-rich product
EV-201	Evaporator
HP-201	Progressive cavity pump
HX-201	Shell and tubes condenser
NF-201	Nanofiltration membrane
NF-202	Nanofiltration membrane
SP-201	squeezing press of TX-201
TX-201	Turbex extractor
UF-201	Ultrafiltration membrane
UF-202	Ultrafiltration membrane
XF-202	Screw feeder
CD-301	Centrifugal decanter of TX-301
CD-302	Centrifugal decanter of proteins product
CD-303	Centrifugal decanter of AX
CF-301	Centrifuge
CF302	Centrifuge
CY-301	Cyclone
D-301	Soaking tanl of TX-301
D-302	Feeding tank of TX-301
D-303	Tank for the solvent recycling of TX-301
D-304	Tank for the solvent of tx-301
D-305	Tank for the preparation of solvent of TX-301
D-306	Buffer tank
D-307	CA acidification tank
D-308	HCl acidification tank
D-309	Tank for the mixing with EtOH
D-310	Buffer tank
D-311	Buffer tank
D-312	Buffer tank
D-313	Buffer tank
DD-301	Drum dryer for AX
DD-302	drum dryer for proteins
F-301	Filter
H-301	Hopper
HP-301	Progressive cavity pump
HP-302	Progressive cavity pump
HP-303	Progressive cavity pump
HP-304	Progressive cavity pump
HP-305	Progressive cavity pump
HX-301	Screw feeder

PL-301	Pneumatic transport line
SC-301	Screw conveyor
SC-302	Screw conveyor
SC-303	Screw conveyor
SC-304	Screw conveyor
SP-301	Squeezing press
SP-302	Squeezing press of TX-301
SS-301	Silo
TX-301	Tubex extractor
W-301	Washer
XF-301	Screw feeder
CC-401	Distillation column
CC-402	Distillation column
CD-401	Centrifugal decanter
D-401	Tank
D-402	Tank of distillate
D-403	Tank of distillate
D-404	Buffer tank
E-401	Ejector
EV-401	Evaporator
EV-402	Evaporator
EV-403	Evaporator
HX-401	Condenser
HX-402	Condenser
RB-401	Reboiler
RB-402	Reboiler
TK-401	Storage tank
TK-402	Storage tank
TK-403	Storage tank
XF-401	Screw feeder
XF-402	Screw feeder
P-201	Centrifugal pump
P-202	Centrifugal pump
P-203	Centrifugal pump
P-204	Centrifugal pump
P-205	Centrifugal pump
P-206	Centrifugal pump
P-207	Centrifugal pump
P-208	Centrifugal pump
P-209	Centrifugal pump
P-210	Centrifugal pump
P-211	Centrifugal pump
P-212	Centrifugal pump
P-213	Centrifugal pump

P-212	Centrifugal pump
P-301	Centrifugal pump
P-302	Centrifugal pump
P-303	Centrifugal pump
P-304	Centrifugal pump
P-305	Centrifugal pump
P-306	Centrifugal pump
P-307	Centrifugal pump
P-308	Centrifugal pump
P-309	Centrifugal pump
P-310	Centrifugal pump
P-311	Centrifugal pump
P-312	Centrifugal pump
P-313	Centrifugal pump
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
VP-201	Vacuum pump

1.17 Line list

1.17.1 Section 1 line list

Stream number	Flowrate kg/h	Temperature, °C	Pressure, bar	Phase(s)	Water	Polyphenols	AX	Proteins	Cellulose	Monosaccharides	Ash an lipids	Air	Methane	N2	Ethanol	Citric acid	HCl
1001	200,00	30	1	S	72,20%	0,15%	8,31%	7,20%	3,46%	1,54%	7,14%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
1002	200,00	30	1	S	72,20%	0,15%	8,31%	7,20%	3,46%	1,54%	7,14%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
1003	200,00	30	1	S	72,20%	0,15%	8,31%	7,20%	3,46%	1,54%	7,14%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
1004	860,70	137	4	S, V	16,78%	0,03%	1,93%	1,67%	0,80%	0,36%	1,66%	76,76%	0,00%	0,00%	0,00%	0,00%	0,00%
1005	200,00	30	1	S	72,20%	0,15%	8,31%	7,20%	3,46%	1,54%	7,14%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
1006	139,00	30	1	S	60,00%	0,21%	11,96%	10,36%	4,98%	2,22%	10,28%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
1007	139,00	30	1	S	60,00%	0,21%	11,96%	10,36%	4,98%	2,22%	10,28%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
1008	598,19	137	4	S, V	13,94%	0,05%	2,78%	2,41%	1,16%	0,52%	2,39%	76,76%	0,00%	0,00%	0,00%	0,00%	0,00%
1009	605,99	137	4	S, V	14,54%	0,05%	2,90%	2,51%	1,21%	0,54%	2,49%	75,77%	0,00%	0,00%	0,00%	0,00%	0,00%
1039	459,19	170	4	V	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%
1010	61,78	35	1	S	10,00%	0,48%	26,90%	23,30%	11,20%	5,00%	23,12%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
1011	340,95	145	4	S, V	1,81%	0,09%	4,87%	4,22%	2,03%	0,91%	4,19%	0,00%	0,00%	81,88%	0,00%	0,00%	0,00%
1012	55,60	35	1	S	10,00%	0,48%	26,90%	23,30%	11,20%	5,00%	23,12%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%

Stream number	Flowrate kg/h	Temperature, °C	Pressure, bar	Phase(s)	Water	Polyphenols	AX	Proteins	Cellulose	Monosaccharides	Ash an lipids	Air	Methane	N2	Ethanol	Citric acid	HCl
1013	340,95	145	4	S, V	1,81%	0,09%	4,87%	4,22%	2,03%	0,91%	4,19%	0,00%	0,00%	81,88%	0,00%	0,00%	0,00%
1014	50,04	35	4	S	10,00%	0,48%	26,90%	23,30%	11,20%	5,00%	23,12%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
1015	61,78	35	1	S	10,00%	0,48%	26,90%	23,30%	11,20%	5,00%	23,12%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
1016	660,70	25	1	V	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%
1017	660,70	170	4	V	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%
1018	660,70	170	4	V	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%
1019	459,19	25	1	V	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%
1020	459,19	170	4	V	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%
1021	505,28	25	1	V	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%
1022	505,28	80	1	V	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%
1023	0,62	25	1	V	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%
1024	505,90	80	1	V	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	99,88%	0,12%	0,00%	0,00%	0,00%	0,00%
1025	588,68	70	1	S, V	14,49%	0,01%	0,28%	0,24%	0,12%	0,05%	0,24%	84,46%	0,00%	0,00%	0,00%	0,00%	0,00%
1026	6,18	35	1	S	10,00%	0,48%	26,90%	23,30%	11,20%	5,00%	23,12%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
1027	7,80	35	1	S	10,00%	0,48%	26,90%	23,30%	11,20%	5,00%	23,12%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%

Stream number	Flowrate kg/h	Temperature, °C	Pressure, bar	Phase(s)	Water	Polyphenols	AX	Proteins	Cellulose	Monosaccharides	Ash an lipids	Air	Methane	N2	Ethanol	Citric acid	HCl
1028	580,88	70	1	V	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%
1029	580,88	70	1	V	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%
1030	1,62	35	0	S,V	14,49%	0,01%	0,28%	0,24%	0,12%	0,05%	0,24%	84,46%	0,00%	0,00%	0,00%	0,00%	0,00%
1031	285,35	145	4	S,V	0,22%	0,01%	0,58%	0,50%	0,24%	0,11%	0,50%	0,00%	0,00%	97,84%	0,00%	0,00%	0,00%
1032	6,18	35	1	S	10,00%	0,48%	26,90%	23,30%	11,20%	5,00%	23,12%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
1033	285,35	145	4	S,V	0,22%	0,01%	0,58%	0,50%	0,24%	0,11%	0,50%	0,00%	0,00%	97,84%	0,00%	0,00%	0,00%
1034	0,06	35	1	S	10,00%	0,48%	26,90%	23,30%	11,20%	5,00%	23,12%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
1035	279,24	170	4	V	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%
1036	279,24	170	4	V	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%
1037	11,74	0	0	S	10,00%	0,48%	26,90%	23,30%	11,20%	5,00%	23,12%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
1038	279,24	170	4	V	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%
1039	61,00	25	1	L	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%

1.17.2 Section 2 line list

Stream Number	Flowrate, kg/h	T, °C	P, bar	Phase	Water, %	Polyphenols, %	AX, %	Proteins, %	Cellulose, %	Monosaccharides, %	Air, %	Methane, %	N2, %	Ethanol, %	Citric Acid, %	HCl, %	NaOH, %
2001	61,8	35	1	S	10,00%	0,48%	26,90%	23,30%	16,20%	23,12%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
2002	61,8	35	1	S	10,00%	0,48%	26,90%	23,30%	16,20%	23,12%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
2003	815,9	35	1	S, L	39,37%	0,04%	0,11%	0,52%	0,86%	0,03%	0,00%	0,00%	0,00%	59,06%	0,00%	0,00%	0,00%
2004	114,2	35	1	S, L	27,06%	0,05%	13,96%	9,86%	14,05%	2,56%	0,00%	0,00%	0,00%	32,47%	0,00%	0,00%	0,00%
2005	66,6	35	1	S, L	13,91%	0,08%	23,93%	16,90%	24,09%	4,39%	0,00%	0,00%	0,00%	16,70%	0,00%	0,00%	0,00%
2006	66,6	35	1	L	45,45%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	54,55%	0,00%	0,00%	0,00%
2007	20,0	35	1	L	45,45%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	54,55%	0,00%	0,00%	0,00%
2008	86,6	35	1	L	45,45%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	54,55%	0,00%	0,00%	0,00%
2009	617,8	35	1	L	40,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	60,00%	0,00%	0,00%	0,00%
2010	565,4	35	1	L	39,34%	0,04%	0,12%	0,56%	0,91%	0,03%	0,00%	0,00%	0,00%	59,00%	0,00%	0,00%	0,00%
2011	250,5	35	1	L	39,34%	0,04%	0,12%	0,56%	0,91%	0,03%	0,00%	0,00%	0,00%	59,00%	0,00%	0,00%	0,00%
2012	570,2	35	1	L	39,54%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	60,46%	0,00%	0,00%	0,00%
2013	570,2	35	1	L	39,54%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	60,46%	0,00%	0,00%	0,00%
2014	565,4	35	1	L	39,34%	0,04%	0,12%	0,56%	0,91%	0,03%	0,00%	0,00%	0,00%	59,00%	0,00%	0,00%	0,00%

Stream Number	Flowrate, kg/h	T, °C	P, bar	Phase	Water, %	Polyphenols, %	AX, %	Proteins, %	Cellulose, %	Monosaccharides, %	Air, %	Methane, %	N2, %	Ethanol, %	Citric Acid, %	HCl, %	NaOH, %
2015	565,4	35	2	L	39,34%	0,04%	0,12%	0,56%	0,91%	0,03%	0,00%	0,00%	0,00%	59,00%	0,00%	0,00%	0,00%
2016	501,5	35	1	L	39,91%	0,04%	0,10%	0,06%	0,01%	0,02%	0,00%	0,00%	0,00%	59,87%	0,00%	0,00%	0,00%
2017	565,4	35	2	L	39,34%	0,04%	0,12%	0,56%	0,91%	0,03%	0,00%	0,00%	0,00%	59,00%	0,00%	0,00%	0,00%
2018	501,5	35	1	L	39,91%	0,04%	0,10%	0,06%	0,01%	0,02%	0,00%	0,00%	0,00%	59,87%	0,00%	0,00%	0,00%
2019	501,5	35	1	L	39,91%	0,04%	0,10%	0,06%	0,01%	0,02%	0,00%	0,00%	0,00%	59,87%	0,00%	0,00%	0,00%
2020	370,7	80	7	0	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%
2021	370,7	80	7	L	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%
2022	370,7	80	7	L	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%
2023	370,7	80	7	L	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%
2024	370,7	80	7	L	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%
2025	370,7	80	7	L	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%
2026	4156,2	25	1	L	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%
2027	4156,2	25	1	L	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%
2028	206,4	158	5	V	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%
2029	206,4	155	5	L	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%

Stream Number	Flowrate, kg/h	T, °C	P, bar	Phase	Water, %	Polyphenols, %	AX, %	Proteins, %	Cellulose, %	Monosaccharides, %	Air, %	Methane, %	N2, %	Ethanol, %	Citric Acid, %	HCl, %	NaOH, %
2030	370,7	80	7	L	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%
2031	371,0	80	7	L	99,89%	0,00%	0,00%	0,00%	0,10%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
2032	370,7	80	7	L	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%
2033	371,0	80	7	L	99,89%	0,00%	0,00%	0,00%	0,10%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
2034	371,0	80	7	L	99,89%	0,00%	0,00%	0,00%	0,10%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
2035	371,0	80	7	L	99,89%	0,00%	0,00%	0,00%	0,10%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
2036	4156,2	25	1	L	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%
2037	4156,2	25	1	L	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%
2038	206,4	158	5	V	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%
2039	206,4	155	5	L	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%
2040	370,7	50	7	L	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%
2041	370,9	50	7	L	99,94%	0,00%	0,00%	0,00%	0,05%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
2042	370,7	50	7	L	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%
2043	370,9	50	7	L	99,94%	0,00%	0,00%	0,00%	0,05%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
2044	370,9	50	7	L	99,94%	0,00%	0,00%	0,00%	0,05%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%

Stream Number	Flowrate, kg/h	T, °C	P, bar	Phase	Water, %	Polyphenols, %	AX, %	Proteins, %	Cellulose, %	Monosaccharides, %	Air, %	Methane, %	N2, %	Ethanol, %	Citric Acid, %	HCl, %	NaOH, %
2045	370,9	50	7	L	99,94%	0,00%	0,00%	0,00%	0,05%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
2046	4156,2	40	1	L	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%
2047	4156,2	40	1	L	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%
2048	7,6	158	5	V	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%
2049	7,6	155	5	L	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%
2050	63,9	35	1	L	34,82%	0,09%	0,30%	4,47%	8,01%	0,07%	0,00%	0,00%	0,00%	52,23%	0,00%	0,00%	0,00%
2051	63,9	35	1	L	34,82%	0,09%	0,30%	4,47%	8,01%	0,07%	0,00%	0,00%	0,00%	52,23%	0,00%	0,00%	0,00%
2052	501,5	35	7	L	39,91%	0,04%	0,10%	0,06%	0,01%	0,02%	0,00%	0,00%	0,00%	59,87%	0,00%	0,00%	0,00%
2053	501,5	35	7	L	39,91%	0,04%	0,10%	0,06%	0,01%	0,02%	0,00%	0,00%	0,00%	59,87%	0,00%	0,00%	0,00%
2054	50,6	35	1	L	39,56%	0,35%	0,14%	0,55%	0,02%	0,03%	0,00%	0,00%	0,00%	59,34%	0,00%	0,00%	0,00%
2055	501,5	35	7	L	39,91%	0,04%	0,10%	0,06%	0,01%	0,02%	0,00%	0,00%	0,00%	59,87%	0,00%	0,00%	0,00%
2056	50,6	35	1	L	39,56%	0,35%	0,14%	0,55%	0,02%	0,03%	0,00%	0,00%	0,00%	59,34%	0,00%	0,00%	0,00%
2057	50,6	35	1	L	39,56%	0,35%	0,14%	0,55%	0,02%	0,03%	0,00%	0,00%	0,00%	59,34%	0,00%	0,00%	0,00%
2058	333,6	80	7	L	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%
2059	333,6	80	7	L	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%

Stream Number	Flowrate, kg/h	T, °C	P, bar	Phase	Water, %	Polyphenols, %	AX, %	Proteins, %	Cellulose, %	Monosaccharides, %	Air, %	Methane, %	N2, %	Ethanol, %	Citric Acid, %	HCl, %	NaOH, %
2060	333,6	80	7	L	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%
2061	333,6	80	7	L	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%
2062	333,6	80	7	L	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%
2063	333,6	80	7	L	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%
2064	4156,2	25	1	L	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%
2065	4156,2	25	1	L	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%
2066	206,4	158	5	V	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%
2067	206,4	155	5	L	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%
2068	333,6	80	7	L	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%
2069	333,9	80	7	L	99,89%	0,00%	0,00%	0,00%	0,10%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
2070	333,6	80	7	L	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%
2071	333,9	80	7	L	99,89%	0,00%	0,00%	0,00%	0,10%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
2072	333,9	80	7	L	99,89%	0,00%	0,00%	0,00%	0,10%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
2073	333,9	80	7	L	99,89%	0,00%	0,00%	0,00%	0,10%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
2074	4156,2	25	1	L	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%

Stream Number	Flowrate, kg/h	T, °C	P, bar	Phase	Water, %	Polyphenols, %	AX, %	Proteins, %	Cellulose, %	Monosaccharides, %	Air, %	Methane, %	N2, %	Ethanol, %	Citric Acid, %	HCl, %	NaOH, %
2075	4156,2	25	1	L	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%
2076	206,4	158	5	V	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%
2077	206,4	155	5	L	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%
2078	333,6	40	7	L	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%
2079	333,9	50	7	L	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%
2080	333,6	50	7	L	99,94%	0,00%	0,00%	0,00%	0,05%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
2081	333,9	50	7	L	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%
2082	333,9	50	7	L	99,94%	0,00%	0,00%	0,00%	0,05%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
2083	333,9	50	7	L	99,94%	0,00%	0,00%	0,00%	0,05%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
2084	4156,2	50	7	L	99,94%	0,00%	0,00%	0,00%	0,05%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
2085	4156,2	40	1	L	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%
2086	7,6	158	5	V	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%
2087	7,6	155	5	L	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%
2088	450,9	35	1	L	39,95%	0,00%	0,09%	0,00%	0,01%	0,02%	0,00%	0,00%	0,00%	59,34%	0,00%	0,00%	0,00%
2089	450,9	35	1	L	39,95%	0,00%	0,09%	0,00%	0,01%	0,02%	0,00%	0,00%	0,00%	59,34%	0,00%	0,00%	0,00%

Stream Number	Flowrate, kg/h	T, °C	P, bar	Phase	Water, %	Polyphenols, %	AX, %	Proteins, %	Cellulose, %	Monosaccharides, %	Air, %	Methane, %	N2, %	Ethanol, %	Citric Acid, %	HCl, %	NaOH, %
2090	34,2	158	5	V	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%
2091	34,2	153	5	L	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%
2092	30,7	80	1	V	3,30%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	96,70%	0,00%	0,00%	0,00%
2093																	
2094	19,9	80	1	L	95,59%	0,90%	0,36%	1,41%	0,05%	0,09%	0,00%	0,00%	0,00%	1,61%	0,00%	0,00%	0,00%
2095	0,6	80	1	S	1,97%	31,49%	12,54%	49,36%	1,63%	2,98%	0,00%	0,00%	0,00%	0,03%	0,00%	0,00%	0,00%
2096	19,3	80	1	L	98,34%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	1,66%	0,00%	0,00%	0,00%
2097	20,7	158	5	V	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%
2098	20,7	153	5	L	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%
2099	50,0	80	1	L	39,99%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	60,01%	0,00%	0,00%	0,00%
2100	50,0	30	1	L	39,99%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	60,01%	0,00%	0,00%	0,00%
2101	0,6	80	1	S	1,97%	31,49%	12,54%	49,36%	1,63%	2,98%	0,00%	0,00%	0,00%	0,03%	0,00%	0,00%	0,00%
2102	1350,5	20	1	L	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%
2103	1350,5	40	1	L	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%

1.17.3 Section 3 line list

Stream number	Flow rate	T, °C	P, bar	Phase	Water, %	Polyphenols, %	AX, %	Proteins, %	Cellulose and other products, %	Monosaccharides, %	Air, %	Methane, %	N2, %	Ethanol, %	Citric acid, %	HCl, %	NaOH, %
3001	138,6	25	1	L	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%
3002	67,0	35	1	S	30,45%	0,08%	23,80%	16,81%	23,96%	4,37%	0,00%	0,00%	0,00%	0,53%	0,00%	0,00%	0,00%
3003	48,0	25	1	S, L	4,62%	0,11%	32,86%	23,21%	33,08%	6,04%	0,00%	0,00%	0,00%	0,08%	0,00%	0,00%	0,00%
3004	18,9	25	1	L	95,90%	0,00%	0,84%	0,59%	0,85%	0,15%	0,00%	0,00%	0,00%	1,66%	0,00%	0,00%	0,00%
3005	48,0	25	1	S	4,62%	0,11%	32,86%	23,21%	33,08%	6,04%	0,00%	0,00%	0,00%	0,08%	0,00%	0,00%	0,00%
3006	288,0	169,8263	4	S, V	0,77%	0,02%	5,48%	3,87%	5,51%	1,01%	0,00%	0,00%	83,00%	0,01%	0,00%	0,00%	0,00%
3007	43,2	25	1	S	4,62%	0,11%	32,86%	23,21%	33,08%	6,04%	0,00%	0,00%	0,00%	0,08%	0,00%	0,00%	0,00%
3008	244,8	169,8263	1	S, V	0,09%	0,00%	0,64%	0,46%	0,65%	0,12%	0,00%	0,00%	97,65%	0,00%	0,00%	0,00%	0,00%
3009	4,8	25	1	S	4,62%	0,11%	32,86%	23,21%	33,08%	6,04%	0,00%	0,00%	0,00%	0,08%	0,00%	0,00%	0,00%
3010	240,0	25	1	V	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%
3011	48,0	25	1	S	4,62%	0,11%	32,86%	23,21%	33,08%	6,04%	0,00%	0,00%	0,00%	0,08%	0,00%	0,00%	0,00%
3012	336,0	25	1	S, L	73,00%	0,02%	4,97%	3,49%	4,91%	0,90%	0,00%	0,00%	0,00%	0,01%	0,00%	0,00%	12,69%
3013	503,8	25	1	L	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%
3014	96,0	25	1	S	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%

Stream number	Flow rate	T, °C	P, bar	Phase	Water, %	Polyphenols, %	AX, %	Proteins, %	Cellulose and other products, %	Monosaccharides, %	Air, %	Methane, %	N2, %	Ethanol, %	Citric acid, %	HCl, %	NaOH, %
3015	599,8	25	1	L	84,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	16,00%
3016	599,8	25	1	L	84,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	16,00%
3017	66,6	25	1	L	84,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	16,00%
3018	666,4	25	1	L	84,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	16,00%
3019	926,2	35	1	L	80,88%	0,00%	1,78%	1,09%	0,00%	0,22%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	14,96%
3020	288,0	35	1	L	80,88%	0,00%	1,78%	1,09%	0,00%	0,22%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	14,96%
3021	76,2	35	1	S, L	73,48%	0,07%	0,25%	2,13%	8,82%	1,26%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	14,00%
3022	40,0	35	1	L	84,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	16,00%
3023	36,2	35	1	S, L	61,87%	0,15%	0,52%	4,48%	18,56%	2,65%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	11,78%
3024	26,7	35	1	L	84,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	16,00%
3025	9,5	35	1	S	0,00%	0,58%	1,96%	16,98%	70,43%	10,04%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
3026	656,9	35	1	L	79,80%	0,00%	2,40%	1,47%	1,42%	0,31%	0,00%	0,00%	0,00%	0,01%	0,00%	0,00%	14,61%
3027	656,9	35	1	L	79,80%	0,00%	2,40%	1,47%	1,42%	0,31%	0,00%	0,00%	0,00%	0,01%	0,00%	0,00%	14,61%
3028	656,9	35	1	L	79,80%	0,00%	2,40%	1,47%	1,42%	0,31%	0,00%	0,00%	0,00%	0,01%	0,00%	0,00%	14,61%
3029	463,1	25	1	L	41,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	59,00%	0,00%	0,00%

Stream number	Flow rate	T, °C	P, bar	Phase	Water, %	Polyphenols, %	AX, %	Proteins, %	Cellulose and other products, %	Monosaccharides, %	Air, %	Methane, %	N2, %	Ethanol, %	Citric acid, %	HCl, %	NaOH, %
3030	1120,0	25	1	L	63,76%	0,00%	1,41%	0,86%	0,83%	0,18%	0,00%	0,00%	0,00%	0,00%	24,40%	0,00%	8,57%
3031	28,2	25	1	S, L	37,89%	0,00%	5,85%	35,44%	3,46%	0,73%	0,00%	0,00%	0,00%	0,00%	11,03%	0,00%	5,59%
3032	1361,1	25	1	L	68,20%	0,00%	1,09%	0,01%	0,65%	0,14%	0,00%	0,00%	0,00%	0,00%	19,85%	0,00%	10,07%
3033	360,7	25	1	L	64,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	36,00%	0,00%
3034	1721,8	25	1	L	67,32%	0,00%	0,86%	0,01%	0,51%	0,11%	0,00%	0,00%	0,00%	0,00%	15,69%	7,54%	7,96%
3035	1721,8	25	1	L	40,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	60,00%	0,00%	0,00%	0,00%
3036	3443,6	25	1	L	53,66%	0,00%	0,43%	0,00%	0,26%	0,05%	0,00%	0,00%	0,00%	30,00%	7,84%	3,77%	3,98%
3037	3416,7	25	1	L	53,92%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	30,15%	7,88%	3,79%	4,00%
3038	26,9	25	1	S, L	20,60%	0,00%	54,72%	0,00%	0,33%	6,84%	0,00%	0,00%	0,00%	11,52%	3,01%	1,45%	1,53%
3039	28,2	25	1	S, L	37,89%	0,00%	5,85%	35,44%	3,46%	0,73%	0,00%	0,00%	0,00%	0,00%	11,03%	0,00%	5,59%
3040	28,2	25	1	S, L	37,89%	0,00%	5,85%	35,44%	3,46%	0,73%	0,00%	0,00%	0,00%	0,00%	11,03%	0,00%	5,59%
3041	10,3	25	1	L	69,51%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	20,23%	0,00%	10,26%
3042	18,0	25	1	S, L	19,86%	0,00%	9,19%	55,65%	5,44%	1,15%	0,00%	0,00%	0,00%	0,00%	5,78%	0,00%	2,93%
3043	18,0	25	1	S, L	19,86%	0,00%	9,19%	55,65%	5,44%	1,15%	0,00%	0,00%	0,00%	0,00%	5,78%	0,00%	2,93%
3044	5,2	158	5	V	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%

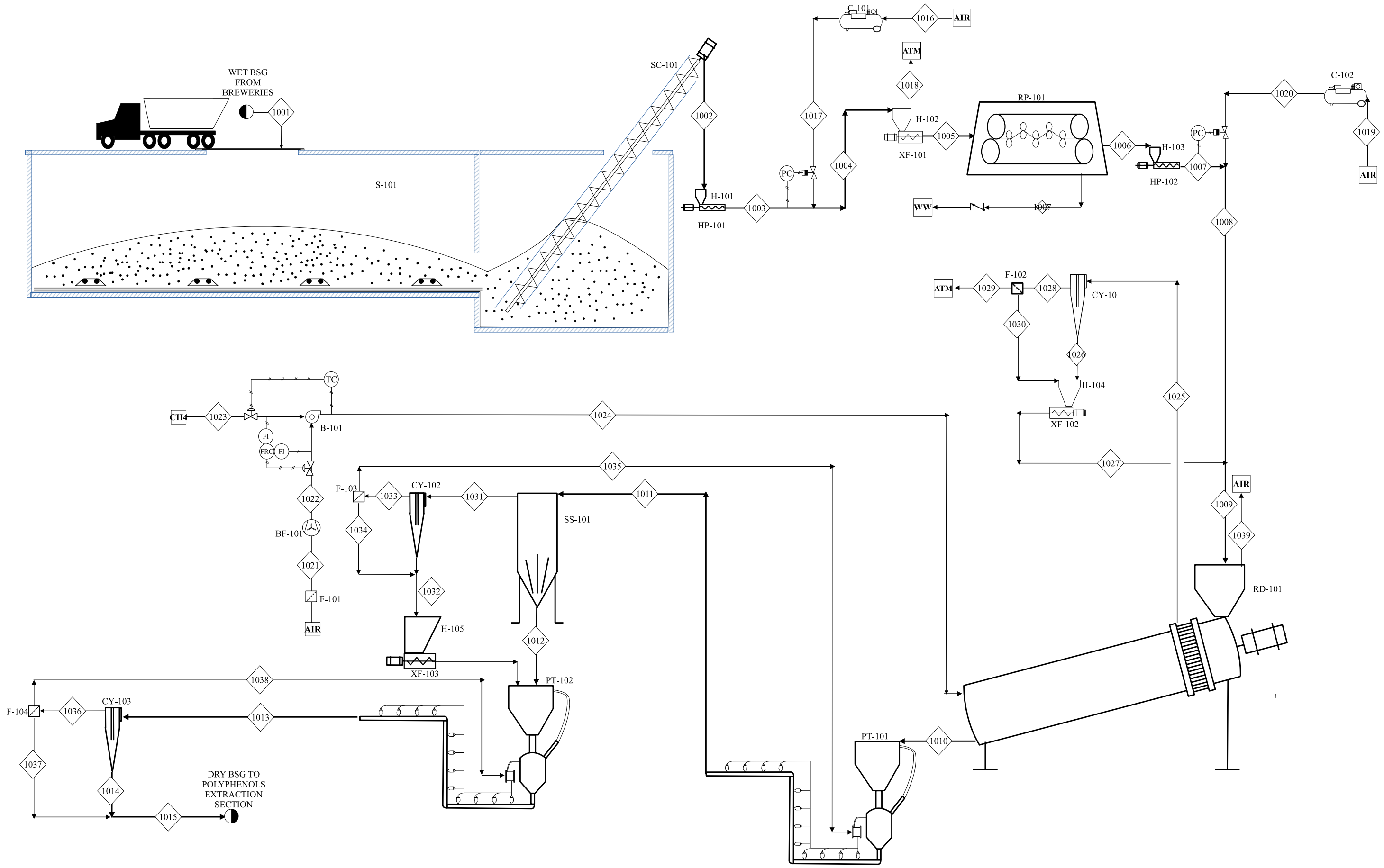
Stream number	Flow rate	T, °C	P, bar	Phase	Water, %	Polyphenols, %	AX, %	Proteins, %	Cellulose and other products, %	Monosaccharides, %	Air, %	Methane, %	N2, %	Ethanol, %	Citric acid, %	HCl, %	NaOH, %
3045	5,2	153	5	L	100,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%
3046	4,9	80	1	V	69,51%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	20,23%	0,00%	10,26%
3047	13,1	80	1	S	1,39%	0,00%	12,61%	76,35%	7,46%	1,58%	0,00%	0,00%	0,00%	0,00%	0,40%	0,00%	0,21%
3048	26,9	25	1	S, L	20,60%	0,00%	54,72%	0,00%	0,33%	6,84%	0,00%	0,00%	0,00%	11,52%	3,01%	1,45%	1,53%
3049	3,6	25	1	L	54,06%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	30,23%	7,90%	3,80%	4,01%
3050	23,3	25	1	S, L	15,45%	0,00%	63,15%	0,00%	0,38%	7,90%	0,00%	0,00%	0,00%	8,64%	2,26%	1,09%	1,15%
3051	23,3	25	1	S, L	15,45%	0,00%	63,15%	0,00%	0,38%	7,90%	0,00%	0,00%	0,00%	8,64%	2,26%	1,09%	1,15%
3052	6,8	158	5	V	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%
3053	6,3	80	1	V	54,06%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	30,23%	7,90%	3,80%	4,01%
3054	120,1	25	1	L	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%
3055	6,3	30	1	L	54,06%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	30,23%	7,90%	3,80%	4,01%
3056	120,1	40	1	L	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%
3057	17,0	80	1	S	0,74%	0,00%	86,64%	0,01%	0,52%	10,83%	0,00%	0,00%	0,00%	0,60%	0,16%	0,08%	0,08%
3058	6,8	153	5	L	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%
3059	138,3	25	1	L	92,21%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	0,00%	7,79%	0,00%	0,00%	0,00%

1.17.4 Section 4 line list

Stream number	Flowrate turnover kg/h	Temperature	Pressure	Phases	Water %	Ethanol %	CA%	HCl %	NaCl %
4001	1593,64	25,00	1,00	L	0,42	0,40	0,10	0,00	0,08
4002	1504,89	109,00	1,00	L	0,45	0,01	0,31	0,00	0,23
4003	972,43	109,00	1,00	L	0,45	0,01	0,31	0,00	0,23
4004	532,46	109,00	1,00	L	0,45	0,01	0,31	0,00	0,23
4005	1565,21	53,58	1,00	L	0,42	0,40	0,10	0,00	0,08
4006	1442,06	54,00	1,00	L	0,45	0,43	0,11	0,00	0,00
4007	793,39	25,00	1,00	L	0,59	0,00	0,41	0,00	0,00
4008	554,18	80,00	1,00	L	0,59	0,00	0,41	0,00	0,00
4009	239,21	80,00	1,00	L	0,59	0,00	0,41	0,00	0,00
4010	205,45	125,33	1,00	L	0,52	0,00	0,48	0,00	0,00
4011	171,70	92,67	0,50	L	0,43	0,00	0,57	0,00	0,00
4012	137,94	60,00	0,25	L	0,29	0,00	0,71	0,00	0,00
4013	1316,93	90,00	1,00	V	0,40	0,60	0,00	0,00	0,00
4014	1316,93	25,00	1,00	L	0,40	0,60	0,00	0,00	0,00
4015	46153,85	20,00	1,00	L	1,00	0,00	0,00	0,00	0,00
4016	46153,85	40,00	1,00	L	1,00	0,00	0,00	0,00	0,00
4017	255,75	25,00	1,00	L	0,40	0,60	0,00	0,00	0,00
4018	1061,19	25,00	1,00	L	0,40	0,60	0,00	0,00	0,00
4019	1740,00	158,00	5,00	V	1,00	0,00	0,00	0,00	0,00
4020	1740,00	153,00	5,00	L	0,00	0,00	0,00	0,00	0,00
4021	700,39	90,00	1,00	V	0,40	0,60	0,00	0,00	0,00

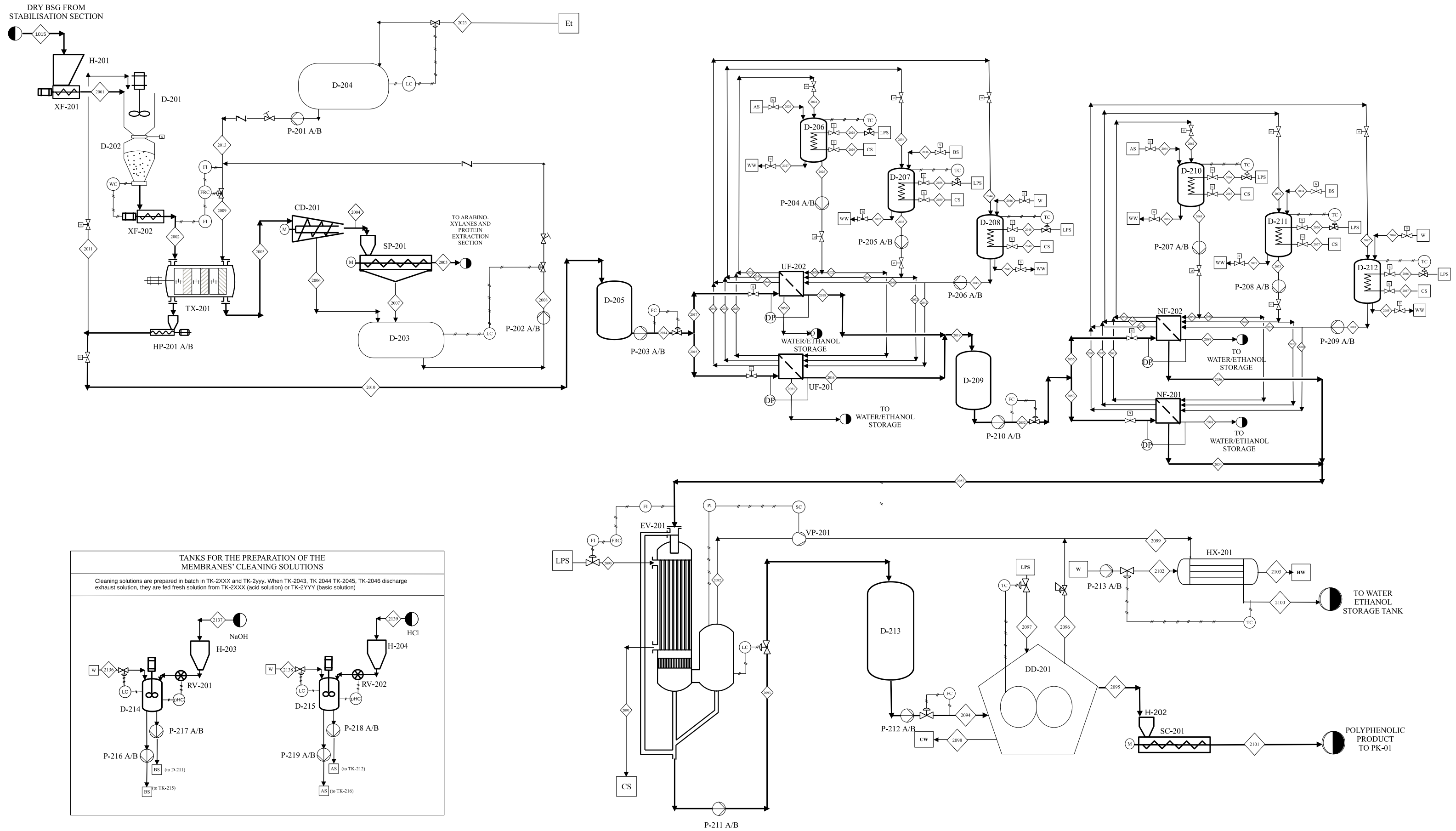
Stream number	Flowrate turnover kg/h	Temperature	Pressure	Phases	Water %	Ethanol %	CA%	HCl %	NaCl %
4022	700,39	25,00	1,00	L	0,40	0,60	0,00	0,00	0,00
4023	27692,31	20,00	1,00	L	1,00	0,00	0,00	0,00	0,00
4024	27692,31	40,00	1,00	L	1,00	0,00	0,00	0,00	0,00
4025	74,37	25,00	1,00	L	0,40	0,60	0,00	0,00	0,00
4026	626,02	25,00	1,00	L	0,40	0,60	0,00	0,00	0,00
4027	969,00	158,00	5,00	V	1,00	0,00	0,00	0,00	0,00
4028	969,00	153,00	5,00	L	1,00	0,00	0,00	0,00	0,00
4029	16,88	158,00	5,00	V	1,00	0,00	0,00	0,00	0,00
4030	27,01	133,50	0,00	V	1,00	0,00	0,00	0,00	0,00
4031	27,01	130,00	0,00	L	1,00	0,00	0,00	0,00	0,00
4032	33,76	92,67	0,00	V	1,00	0,00	0,00	0,00	0,00
4033	33,76	90,00	0,00	L	1,00	0,00	0,00	0,00	0,00
4044	27,01	85,00	1,00	L	1,00	0,00	0,00	0,00	0,00
4045	23,63	90,00	0,50	L	1,00	0,00	0,00	0,00	0,00
4046	33,76	88,00	1,00	L	1,00	0,00	0,00	0,00	0,00
4047	84,39	0,00	0,50	L	1,00	0,00	0,00	0,00	0,00
4049	23,63	92,67	0,50	V	1,00	0,00	0,00	0,00	0,00
4050	10,13	92,67	1,00	V	1,00	0,00	0,00	0,00	0,00
4051	0,00	25,00	1,00	S	0,00	0,00	1,00	0,00	0,00
4052	0,00	25,00	1,00	L	0,40	0,60	0,00	0,00	0,00
4053	1032,76	25,00	1,00	L	0,40	0,60	0,00	0,00	0,00
4054	123,16	25,00	1,00	S	0,00	0,00	0,00	0,00	0,98

1.18PFD



EQUIPMENT Legend	UTILITIES Legend	CONTROL SYSTEMS Legend	VALVES AND LINE SYMBOLS Legend
B – Inline burner BF Blowing fan C Air compressor CC Distillation column CD Centrifugal decanter CY Cyclone D Process tank DC Static decanter DD Drum dryer F Air filter EV Evaporator H Hopper HP Progressive cavity pump LP Rotatory lobe pump LF Liquid filter MF Microfiltration membrane NF Nanofiltration membrane RP Roll press filter S Undergound storage tank SC Screw conveyor SP Squeezing press SS Blender silo PT ens phase penumatic transport line VP Volumetric pump TK Storage tank TX Turbex cavitational extractor XF Feeder	AS acid solution for membranes' regeneration BS basic solution for membranes' regeneration CA Citric acid solution CS Condensend steam return Et Ethanolic aqueous solution 60% w/w NaOH *X* M sodium hydroxide solution HCl Chloridric acid solution LPS Low pressure steam W Water WW Waste water sewage disposal	CC Composition control FC Flowrate control KS time-activated control PC Pressure control PHC pHC control TC Temperature control WC Weight control	Throttling valve ON/OFF valve Rotary valve Main process line Secondary process line Control system line

AUTHOR: Roberto Chighine	DATE 17/08/2021
LOCATION: Unknown	DESCRIPTION
UNIT: BSG Stabilisation	DOCUMENT BSG-FS-001
PLANT: BSG 2000 ton/y	SHEET 1 of 4
JOB: Polyphenols, Proteins, Arabinoxylans extracts from BSG	REVISION: 2



EQUIPMENT Legend

B – Inline burner
BF Blowing fan
C Air compressor
CC Distillation column
CD Centrifugal decanter
CY Cyclone
D Process tank
DC Static decanter
DD Drum dryer
F Air filter
EV Evaporator
H Hopper
HP Progressive cavity pump
LP Rotatory lobe pump
LF Liquid filter
MF Microfiltration membrane
NF Nanofiltration membrane
RP Roll press filter
S Undergound storage tank
SC Screw conveyor
SP Squeezing press
SS Blender silo
PT ens phase penumatic transport line
VP Volumetric pump
TK Storage tank
TX Turbex cavitational extractor
XF Feeder

UTILITIES Legend

AS acid solution for membranes' regeneration
BS basic solution for membranes' regeneration
CA Citric acid solution
CS Condensend steam return
Et Ethanolic aqueous solution 60% w/w
NaOH *X* M sodium hydroxide solution
HCl Chloridric acid solution
LPS Low pressure steam
W Water
WW Waste water sewage disposal

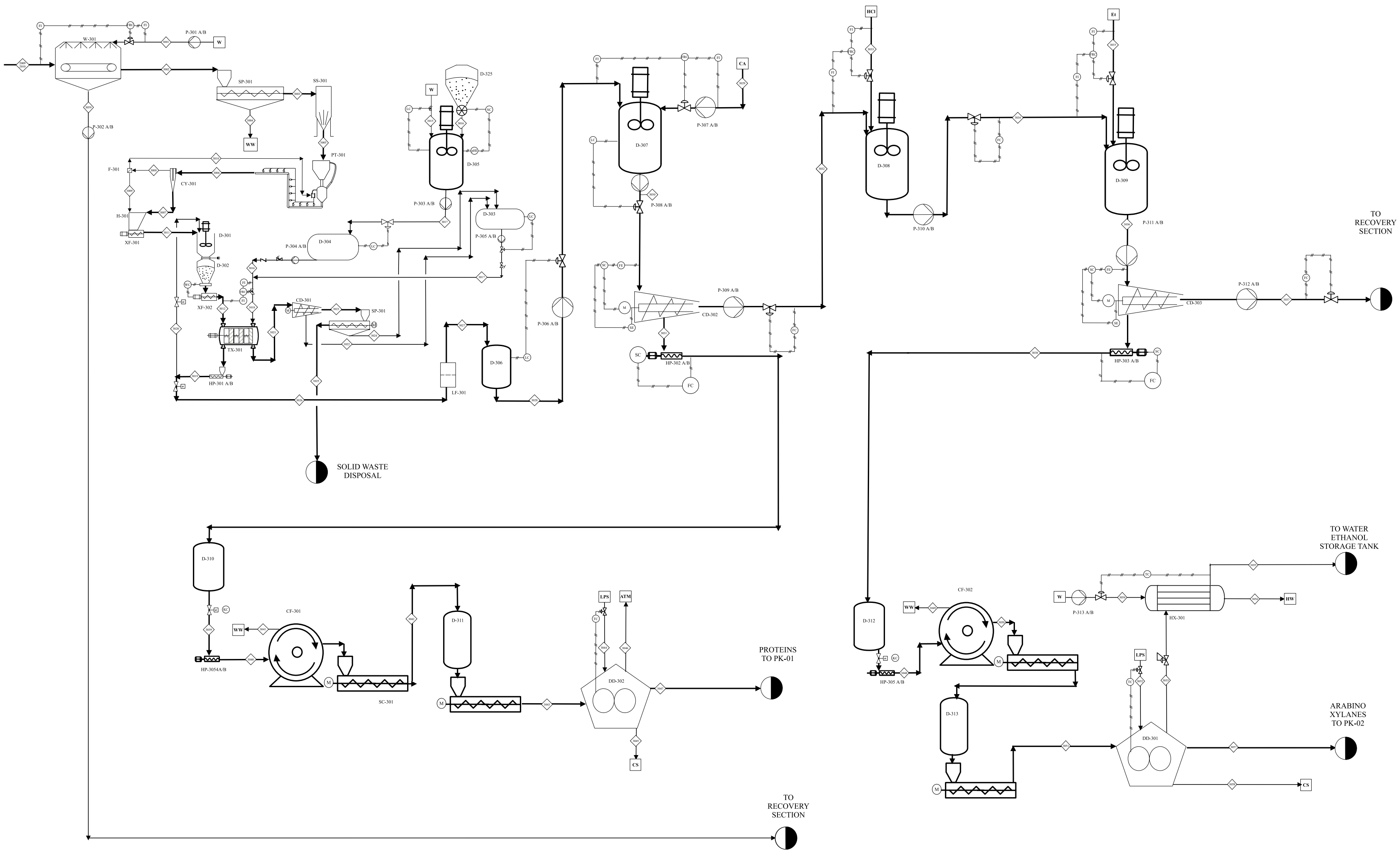
CONTROL SYSTEMS Legend

CC Composition control
FC Flowrate control
KS time-activated control
PC Pressure control
PHC pHC control
TC Temperature control
WC Weight control

VALVES AND LINE SYMBOLS Legend

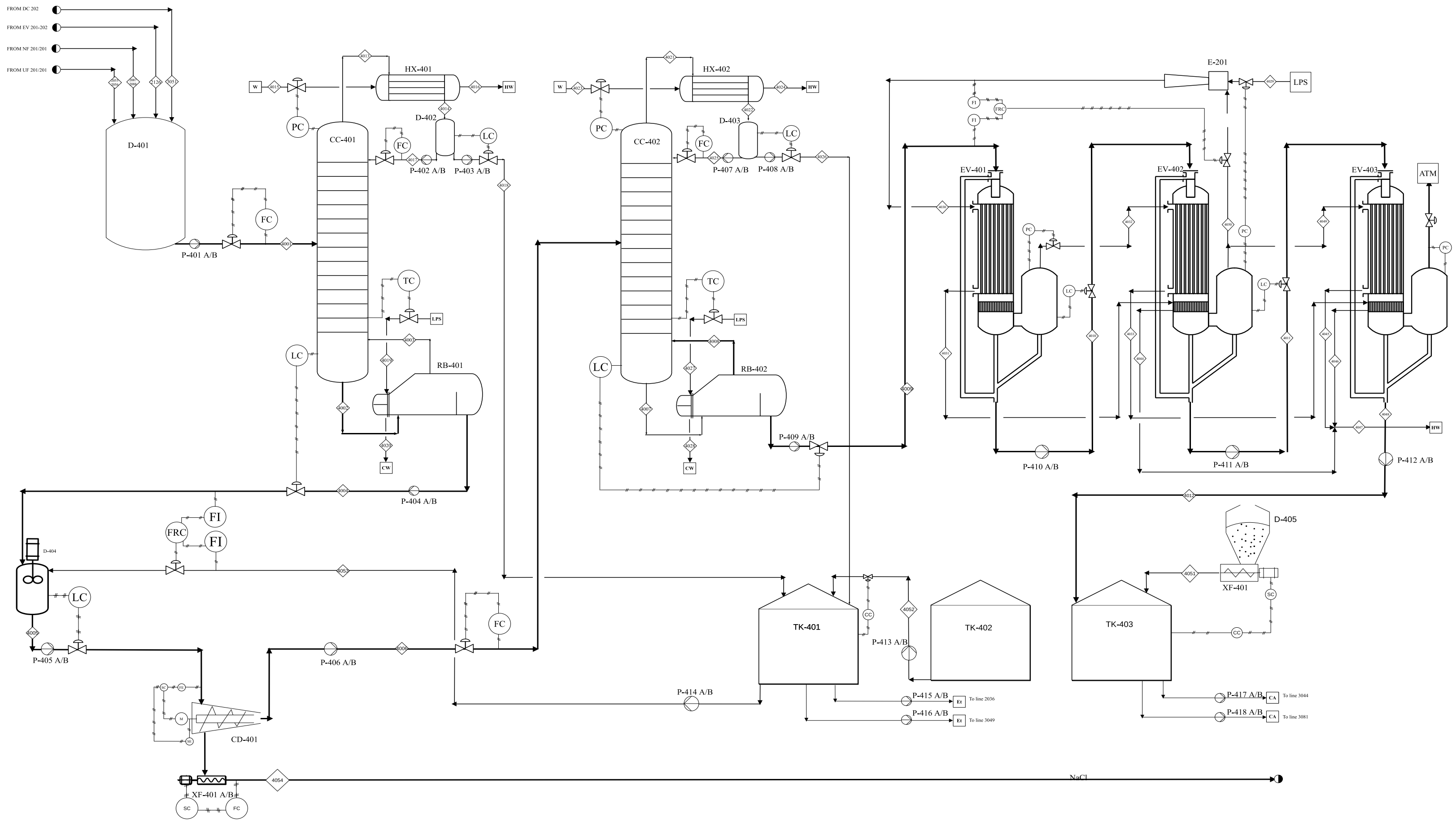
Throttling valve
ON/OFF valve
Rotary valve
Main process line
Secondary process line
Control system line

AUTHOR: Roberto Chighine	DATE 17/08/2021
LOCATION: Unknown	DESCRIPTION
UNIT: Polyphenols extraction	DOCUMENT BSG-FS-001
PLANT: BSG 2000 ton/y	SHEET 2 of 4
JOB: Polyphenols, Proteins, Arabinoxylans extracts from BSG	REVISION: 2



EQUIPMENT Legend	UTILITIES Legend	CONTROL SYSTEMS Legend	VALVES AND LINE SYMBOLS Legend
RP Roll press filter S Undergound storage tank SC Screw conveyor SP Squeezing press SS Blender silo PT ens phase penumatic transport line VP Volumetric pump TK Storage tank TX Turbex cavitational extractor XF Feeder	AS acid solution for membranes' regeneration BS basic solution for membranes' regeneration CA Citric acid solution CS Condensend steam return Et Ethanolic aqueous solution 60% w/w NaOH *X* M sodium hydroxide solution HCl Chloridric acid solution LPS Low pressure steam W Water WW Waste water sewage disposal	CC Composition control FC Flowrate control KS time-activated control PC Pressure control PHC pHC control TC Temperature control WC Weight control	Throttling valve ON/OFF valve Rotary valve Main process line Secondary process line Control system line

AUTHOR: Roberto Chighine	DATE 17/08/2021
LOCATION: Unknown	DESCRIPTION
UNIT: Protein and arabino-xylanes extraction	DOCUMENT BSG-FS-001
PLANT: BSG 2000 ton/y	SHEET 3 of 4
JOB: Polyphenols, Proteins, Arabinoxylans extracts from BSG	REVISION: 2



EQUIPMENT Legend	UTILITIES Legend	CONTROL SYSTEMS Legend	VALVES AND LINE SYMBOLS Legend
B – Inline burner BF Blowing fan C Air compressor CC Distillation column CD Centrifugal decanter CY Cyclone D Process tank DC Static decanter DD Drum dryer F Air filter EV Evaporator H Hopper HP Progressive cavity pump LP Rotatory lobe pump LF Liquid filter MF Microfiltration membrane NF Nanofiltration membrane RP Roll press filter S Undergound storage tank SC Screw conveyor SP Squeezing press SS Blender silo PT ens phase penumatic transport line VP Volumetric pump TK Storage tank TX Turbex cavitational extractor XF Feeder	AS acid solution for membranes' regeneration BS basic solution for membranes' regeneration CA Citric acid solution CW Condensend steam return Et Ethanolic aqueous solution 60% w/w NaOH sodium hydroxide solution HCl Chloridric acid solution LPS Low pressure steam W Water HW Heated water return WW Waste water sewage disposal	CC Composition control FC Flowrate control KS time-activated control PC Pressure control PHC pHC control SC Drive speed control TC Temperature control WC Weight control	Throttling valve ON/OFF valve Rotary valve Main process line Secondary process line Control system line

AUTHOR: Roberto Chighine	DATE 17/08/2021
LOCATION: Unknown	DESCRIPTION
UNIT: Ethanol and citric acid recovery	DOCUMENT BSG-FS-001
PLANT: BSG 2000 ton/y	SHEET 4 of 4
JOB: Polyphenols, Proteins, Arabinoxylans extracts from BSG	REVISION: 2

1.19Control system description

1.19.1 BSG stabilisation section control system description

- On stream 1003: a shut-off valve is open when the pressure in the stream falls below the setpoint, letting in the air coming from C-101
- On stream 1007: a shut-off valve is open when the pressure in the stream falls below the setpoint, letting in the air coming from C-102
- The flowrate of methane in stream 1023 is regulated on the basis of the temperature in stream 1024 out of burner B-101
- The flowrate of air in stream 1022 is regulated by a throttoling valve, with a flow ratio control reading the flowrate in 1022 and 1023

1.19.2 Polyphenols extraction section control system description

- The flowrate out of D-202 is regulated by a weight reading cell
- The flowrate of solvent in stream 2009 iss regulated by a throttoling valve, with a flowrate ratio control reading the flowrate in stream 2009 and comparing it with the signal from the loading cell of D-202
- A level controller reads the level in D-204 and regulates the solvent feed to the tank, stream 2023
- A level controller reads the level in D-203 and regulates the outlet flowrate, stream 2008
- The flowrate on stream 2014, out of D-205, is regulated reading the flowrate on said stream and operating a throttling valve on the same stream.
- The opening of the shut-off valves on stream 2028 and 2029 (inlet and outlet of LPS and condense to D-206) are time operated, opening before the membranes regeneration routine.
- The flowrate of LPS to D-206 (stream 2028) is regulated by a throttling valve based on the temperature reading in D-206.
- The opening of the shut-off valves on stream 2026 and 2027 (inlet and outlet of acid solution to D-206) are time operated.
- The opening of the shut-off valves on stream 2038 and 2039 (inlet and outlet of LPS and condense to D-207) are time operated, opening before the membranes regeneration routine.
- The flowrate of LPS to D-207 (stream 2038) is regulated by a throttling valve based on the temperature reading in D-209.
- The opening of the shut-off valves on stream 2036 and 2037 (inlet and outlet of basic solution to D-207) are time operated

- The opening of the shut-off valves on stream 2048 and 2049 (inlet and outlet of LPS and condense to D-208) are time operated, opening before the membranes regeneration routine.
- The flowrate of LPS to D-208 (stream 2048) is regulated by a throttling valve based on the temperature reading in D-208.
- The opening of the shut-off valves on stream 2046 and 2047 (inlet and outlet of water to D-208) are time operated
- The flowrate on stream 2052, out of D-209, is regulated reading the flowrate on said stream and operating a throttling valve on the same stream.
- The opening of the shut-off valves on stream 2066 and 2067 (inlet and outlet of LPS and condense to D-210) are time operated, opening before the membranes regeneration routine.
- The flowrate of LPS to D-210 (stream 2066) is regulated by a throttling valve based on the temperature reading in D-210.
- The opening of the shut-off valves on stream 2064 and 2065 (inlet and outlet of acid solution to D-210) are time operated.
- The opening of the shut-off valves on stream 2076 and 2077 (inlet and outlet of LPS and condense to D-211) are time operated, opening before the membranes regeneration routine.
- The flowrate of LPS to D-211 (stream 2076) is regulated by a throttling valve based on the temperature reading in D-211.
- The opening of the shut-off valves on stream 2074 and 2075 (inlet and outlet of basic solution to D-211) are time operated
- The opening of the shut-off valves on stream 2086 and 2087 (inlet and outlet of LPS and condense to D-212) are time operated, opening before the membranes regeneration routine.
- The flowrate of LPS to D-212 (stream 2086) is regulated by a throttling valve based on the temperature reading in D-212.
- The opening of the shut-off valves on stream 2084 and 2085 (inlet and outlet of water to D-212) are time operated
- The flowrate of LPS feed to EV-201, stream 2090 is regulated by a throttling valve, with a flowrate ratio control which reads the flowrated in streams 2057 and 2090.
- The velocity of the vacuum pump VP-201 on current 2092 is regulated on the basis of the pressure in EV-201
- The flowrate of 2093 is regulated on the basis of a level control reading the level on the disengagement tank of EV-201
- The flowrate on stream 2094, out of D-213, is regulated reading the flowrate on said stream and operating a throttling valve on the same stream.
- The flowrate of the LPS feed of DD-201, stream 2097, is regulated on the basis of the cylinders' skin temperature of DD-201

- The flowrate of cooling water to HX-201, stream 2102, is regulated on the basis of the temperature of the condensated liquid out of HX-201, stream 2100
- The water feed to tank D-214 is regulated on the basis of the level in the same tank.
- The velocity of the rotary valve RV-201 ir resgulated on the basis of the pH in tank D-214
- The water feed to tank D-215 is regulated on the basis of the level in the same tank.
- The velocity of the rotary valve RV-202 ir resgulated on the basis of the pH in tank D-214

1.19.3 Ax and proteins extraction section control system description

- The water feed to W-301, stream 3001, is regulated by a flowrate ratio control, reading the flowrate in the same stream and in streams 2009/2010
- The flowrate out of D-302 is regulated by a weight reading cell
- The flowrate of solvent in stream 3018 is regulated by a throttoling valve, with a flowrate ratio control reading the flowrates in stream 3018 and the signal from the load cell of D-302
- The feed of water to D-305 is regulated on the basis of the level in the same tank
- The velocity of the rotary valve RV-301 is reguated on the basis of the pH in D-305
- A level controller reads the level in D-304 and regulates the solvent feed to the tank, stream 3017
- A level controller reads the level in D-303 and regulates the outlet flowrate, streams 3017
- The flowrate on stream 3028, out of D-306, is regulated reading the flowrate on said stream and operating a throttling valve on the same stream.
- The feed flowrate of citric acid to tank D-307, stream 3029, is regulated on the basis of a flowrate ratio control, reading the flowrated in stream 3028 and 3029
- The flowrate out of D-307, stream 3030, is regulated by a throttling valve based on the level in tank D-307
- The speed of the progressive cavity pump HP-302 is regulated based on the flowrate of its stream
- The flowrate on stream 3032, out of CD-302, is regulated reading the flowrate on said stream and operating a throttling valve on the same stream.

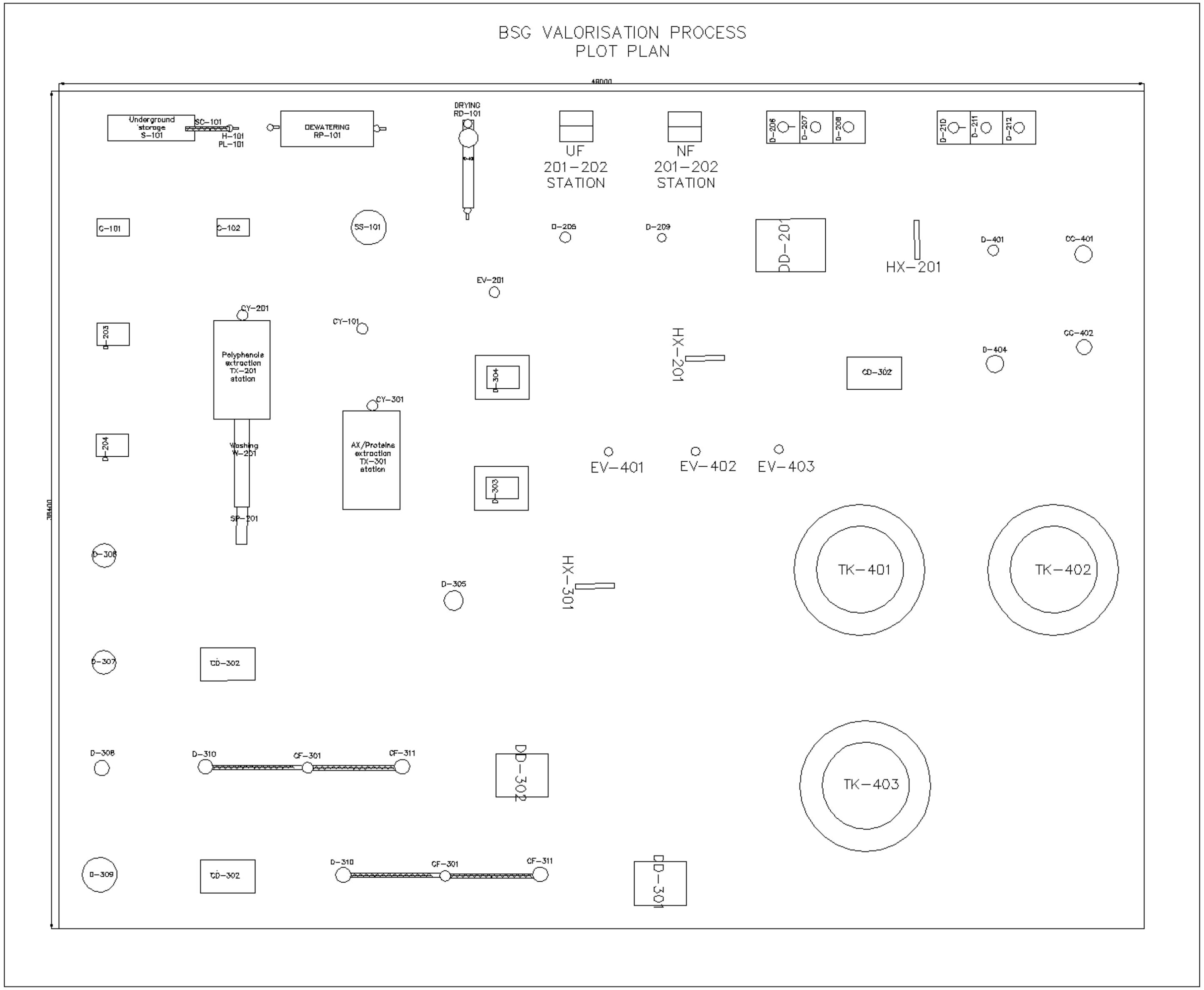
- The feed flowrate of alkaline solution to tank D-308, stream 3033, is regulated on the basis of a flowrate ratio control, reading the flowrated in stream 3032 and 3033
- The flowrate on stream 3034, out of D-308, is regulated reading the flowrate on said stream and operating a throttling valve on the same stream.
- The feed flowrate of ethanolic solution to tank D-309, stream 3035, is regulated on the basis of a flowrate ratio control, reading the flowrated in stream 3034 and 3035
- The speed of the progressing cavity pump HP-303 is regulated based on the flowrate on stream 3038
- The flowrate on stream 3037, out of CD-303, is regulated reading the flowrate on said stream and operating a throttling valve on the same stream.
- The flowrate of the LPS feed of DD-302, stream 3044, is regulated on the basis of the cylinders' skin temperature of DD-302
- The flowrate of the LPS feed of DD-301, stream 3052, is regulated on the basis of the cylinders' skin temperature of DD-301
- The cooling water feed flowrate to HX-301, stream 3054, is regulated on the basis on the temperature of the condensed liquid out of HX-301, stream 3055

1.19.4 Recovery section control system description

- The flowrate out of D-401, stream 4001, is regulated reading the flowrate on said stream and operating a throttling valve on the same stream.
- The LPS feed flowrate to RB-401, stream 4019, is regulated on the basis of the temprature on a pilot tray in CC-401
- The flowrate out of RB-401 is regulated by a throttling valve on the basis of the level reading on the bottom of the column CC-401
- The cooling water feed flowrate to HX-401, stream 4015, is regulated on the basis of the pressure reading on the top oc the column CC-401
- The reflux flowrate to column CC-401, stream 4017, is regulated by a throttling valve on the basis of the flowrate reading in the same stream
- The flowrate of the outlet stream of D-402, stream 4018, is regulated based on the the level reading in D-402
- The flowrate out of the buffer tank D-403, stream 4005, is regulated by a throttling valve on the same stream, on the basis of the level read in d-403
- The speed of the solids' screw conveyor XF-401, stream 4054, is based on a flowrate reading on the same stream
- The LPS feed flowrate to RB-402, stream 4027, is regulated on the basis of the temprature on a pilot tray in CC-402

- The flowrate out of RB-402 is regulated by a throttling valve on the basis of the level reading on the bottom of the column CC-402
- The cooling water feed flowrate to HX-402, stream 4023, is regulated on the basis of the pressure reading on the top oc the column CC-402
- The reflux flowrate to column CC-402, stream 4025, is regulated by a throttling valve on the basis of the flowrate reading in the same stream
- The flowrate of the outlet stream of D-403, stream 4026, is regulated based on the the level reading in D-403
- The LPS feed flowrate to the ejector E-201, stream 4029, is regulated on the basis of the pressure in the second stage of the multiple effect evaporator, EV-402
- The gas flowrate out of EV-401, stream 4032, is regulated basis on the pressure in EV-401
- The liquid flowrate out of EV-401, stream 4010, is regulated by a throttling valve on the basis on the level read in EV-401
- The flowrate of the spilled vapour out of EV-402, stream 4050, is regulated by a flowrate ratio control, reading the flowrates in streams 4050 and 4009 (feed of EV-401)
- The flowrate of the liquid out of EV-402, stream 4011, is regulated by a throttling valve, on the basis of the level read in EV-402
- The speed of the screw-feeder XF-401 is regulated on the basis of the pH in the citric acid tank TK-403
- The flowrate of stream 4052, out of TK-402, is regulated by a throttling valve on the basis of the ethanol fraction in TK-401
- The flowrate of stream 4053 (ethanolic solution used for the precipitation of NaCl), is regulated by a throttling valve on the basis of the flowrate ratio between stream 4053 and 4004

Plot plan



BSG VALORISATION PROCESS
PLOT PLAN
Underground storage S-101
SC-101
H-101
PL-101
DEWATERING RP-101
DRYING RD-101
UF 201-202 STATION
NF 201-202 STATION
C-101
C-102
SS-101
D-206
D-209
DD-201
HX-201
D-401
CC-401
EV-201
CY-201
CY-101
D-203
Polyphenols extraction TX-201 station
HX-201
CD-302
D-404
CC-402
CY-301
D-304
D-204
Washing W-201
AX/Proteins extraction TX-301 station
D-303
EV-401
EV-402
EV-403
SP-201
TK-401
TK-402
D-305
HX-301
D-307
CD-302
D-308
D-310
CF-301
CF-311
DD-302
TK-403
D-309
CD-302
D-310
CF-301
CF-311
DD-301

1.20 CAPEX and OPEX preliminary estimation

Having defined the equipment necessary for the process, it is possible to carry out a preliminary CAPEX and OPEX estimation of the plant. For the evaluation of the CAPEX of the plant, it is necessary to estimate the cost of each equipment. For most equipment, this was done according to Guthrie's method: the method allows for the estimation of the cost of certain pieces of equipment based on certain characteristics (e.g. height and diamter for tanks, exchange area for heat exchangers, and so on). The costs obtained need to be actualised. This has been done with the CEPCI indexes of year 1969 and year 2019. When a relationship for the estimate of the cost was not available, the price was obtained by obtaining estimates from vendors for equipment with a similar performance. The summation of all the equipment costs allows for the calculation of the installed cost. On its basis, the other costs that define the opex are calculated as a certain percentage of the installed cost. The CAPEX of the plant is reported in table 6.13.1

Direct costs	**euros**
Installed cost	3440000
Piping and insulation	1070000
Electrical facilities	345000
Buildings	999000
Yard improvements	413000
Total direct costs	6267000
Indirect costs	
Engineering	815000
Construction	627000
Legal and contrctors fees	440000
Project continjency	251000
Total indirect costs	2133000
TPI	8400000
Working capital	1260000
CAPEX	9660000

For the calculation of the OPEX, the consumption of utilities and raw materials have been calculated with the process flowsheeting, and the price of each was researched through vendors on the internet for raw materials, and from a reference in the case of the utilities. The unitary cost of the utilities and their consumption is reported in chapter 6.2.3. For an estimation of the operating labour, the number of operators for shift was estimated based on the quipment installed in the plant (Table 6.13.2)

Equipment	Operators per shift

Conveyors	0,1
Crushers, mills, grinders	0,5
Drives and power recovery machines	0,4
Evaporators	0,3
Vaporizers	0,1
Furnaces	0,3
Fans	0,1
Blowers and Compressors	0,4
Gas solid contacting equipment	0,4
Heat exchangers	0,1
Mixers	0,2
Towers	0,2
Drums	0,0
Pumps	0,0
Reactors	0,3
Clarifiers and thickeners	0,1
Centrifugal separators and filters	0,1
Cyclones	0,0
Bag filters	0,1
Electrostatic precipitators	0,1
Rotary and belt filters	0,1
Plat and frame, shell and leaf filters	0,6
Expression equipment	0,1
Screens	0,1
Size/englargement equipment	0,1
Storage vessels	0,0

The estimated average yearly cost per operator is 40.000 euros/year. Based on the utilities and raw materials consumption, as well as the operating labour estimation, the OPEX of the plant was calculated, and it is reported in table 6.13.3

	euro/year
Raw materials	240000
Utilities:	
Electrical energy	102000
LPS	121000
Chilled water	14000
Operating labor	240000
Clerical labor	48000
Maintenance	120000
Operating supplies	18000
Laboratory charges	24000
Patents	18000

Tot. ADME	945000
Overhead	264000
local taxes	180000
Insurance	51000
Tot. Aime	495000
Tot. Ame	1440000
Administrative costs	66000
Distribution and selling	63000
Research and development	24000
Tot. General expenses	153000
Depreciation	420000
Total expenses	2013000

1.21 Calculation of economical indexes

To carry out an economical evaluation of the plant, the following indexes where calculated:

- ROI: The return on investement has been calculated on a yearly base, as the ratio between the yearly net cash flow before taxes and the total investement of the plant.

$$ROI = \frac{867.000\ euros/year}{9660000\ euros} = 8.98\%$$

- IRR: The IRR has been calculated by equaling to 0 the NPV and searching for the value of IRR that satisfied the equation:

$$NPV = 0 = \sum_{i=1}^{20} \frac{Cash\ flow\ of\ year\ i}{(1 + IRR)^i} - initial\ investement$$

  The search yielded a value for the IRR of 6.36% considering 20 years of lifespan of the plant

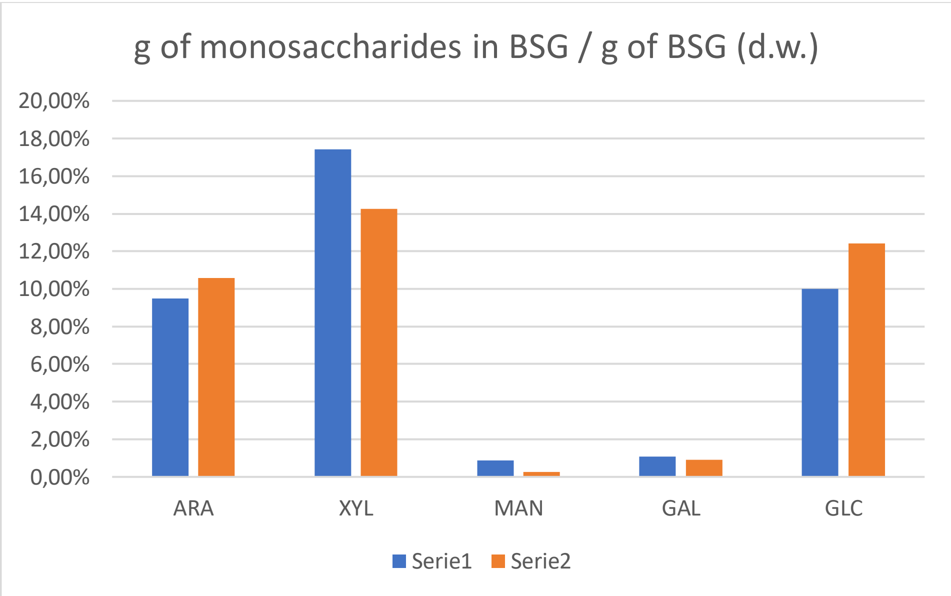
- PBP: The payback period has been calculated as the ratio between the investement and the yearly net cash flow, with 1 more year added to account for the design, procurement and construction of the plant, preliminary to the start of the productivity. The PBP of the plant has been estimated in 12 years.
- BEP: Given the heterogeneity of the price of the 3 products and the byproduct of the plant, the BEP has been calculated instead on the basis of treated BSG.

$$BEP = \frac{Fixed\ Costs}{Profit\ per\ ton\ of\ processed\ BSG\ - variable\ expenses\ per\ ton\ of\ processed\ BSG}$$

To break even, it results that the minimum quantity of processed BSG must be 1550 ton/year.

7. Conclusions

The composition of the BSG in terms of monosaccharides and protein content has been carried out. The study has found values in accordance to the ones found in the literature.



The percantage of the main monosaccharides (Arabinose, Xylose, Mannose, Galactose and Glucose) in the BSG is reported in the previous graph. The average protein content, measured with an elemental analysis using the Kjeldahl method allowed for the estimation of the protein content in the BSG evaluated to be around 23% in mass on a dry BSG sample basis. The process as devised in (Coimbra Rodrigues da Silva, et al., 2012) has been modified, with two preliminary polyphenols extraction, one with an ethanolllic solution and one with pure water. The products obtained have been analysed, in terms of monosaccharides composition and protein content, and for the polyphenolic products also in terms of phenolic content, according to the Folin-Ciocalteu method. The detailed compositions obtained, as well as the resultant yields, are reported in detail in the chapters regarding the experimental results. A preliminary granulometry of the BSG has been also carried out, but needs more work to be more precisely characterised. The yields data in the polyphenols extraction have been compared with the results of a test carried out using the Turbex cavitational extractor patentend by G. Cavaglià. The extraction performance of the Turbex has been compared to the ones obtained with conventional extraction means. Especially in the case of extraction with ethanolic solution, the yield of polyphenols increased dramatically, being of over 90%

against a yield of 58% obtained with conventional extractors. The data obtained experimentally allowed for an evaluation of the Turbex performance, as well as the design of a pilot-scale process able to treat 2000 tons per year of matrix. The process, as conceived, is in part different from the laboratory-scale one: namely, it has been deemed not necessary to proceed with 3 subsequent extractions of AX and proteins with increasingly alkaline solutions: the ability of the Turbex to realise a multiple ideal equilibrium stage extraction would have made such a choice redundant. Also, as explained, this process features the addition of a preliminary polyphenols extraction with ethanolic solution, which was absent in the original process. The process design included the process flowsheeting, the process description, the design basis, the PFD and P&ID redaction, as long as the line list, the instrument list and the equipment list. The definition of the equipment has allowed for the calculation of preliminary CAPEX and OPEX of the process, and economical evaluators to assess the economical viability of the process. The process generates a net yearly income of 867.000 euros, for a yearly ROI of 8.98% and IRR along the 20 years lifespan of the plant of 6.36%. The process's payback period is expected to be 12 years. The process as designed is able to work with a fluctuating supply of BSG, according to the seasonality of the beer production. The process is able to produce 4.7 tons per year of polyphenols-rich product, 110 tons per year of proteini product and 143 tons per year of arabino-xylanes rich product.

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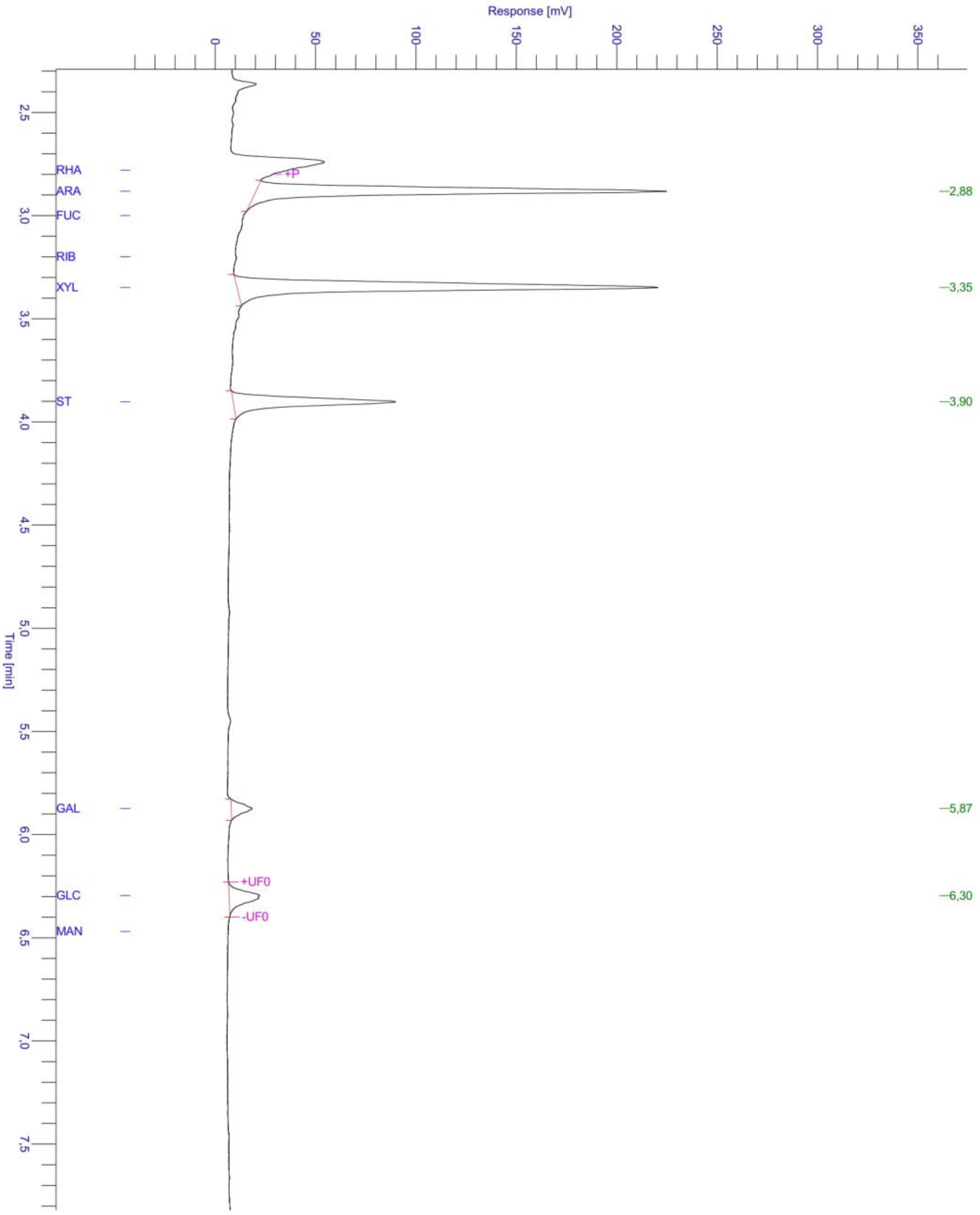
Appendix A – Sugar analysis chromatograms

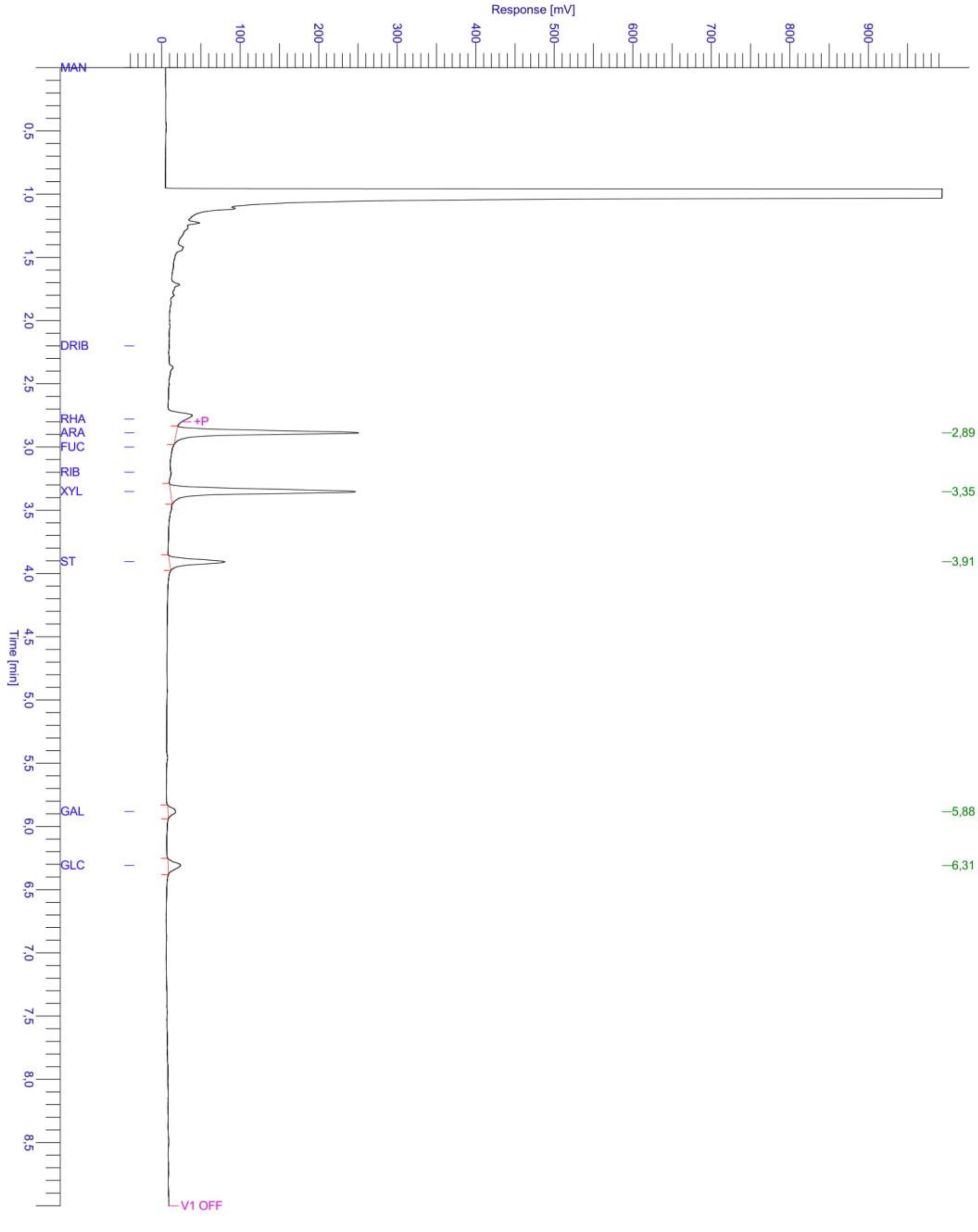
Chromatogram

Sample Name : AX1_R1 Sample #: 001 Page 1 of 1
FileName : C:\Users\manager\Desktop\Data\RobertoChighine\Raw\AX1_R1_001.raw
Date : 28/05/2021 14:15:25
Method : acucares_automatico Time of Injection: 28/05/2021 12:37:15
Start Time : 2,29 min End Time : 7,82 min Low Point : -47,27 mV High Point : 360,85 mV
Plot Offset: -47,27 mV Plot Scale: 408,1 mV



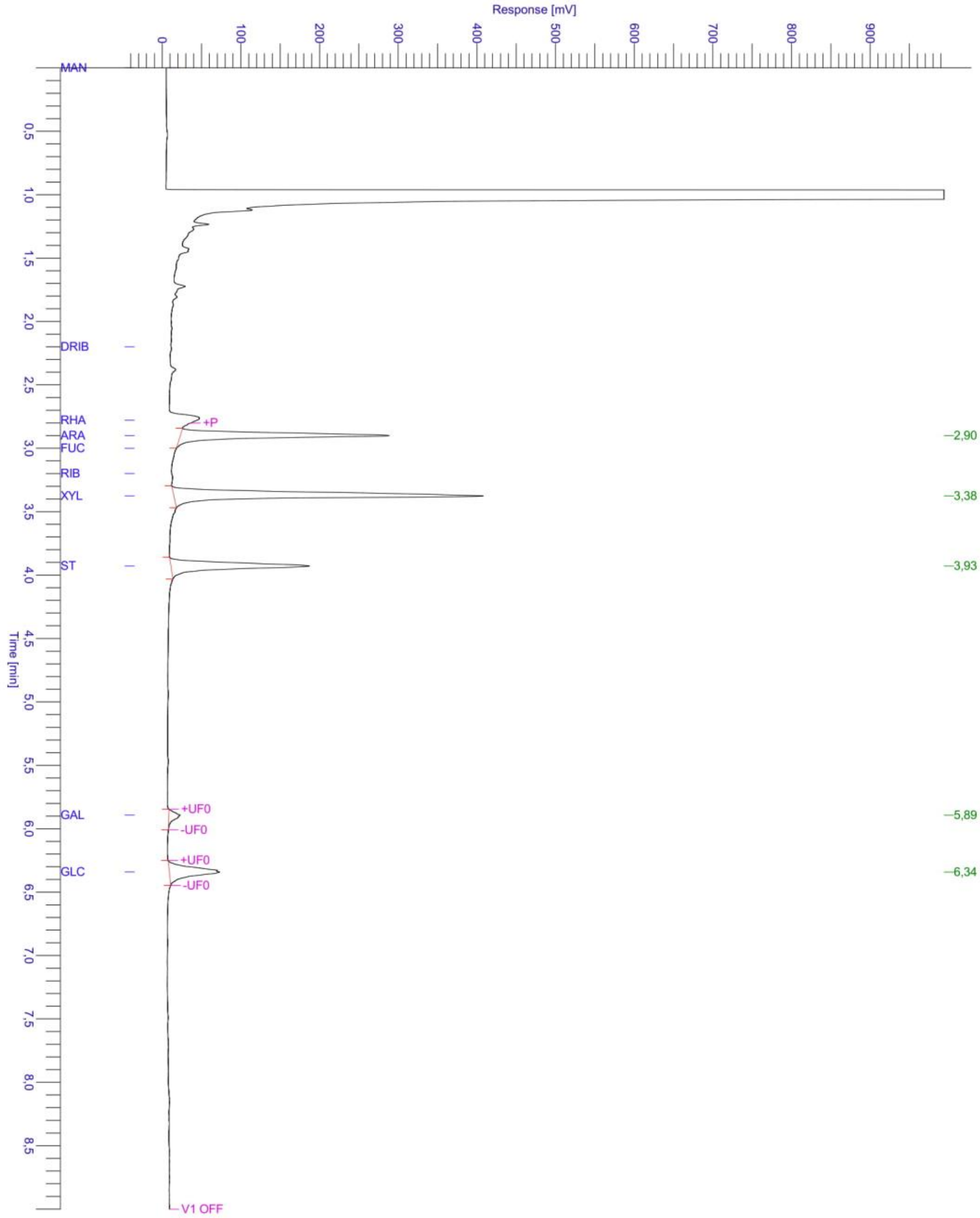
Chromatogram

Sample Name : AX1_R2 Sample #: 002 Page 1 of 1
FileName : C:\Users\manager\Desktop\Data\RobertoChighine\Raw\AX1_R2_002.raw
Date : 28/05/2021 14:17:00
Method : acucares_automatico Time of Injection: 28/05/2021 12:50:00
Start Time : 0,00 min End Time : 9,00 min Low Point : -47,40 mV High Point : 993,88 mV
Plot Offset: -47,40 mV Plot Scale: 1041,3 mV



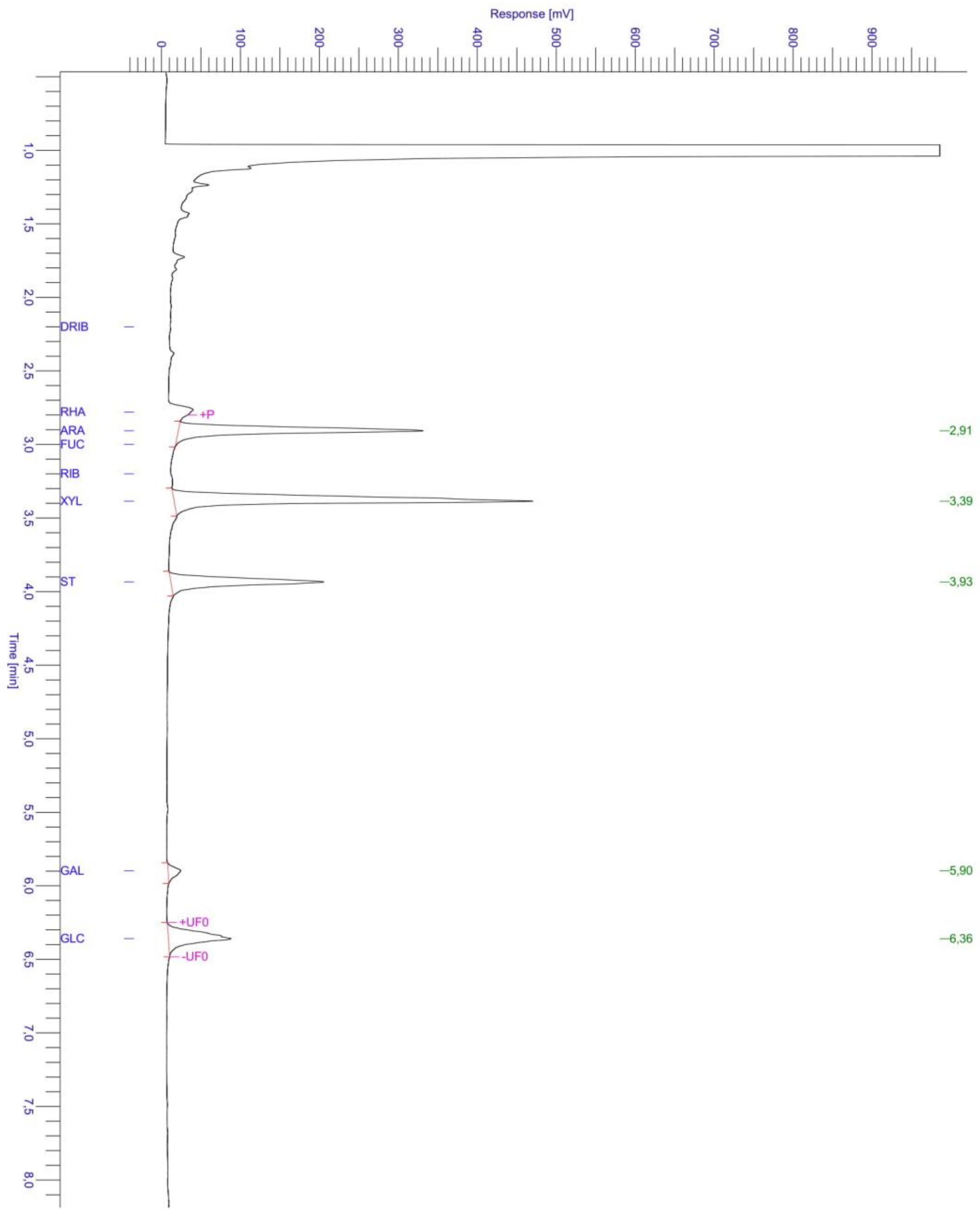
Chromatogram

Sample Name : AX2_R1 Sample #: 003 Page 1 of 1
FileName : C:\Users\manager\Desktop\Data\RobertoChighine\Raw\AX2_R1_003.raw
Date : 28/05/2021 14:19:01
Method : acucares_automatico Time of Injection: 28/05/2021 13:02:42
Start Time : 0,00 min End Time : 9,00 min Low Point : -47,68 mV High Point : 993,88 mV
Plot Offset: -47,68 mV Plot Scale: 1041,6 mV



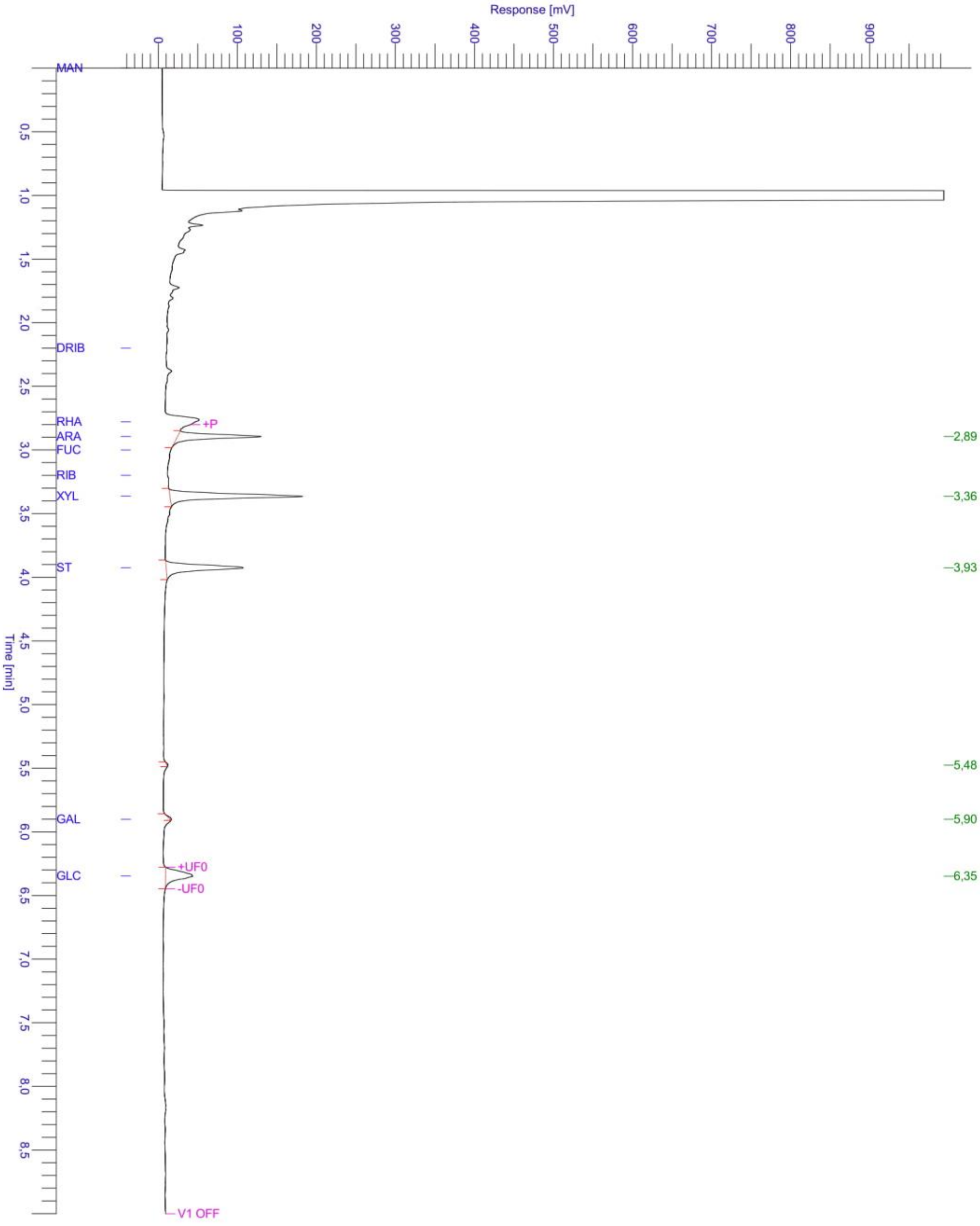
Chromatogram

Sample Name : AX2_R2 Sample #: 004 Page 1 of 1
FileName : C:\Users\manager\Desktop\Data\RobertoChighine\Raw\AX2_R2_004.raw
Date : 28/05/2021 14:20:20
Method : acucares_automatico Time of Injection: 28/05/2021 13:15:25
Start Time : 0,47 min End Time : 8,19 min Low Point : -47,28 mV High Point : 985,84 mV
Plot Offset: -47,28 mV Plot Scale: 1033,1 mV



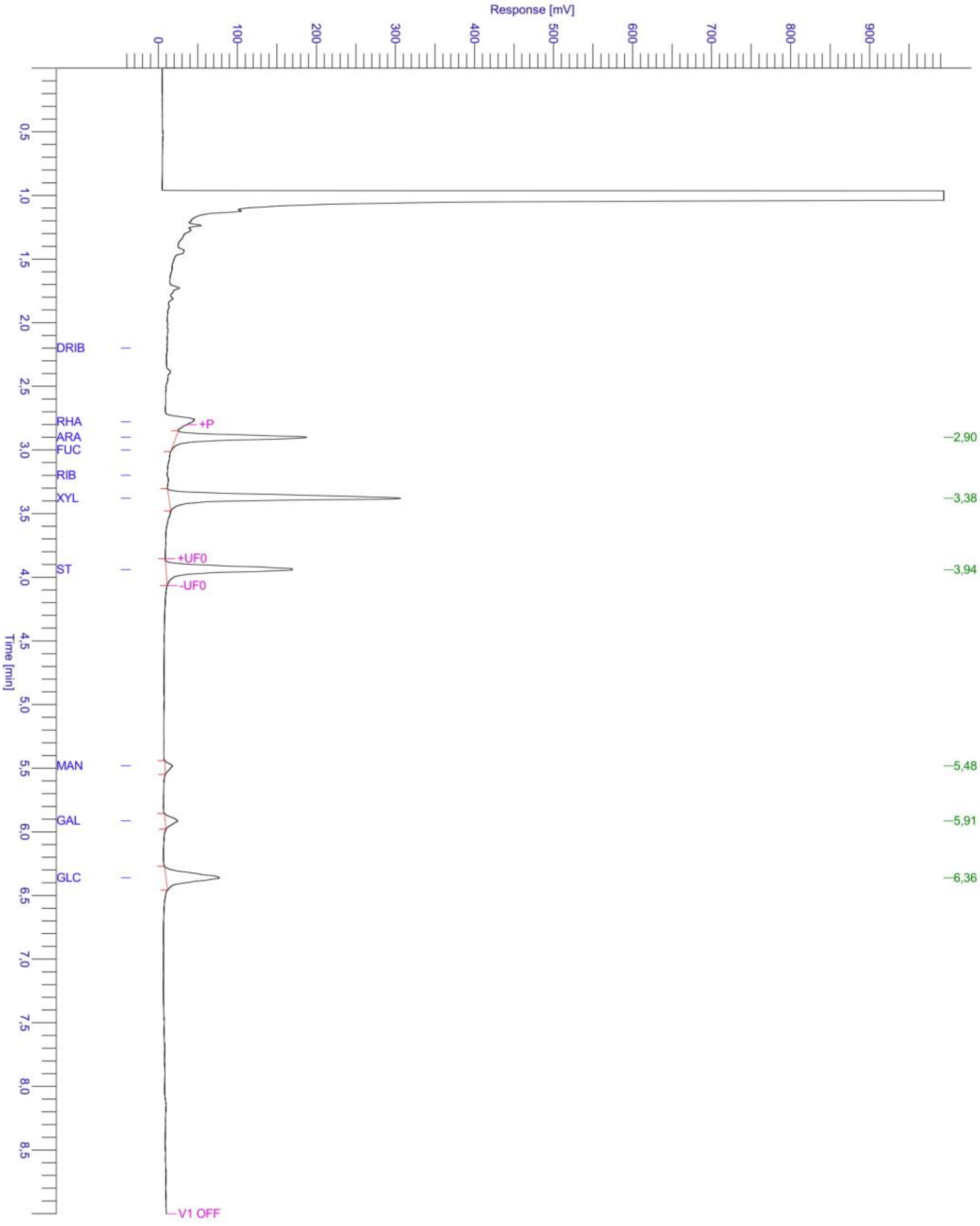
Chromatogram

Sample Name : AX3_R1 Sample #: 005 Page 1 of 1
FileName : C:\Users\manager\Desktop\Data\RobertoChighine\Raw\AX3_R1_005.raw
Date : 28/05/2021 14:21:35
Method : acucares_automatico Time of Injection: 28/05/2021 13:28:10
Start Time : 0,00 min End Time : 9,00 min Low Point : -47,31 mV High Point : 993,88 mV
Plot Offset: -47,31 mV Plot Scale: 1041,2 mV



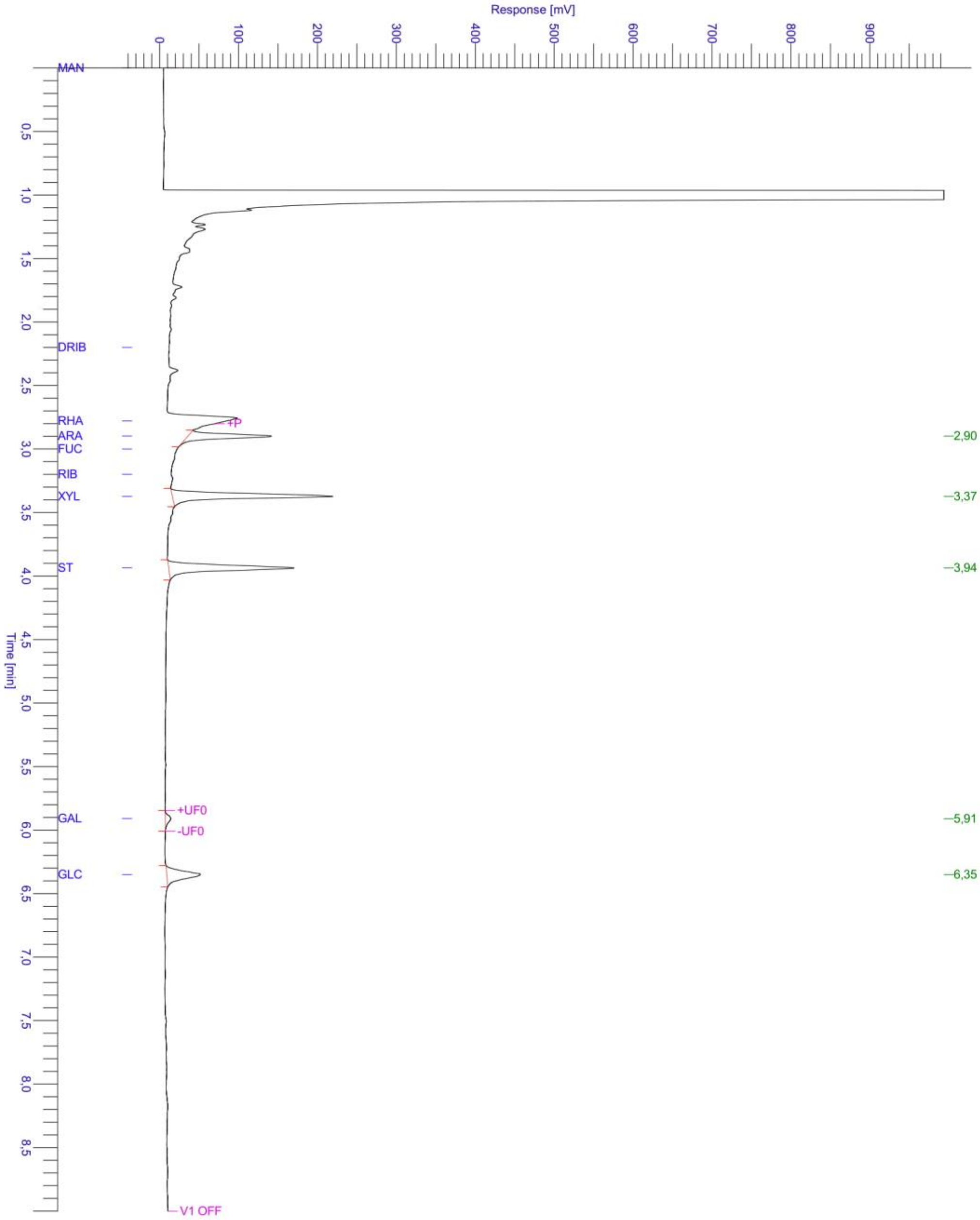
Chromatogram

Sample Name : AX3_R2 Sample #: 006 Page 1 of 1
FileName : C:\Users\manager\Desktop\Data\RobertoChighine\Raw\AX3_R2_006.raw
Date : 28/05/2021 14:23:07
Method : acucares_automatico Time of Injection: 28/05/2021 13:40:58
Start Time : 0,00 min End Time : 9,00 min Low Point : -47,40 mV High Point : 993,88 mV
Plot Offset: -47,40 mV Plot Scale: 1041,3 mV



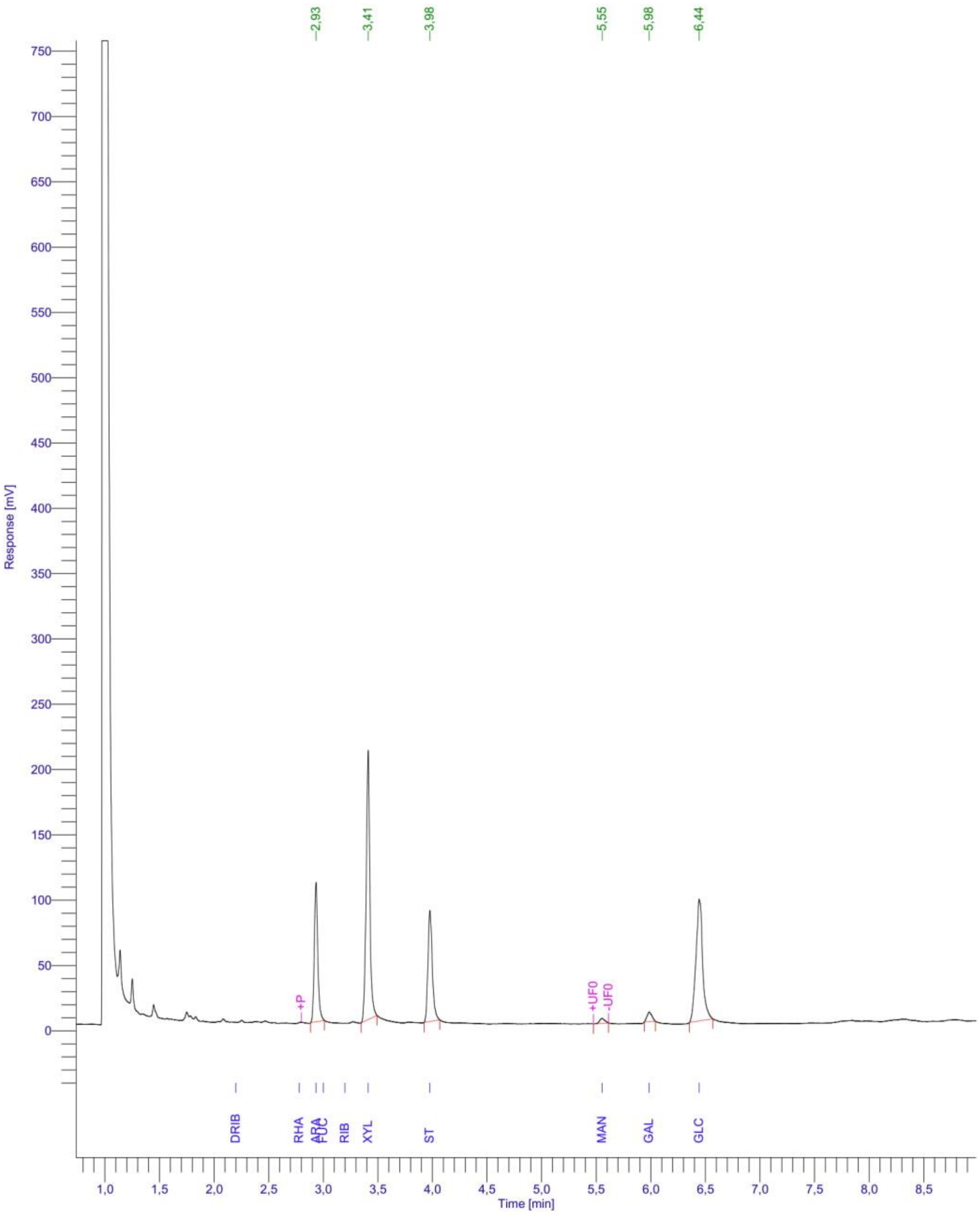
Chromatogram

Sample Name : AX4_R1 Sample #: 007 Page 1 of 1
FileName : C:\Users\manager\Desktop\Data\RobertoChighine\Raw\AX4_R1_007.raw
Date : 28/05/2021 14:24:34
Method : acucares_automatico Time of Injection: 28/05/2021 13:53:46
Start Time : 0,00 min End Time : 9,00 min Low Point : -47,48 mV High Point : 993,88 mV
Plot Offset: -47,48 mV Plot Scale: 1041,4 mV



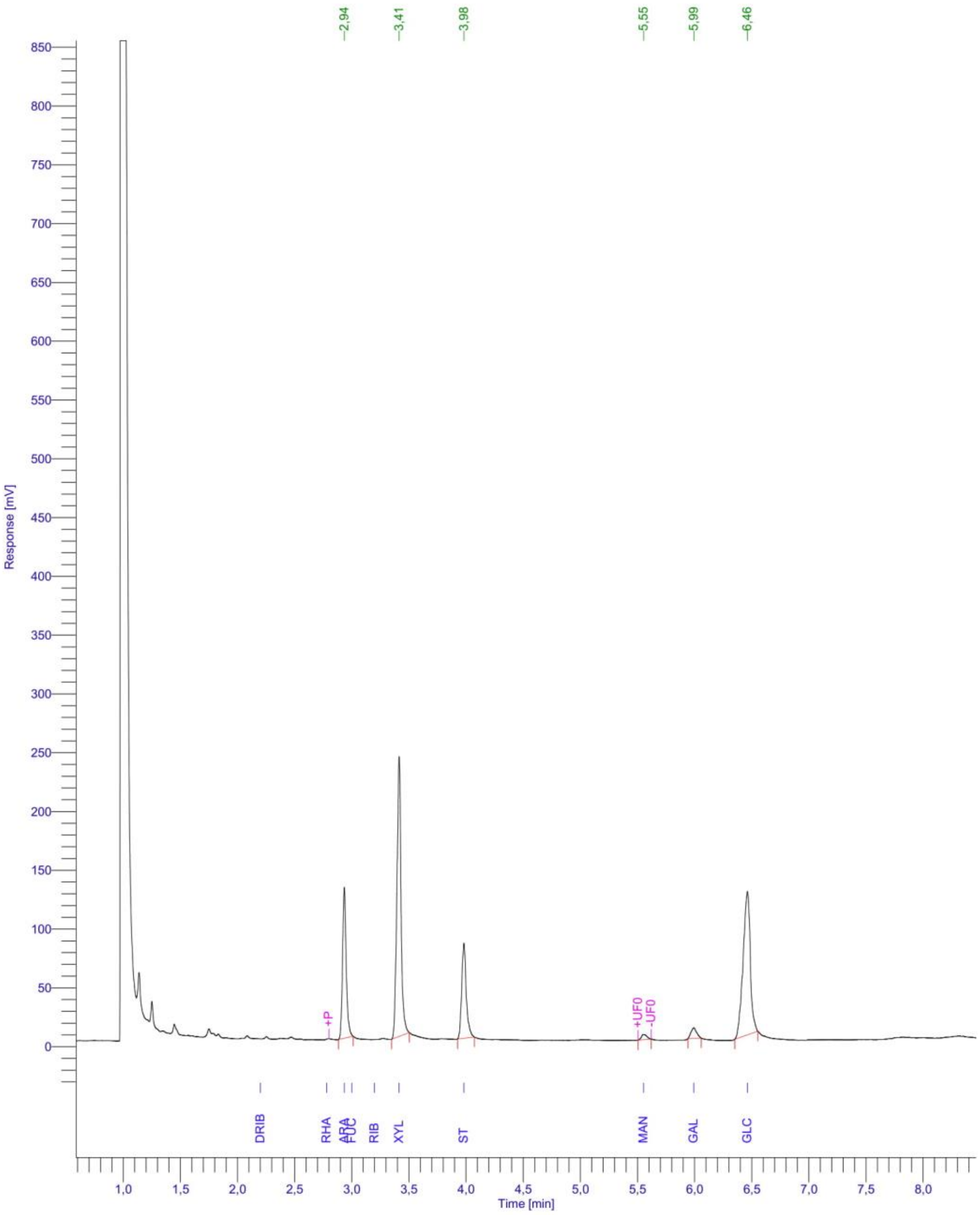
Chromatogram

Sample Name : BSG_C1 Sample #: 017 Page 1 of 1
FileName : C:\Users\manager\Desktop\Data\RobertoChighine\Raw\BSG_C1_017.raw
Date : 24/05/2021 14:01:59
Method :
Time of Injection: 21/05/2021 16:11:07
Start Time : 0,74 min End Time : 8,98 min Low Point : -47,43 mV High Point : 758,04 mV
Plot Offset: -47,43 mV Plot Scale: 805,5 mV



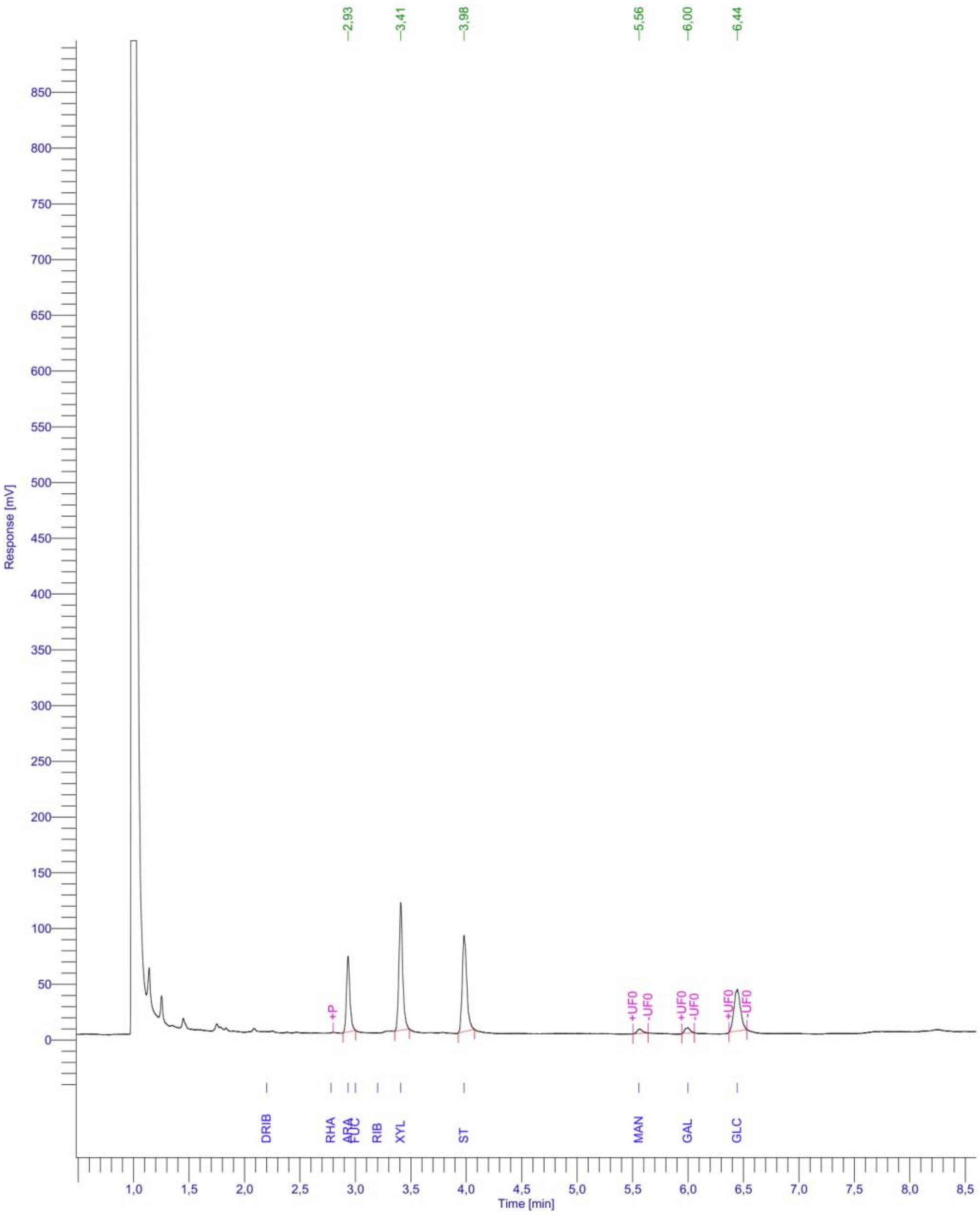
Chromatogram

Sample Name : BSG_C2 Sample #: 018 Page 1 of 1
FileName : C:\Users\manager\Desktop\Data\RobertoChighine\Raw\BSG_C2_018.raw
Date : 24/05/2021 14:08:38
Method : Time of Injection: 21/05/2021 16:23:55
Start Time : 0,59 min End Time : 8,46 min Low Point : -39,20 mV High Point : 855,57 mV
Plot Offset: -39,20 mV Plot Scale: 894,8 mV



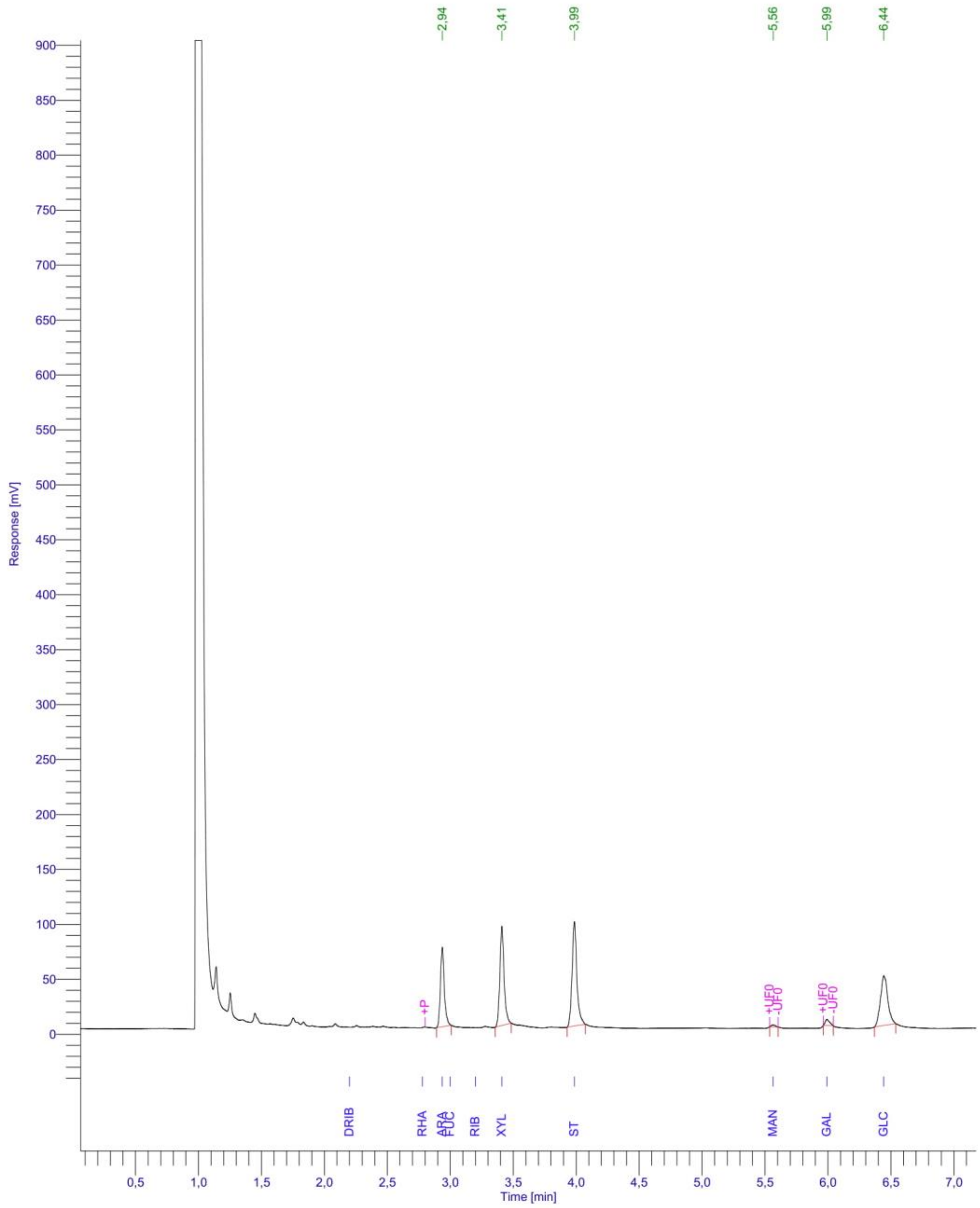
Chromatogram

Sample Name : BSG_NC_1 Sample #: 019 Page 1 of 1
FileName : C:\Users\manager\Desktop\Data\RobertoChighine\Raw\BSG_NC_1_019.raw
Date : 24/05/2021 14:11:53
Method : Time of Injection: 21/05/2021 16:36:59
Start Time : 0,48 min End Time : 8,60 min Low Point : -47,29 mV High Point : 896,36 mV
Plot Offset: -47,29 mV Plot Scale: 943,6 mV



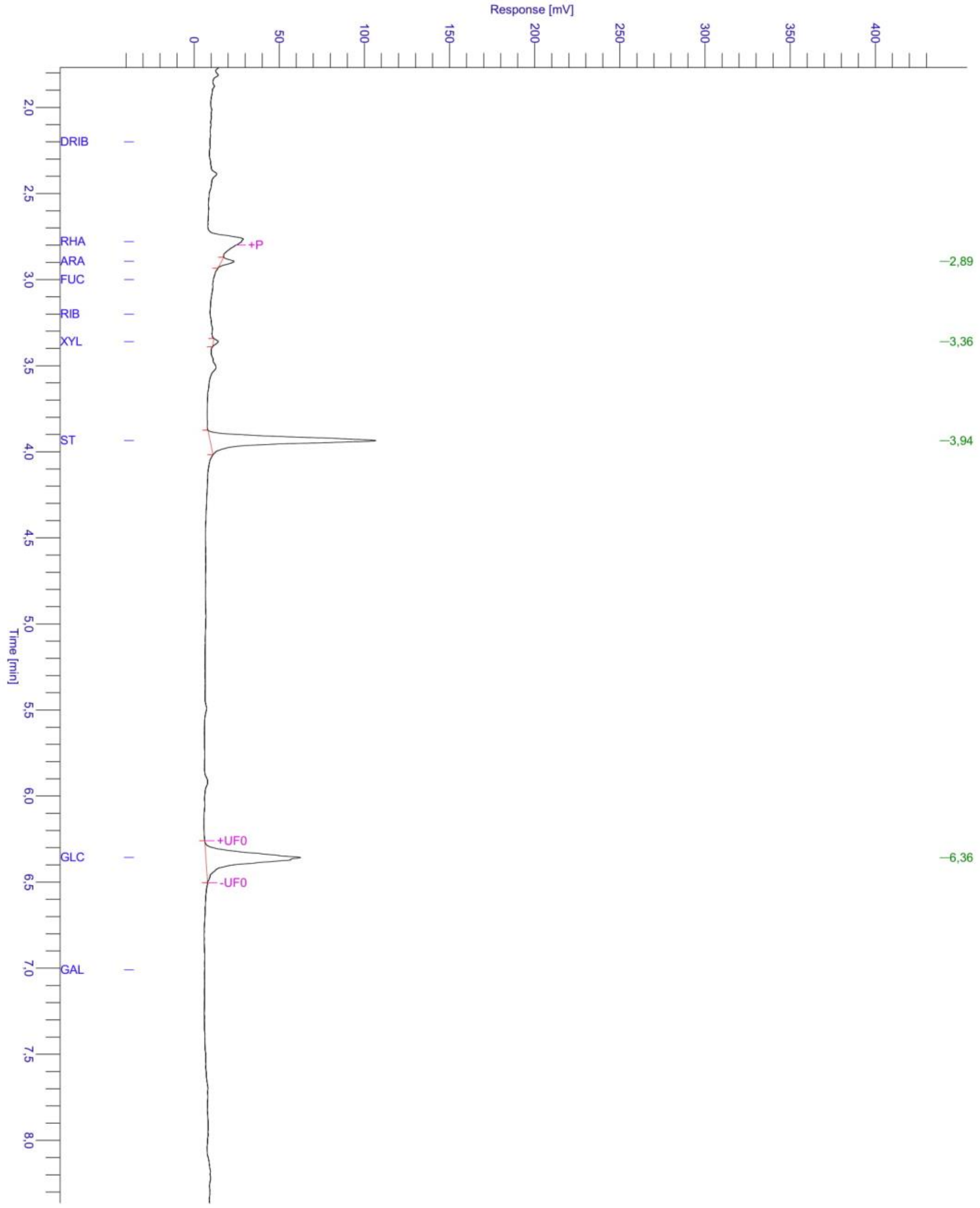
Chromatogram

Sample Name : BSG_NC_2 Sample #: 020 Page 1 of 1
FileName : C:\Users\manager\Desktop\Data\RobertoChighine\Raw\BSG_NC_2_020.raw
Date : 24/05/2021 14:14:56
Method : Time of Injection: 21/05/2021 16:49:49
Start Time : 0,06 min End Time : 7,18 min Low Point : -47,48 mV High Point : 904,39 mV
Plot Offset: -47,48 mV Plot Scale: 951,9 mV



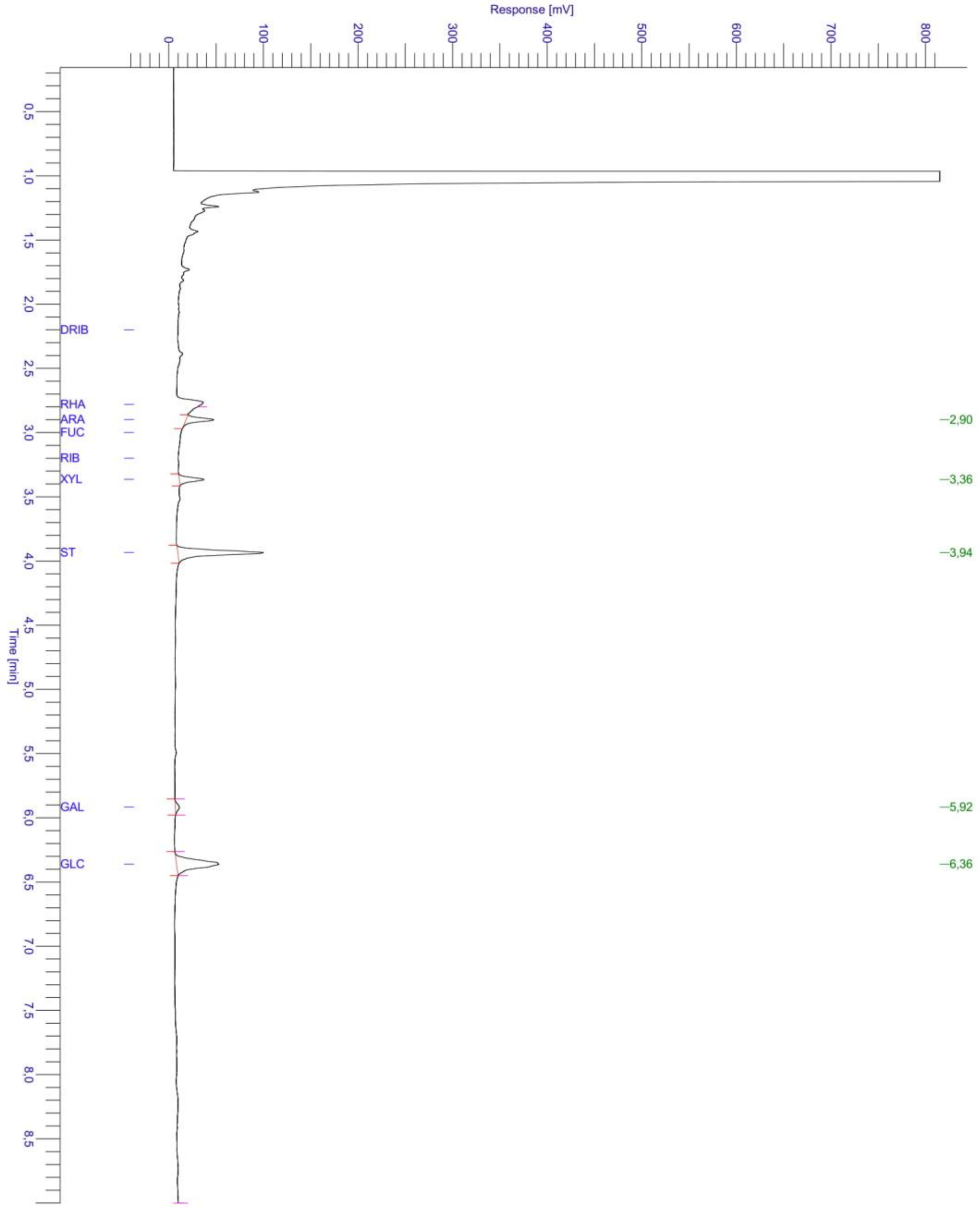
Chromatogram

Sample Name : EtOH Sample #: 008 Page 1 of 1
FileName : C:\Users\manager\Desktop\Data\RobertoChighine\Raw\EtOH_008.raw
Date : 28/05/2021 14:27:18
Method : acucares_automatico Time of Injection: 28/05/2021 14:06:30
Start Time : 1,77 min End Time : 8,36 min Low Point : -41,08 mV High Point : 437,88 mV
Plot Offset: -41,08 mV Plot Scale: 479,0 mV



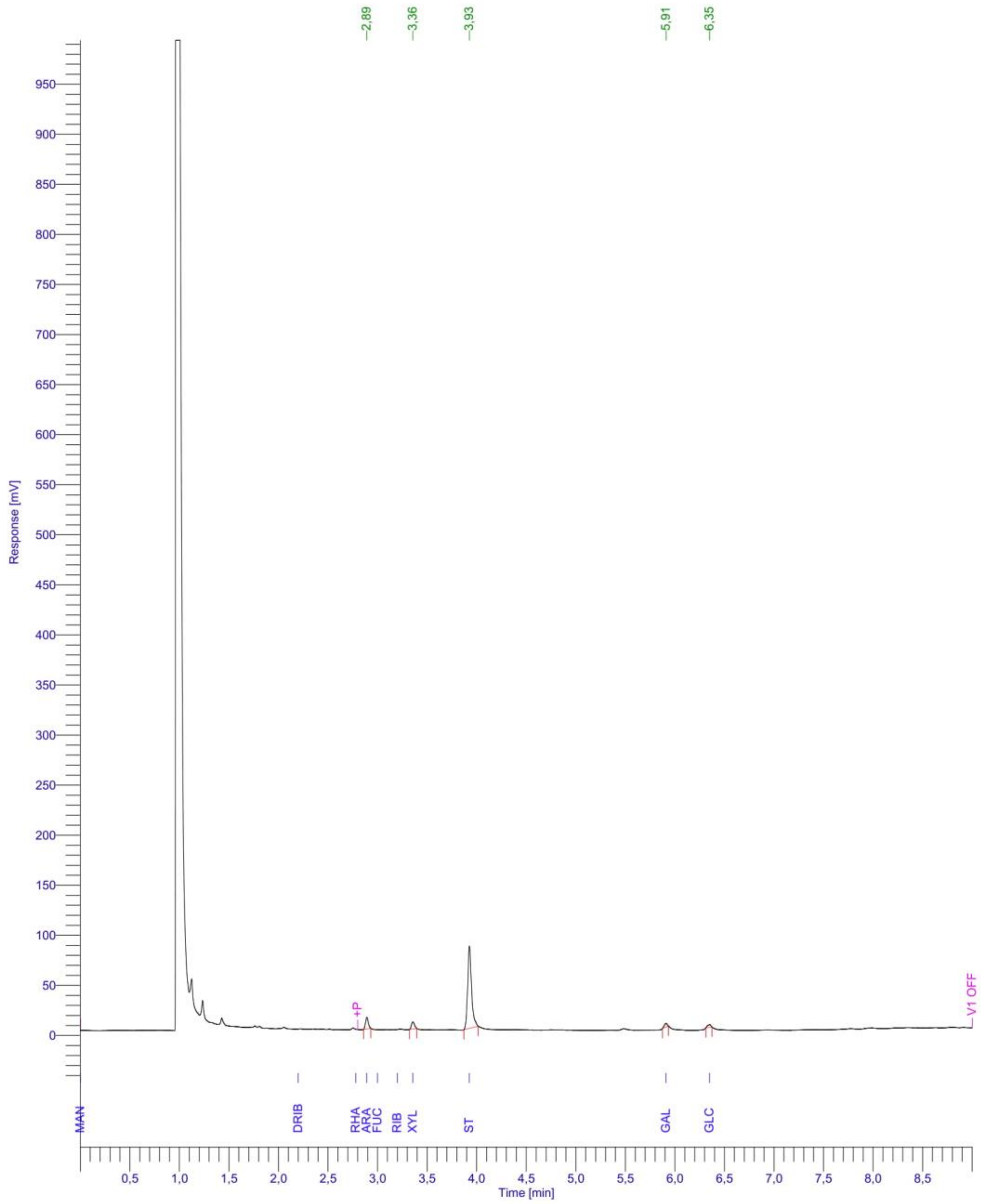
Chromatogram

Sample Name : H2O Sample #: 009 Page 1 of 1
FileName : C:\Users\manager\Desktop\Data\RobertoChighine\Raw\H2O_009.raw
Date : 28/05/2021 14:32:09
Method : acucares_automatico Time of Injection: 28/05/2021 14:19:14
Start Time : 0,16 min End Time : 9,00 min Low Point : -47,15 mV High Point : 815,06 mV
Plot Offset: -47,15 mV Plot Scale: 862,2 mV



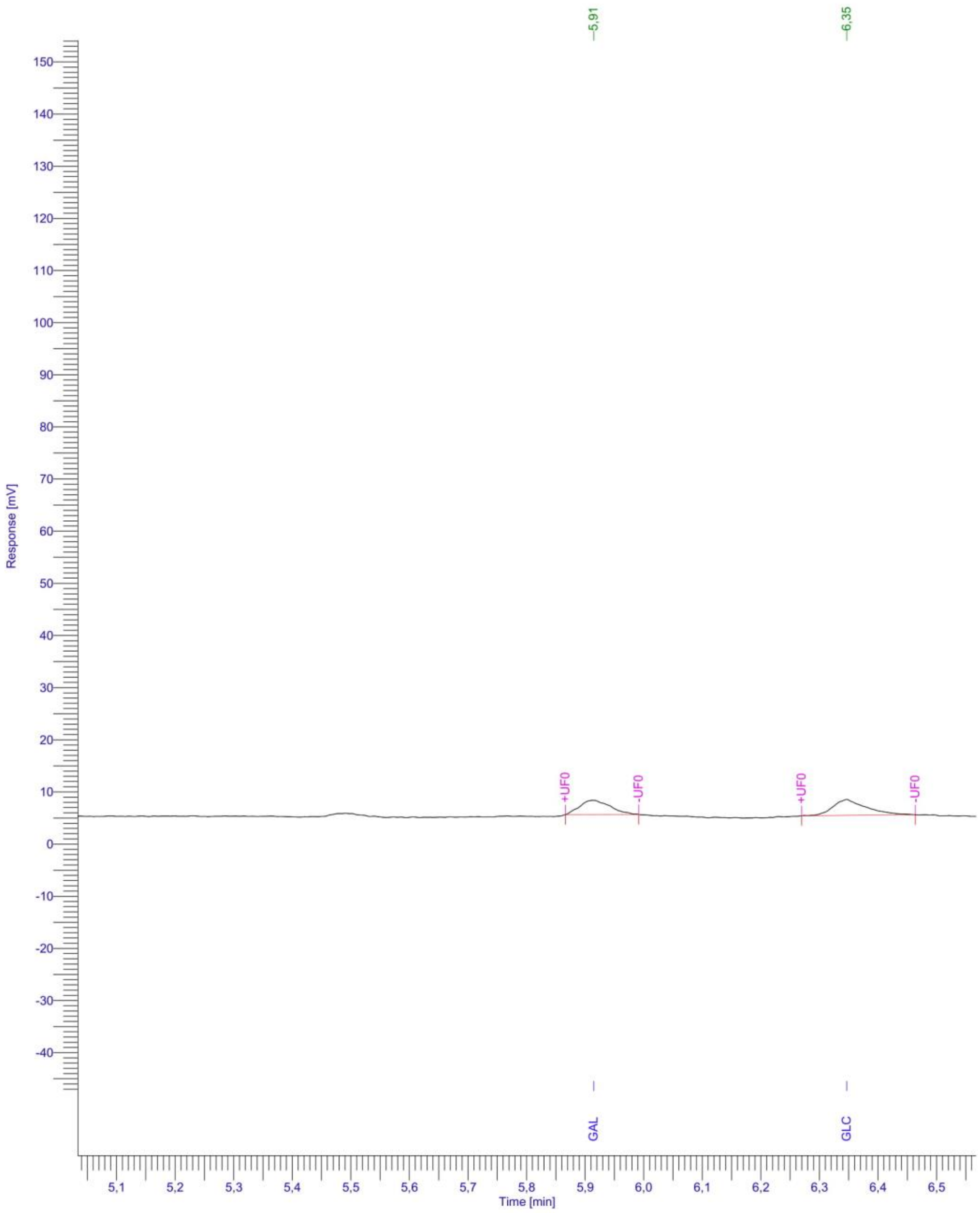
Chromatogram

Sample Name : PP1_1 Sample #: 001 Page 1 of 1
FileName : C:\Users\manager\Desktop\Data\RobertoChighine\Raw\PP1_1_001.raw
Date : 24/05/2021 14:17:01
Method : Time of Injection: 21/05/2021 12:46:58
Start Time : 0,00 min End Time : 9,00 min Low Point : -47,32 mV High Point : 993,88 mV
Plot Offset: -47,32 mV Plot Scale: 1041,2 mV



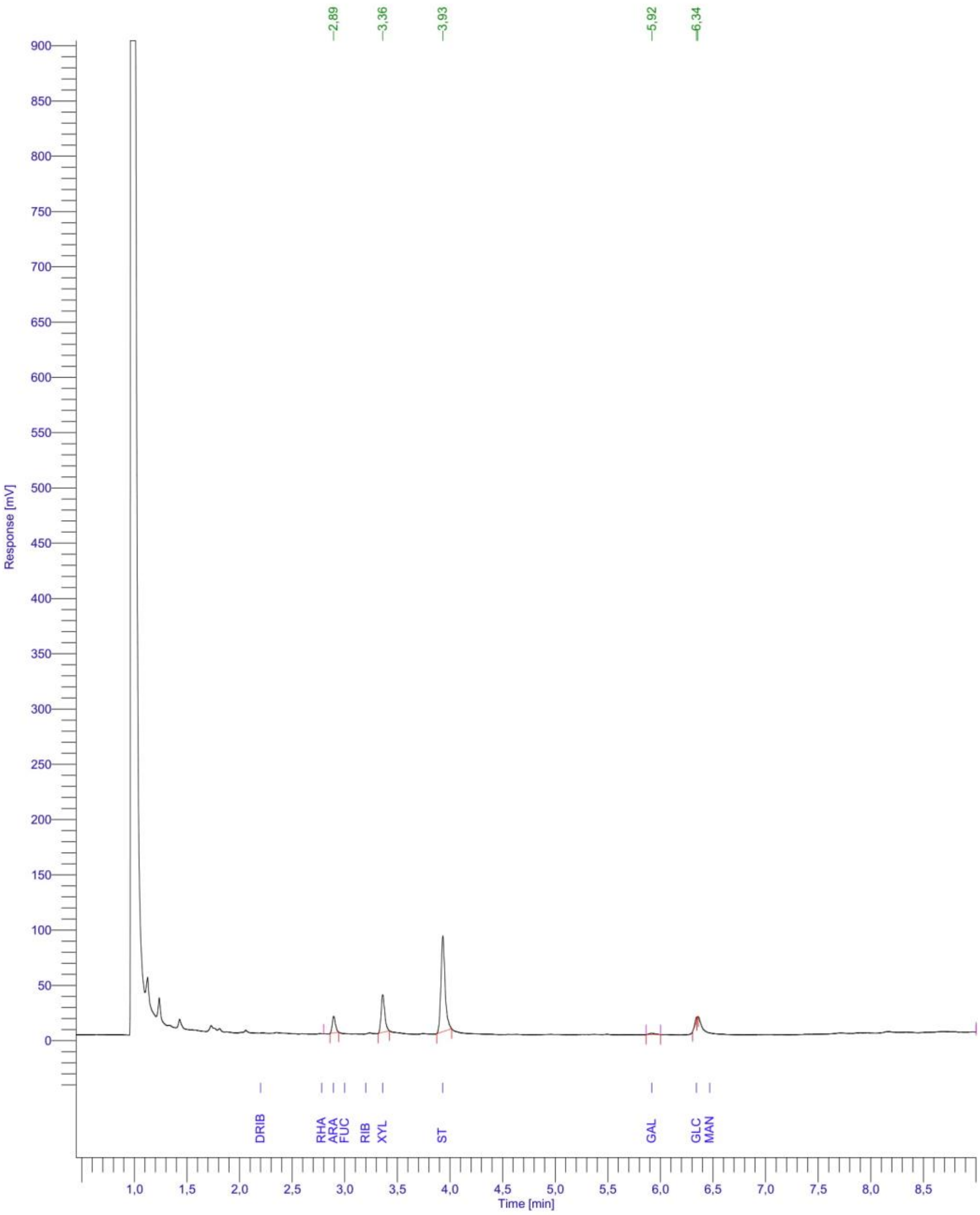
Chromatogram

Sample Name : PP1_2 Sample #: 002 Page 1 of 1
FileName : C:\Users\manager\Desktop\Data\RobertoChighine\Raw\PP1_2_002.raw
Date : 24/05/2021 14:18:49
Method : Time of Injection: 21/05/2021 12:59:42
Start Time : 5,03 min End Time : 6,57 min Low Point : -47,34 mV High Point : 154,08 mV
Plot Offset: -47,34 mV Plot Scale: 201,4 mV



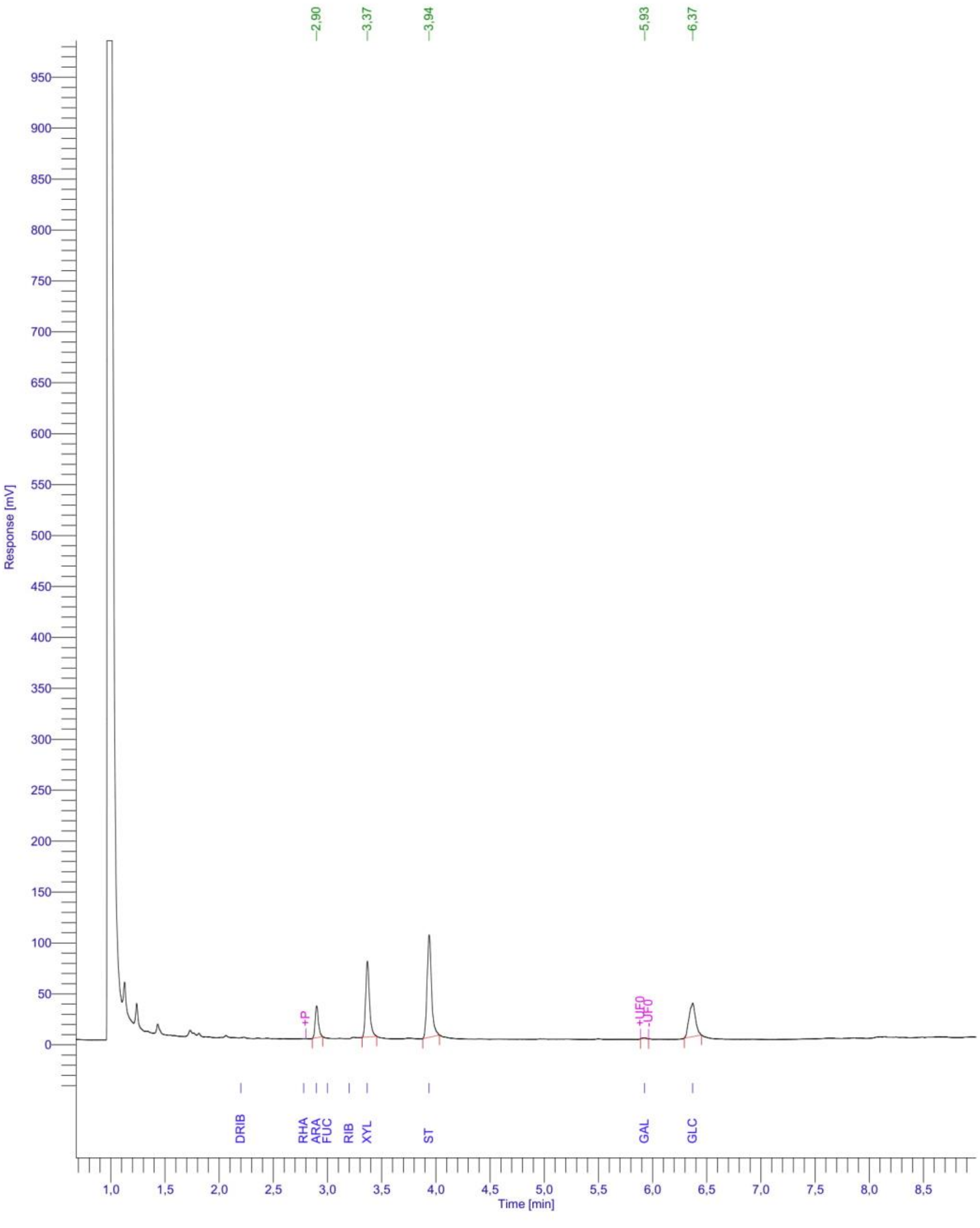
Chromatogram

Sample Name : PP2_1 Sample #: 003 Page 1 of 1
FileName : C:\Users\manager\Desktop\Data\RobertoChighine\Raw\PP2_1_003.raw
Date : 24/05/2021 14:20:48
Method : Time of Injection: 21/05/2021 13:12:25
Start Time : 0,45 min End Time : 9,00 min Low Point : -47,11 mV High Point : 904,55 mV
Plot Offset: -47,11 mV Plot Scale: 951,7 mV



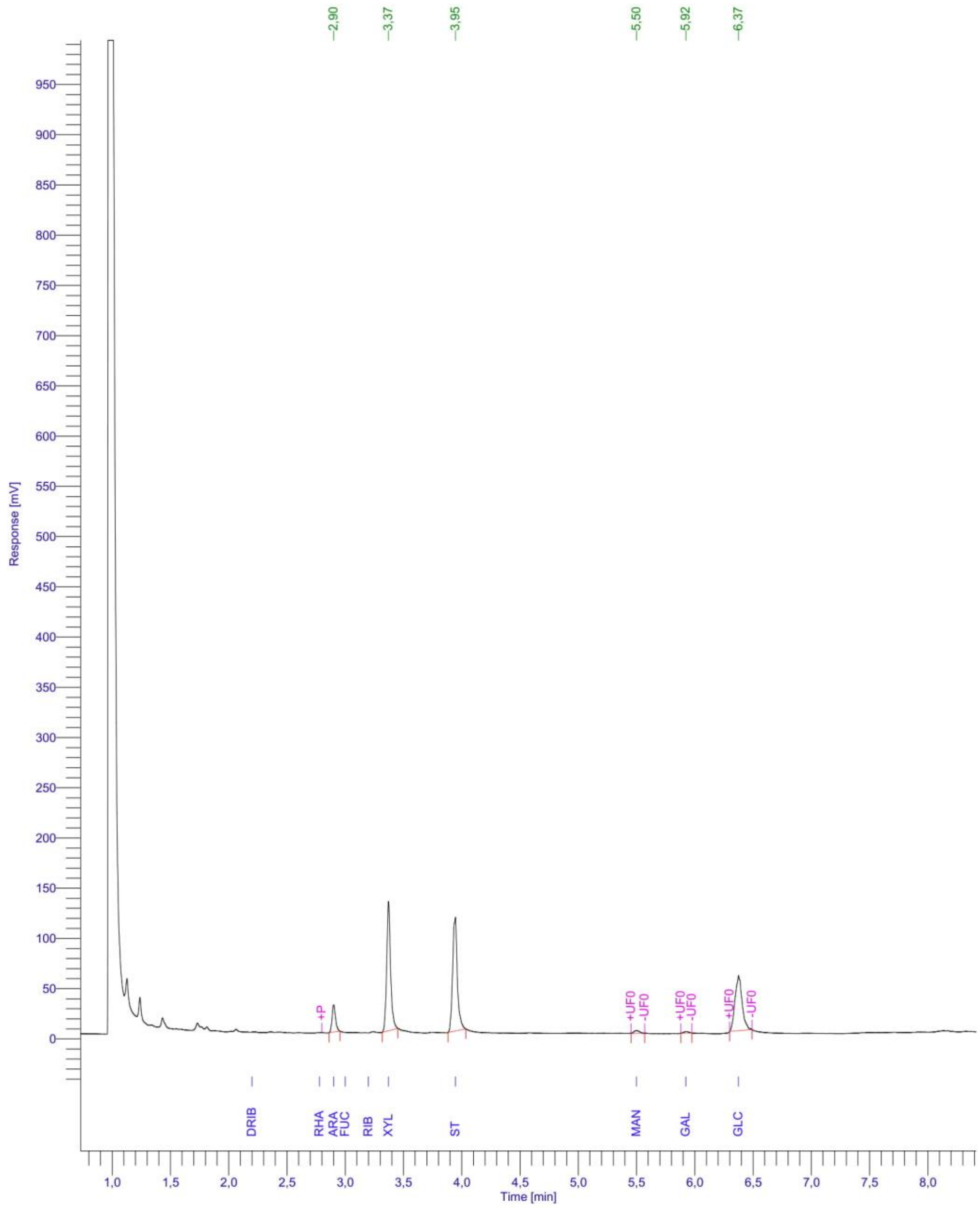
Chromatogram

Sample Name : PP2_2 Sample #: 004 Page 1 of 1
FileName : C:\Users\manager\Desktop\Data\RobertoChighine\Raw\PP2_2_004.raw
Date : 24/05/2021 14:22:23
Method : Time of Injection: 21/05/2021 13:25:20
Start Time : 0,68 min End Time : 8,99 min Low Point : -47,31 mV High Point : 985,84 mV
Plot Offset: -47,31 mV Plot Scale: 1033,2 mV



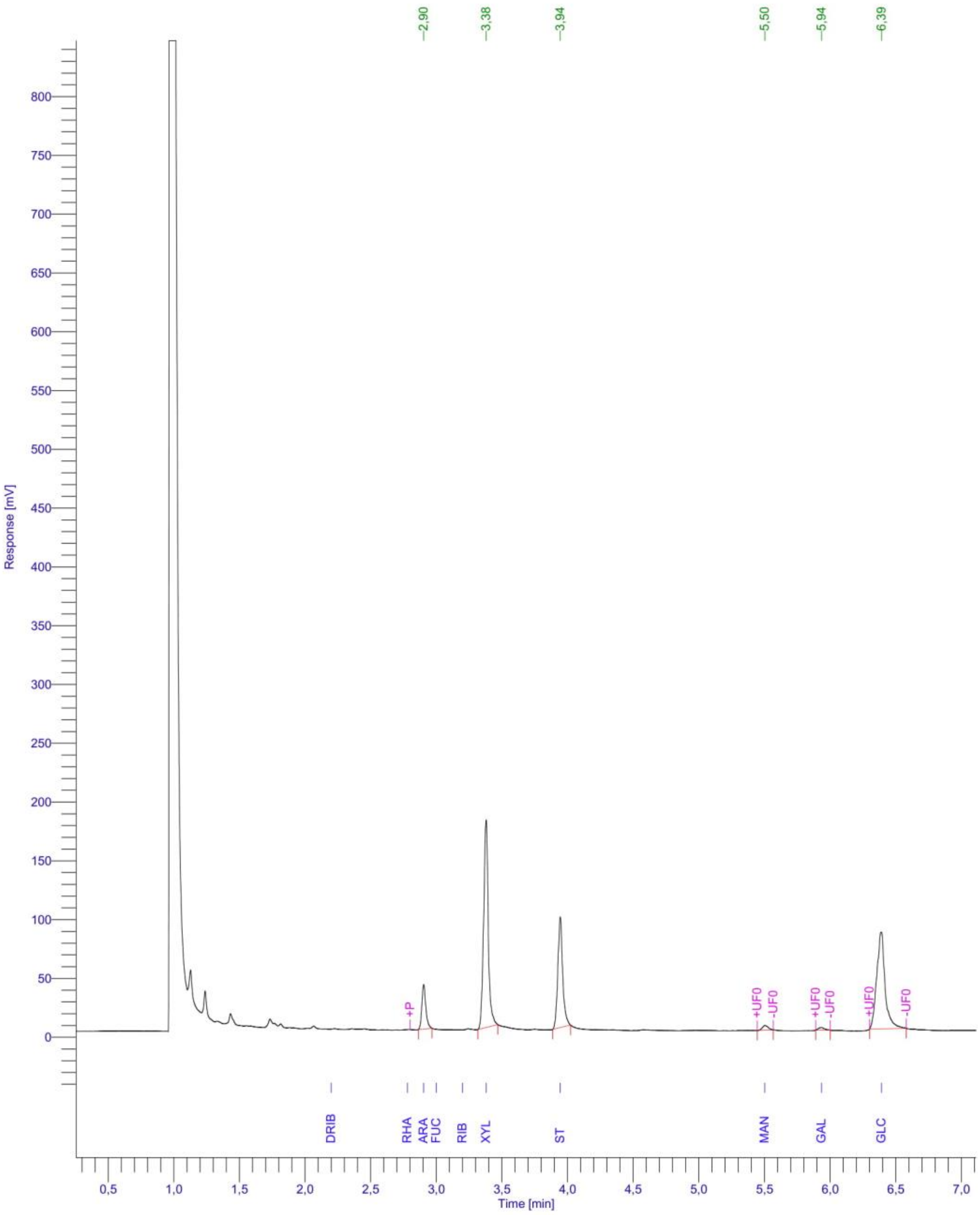
Chromatogram

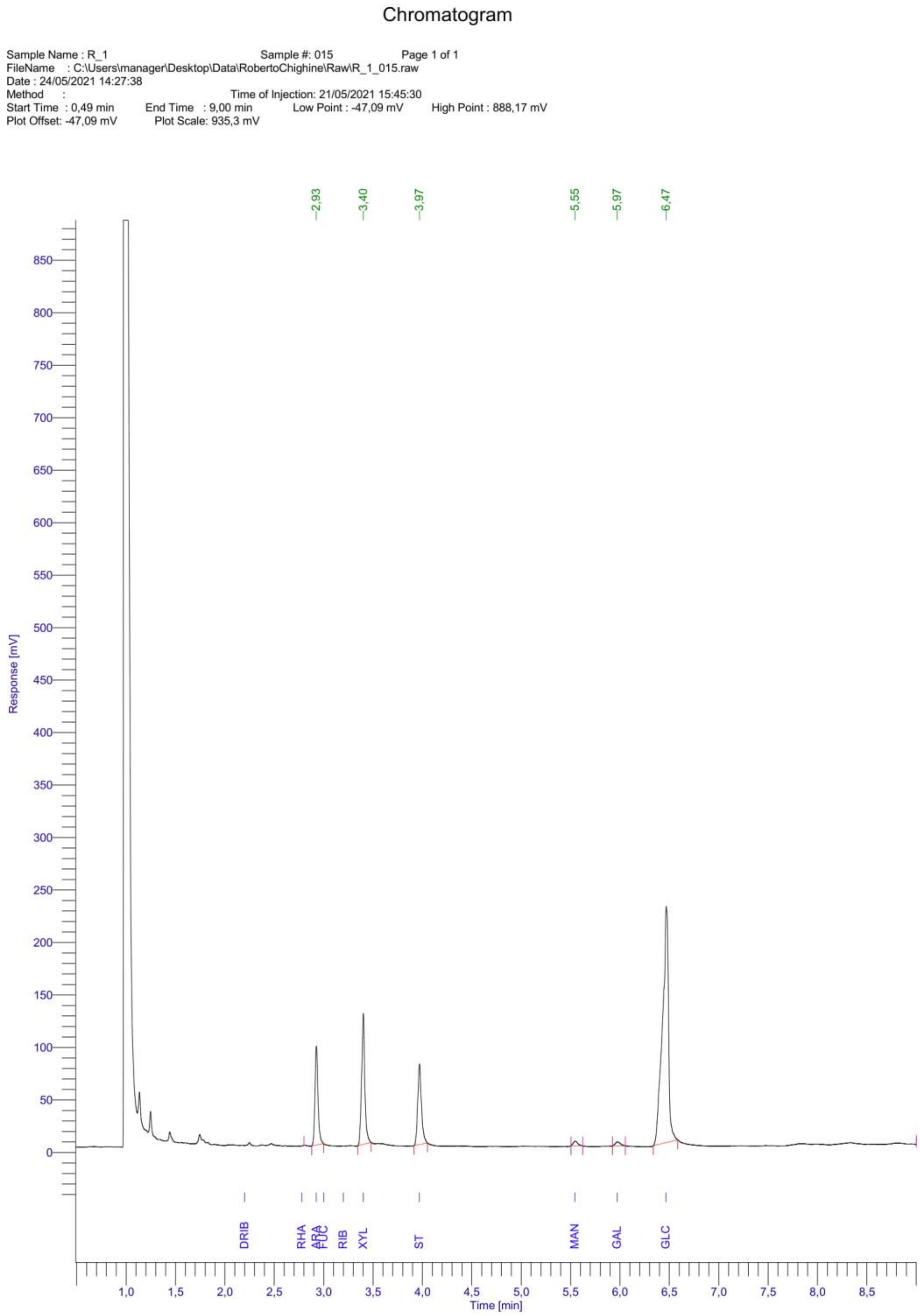
Sample Name : PP3_1 Sample #: 005 Page 1 of 1
FileName : C:\Users\manager\Desktop\Data\RobertoChighine\Raw\PP3_1_005.raw
Date : 24/05/2021 14:24:13
Method : Time of Injection: 21/05/2021 13:38:05
Start Time : 0,73 min End Time : 8,41 min Low Point : -47,30 mV High Point : 993,88 mV
Plot Offset: -47,30 mV Plot Scale: 1041,2 mV



Chromatogram

Sample Name : PP3_2 Sample #: 006 Page 1 of 1
FileName : C:\Users\manager\Desktop\Data\RobertoChighine\Raw\PP3_2_006.raw
Date : 24/05/2021 14:26:09
Method : Time of Injection: 21/05/2021 13:50:47
Start Time : 0,26 min End Time : 7,11 min Low Point : -47,23 mV High Point : 847,53 mV
Plot Offset: -47,23 mV Plot Scale: 894,8 mV




Chromatogram
Sample Name : R_1
Sample #: 015
Page 1 of 1
FileName : C:\Users\manager\Desktop\Data\RobertoChighine\Raw\R_1_015.raw
Date : 24/05/2021 14:27:38
Method :
Time of Injection: 21/05/2021 15:45:30
Start Time : 0,49 min
End Time : 9,00 min
Low Point : -47,09 mV
High Point : 888,17 mV
Plot Offset: -47,09 mV
Plot Scale: 935,3 mV
2,93
3,40
3,97
5,55
5,97
6,47
Response [mV]
DRIB
RHA
ARA
FUC
RIB
XYL
ST
MAN
GAL
GLC
Time [min]

9

9. Appendix C: Instrument list

1.22 Level instruments

TAG	SERVICE	FLUID	NOTES
LE 101	S-101		Ultrasound level reader
LI 101	S-101		
LT 101	S-101		
LE 102	H-101		Ultrasound level reader
LT 102	H-101		
LE 103	H-103		
LT 103	H-103		
LE 104	RP-101	L	
LC 101	RP-101		
LAH 101	RP-101		
LAHH 101	RP-101		
LT 201	D-203	L	
LIC 201	D-203		
LAH 201	D-203		
LAHH 201	D-203		
LAL 201	D-203		
LALL 201	D-203		
LT 202	D-204		
LIC 202	D-204		
LAH 202	D-204		
LAHH 202	D-204		
LAL 202	D-204		
LALL 202	D-204		
LT 203	D-205	L	
LAH 203	D-205		
LAHH 203	D-205		
LAL 203	D-205		
LALL 203	D-205		
LT 204	D-206	L	
LAH 204	D-206		
LAHH 204	D-206		
LAL 204	D-206		
LALL 204	D-206		
LT 205	D-207	L	
LAH 205	D-207		
LAHH 205	D-207		
LAL 205	D-207		
LALL 205	D-207		
LT 206	D-208	L	
LAH 206	D-208		
LAHH 206	D-208		

LAL 206	D-208		
LALL 206	D-208		
LT 207	D-209	L	
LAH 207	D-209		
LAHH 207	D-209		
LAL 207	D-209		
LALL 207	D-209		
LT 208	D-210	L	
LAH 208	D-210		
LAHH 208	D-210		
LAL 208	D-210		
LALL 208	D-210		
LT 209	D-211	L	
LAH 209	D-211		
LAHH 209	D-211		
LAL 209	D-211		
LALL 209	D-211		
LT 210	D-212	L	
LAH 210	D-212		
LAHH 210	D-212		
LAL 210	D-212		
LALL 210	D-212		
LT 211	EV-201	L	
LAH 211	EV-201		
LAHH 211	EV-201		
LAL 211	EV-201		
LALL 211	EV-201		
LIC 203	EV-201		
LT 212	D-213		
LAH 212	D-213		
LAHH 212	D-213		
LAL 212	D-213		
LALL 212	D-213		
LT 301	D-303	L	
LC 301	D-303		
LAH 301	D-303		
LAHH 301	D-303		
LAL 301	D-303		
LALL 301	D-303		
LT 302	D-304	L	
LC 302	D-304		
LAH 302	D-304		
LAHH 302	D-304		
LAL 302	D-304		
LALL 302	D-304		
LT 303	D-305	L	

LC 303	D-305		
LAH 303	D-305		
LAHH 303	D-305		
LAL 303	D-305		
LALL 303	D-305		
LT 304	D-306	L	
LAH 304	D-306		
LAHH 304	D-306		
LAL 304	D-306		
LALL 304	D-306		
LT 305	D-307	L	
LAH 305	D-307		
LAHH 305	D-307		
LAL 305	D-307		
LALL 305	D-307		
LT 306	D-308	L	
LAH 306	D-308		
LAHH 306	D-308		
LAL 306	D-308		
LALL 306	D-308		
LE 307	D-309	L	
LT 307	D-309		
LAH 307	D-309		
LAHH 307	D-309		
LAL 307	D-309		
LALL 307	D-309		
LT 308	D-310	L	
LAH 308	D-310		
LAHH 308	D-310		
LAL 308	D-310		
LALL 308	D-310		
LT 309	D-312	L	
LAH 309	D-312		
LAHH 309	D-312		
LAL 309	D-312		
LALL 309	D-312		
LT 401	D-401	L	
LAH 401	D-401		
LAHH 401	D-401		
LAL 401	D-401		
LALL 401	D-401		
LIC 401	CC-401		
LE 402	CC-401	L	
LT 402	CC-401		
LAH 402	CC-401		
LAHH 402	CC-401		

LAL 402	CC-401		
LALL 402	CC-401		
LE 403	CC-401	L	
LT 403	CC-401		
LT 404	D-402	L	
LAH 403	D-402		
LAHH 403	D-402		
LAL 403	D-402		
LALL 403	D-402		
LIC 402	D-402		
LT 405	D-403	L	
LAH 404	D-403		
LAHH 404	D-403		
LAL 404	D-403		
LALL 404	D-403		
LIC 403	D-403		
LT 406	DC-401	L	
LAH 405	DC-401		
LAHH 405	DC-401		
LAL 405	DC-401		
LALL 405	DC-401		
LE 407	CC-402	L	
LT 407	CC-402		
LIC 404	CC-402		
LE 408	CC-402	L	
LT408	CC-402		
LAH 406	CC-402		
LAHH 406	CC-402		
LAL 406	CC-402		
LALL 406	CC-402		
LT 409	D-403	L	
LAH 407	D-403		
LAHH 407	D-403		
LAL 407	D-403		
LALL 407	D-403		
LIC 405	D-403		
LT 410	EV-401	L	
LAH 408	EV-401		
LAHH 408	EV-401		
LAL 408	EV-401		
LALL 408	EV-401		
LIC 406	EV-401		
LT 411	EV-402	L	
LAH 409	EV-402		
LAHH 409	EV-402		
LAL 409	EV-402		

LALL 409	EV-402		
LIC 407	EV-402		
LT 412	EV-403	L	
LAH 410	EV-403		
LAHH 410	EV-403		
LAL 410	EV-403		
LALL 410	EV-403		
LT 413	TK-403	L	
LAH 411	TK-403		
LAHH 411	TK 403		
LAL 411	TK-403		
LALL 411	TK-403		
LT 414	TK-402	L	
LAH 412	TK-402		
LAHH 412	TK 402		
LAL 412	TK-402		
LALL 412	TK-402		
LT 415	TK-401	L	
LAH 413	TK-401		
LAHH 413	TK 401		
LAL 413	TK-401		
LALL 413	TK-401		

1.23 Flow instruments

TAG	SERVICE	FLUID	NOTES
FE 201	TX-201	L	Reads soaked BSG flow
FT 201	TX-201		
FI 201	TX-201		
FE 202	TX-201	L	Reads solvent flow
FT 202	TX-201		
FI 202	TX-201		Displays soaked BSG flow
FI 202	TX-201		Displays solvent flow
FRC 201	TX-201		
FE 203	UF-201 / UF 202	L	
FIC 201	UF-201 / UF 202		
FE 204	NF-201 / NF 202	L	
FIC 202	NF-201 / NF-202		
FE 205	EV-201	LPS	
FT 203	EV-201		
FE 206	EV-201	L	
FT 204	EV-201		

FRC 202	EV-201		
FE 207	DD-213	L	
FIC 203	DD-213		
FE 301	W-301	L	
FT 301	W-301		
FE 302	W-301	L	
FI 302	W-301		
FRC 301	W-301		
FE 303	TX-301	L	
FT 303	TX-301		
FI 303	TX-301		
FRC 302	TX-301		
FE 304	D-305		
FT 304	D-305		
FIC 301	D-305		
FE 305	D-306		
FT 305	D-306		
FIC 302	D-306		
FE 306	D-307		
FT 306	D-307		
FE 307	D-307		
FT 307	D-307		
FRC 303	D-307		
FE 308	CD-301		
FT 308	CD-302		
FIC 303	CD-302		
FE 309	HP-303		
FT 309	HP-303		
FE 310	D-308		
FT 310	D-308		
FIC 305	D-308		
FE 311	D-308		
FT 311	D-308		
FE 312	D-308		
FT 312	D-308		
FRC 304	D-308		
FE 314	D-309		
FT 314	D-309		
FIC 306	D-309		
FE 314	D-309		
FT 314	D-309		
FE 315	D-309		
FT 315	D-309		
FRC 305	D-309		
FE 316	CD-303		
FT 316	CD-303		

FIC 307	CD-303		
FE 317	HP-304		
FT 317	HP-304		
FE 318	CD-302		
FT 318	CD-302		
FE 319	CD-303		
FT 319	CD-303		
FE 401	CC-401	L	
FT 401	CC-401		
FIC 401	CC-401		
FE 402	CC-401	L	
FT 402	CC-401		
FIC 402	CC-401		
FE 403	SIC 401	L	
FT 403	SIC 401		
FE 404	CC-402	L	
FT 404	CC-402		
FIC 403	CC-402		
FE 405	CC-402	L	
FT 405	CC-402		
FIC 404	CC-402		
FE 406	EV-401	V	
FT 406	EV-401		
FE 407	EV-401	L	
FT 407	EV-401		
FRC 401	E-401		

1.24 Temperature instruments

TAG	SERVICE	FLUID	NOTES
TE 101	B-101	G	
TC 101	B-101		
TAH 101	B-101		
TAHH 101	B-101		
TAL 101	B-101		
TALL 101	B-101		
TE 201	D-206	L	
TT 201	D-206		
TIC 201	D-206		
TAH 201	D-206		
TAHH 201	D-206		
TE 202	D-207	L	
TT 202	D-207		
TIC 202	D-207		
TAH 202	D-207		
TAHH 202	D-207		
TE 203	D-208	L	

TT 203	D-208		
TIC 203	D-208		
TAH 203	D-208		
TAHH 203	D-208		
TE 204	D-210	L	
TT 204	D-210		
TIC 204	D-210		
TAH 204	D-210		
TAHH 204	D-210		
TE 205	D-211	L	
TT 205	D-211		
TIC 205	D-211		
TAH 205	D-211		
TAHH 205	D-211		
TE 206	D-212	L	
TT 206	D-212		
TIC 206	D-212		
TAH 206	D-212		
TAHH 206	D-211		
TE 207	D-211	L	
TT 207	D-211		
TI 207	D-211		
TAH 207	D-211		
TAHH 207	D-211		
TE 208	DD-201	*	*On the dryer's roll skin
TT 208	DD-201		
TIC 208	DD-201		
TE 401	CC-401	L	On a pilot tray in the stripping section of the column
TT 401	CC-401		
TIC 401	CC-401		
TE 402	CC-401	V	On a pilot tray in the rectification section of the column
TT 402	CC-401		
TI 401	CC-401		
TAH 401	CC-401		
TAHH 401	CC-401		
TAL 401	CC-401		
TALL 401	CC-401		
TE 403	CC-402	L	On a pilot tray in the stripping

			section of the column
TT 403	CC-402		
TIC 402	CC-402		
TE 404	CC-402	V	On a pilot tray in the rectification section of the column
TT 404	CC-402		
TI 402	CC-402		
TAH 402	CC-402		
TAHH 402	CC-402		
TAL 402	CC-402		
TALL 402	CC-402		
TE 405	EV-402	L	
TT 405	EV-402		
TI 403	EV-402		
TAH 403	EV-402		
TAHH 403	EV-402		
TE 406	EV-403	L	
TT 406	EV-403		
TAH 404	EV-403		
TAHH 404	EV-403		

1.25 Speed instruments and inverters

TAG	SERVICE	NOTES
SC-101	HP-101	
SC-102	XF-101	
SC-103	HP-102	
SC-104	XF-102	
SC-105	XF-103	
SC-106	XF-104	
SIC 201	XF-201	
SE 201	XF-201	
ST 201	XF-201	
I 201	XF-201	
SC-301	XF-301	
SE 301	XF-302	
ST 301	XF-302	
SIC 302	XF-302	
I 301	XF-302	
SE 302	RV-301	
ST 302	RV-301	

SIC 303	RV-301	
I 302	RV-301	
SE 303	HP-302	
ST 303	HP-302	
SIC 304	HP-302	
I 304	HP-302	
SE 304	HP-303	
ST 304	HP-303	
SIC 305	HP-303	
I 305	HP-303	
SE 305	CD-302	
ST 305	CD-302	
SIC 306	CD-302	
SE 306	CD-304	
ST 306	CD-304	
SIC 307	CD-304	
I 306	HP-304	
I 307	CF-301	
I 308	SC-301	
I 309	SC-302	
I 310	DD-302	
I 311	HP 305	
I 312	CF-302	
I 313	SC-303	
I 314	SC-304	
I 315	DD-301	
I 316	CD-302	
I 317	CD-303	

1.26Pressure instruments

PE 101	C-101	Air	
PT 101	C-101		
PS 101	C-101		
PSV 201	D-203	VOC	
PSV 202	D-204	VOC	
PSV 203	D-204	VOC	
DPE 201	UF 201	L	
DPT 201	UF 201		
DPSH 201	UF 201		
DPAH 201	UF 201		
DPE 202	UF 202	L	
DPT 202	UF 202		
DPSH 202	UF 202		
DPAH 202	UF 202		
PSV 204	D-209	VOC	

DPE 203	NF 201	L	
DPT 203	NF 201		
DPSH 203	NF 201		
DPAH 203	NF 201		
DPE 204	NF 201	L	
DPT 204	NF 201		
DPSH 204	NF 201		
DPAH 204	NF 201		
PSV 204	EV-201	V	
PE 201	EV-201	V	
PT 201	EV-201		
PIC 201	EV-201		
PSV 206	D-211	VOC	
PSV 301	D-303	VOC	
PSV 302	D-304	VOC	
PSV 303	D-306	VOC	
PSV 304	D-307	VOC	
PSV 305	D-308	VOC	
PSV 306	D-309	VOC	
PSV 401	D-401	VOC	
PE 401	CC-401	V	
PT 401	CC-401		
PI 401	CC-401		
PE 402	CC-401	V	
PT 402	CC-401		
PIC 401	CC-401		
PSV 402	D-403	VOC	
PE 403	CC-402	V	
PT 403	CC-402		
PI 402	CC-402		
PE 404	CC-402	V	
PT 404	CC-402		
PIC 402	CC-402		
PSV 403	EV-401	V	
PE 405	EV-402	V	
PT 405	EV-402		
PIC 403	EV-402		
PSV 404	EV-402	V	
PE 406	EV-403	V	
PT 406	EV-403		
PIC 404	EV-403		
PSV 405	EV-403	V	
PE 407	EV-403	V	
PT 407	EV-403		
PIC 405	EV-403		

1.27Weight instrumensts

TAG	SERVICE	NOTES
WE 101	H-102	
WT 101	H-102	
WE 102	H-104	
WT 102	H-104	
WE 103	H-105	
WT 103	H-105	
WE 104	H-106	
WT 104	H-106	
WT 201	H-201	
WE 201	XF-201	
WE 202		
WT 202	XF-203/XF-204	
WE 301	XF-301	
WT 301	XF-301	
WE 302	XF-302	
WT 302	XF-302	

1.28Time-actionated instruments

TAG	SERVICE	FLUID	NOTES	ACTIONS
KS 201	D-201	L		Open XV-276
KIC 201	UF 201/ UF 202	L		If UF-201 needs regeneration: Close XV-204. XV-206, XV-207, XV-209, XV-212. XV-213, XV-215, XV-218; Open XV-205, XV-208, XV-210, XV-211, XV-214, XV-216, XV-217, XV 219; opposite actions if UF-202 needs regeneration In both cases, open XV-222, XV-224, XV-228, XV-230,

				XV-233, XV-235
KIC 202	NF-201 / NF-202	L		If UF-201 need regeeration: Close XV-239. XV-241, XV-242, XV-244, XV-247. XV-248, XV-250, XV-253; Open XV-240, XV-243, XV-245, XV-246, XV-249, XV-251, XV-252, XV 254; opposite actions if UF-202 needs regeneration In both cases, open XV-257, XV-259, XV-262, XV-264, XV-266, XV-268
KIC 203	D-206	L		Open XV-225
KIC 204	D-206	L		Open XV-221
KIC 205	D-207	L		Open XV-231
KIC 206	D-207	L		Openv XV-226
KIC 207	D-208	L		Open XV-236
KIC 208	D-210	L		Open XV269
KIC 209	D-210	L		Open XV-255
KIC 210	D-211	L		Open XV-270
KIC 211	D-211	L		Open XV260
KIC 212	D-212	L		Open XV-271
KS 303	D-314	L		Starts invertes I 305 to I 307
KS 304	D-322	L		Starts invertes I 308 and I 309
KS 305	D-320	L		Starts inverters I310 to I 312
KS 306	D-321	L		Starts inverters I 313 and I 314

1.29Composition and pH instruments

TAG	SERVICE	NOTES
pHE 301	D-305	
pHT 301	D-305	
pHC 301	D-305	
CE 401	TK-402	
CT 401	TK-402	
CIC 401	TK-402	

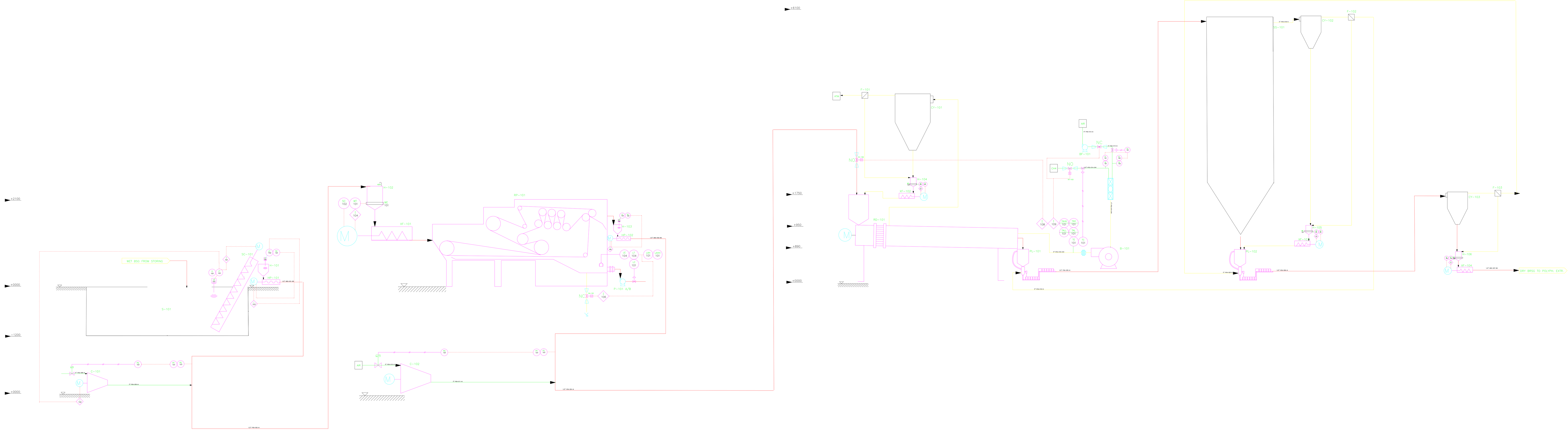
10. Appendix D: Interlock description

TAG	FUNCTION	EQUIPMENT	ACTION
I 101	LAL, LALL	S-101	Stop SC-101
I 102	LAL, LALL	S-101	Stop C-101
I 103	LAL, LALL	S-101	Stop HP-101
I 104	WAL, WALL	H-102	Stop XF-101
I 105	LAL, LALL	H-103	Stop HP-103
I 106	LAH, LAHH	RP-101	Open XV-101
I 107	WAL, WALL	H-104	Stop XF-102
I 108	TAH, TAHH	B-101	Close XV-102 (solid feed to RD-101
I 109	TAH, TAHH	B-101	Close XV-103 (methane feed to B-101
I 110	WAL, WALL	H-105	Stop XF-103
I 111	WAL, WALL	H-106	Stop XF-104
I 201	WAL, WALL	H-201	Stop XF-201
I 202	WAL, WALL	D-202	Stop XF-203
I 203	LAH, LAHH	D-203	Open XV-201
I 204	LAH, LAHH	D-204	Open XV-202
I 205	LAH, LAHH	D-205	Open XV-203
I 206	LAH, LAHH	D-206	Open XV-220
I 207	TAH, TAHH	D-206	Close XV-224
I 208	LAH, LAHH	D-207	Open XV-227
I 209	TAH, TAHH	D-207	Close XV-229
I 210	LAH, LAHH	D-208	Open XV-232
I 211	TAH, TAHH	D-208	Close XV-234
I 212	LAH, LAHH	D-209	Open XV-238
I 213	LAH, LAHH	D-210	Open XV-256
I 214	TAH, TAHH	D-210	Close XV-258

I 215	LAH, LAHH	D-211	Open XV-261
I 216	TAH, TAHH	D-211	Close XV-263
I 217	LAH, LAHH	D-212	Open XV-265
I 218	TAH, TAHH	D-212	Close XV-267
I 219	TAH, TAHH	EV-201	Close XV-272
I 220	LAH, LAHH	EV-201	Close XV-273
I 221	LAH, LAHH	D-213	Open XV-274
I 222	TAH, TAHH	DD-201	Close XV-275
I 301	WAL, WALL	H-301	Stop XF-301
I 302	WAL, WALL	D-301	Stop XF 302
I 303	LAH, LAHH	D-303	Open XV-302
I 304	LAH, LAHH	D-304	Open XV-303
I 305	LAH, LAHH	D-305	Open XV-304
I 306	LAH, LAHH	D-306	Open XV-305
I 307	LAH, LAHH	D-307	Open XV-306
I 308	LAH, LAHH	D-308	Open XV-307
I 309	LAH, LAHH	D-309	Open XV-308
I-310	LAH, LAHH	D-310	Open XV-309
I 312	LAH, LAHH	D-312	Open XV-316
I 313	TAH, TAHH	DD-302	Close XV-314
I 315	TAH, TAHH	DD-301	Close XV-318
I 401	LAH, LAHH	D-401	Open XV-401
I 402	LAH, LAHH	D-403	Open XV-402
I 403	LAH, LAHH	DC-401	Open XV-403
I 404	LAH, LAHH	EV-401	Open XV-404
I 405	LAH, LAHH	EV-402	Open XV-405
I 406	LAH, LAHH	EV-403	Open XV-406
I 407	LAH, LAHH	TK-403	Open XV-407

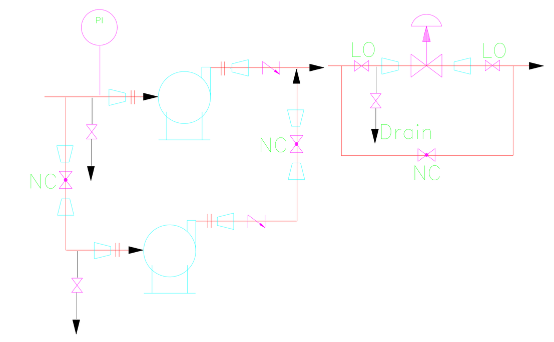
11. Appendix F Preliminary P&ID

BSG PRE-TREATMENT SECTION (SECTION 1)

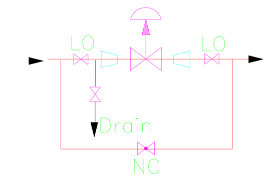


General Notes

Note 1:Valid for all regulated centrifugal pumps unless otherwise specified



Note 2:Valid for all regulation valve unless otherwise specified



Equipment nomenclature	
B-XXX	Inline burner
BF-XXX	Blowing fan
C-XXX	Air compressor
CC-XXX	Distillation column
CD-XXX	Centrifugal decanter
CY-XXX	Cyclone
D-XXX	Process tank
DC-XXX	Static decanter
DD-XXX	Drum dryer
F-XXX	Air filter
EV-XXX	Evaporator
H-XXX	Hopper
HP-XXX	Progr. cavity pump
LP-XXX	Rotatory lobes pump
LF-XXX	Liquid filter
UF-XXX	Ultrafiltration m.
NF-XXX	Nanofiltration m.
RP-XXX	Roll press filter
S-XXX	Underground st. tank
SC-XXX	Screw conveyor
SP-XXX	Squeezing press
SS-XXX	Blender silo
PL-XXX	Dense ph. pneum. tr.
TK-XXX	Storage tank
TX-XXX	TURBEX
XF-XXX	Screw feeder

Primary process line

Secondary process line

Utility line

Pneumatic signal

Electrical signal

No.	Revision/Issue	Date

Drawing Name

BSG PRE-TREATMENT SECTION (SECTION 1)

Project Name and Address

BSG VALUATION PROCESS

Area

SECTION 1

Stamp

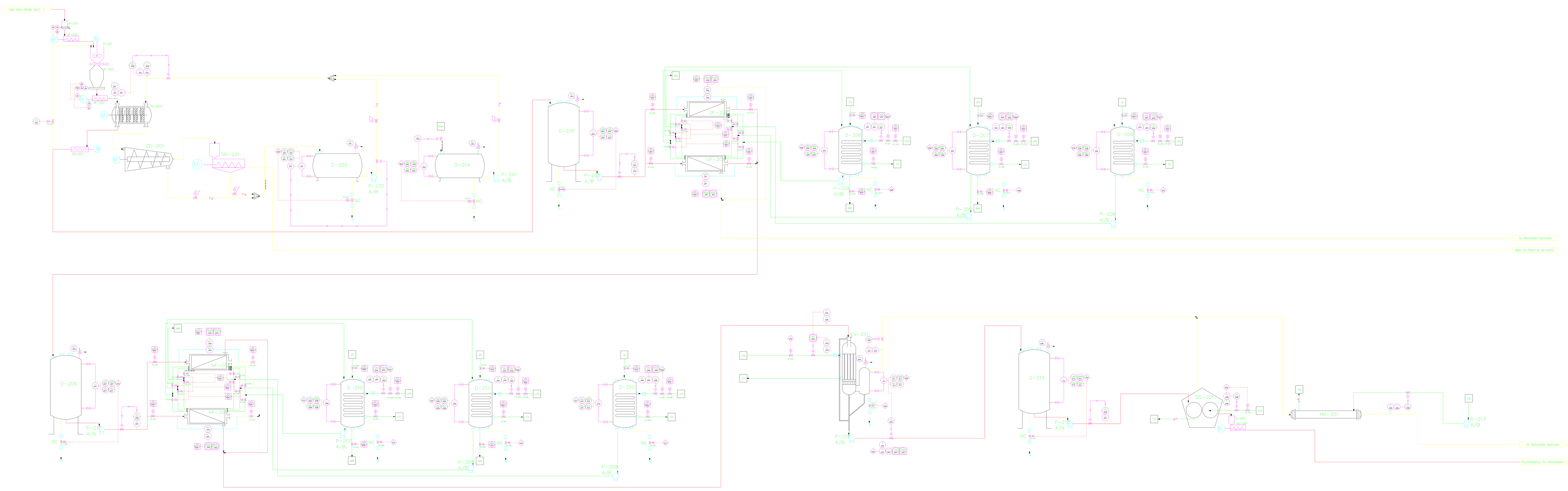
Drawing Number

1 OF 4

Author

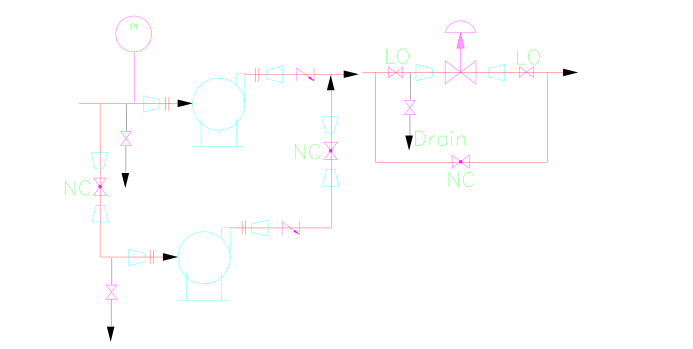
Roberto Chignine

POLYPHENOLS-RICH PRODUCT EXTRACTION SECTION (SECTION 2)



General Notes

Note 1:Valid for all regulated centrifugal pumps unless otherwise specified



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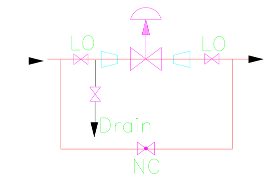

Equipment nomenclature	
B-XXX	Inline burner
BF-XXX	Blowing fan
C-XXX	Air compressor
CC-XXX	Distillation column
CD-XXX	Centrifugal decanter
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DD-XXX	Drum dryer
F-XXX	Air filter
EV-XXX	Evaporator
H-XXX	Hopper
HP-XXX	Progr. cavity pump
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UF-XXX	Ultrafiltration m.
NF-XXX	Nanofiltration m.
RP-XXX	Roll press filter
S-XXX	Underground st. tank
SC-XXX	Screw conveyor
SP-XXX	Squeezing press
SS-XXX	Blender silo
PL-XXX	Dense ph. pneum. tr.
TK-XXX	Storage tank
TX-XXX	TURBEX
XF-XXX	Screw feeder

Primary process line

Secondary process line

Utility line

Pneumatic signal

Electrical signal

No.	Revision/Issue	Date

Drawing Name

POLYPHENOLS EXTRACTION SECTION

Project Name and Address

BSG VALUATION PROCESS

Area

SECTION 2

Stamp

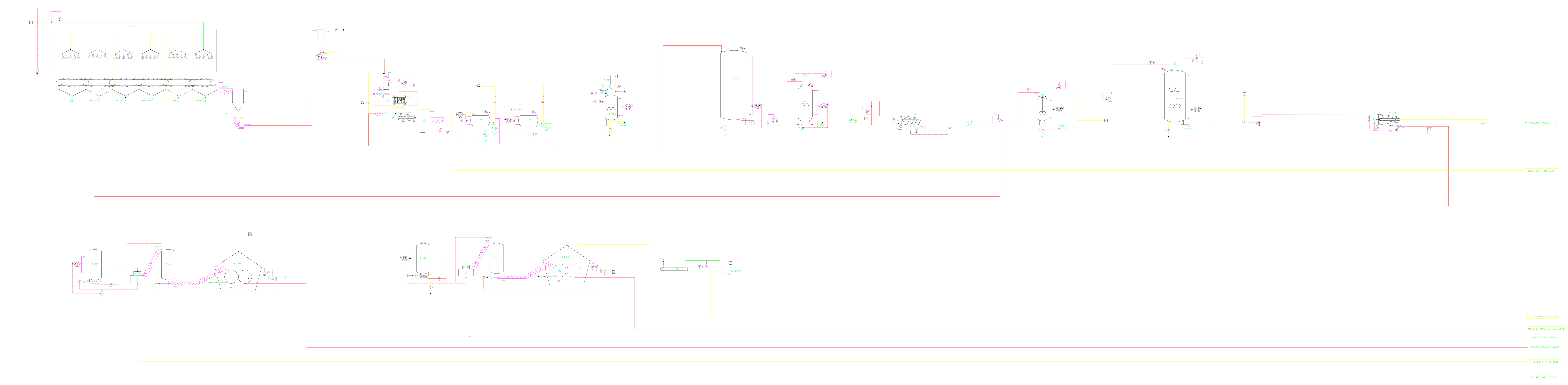
Drawing Number

2 OF 4

Author

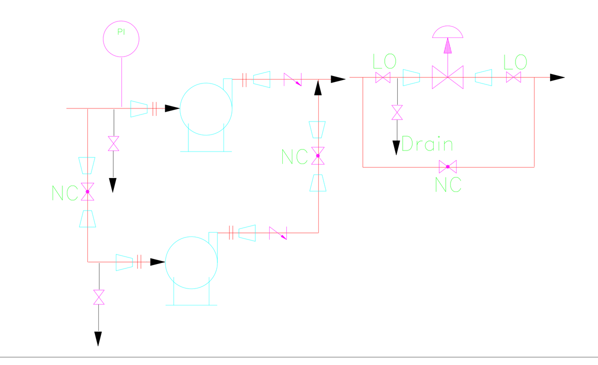
Roberto Chighine

ARABINO-XYLANES AND PROTEINS EXTRACTION SECTION (SECTION 3)



General Notes

Note 1:Valid for all regulated centrifugal pumps unless otherwise specified



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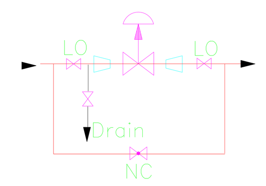


Equipment nomenclature	
B-XXX	Inline burner
BF-XXX	Blowing fan
C-XXX	Air compressor
CC-XXX	Distillation column
CD-XXX	Centrifugal decanter
CY-XXX	Cyclone
D-XXX	Process tank
DC-XXX	Static decanter
DD-XXX	Drum dryer
F-XXX	Air filter
EV-XXX	Evaporator
H-XXX	Hopper
HP-XXX	Progr. cavity pump
LP-XXX	Rotatory lobes pump
LF-XXX	Liquid filter
UF-XXX	Ultrafiltration m.
NF-XXX	Nanofiltration m.
RP-XXX	Roll press filter
S-XXX	Underground st. tank
SC-XXX	Screw conveyor
SP-XXX	Squeezing press
SS-XXX	Blender silo
PL-XXX	Dense ph. pneum. tr.
TK-XXX	Storage tank
TX-XXX	TURBEX
XF-XXX	Screw feeder

Primary process line

Secondary process line

Utility line

Pneumatic signal

Electrical signal

No.	Revision/Issue	Date

Drawing Name

ARABINO-XYLANES AND PROTEINS EXTRACTION SECTION

Project Name and Address

BSG VALUATION PROCESS

Area

SECTION 3

Stamp

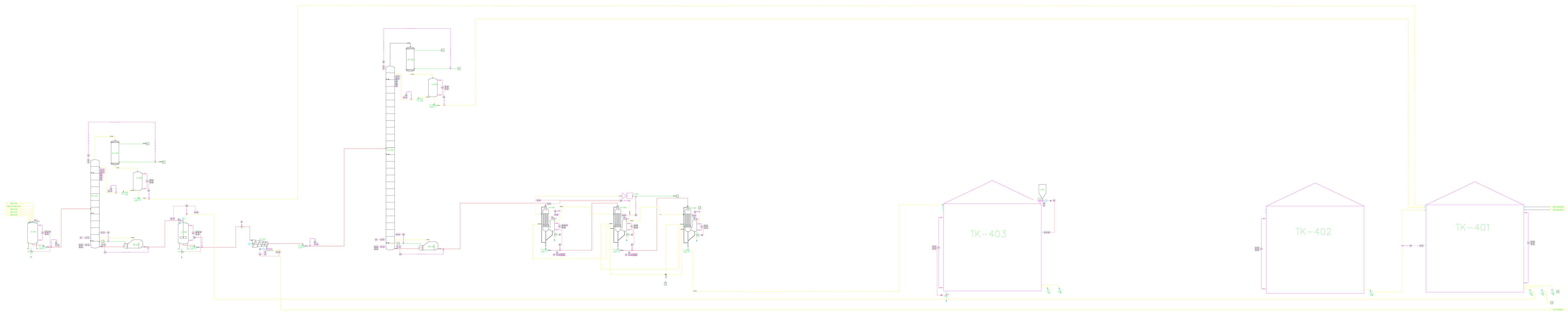
Drawing Number

3 OF 4

Author

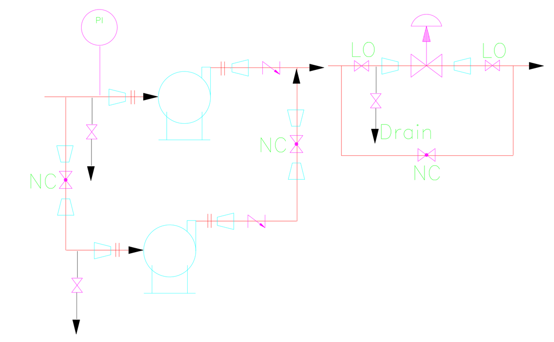
Roberto Chighine

SOLVENT AND CHEMICALS RECOVERY SECTION (SECTION 4)

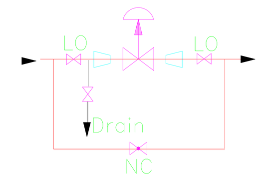


General Notes

Note 1:Valid for all regulated centrifugal pumps unless otherwise specified



Note 2:Valid for all regulation valve unless otherwise specified



Equipment nomenclature	
B-XXX	Inline burner
BF-XXX	Blowing fan
C-XXX	Air compressor
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CD-XXX	Centrifugal decanter
CY-XXX	Cyclone
D-XXX	Process tank
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DD-XXX	Drum dryer
F-XXX	Air filter
EV-XXX	Evaporator
H-XXX	Hopper
HP-XXX	Progr. cavity pump
LP-XXX	Rotatory lobes pump
LF-XXX	Liquid filter
UF-XXX	Ultrafiltration m.
NF-XXX	Nanofiltration m.
RP-XXX	Roll press filter
S-XXX	Underground st. tank
SC-XXX	Screw conveyor
SP-XXX	Squeezing press
SS-XXX	Blender silo
PL-XXX	Dense ph. pneum. tr.
TK-XXX	Storage tank
TX-XXX	TURBEX
XF-XXX	Screw feeder

Primary process line

Secondary process line

Utility line

Pneumatic signal

Electrical signal

No.	Revision/Issue	Date

Drawing Name

RECOVERY SECTION

Project Name and Address

BSG VALUATION PROCESS

Area

RECOVERY SECTION

Stamp

Drawing Number

4 of 4

Author

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