POLITECNICO DI TORINO

Master's Degree in Environmental and Land Engineering
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Master's Thesis

Characterization of Cutting Sludge from Ornamental Stone and Possible Recovery Solutions for Circular Economy in Different Scenarios

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Abstract

The current global industrial sector is desperately searching for a new material that can replace the expensive and, often, rare raw material. Furthermore, the problem of Greenhouse Gases by pollution is an actual emergency that especially affects cement production plants and they must find new ways to decrease emissions and find sources that can be more sustainable. The study aims to recover a typical waste of ornamental stone plants, which is the sludge produced by cuttings operated by diamond tools, in rock types such as diorite and marble. Both stones have different mechanical behaviors; however, the research wants to analyze, with typical soil and stone characterization, the content of the waste, and then find new applications that can be suitable based on the results obtained. The research has been performed with two different wastes. Different experimental evaluations were carried out to find some matches as byproducts. The characterization is comprehensive of: preliminary analysis, geotechnical evaluations, load test in laterally confined socket, XRD and XRF, and compression stress after a certain curing time. Some of the quoted evaluations were carried out comparing the behavior with the addition of a percentage, equal to 10%, of cement and samples at different conditions, such as samples dried overnight and others stabilized for one hour. Using literature and British standard references, the study aims to find how ornamental stone cuttings could replace cement in certain aspects by comparing mechanical, chemical, and physical properties.

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Chapter 1

Introduction

In recent years, every industrial sector is began a new type of economy and approach of the production chain from the raw material to the final product. That new concept is called **Circular Economy** where every material, every waste coming from different sectors is probably valuable in other production facilities. The current thesis study wants to analyze the recent critical sector of stone cuttings, specifically here **Ornamental Stone Cuttings** coming from the granite processing. In Italy there are 10,373 companies that extract and process stone, of which 9313 deal with natural stone [1]. Among those numbers: Turin, Cuneo and Verbania are the locations with the most production facilities of natural stone on the entire Piedmont region [1]. They account for 97% of the total production plant in the region and in the last 5 years they have extracted on average 1009 tons of raw material. Although, the resulting amount of sludge produced is about 37,000 tons [1].

It is usually classified as **Waste** and it is sent to landfill. Therefore, to the Circular Economy approach, it is necessary to change the life cycle of the product and, since granite could have great mechanical properties, the study wants to discover new paths for recovering and reusing the valuable material. One of possible solutions is in the road subgrade in which the foundation layer is made of bound mixture, granular mixture or hydraulic mixture [2]. It is important to specify it is not a high module layer, namely, the largest amount of load is supported by the superficial layers like the binder layer or the base layer [2].

Another important feature that road subgrade must have is the low plasticity index [3]. It must be under 30% and if the soil was of low compressibility, the requirements are fulfilled [3]. To start the experimental evaluation, a natural stone facility was selected in Turin province.

The selected company was Tomaino Graniti s.r.l located at Castellamonte. It owns a quarry, located in Traversella, where it extracts diorite plutonic rocks, and monthly it produces about 3-4 m^3 of cutting stone sludge depending on the demand. The quarry is stretched out for about two hectares on the left bank of Bersella stream

valley in the western area of the dioritic pluton that characterizes the location [4]. The Traversella mineralizations are strictly related to the homonymous pluton, an intrusive body of quartz-diorite to monzodiorite composition of Oligocene Age (about 30 million years). The mechanism of its formation is the pluton intrusion of a tectonic unit belonging to the Austroalpine Domain, mainly composed of micaschist and gneiss, with minor metabasite bodies [5].

In that quarry *Diorite chiara* is extracted, and its texture is characterized by gray (that is typical of quartz-rich rocks), medium and large particles which give a high level of hardness [4]. In the production facility, Tomaino Graniti uses two kinds of cutting tools:

- Multi-Wire Machine to quickly cut the block and to produce flagstones
- Bridge milling machine with 1,600 discs

In the literature, many studies regarding performances of those machines can be found. Emre [6] evaluated the different performances of a mono-wire cutting system by analyzing mechanical and energy parameters. The paper studied the evolution of the **Unit Wear**, which is useful to understand the friction and consumption of diamond wire (*Kerf loss*). By adjusting the peripheral speed (which is the rotational speed applied to the wire) and the cutting speed (that is related to the descending motion along the block), the Unit Wear changes by quadratic or cubic dependencies [6].

Spriano [7] investigated the consumption of diamond beads according to the type of stone cut. The analysis included a mineralogical characterization of stone debris in order to evaluate the influence of the instrument. The study assumed to use 7 mm diameter wires, which is the reference standard for stone slabbing (usually granite stone). Spriano [7] stated:

> "*[...] deep knowledge of interaction between the diamond beads and stones turns into a whole comprehension of the cutting process. This will allow us to optimize cutting action: a double examination (stones and beads) represents a complete approach to the comprehension of the wear processes during stone cutting.[...]*"

The cutting mud, resulting from using diamond wire tools, has great variability in particle size, shape and conformation [7]. The mineralogical analysis has allowed the discovery of metal powder and super-abrasive grains in the waste composition. Hence, Spriano [7] states that the chemical composition of the metal diamond bead matrix directly influences the efficiency of the cutting action, and diamonds must remain in the diamond wire as much as possible until they show cutting ability. However, another important feature discovered is that iron-based stones drastically reduce the lifetime of diamond wire [7]. Since dioritic rocks have many

Fe-based grains, it is a feature to be evaluated during wire choice. It is, henceforth, demonstrated that granite rocks provide high hardness, and as quoted so far, the sludge coming from cutting blocks is valuable.

Chapter 2

Legislative framework on National and regional level

2.1 Italian framework

2.1.1 Italian law: Environmental Code D. Lgs. April 3rd 2006, No 152

The primary regulatory reference regarding municipal and industrial waste is the **Environmental Code** (Legislative Decree April 3rd 2006, n. 152), whose articles define treatment protocols, recovery and recycling strategies, destinations, and relative emissions for any compound classified as waste. According to *Article 183, subpara.1a* of the Act, **waste** is defined:

> "*any substance or object which the holder discards or intends or is required to discard.*"[8]

In the same article, *subpara.6f* defines the **waste producer** as:

> "*the person whose activity produces waste and the subject to whom said production is legally attributable (initial producer) or anyone who performs pre-treatment, mixing, or other operations that have modified the nature or composition of said waste (new producer).*"[8]

Article 183 further clarifies several key technical expressions:[8]

- **Preparation for reuse** (*Art. 183, subpara.6q*): operations such as checking, cleaning, or repairing, through which products or components that have become waste are prepared to be reused without further pre-treatment
- **Reuse** (*Art. 183, subpara.6r*): any operation by which products or components that are not waste are used again for the same purpose for which they were originally conceived

- **Treatment** (*Art. 183, subpara.6s*): recovery or disposal operations, including preparation before such actions
- **Recovery** (*Art. 183, subpara.6t*): any operation whose main result is to allow waste to serve a useful purpose by replacing other materials
- **Recovery of matter** (*Art. 183, subpara.6t-bis*): any recovery operation other than energy recovery; this includes recycling and back-filling
- **Recycling** (*Art. 183, subpara.6u*): recovery operations where waste is processed into products or substances for their original or other purposes (excluding energy recovery)
- **Back-filling** (*Art. 183, subpara.6u-bis*): a recovery operation where suitable non-hazardous waste is used for reclamation in excavated areas or for engineering purposes in landscaping

The present decree defines another important product: **Byproduct (Art. 184-bis, subpara.1**: it's a byproduct and not a waste regarding the *Art. 183, subpara.1a*, any substance or object which foresees the following conditions:[8]

- The substance or the object is manufactured by a production process, which is a part of it, and the primary purpose of which is not the production of that substance or object;
- the substance or object will certainly be used, in the course of the same or a subsequent production or utilization process, by the producer or third parties;
- the substance or object can be used directly without any further treatment other than normal industrial practice;
- the further use is legal, that is, the substance or object meets, for the specific use, all relevant requirements concerning the products, the protection of health, and the environment, and it will not lead to overall adverse impacts on the environment or human health

In the same article, *subpara.2* defines the reasons for including waste among the by-products "*guaranteeing a high level of protection of the environment and human health, also promoting the careful and rational use of natural resources, giving priority to replicable practices of industrial symbiosis*"[8] A waste ceases to be defined in this way, according to Article 184-ter, when "*it has been undergone a recovery operation, including recycling, and meets the specific criteria*"[8], by listing other specifications. In general, to be included in a production chain, waste must "be subjected to a recovery operation, including recycling, and meet the specific criteria" be respected with the following conditions:[9]

- The material must be used on other industrial sector or different "specific purpose"
- The existence of a market and trade for the substance
- The material meets the technical and regulatory requirements for its specific market
- There is no negative environmental or health impact

Pursuant to Article 2 of the decree, recovery operations are contingent upon a verification process ensuring that the waste materials strictly adhere to the stipulated criteria. Conversely, the subsequent paragraph clarifies that in instances where specific criteria have not been predefined, recovery may still proceed via case-by-case authorizations granted by the competent authorities, including: [8]

- Entry of eligible material for the recovery operation
- Allowed process and techniques
- Quality criteria obtained by recovery operation and fulfillment of limit values
- Requirements for compliance with refusal qualification termination criteria that control systems must demonstrate (self-monitoring)
- Agreement claims

In Annex V Title V Part IV is summarized an important table regarding contaminant thresholds in soil, subsoil and underground reservoir according to the specific destination of the site: public or industrial. Table 2.1 shows thresholds for some of the contaminants that are interesting for the thesis study. Since the Tomaino Graniti plant produces an industrial wastewater that contains part of the water removed from the sludge in the dewatering section, Table 2.2 displays the threshold limits that must comply.

2.1.2 Italian law: D. Lgs. May 30th, 2008, No 117

Furthering the study on another important regulation, Legislative Decree 3 April 2006 n° 152 defines waste treatments and destinations from a general point of view, Legislative Decree 117/2008 accurately defines the destinations, strategies, and treatments of excavation waste and the mineral resources resulting from the processing and extraction of rocks in quarries.[9]. In detail, in *art. 3 subpara.f* defines the mineral or mineral resource as "*a natural deposit in the Earth's crust of organic or inorganic substances, such as energy fuels, metallic minerals, industrial minerals and minerals for construction, excluding water*"[9]. In the following

Table 2.1: Annex V Table 1 regarding thresholds in soil, subsoil or underground reservoir

Metal	**Column A Green/Public site [mg/kg ss]**	**Column B Industrial site [mg/kg ss]**
Sb	10	30
As	20	50
Cd	2	15
Co	20	250
Ni	120	500
Pb	100	1000
Cu	120	600
Zn	150	1500
Hg	1	5
Cr Total	150	800
Cr IV	2	15

Table 2.2: Threshold limits for Sewage Network discharge of industrial wastewater (Annex V, Title V, Part 3)

Parameter	**Threshold (mg/L)**
Al	2.0
As	0.5
Ba	—
B	4.0
Cd	0.02
Total Cr	4.0
Cr VI	0.2
Fe	4.0
Mn	4.0
Hg	0.005
Ni	4.0
Pb	0.3
Cu	0.4
Se	0.03
Sn	—
Zn	1.0

paragraph, it also defines what industries, engaged in the transformation of mineral

resources, are *extractive industries*, that is

> "*all establishments and enterprises engaged in the extraction, surface or underground, of mineral resources for commercial purposes, including drilling, extraction or the treatment of extracted material*" [9]

It is useful to point out that in *art. 5 subpara.1* an important aspect is defined regarding the operator's responsibility and fundamental task

> "*The operator shall draw up a management plan for extraction waste for the minimization, treatment, recovery and disposal of the waste itself, in compliance with the principle of sustainable development*" [9]

The operator has the fundamental task of ensuring sustainable development during waste management and, as *para 2a* of the same article states, is aimed at "*preventing or reducing the production of extraction waste and its dangerousness*" [9] Furthermore, *subpara.2b* establishes the ultimate purpose for this type of liability, definitively establishing the reasons why sustainable waste management is required[9]

> "*incentivize the recovery of extraction waste through recycling, reuse or remediation of the extraction waste concerned, if these operations do not pose risks to the environment, in accordance with current environmental standards and, where relevant, to the requirements of this decree.*"

Subpara.3 of art. 5 sets out some points on the study of the composition of waste and its classification, including a careful study of the storage, where present, to be described on the management plan, namely[9]

> "*it establishes the need to provide the characterization of waste and the quantity at which it is produced, a description of the operations for which waste is produced and, where present, the processing operations, a classification of the structure of the repository containing the waste, a description of the manner in which adverse effects on the environment and human health may arise, the control and monitoring procedures, the proposed plan for closing the site, measures to prevent water deterioration, a description of the structure of the repository*"

To characterize the waste, this decree requires, in *Annex I*, all the indications and requests that a plant must provide in the management plan. Annex I quotes as follows: "Extraction waste to be deposited in a storage facility must be characterized in such a way as to ensure the long-term physicochemical stability of the warehouse that accommodates it and prevent the occurrence of major accidents. The characterization shall include, if appropriate and according to the category of the extraction waste storage facility, the following elements:[9]

- description of the expected physical and chemical characteristics of the mining waste to be deposited in the short and long term, with particular reference to its stability to surface atmospheric/meteorological conditions, taking into account the type of ore or minerals extracted and the nature of the overburden and/or gangue minerals that will be removed during mining operations
- classification of extraction waste under the relevant heading of Decision 2000/532/CE, with particular regard to hazard characteristics
- description of the chemicals to use during the treatment of the mineral resources and their relative stability
- description of the storage method
- transport system adopted to move the mining waste

2.1.3 Italian law: D.P.R. September 8th, 1997, No 357

Italian law imposes various regulations regarding the conservation of the natural environment, defining several frameworks for the restoration and maintenance of the excavation site. In particular, in *art. 2* of *Presidential Decree 357/1997* it defines **Conservation status of a natural habitat**[10] (*subpara.1e*):

> "the effect of the sum of the factors that influence the natural habitat as well as the typical species found in it, which can alter, in the long term, its natural distribution, structure and functions, as well as the survival of its typical species"[10]

Subpara.1e suggests some guidelines on what a *natural habitat* should look like by definition at a satisfactory level, therefore, what the distribution area, structure, and specific functions suitable for its maintenance and the conservation status of typical species should look like. [10]

2.1.4 Italian law: Ministerial decree June 28th,2024, No 127

With reference to the Environmental Code, in particular Article 184-ter, paragraph 2, quoted at subsection 2.1.1, in 2024 the Italian Parliament issued this decree regarding the "Regulation for the Termination of the Classification of Waste from Construction, Demolition and Other Inerts of Mineral Origin" by which it establishes the actual procedure and limits of concentrations of pollutants contained within processing waste and present on construction sites that can be revalued as a by-product in other industrial or tertiary sectors. [11] Furthermore, with respect to the definitions cited in subsection 2.1.1, the aforementioned decree describes [11]

- **Inert waste from construction and demolition activities**: waste from construction and demolition operations identified in Chapter 17 of the European Waste List referred to in Commission Decision 2000/532/CE of May 3, 2000, where listed in Annex 1, Table 1, point 1
- **Reclaimed aggregate**: mineral aggregate derived from waste produced by the construction sector (*recycled aggregate*) or mineral aggregate that results from waste obtained from modifications of the source material (*artificial aggregate*) which has ceased to be such as a result of one or more recovery operations in compliance with the conditions listed in Article 184-ter, quoted in subsection 2.1.1, and the provisions of this Regulation
- **Inert waste**: solid waste resulting from construction and demolition activities and other waste of mineral origin that does not undergo any significant physical, chemical or biological transformation, that doesn't dissolve, burn, it isn't subjected to other physical or chemical reactions, it isn't biodegradable, and, in case of contact with other materials, it doesn't cause harmful effects such as to cause environmental pollution or harm to human health

Table 2.3: Chapter 17 waste class distinguished by their EER Code

EER code	**Waste class**
170101	Cement
170102	Bricks
170103	Tiles and ceramics
170107	Mixtures or slag of cement, bricks, tiles, and ceramics, other than those referred to in heading 170106
170302	Bituminous mixtures other than those falling within heading No 170301
170504	Excavated soil and rocks, other than those falling within heading No 170503 excluding those coming from contaminated sites subject to remediation
170508	Steel for railway ballast, other than that referred to in heading 170507
170904	Mixed waste from construction and demolition activities, other than those falling within headings 170901,170902 and 170903

From this last definition, we can deduce the type of waste resulting from rock cutting, and therefore *Annex 1* lists all waste, also classified with its EER codes, which can be revalued in the form of other materials. Table 2.3 shows inert waste derived from construction activities, which is listed in Chapter 17 of the European

Waste List. Furthermore, table 2.4 shows all other inert wastes that are not listed in Chapter 17 of the European Waste List and therefore are inert materials of mineral origin. [11] *Art. 3* describes, pursuant to articles *184-ter* and *art.1* of the aforementioned decree, that

> "inert waste from construction and demolition activities and other inert waste of mineral origin cease to be classified as waste and are classified as recovered aggregate if the recycled or artificial aggregate from the recovery treatment complies with the criteria set out in Annex 1"[11]

Therefore, "*underground waste*" and waste classified with the EER code 170504 for the production of recovered aggregate are not allowed by this legislation.

Table 2.4: Waste class outside chapter 17 distinguished by their EER code

EER code	**Waste class**
010408	Gravel and crushed stone waste, other than that falling within heading No 010407
010409	Sand and clay waste
010410	Powders and related residues, other than those falling within heading No 010407
010413	Waste produced by cutting and sawing stone, other than that referred to in heading 010407
101201	Preparation mixture residues not subjected to thermal treatment
101206	Waste molds consisting exclusively of sphridia and waste of glazed and fired raw ceramic products oda sphridia of fired brick and expanded clay possibly covered with concentrated raw glaze <10% by weight
101208	Ceramic waste, bricks, tiles and building materials (subject to heat treatment)
101311	Waste from the production of cement-based composite materials, other than those falling within headings 101309 and 101310
120117	Residues of sandblasting material, other than those referred to in 120116 consisting exclusively of waste abrasive sands
191209	Minerals (e.g. sand, rocks, aggregates)
200301	Undifferentiated municipal waste, limited to the inert fraction of abandoned waste from construction and demolition activities.

2.1.5 Italian law: D.P.R. June 13th, 2017, No 120

Italian law provides for various regulations regarding the simplified discipline of excavated soils and rocks management. In particular, *art. 2* of the *Presidential Decree 120/2017* defines **Excavated soils and rocks** (*subpara.1c*):[12]

> "excavated soil deriving from activities aimed at the construction of a work, including: general excavations (earthmoving, foundations, trenches); drilling, boring, piling, consolidation; infrastructural works (tunnels, roads); removal and leveling of earthworks (; sediments deriving from emptying, desilting, and degravelling operations))."

Excavated soils and rocks are defined as **By-products** according to *art. 4* (*subpara.2*): [12]

> "excavated soils and rocks, to be qualified as by-products, must satisfy the following requirements: a) they are generated during the construction of a work, of which they constitute an integral part and whose primary purpose is not the production of such material; b) their use complies with the provisions of the utilization plan *[...]*, c) they are suitable for direct use, i.e., without any further treatment other than normal industrial practice; d) they satisfy the environmental quality requirements *[...]*."

Art. 2 defines **Normal industrial practice** (*subpara.o*): [12]

> "operations, even if not conducted individually, to which excavated soils and rocks may be subjected, aimed at improving their merceological characteristics to make their use more productive and technically effective. Subject to compliance with the requirements provided for by-products and environmental quality requirements, normal industrial practice treatment guarantees the use of excavated earth and rocks in accordance with the technical criteria established by the project."

subpara.c of *art. 2* establishes to which type of operations excavated soils and rocks may be subjected, to acquire the status of by-product and being reused. Finally, **waste** is defined according to *art. 3* (*subpara.2*):[12]

> "Waste deriving directly from the execution of demolition interventions of buildings or other pre-existing structures is excluded from the scope of application of this regulation *[...]*."

2.1.6 Italian law: D. Lgs. June 25th, 2024, No 84

Art. 9 of Legislative Decree 25 June 25 2024 n°84 provides the regulations for the recovery of mineral resources from extractive waste. In particular, it introduces the concept of valorization of historical deposits (*subpara.1*): [13]

> "Given the significant quantity of extraction waste in closed storage facilities and the related potential in terms of critical raw materials *[...]*, for the recovery of mineral resources from closed extraction waste storage facilities, including abandoned ones *[...]*, Royal Decree 29 July 1927 n°1443, shall apply, insofar as compatible."

subpara.1 of *art. 9* promotes the extraction of critical raw materials from pre-existing extraction waste. *Art. 9* also introduces a series of amendments to Legislative Decree 30 May 30 2008 n°117. In particular, *subpara.2.b* includes the following definitions: [13]

- **Historical extraction waste** (*Art. 9, subpara.2.b.1.d-bis*): extraction waste attributable to mining activities closed or abandoned prior to the date of entry into force of this decree
- **Recovered mineral resource** (*Art. 9, subpara.2.b.2.f-bis*): raw materials recovered from an anthropogenic deposit, composed of extraction waste from previous extractive activities.
- **Historical extractive waste deposit** (*Art. 9, subpara.2.b.2.f-ter*): deposit of mineral elements, consisting of extractive waste, which is a potential source of secondary raw materials from the recovery of mine waste and those deriving from processing.

Art. 9 introduces in *subpara.2.c* a further amendment to Legislative Decree 30 May 30 2008 n°117, defining the **Recovery Plan for raw materials from historical extraction waste** (*art. 5-bis*): [13]

> "The extraction of mineral substances in closed or abandoned extractive waste storage facilities, for which the mining license is no longer in force, may only be granted following the development, by the prospective licensee, of a specific 'Recovery Plan for raw materials from historical extraction waste'. The Recovery Plan must demonstrate the economic and environmental sustainability of the entire life cycle of the operations, including the management of processing tailings."

Art. 5-bis defines the Recovery Plan to which the extractive activity from extractive waste deposits must be subjected. It introduces the concept of economic and environmental sustainability to be guaranteed throughout the entire extractive process, also considering the management of tailings. [13]

2.2 Piedmont Framework

2.2.1 Regional framework November 17th 2016, No 23

The subject matter to which the decree refers is: "activities involving changes in the physical state of the soil and subsoil, directed towards the extraction, for processing, selection or otherwise use and marketing purposes, of materials belonging to the second category of Article 2 of Royal Decree No 1443 of July 29th, 1927" [14]. As it focuses primarily on rock processing, the framework quotes the desire to pursue "environmental and landscape protection"[14] as the goal of soil processing and transformation. *Art. 2, subpara.2* defines the purposes of the decree, but above all, as it states, the Region also pursues the following purposes:

- addressing mining activities towards a better balance in industrial production and the optimization of interventions for the purpose of environmental recovery, redevelopment, and valorization of abandoned sites [14]

- reducing soil damage, to limit its consumption, through recycling compatible waste and residues from quarry, using recovered inert aggregates from construction and demolition activities, and alternative materials to quarry products [14]

- encouraging the protection and valorization of solid quarry minerals and related activities, with a view to enhancing the disused mining heritage [14]

- improving safety to professionals during the activity, promoting effective prevention actions [14]

Art. 3 introduces the benchmark on the subject of the entire rock supply chain, from the extraction site to its processing destination, defining it as “the unitary reference framework of activities” [14], the **PRAE**. It provides some information regarding: basin planning and Optimal Territorial Areas (ATOs). The same article establishes that the borders are delimited within the Metropolitan City of Turin and the relevant provinces. The plan is made up of several tabs, distinguishing them between: basin cards, pole sheet, and subsequently all the cartographic tables that most accurately describe the site. *Art. 4, subpara.2*, points out the objectives that the PRAE pursues to carry out sustainable extraction that does not degrade the natural environment and respects it ecologically. Below, the letters are mentioned: [14]

- defining the lines for a correct balance between territorial values, such as land, environment, and landscape, mining activity, and the reference market

- protecting and safeguarding deposits under cultivation, recognized ones, and related resources, considering mineral deposits and mining activity as primary sources for the socio-economic development of the territory
- enhancing the value of the cultivated materials through their integral use and specific characteristics
- standardizing the exercise of mining activity throughout the region
- addressing mining activities towards a better balance in industrial production and the optimization of interventions for the purpose of environmental recovery, redevelopment, and valorization of abandoned sites
- promoting, protecting, and qualifying work and businesses
- fostering the recovery of inert aggregates from construction and demolition activities, as well as the use of inert materials for recycling
- ensuring monitoring of mining activities
- addressing environmental and economic synergies resulting from the arrangement and maintenance of riverbanks and hydroelectric basins;
- providing guidance for the procurement of materials necessary for the construction of public works

Letter g, which is linked to *Art. 2, subpara.2, letter b*, is of great importance since it states the commitment by mining companies to maximize the revaluation of waste, debris, and inert materials produced during processing, and which often still contain material as valuable as virgin rock. [14] In Piedmont, there are 3 main extraction sectors (defined by *Art. 4, subpara.4*): aggregates for construction and facility, ornamental stones, and industrial materials. [14] Another important article is *Art. 13* entitled "*Plan for the recovery and management of lithoid materials required for the construction of public works*" [14], which mentions further planning for the recovery of waste in the extractive activities chain. That is, in *subpara.1* "the actuator of the public work" [14] is mentioned, who develops "*a plan of sourcing and management of materials*" [14] which also takes into account "*the use of scrap resulting from mining activity*". [14] In *Art. 13, subpara.2-bis*, a very important concept is introduced regarding sustainability, that is, as it quotes:

> "*The plan referred to in subpara.2 guarantees at least 50 percent of the required needs with the following materials*" [14]

namely, a process must be planned that guarantees a flow of extracted material. It must be exported to companies, with at least a fraction, equal to half of the

requirements, made up of recycled material. Therefore, it lists some of the materials that may fall within this fraction:

- slurries from mining activities
- material available from inert quarries
- materials from recovery activities that include recycling and preparation for reuse

2.2.2 Regional guideline March 25th 2022, No 3

In response to *Regional Law 23/2016*, regarding rock processing, *Regional Regulation March 25th 2022 No 3* explores some environmental issues, listed in *Art. 1, subpara.1*: [15]

- protection of groundwater quality
- precaution and correction at the source of damage to the environment
- quality of the environment
- protection of human health
- traceability of the materials supplied and their location

The key points are always sustainability and prevention aspects, fundamental aspects regarding waste, which are also applied in quarries, rock extraction, and the processing of the extracted material. The framework was defined following the provisions of *Environmental Code* and *Art. 3 of Legislative Decree 117/2008*, although the latter could be repealed. In *letter a of Art. 3 subpara.1* is introduced the expression "*mining treatment*" [15] which is

> "*the process or combination of mechanical, physical, biological, thermal, or chemical processes carried out on mineral resources, including the exploitation of quarries, to extract ores, including size modification, classification, separation and leaching, and reprocessing of previously discarded mining waste; melting and thermal processes are excluded*" [15]

The quote includes processes other than extraction alone, thus it generalizes the concept to mechanical, physical treatments, etc, considering activities downstream the quarry too. The wide use of waste in quarries is to fill voids. It is a key application since it allows reuse in dams, for building routes in quarries to reach the extraction site, or for creating/filling unused cultivation areas. *Letter f of Art. 3, subpara.1*, defines "*void filling*" [15] as:

> "*the set of operations and processes aimed at filling voids, for their use or for the restoration of areas. [...] Waste used for filling must replace materials that are not waste, be suitable for the aforementioned purposes, and be limited to the quantity strictly necessary to pursue those purposes. Recovery with waste is possible in compliance with the conditions laid down in Article 5 "Environmental recovery" of the Ministerial Decree of 5 February 1998*" [15]

The article states the procedures for using waste for filling. Therefore, the category of waste, mentioned in the decree, is non-hazardous or a different material than quarry waste. "*Environmental recovery*", defined in *Art. 5 of the Ministerial Decree February 5th 1998*, is described as

> "*restitution of degraded areas to productive or social uses through morphological remodeling*" [15]

The same article establishes, in *subpara.2*, the characteristics whose waste must own: [15]

- it must not be hazardous
- it is foreseen and ruled by a specific project approved by the competent authority
- it is carried out in compliance with the technical standards and specific conditions set out by the decree for each class of waste used; furthermore, the compliance is referred to the project too
- it is compatible with the chemical-physical, hydrogeological, and geomorphological characteristics of the area to be recovered

It is particularly important that the classification and parameters, whose residue must be owned, fall within the legal threshold, and such waste must be approved in the draft by competent authorities. At the time of filling, *Art. 4, subpara.3 of Regional Regulation No 3, March 22nd 2022*, sets the conditions under which it is allowed to do: [15]

- environmental and health protection is guaranteed at all stages of filling
- the stability of the materials used is provided
- no harm or worsening of soil and water conditions is caused
- the traceability and monitoring of either materials or waste used is ensured.

At the *Art 5, subpara.1*, a list of some materials that can be recovered and that can be used as void-filling materials is introduced. It specifies that the material quoted must be aimed only at improving the morphological conditions of the site. The checklist is structured to establish a hierarchy of priorities among the materials; sure enough, at *Let.a* it is stated: *"the extraction waste, produced by the same or another extraction site"* [15]. *Let. d* is another important statement because, here, the regional regulation quotes "*by-products*" (also cited and defined at 2.1.1) for which it is crucial to introduce the material directly without any further transformation.

Chapter 3

State of art about Ornamental Stone Recovery

After the legislative framework, described in the previous Chapter 2, it is important to understand the *State of Art* about Ornamental Stone Recovery. In the next sections different application areas are discovered and they will be analyzed based on the scientific literature.

3.1 Geopolymerization

Historically, geopolymers are not a new technology. As [16] describes, in early 70's many research about non-flammable plastics were performed. Thus, ceramic-base plastics started to be studied and manufactured. Originally, the main reaction to produce geopolymers, at the beginning, was by using alumino-silicates, like kaolinite, and NaOH at about 100°C. Though an intermediary compound (hydrosolidate) is produced [16]. However, calcined kaolin was preferred to kaolinite because thei reaction avoids the hydrosolidate precipitate formation. The first applications were in 1973-1976 on producing fire-resistant chip-board panels that are 3-layer objects made of a wooden core and two layers of geopolymer coatings [16]. An interesting insight of that production was the one-step process to produce the panels [16]. The definition of *geopolymers* is:

"*mineral polymers resulting from geochemistry or geosynthesis*" [16]

The clear characteristic that arises from the quote is it needs **Chemistry** to produce geopolymers. On contrary to the usual definition of polymers, in this case no organic material is used, instead, inorganic compounds such as minerals and sometimes chemicals. Currently, the main sources are metakaolin, which provides aluminum and silicon, and sodium hydroxide [17]. However, the sodium hydroxide is a powerful chemicals, which is dangerous to human, so new sources are needed to produce less hazardous product. Recent studies are demonstrated the use of calcium-rich materials in geopolymerization [17]. Palmero et al.[18] tried to use mineral sludge containing some interesting compounds: quartz, feldspar, biotite

and dolomite cured at 80°C for 48 h [18]. Sodium hydroxide, sodium silicate, and silica were used as alkali-activated substances [18]. Geopolymerization is defined as:

> "*a chemical reaction between aluminosilicate materials and alkali metal silicates, under strong alkaline conditions*" [18]

and the traditional procedure is the following: [18]

1. Dissolution of aluminum and silica in the alkali solution
2. Transportation of dissolved species
3. Polycondensation of alumina and silica where they are distributing themselves in a tetrahedron structure

The study of Palmero used a alkali-activated samples derived by granite rocks processed by diamond cutting disks. The study has carried out a comparison among a mixture aged for one month and another aged for one year. After a drying process the compound was milled to reach 125 mesh [18] (the length of a sieve's hole is about 0.125 mm). The next step was a qualitative analysis of the composition, through a X-ray fluorescence. However, the results obtained by Palmero et al[18] are different from what will be described in Subsection 5.2. Although the XRF analysis can't be compared, the X-ray diffraction is feasible and comparable to what will be introduced in Section 5.2. Figure 3.1 and Table 3.1 denote the results of the XRD analysis. Palmero's study went beyond and it performed the mineralogical

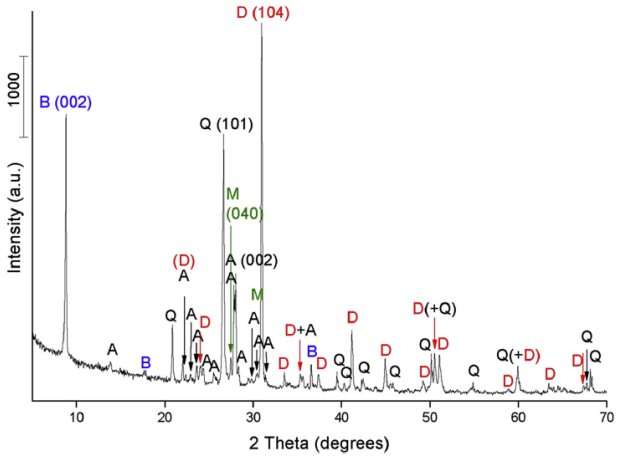

Figure 3.1: XRD pattern obtained by Palmero et al [18]: Q=quartz, D=dolomite, A=albite, B=biotite, M=microcline

Table 3.1: XRD phase quantification by Palmero et al [18]

Phase	Concentration (Wt%)
Dolomite	31.72 ±0.23
Microcline	25.67 ±0.23
Albite	20.51 ±0.15
Quartz	14.30 ±0.10
Biotite	5.42 ±0.18
Amorphous	2.39 ±0.017

characterization of the sample at the SEM. As it will be stated in Section 4.2 the SEM results isn't necessary to be discussed. In the analyzed material, the prevalent minerals are alkaline feldspars, while the minor contribution by the biotite phase is only 5.4%. The contribution of dolomite and quartz is relevant, with respectively 32% and 14% on weight. Despite the alumino-silicate is one of the most abundant fraction in the material, there is no denying that beside it some other mineral are present. In fact, dolomite is quite strange to find together [18], nevertheless, in the same plant other stones can be cut. Amorphous material is in the mixture too, for the same reason. Palmero's [18] study revealed that alumino-silicate waste can produce a novel alkali-activated material. Despite geopolymers are traditionally produced by amorphous or semi-crystalline raw materials, the study analyzed how the process can be enhanced, from a sustainability point of view, by waste powder from ornamental stone cuttings. The waste is surprisingly active as the amorphous one, and it hypothesized that by dissolving some alumino-silicate particles, coming from the waste, inside a strong alkaline condition, the polymerization can occur [18]. The experiment performed by Palmero's study suggested the following phenomenon:

> "*the particles generates an alumino-silicate gel that spreads in larger interstitial spaces among particles and when the gel hardens the alumino-silicate is bound, whereas the solid fraction behaves like a matrix reinforcement*" [18]

The final compound, stated, is a *dense geopolymer* [18] using granite mud, contaminated by dolomite, which owns great mechanical properties that suggest the key potential of alumino-silicate to strength the product. The final conclusion is a possibility of exploiting the byproduct to manifacture dense and lightened products and providing a different destination instead of stone mud landfilling.

3.2 Tunnel spoil, with lime addition, for muck reuse

The material which the thesis refers could have similarities to the following study. Oggeri & Fenoglio [19] assessed possible muck recovery alternatives for spoil management. Following the In-Country legal framework, the study focused to provide guidance and different scenarios, with real case study, to better manage the stone residues that are produced during the entire excavation site. During the methodology part, the study [19] states the importance to classify the material, since the excavation is different among the machines employed. Moreover, given the wide variety of residues produced, each of them has different geotechnical and physical properties. Oggeri & Fenoglio mentioned the following excavation methods applied that produce spoil residues:[19]

- Drill and blast
- Mechanised excavation partialised
- Mechanised excavation
- Mechanised excavation and soil conditioning
- Grouting or reinforcing of the ground in the above listed methods

The samples, withdrawn from the previous excavation methods, are distinguished by the next ground type:

- Rock
- Soft rock or hard soil
- Soil, which is classified as:
  - Coarse
  - Fine

Collecting the samples, Oggeri & Fenoglio did a step up and they found different destination for each material. Below, the destinations chose are listed:

- Aggregates for constructions
- Road works/embankments
- Raw material for industry
- Environmental or land reclamation

- Backfilling

In the paper [19], the technical issue is the aspect discussed regarding the muck management. Therefore, the aspect analyzed during the Sections 4.1 and 5.1 in the current thesis. To study, thoroughly, the sample's performances the following approaches are adopted to understand possible **High Value Reuse Strategy**: [19]

- Muck moisture content and consistency (according to in-situ influences)
- Material compaction process of both excavated and lime addiction (0.5-1%)
- Strain and compressive strength to evaluate mechanical behaviour

With the aforementioned approaches, Oggeri & Fenoglio's paper had two objectives:

1. Mechanical parameter assessment of possible good quality muck
2. Lime stabilization for poor quality muck

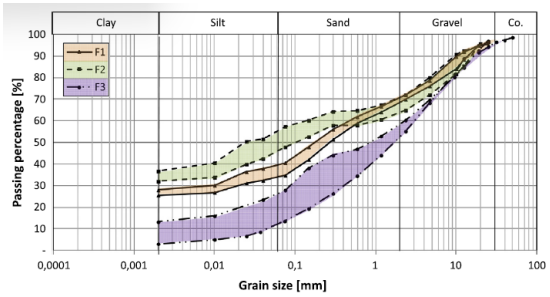

Figure 3.2: Grain distribution of F1, F2, F3 samples obtained by Oggeri & Fenoglio's paper [19]

Afterwards, the paper explains the *Materials and methods* in which it was decided to add limestone at different percentages, in each class of samples (just for handling improvement purposes) [19].

On the *Physical parameters* analysis: grain size distribution (shown in Figure 3.2, consistency indeces, slump test (AASHTO reference), deformation and compressive strength for all trials. The last analysis of mechanical strength was divided into two tests:

- Confined Compression Test: behaviour investigations when the material is subjected to loads in confined conditions
- Unconfined Compression Test: samples are compacted and then extruded at different curing times

Table 3.2: Results of the Consistency Indeces by Oggeri & Fenoglio's paper [19]

Physical parameters	**F1**	**F2**	**F3**
Moisture content w [%]	20.88	20.45	14.5
Passing at 2 mm sieve [%]	70	68	58
Passing at 75 lm sieve [%]	41	53	28
Passing at 2 lm sieve [%]	29	34	13
Liquid limit LL [%]	35.4	34.8	26.9
Plastic limit PL [%]	20.7	22	16.1
Plasticity index PI [%]	14.7	12.8	10.8
Consistency index CI [%]	0.99	1.12	1.15
Liquid limit LL % (lime 2%)	49.0 (↑38%)	62.0 (↑78%)	–
Plastic limit PL % (lime 2%)	24.8 (↑20%)	31.9 (↑45%)	–
Plasticity index PI % (lime 2%)	24.2 (↑65%)	30.1 (↑135%)	–
Consistency index CI % (lime 2%)	1.16 (↑17%)	1.38 (↑23%)	–
Liquid limit LL % (lime 4%)	47.7 (↑35%)	67.5 (↑94%)	–
Plastic limit LP% (lime 4%)	31.3 (↑51%)	39.4 (↑79%)	–
Plasticity index IP % (lime 4%)	16.4 (↑12%)	28.1 (↑120%)	
Consistency index CI % (lime 4%)	1.64 (↑66%)	1.67 (↑49%)	–

Table 3.2 denotes all the results about Consistency Index evaluation obtained by Oggeri & Fenoglio's paper. It is noticeable that additivating a fraction of limestone the indexes change significantly compared with the original material. In all indices, the values increase; however, the *Plasticity Index* doesn't increase with the addition of more limestone. F1 and F2 weren't suitable for mechanical characterization since they were weaker than F3 sample. Furthermore, F3 seemed to comply AASHTO geotechnical classification reference. The paper conclusions denoted the influence of the excavation technical choice employed. Chemical assessments must be performed to determine whether the material is contaminated by pollutants or not (it has underlined the compliance of national legal limits). [19] Oggeri & Fenoglio's study demonstrated that F1 and F2 samples (respectively argillites and highly clayish sandstones) were stabilized by limestone addition. Therefore F3 could be applied in embankment construction, since the mechanical evaluation provides promising results.

Table 3.3: Mechanical characterization of the F3 sample

Label	ρ_d	**Confined modulus**		**Unconfined modulus**		**UCS**
sample	(g/cm3)	tan (MPa)	sec (MPa)	tan (MPa)	sec (MPa)	(kPa)
C1	1.94	45.95	23.222	22.379	16.085	677
C2	1.98	44.147	30.739	14.044	11.619	486
C3	2.19	61.962	44.804	15.335	10.355	285
C4	2.13	57.926	40.953	21.52	12.482	326
C5	1.96	–	–	0.868	0.649	53
C6	1.96	–	–	1.543	1.048	66
C7	2.2	–	–	24.467	14.439	384
C8	2.19	–	–	21.495	11.77	371
C9	2.2	–	–	42.889	16.981	533
C10	2.02	–	–	45.465	12.56	289
C11	2.02	–	–	75.312	24.914	515
C12	1.98	–	–	20.861	10.377	269
C13	2.01	–	–	36.742	26.215	536
NC1	2.08	42.099	25.649	42.232	22.208	809
NC2	2.04	37.206	19.612	59.105	21.899	443
NC3	2.13	49.461	33.311	57.001	20.548	737
NC4	2.14	44.209	28.513	41.968	24.181	929
NC5	2.14	61.833	36.049	16.819	9.921	442
NC6	2.13	66.269	38.443	26.711	12.22	429

3.3 Fine Waste and Slurry Replacement in Concrete and Civil Production

Since the cutting sludge is a fine material, many applications in the concrete production can be applied. Researches suggest many opportunities for replacing the expensive and highly pollutant concrete with the slurry. Al-Zboon and Al-Zou'by [20] proposed to replace the potable mixing water with a cutting slurry from a stone processing in a concrete and mortar production plant. The sludge was characterized and the results are the following:

- The composition is 95.2% of water and 4.8% of solid particles [20]
- The mineralogy is mostly limestone with a composition of 97% of total solids; the lime content is 54%

- The value of D_{50} is 10 μm

The mix design is made by: Portland cement, aggregates, slurry, silica sand and tap water. Al-Zboon and Al-Zou'by [20] analyzed with different mix among them and Table 3.4 displays the composition. The slurry-water ratios span from 0 to

Table 3.4: Mix design proportions in Al-Zboon and Al-Zou'by [20] study varying slurry-to-water ratios

Sample	Aggreg. & Silica (kg)	Slurry (kg)	Tap water (kg)	Cement (kg)	Slurry/Water (wt%)	Total weight (kg)
C_1^a	57.15	0.000	7.250	9	0	73.4
C_2	57.15	1.812	5.437	9	25	73.4
C_3	57.15	3.625	3.625	9	50	73.4
C_4	57.15	5.437	1.812	9	75	73.4
C_5	57.15	7.250	0.000	9	100	73.4

100%, therefore a complete sensitivity analysis is performed to see which is the best composition for the company target. Al-Zboon and Al-Zou'by [20] stated that the compressive strength required by the plant is 20.5 MPa and the slump must be 200 mm. Table **??** displays the methods for preparing and later testing each sample used by Al-Zboon and Al-Zou'by [20]. In the end, the research has

Table 3.5: Experimental program by Al-Zboon and Al-Zou'by [20]

Parameter	Preparing method	Number of samples	Curing period	Testing method
Slump	ASTM C 192-07	15	Fresh	ASTM C 143-10
Compressive strength	ASTM C 192-07	24	7, 28 days	BS 1881: part 111
Tensile strength	ASTM C 192-07	24	7, 28 days	ASTM C 496-10
Flexure strength	ASTM C 192-07	24	7, 28 days	ASTM C 293-10

demonstrated that the replacement of slurry can enhance the mechanical properties of the concrete. However, the best mix was discovered at 50% of Slurry-Water ratio because slump dropped consistently when slurry content increased [20]. That sample achieved a compressive strength of 26.7 Mpa and a slump of 115 mm (the

workability is a little bit far from the target). Al-Zboon and Al-Zou'by [20] said that cutting sludge acts as **micro-filler** filling voids between aggregates. Almeida [21] tested the performance of concrete by analyzing the mechanical parameters in different sand content samples. Here, the fine aggregate is replaced by dry stone cutting waste. The material comes from stone slurry which was dried to gain dry stone dust and it had the following characteristics:

- The lime content is about 54% confirming it is a limestone type waste
- D_{50} is 5 μm which was smaller than Portland Cement particle size
- No pozzolanic and hydraulic activity was not found

The samples were divided according to the Slurry-San ratio, starting from 0% slurry content (that was the reference point) to 100%. Almeida [21] observed some interesting features ranging the sand substitution percentage:

- Between 0-10% better workability and packing with an aesthetic improvement
- Between 10-20% the higher specific surface area (since the dust is smaller than sand) requires higher water demand
- Between 20-100% the water demand is needed because the workability is compromised

For Almeida study the mix design which led to the best performance in terms of mechanical strength, workability and physical properties is the the sample with 5% dust substitution. The main insights are: [21]

- Compressive strength at age 7 days 66.5 MPa, at 28 days 91.1 MPa
- Modulus of elasticity 43 GPa
- The Water-Cement ratio decreased by 16% after 28 days that is interesting since it means that the hydration reaction is enhanced

Almeida [21] stated that using marble and limestone in white concrete can improve cohesion and it can reduce the bleeding effect on slab surfaces. From the aesthetic point of view the the dust acts as colour tone regulator which changes the chromatic scale and it can be associated as a natural pigment in white concrete. A 5% dry dust can be suitable for substituting sand in high-performance white architectural concrete.

Chapter 4

Method and analysis of data

4.1 Geotechnical characterization

4.1.1 Sampling

The sampling was performed on January 12, and the points at which the material was taken were:

- directly from the bottom of the silos in which the sludge is stored (from now to the end, the sample will be called SS, that stays for "Sludge Silos")
- beside the production plant where usually the sludge in excess is discharged to enlarge the storage area of the products inside the plant area (as SS, this sample will be called SE, that stays for "Sludge External")

The samples were stored inside sealed plastic bags (as Figure 4.1 shows) to maintain the water content and to not make contact with other compounds or atmospheric conditions. Here, the aim is to maintain the samples, as much as possible, in an unaltered state because, for future analysis, it is crucial to work with undisturbed material.

4.1.2 Preliminary analysis

The first step was evaluating the water content in the material, which was enough to perform the other analyses, described in the following subsection. The material has been put inside a baking pan and spread uniformly. Afterwards, the material has been weighed and dried for 24 hours in the oven at 110°C. With all the material dried, a weighing was performed to determine how much water was lost. The results are reported in Table 5.1. The moisture content was computed with the expression in Equation 4.1

$$WT = \frac{W_{pre-drying} - W_{post-drying}}{W_{post-drying}} \cdot 100 \tag{4.1}$$

Figure 4.1: Plastic bags of the SS

4.1.3 Grain size analysis

The first insight studied is the grain size distribution. To evaluate the specific grain class featured in the material, sieves are needed to classify the particles by grain size. According to the study carried out by Zichella [22], the mesh size of each sieve must be: 0.212 mm, 0.106 mm, 0.063 mm, and 0.038 mm. A *Sample reduction by fractional shovelling* was performed, following the British Standard EN 932-1 1996 [23], to sample the material and perform a wet sieving. Although the material could be sieved in a dry state, the quoted study suggested performing a wet procedure, as the material is very fine and vibration would have spread dust, consequently losing some material. In the laboratory, a sieve test was done according to the pass towards descendant size, as shown in the Figure 4.2.

The bottom portion was gathered inside a tank and filtered with paper filters to determine the amount of particles below 0.038 mm. The filters used are of the type Prat Dumas ® with reference 110. That means the particle retention is about 6-10 μm. In Figure 5.1, which reports the resulting grain size distribution of SS and SE, the point at the smallest retention value is 0.008 mm, and the resulting value of cumulative passing.

Figure 4.4 displays the system used for filtering the bottom part of the sieving process. It is configured to use water flow to make the vacuum inside the beuta. On top, a bottleneck guests the paper filter, and after placing it, the vacuum is set. In appendix A, a summary of measurements related to the distribution evaluation is shown in table A.1.

4.1.4 Consistency Indices

To characterize the behavior of the material from a geotechnical point of view, the study focused as a first approach to the *Atterberg limits*. A thorough procedure

Figure 4.2: Sieve system to analyse the grain size

Figure 4.3: Baking sheets in which the sieved material was collected

is indicated in the ASTM standard D4318-10 [24], in which it specifies how to perform the *Liquid Limit Test* and the *Plastic Limit Test* (also all calculations needed to reach the full characterization).

Liquid Limit

For both samples, an evaluation with Casagrande's Spoon was performed to analyze the *Liquid Limit Test*. As Figure 4.5 shows on the right side, there is the actual equipment to evaluate the liquidity of the material. The procedure is simple:

1. Put the sample on a wooden table
2. Mix the material for a while to enhance the loss of water
3. Harvest a portion and put it on the spoon

Figure 4.4: Vacuum Filtration System used for evaluating < 0.038 mm grain size

Figure 4.5: Setup of Casagrande's Spoon

4. Spread on the spoon in such a way that the material is on the same level as the edge
5. Dig a groove in the sample in the middle of the spoon
6. Rotate the crank and count the number of hits until the sample covers the groove with a distance of about 2 cm
7. Repeat the procedure until the number of hits reaches a satisfactory value for the scope of the experiment

Collecting all the trials, in Table 5.2, a summary of the number of blows and the relative evaluation for the moisture contents is sketched out. Afterwards, a linear fit analysis of the points is performed comparing the Moisture content and the Blows done. The resulting linear fit is distributed as Equation 4.2 suggests:

$$y = Slope \cdot log_{10}(x) + Intercept \tag{4.2}$$

The *Slope* and *Intercept* values were found using the *Polyfit* algorithm through a Python code (which is given in the Appendix A Listing A.2). The output provided is always a list of values, in which at each different position, there are the polynomial

coefficients resulting from the fitting. Since it is a linear fitting, the polynomial fitting is evaluated at order 1. However, the presence of the log_{10} in the fitting equation is explained by the type of distribution of the scatter plot, which is a semilogarithmic graph on the x-axis.

Plastic Limit and Plasticity Index

On the other hand, the *Plastic Limit Test* is the evaluation of one single value of moisture content (as reported by Table 5.3), which corresponds to the *Plastic Limit*. The aforementioned parameter was computed by an average of the resulting Moisture Content of each trial. The procedure to get knowledge about *Plastic limit* is:

1. Take a fraction of the sample and let it sit on a surface that doesn't absorb water
2. Mix the sample with a touch of water to homogenize the moisture
3. Withdraw a small part of the sample and dry it out on an absorbing surface by making a tiny cylinder-like shape
4. Continue to dry it until it immediately starts to break
5. Weigh all the material dried and put it in the oven at 110°C for a couple of hours

The drying is crucial to estimate the Moisture Content of each sample, which is equivalent to the *Plastic Limit* Then, it is important to compute the *Plasticity Index (PI)* which is defined by the Equation 4.3

$$PI = W_L - W_P \tag{4.3}$$

Afterwards, knowing the *Liquid Limit* and the *Plasticity Index*, in Figure 4.6 (produced with the code at Appendix A Listing **??**) is introduced the Casagrande's Plasticity chart that classifies the behaviour of a soil distinguishing it by: its plasticity (type of soil) and its compressibility. As well as the reference [25] introduces the "*A Line*" divides the clay and the silt class, namely: under the line the soil is a silt, on the other hand, it is a clay. The actual meaning of each region is the following

1. Inorganic silt of low compressibility
2. Inorganic silt of medium compressibility and organic silt
3. Inorganic silt of high compressibility and organic clay

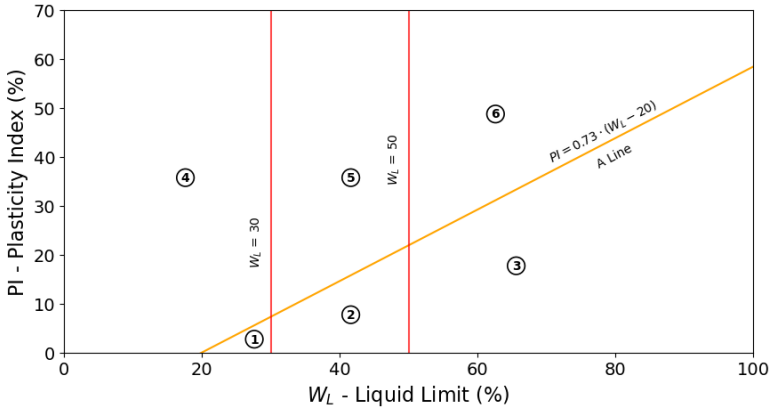

Figure 4.6: Casagrande's Plasticity chart [25]

4. Inorganic clay of low compressibility
5. Inorganic clay of medium compressibility
6. Inorganic clay of high compressibility

4.1.5 Laterally Confined In-Grip Load Test

The next step of the experimental experience is the Latrally Confined In-Grip Load test, in which two different evaluations were performed:

- Unaltered state test
- Unaltered state sample plus 10% of Portland cement as additive
- Dried Overnight for only the Sludge Silos sample
- Constipation and stabilization for 1 hour for samples Sludge Silos with and without additive

The analysis focused on studying the behaviour of the material when it is left to stabilize itself for one hour. The purpose is to make the material stronger because the compound isn't very stable at the beginning. These trials are important to understand whether the compound is able to stabilize its structure (with and without the additivation of cement) or not. The actual specification of Portland cement is **Portland 42,5 R II A** which means and, for the purpose of the study, the additivation of a certain amount of cement is able to enhance the performance of the material. The next step was the preparation of the sample inside the cell, as Figure 4.7 introduces. The procedure to set the sample is the following:

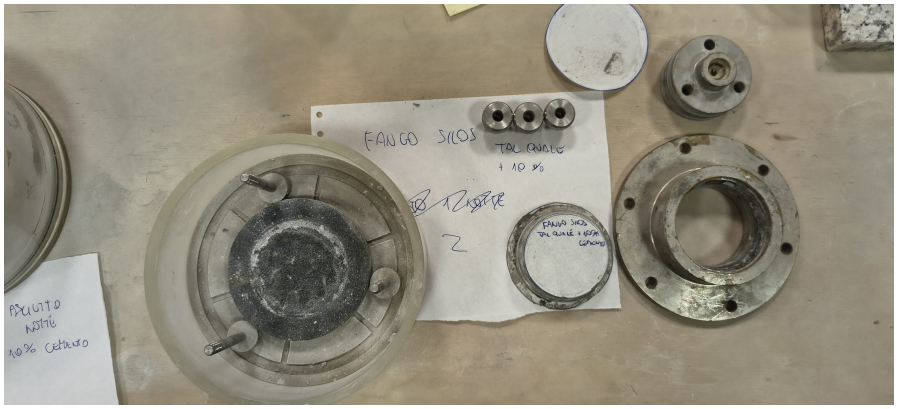

Figure 4.7: Setup of the edometric cell to prepare the sample

- Cut a paper filter to place it, afterwards, on the porous medium and over the ring.
- Fill the ring with the wanted sample
- Put the filled ring on the paper filter located above the porous stone
- Close the system with the piston and the three bolts
- Insert the cell in the edometer (sketched in Figure 4.8)

The paper filter above the porous medium is useful to prevent clogging phenomena in the hole; indeed, the issue will hinder the water from flowing away from the trial. Same situation in the piston, since the contact surface with the sample is made by a porous medium too. Settle the equipment, the sample is ready to evaluate the load test by taking notes of: load placed (by using a calibrated disk), settlement, and time of taking the earlier parameters. Since some trials were conducted for more than one day, a time set in hours was preferred to appreciate the distribution in each test. The settlement was measured by using a datalogger, as shown in Figure 4.9 connected by two different sensors which give the value of the lengthening and shortening of their branch. The total offset of each sensor is 20 mm, and as Figure 4.9 exhibits, it reaches the sensitivity of $\pm$ 0.01 mm.
The test was performed in two phases:

- In the first part, there is the *Loading phase* in which the load is set by increasing twice the previous weight each time.
- After reaching a satisfying value of load, the second phase is the *Unloading phase* where each plate is removed, and by letting the first plate, a checking of the settlement difference is verified and noted

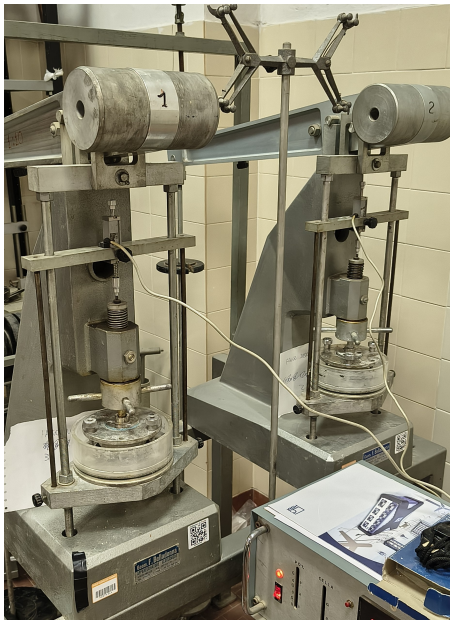

Figure 4.8: Edometer used for the laterally confined in-grip load test

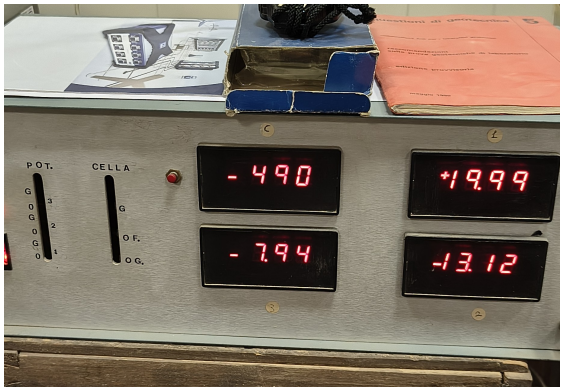

Figure 4.9: Datalogger to check the settlement

In Table A.2 there is a summary of the values registered for the first three types of trials, while in Table A.3 there are the samples let stabilizing for one hour. Given all the parameters reported in Tables A.2 and A.3, the evaluation continues by crafting different graphs for different purposes. Before plotting, different calculations were performed to use the most proper parameters.

$$\sigma_z = \frac{load \cdot g \cdot 10 \cdot 1000}{A} \tag{4.4}$$

The most important value is *Stress* and in Equation 4.4 is described how it was

computed. To obtain a measure on the order of kPa , Equation 4.4 has different insights:

- The loading area A, which is the cross-sectional area of the edometric ring, has a diameter equal to 50 mm; so the value will be in mm^2
- The coefficient *10* is a scale related to a momentum force, to multiply at the load, between the weighted plate and the point at which the force is actually applied
- Since a force defined in N (newton) unit divided by an area defined in mm^2 represents a stress with MPa unit, it is suitable to multiply by 1000 to obtain kPa unit of measurements

Another important parameter to study during the geotechnical characterization is the *Settlement* ϵ_z defined in Equation 4.5

$$\epsilon_z = \frac{H - H_0}{H_0} = \frac{\Delta H}{H_0} \tag{4.5}$$

To be comfortable during the calculation, the difference ΔH was preferred since the sensor can't register the height of the ring every time but only the lengthening or shortening of the sensor's branch. The types of charts evaluated in Section 5.1 are three:

- Analysis of the *Settlement* compared to the Time and Log(Time)
- Evolution of the Settlement compared to the vertical stress (σ_z) during the Loading phase and the Unloading phase
- Variation of the Edometric module compared to the stress applied

The code with which the charts in Section 5.1 were performed is located in Appendix A Listing A.3

4.1.6 Workability of the sludge

To better know the best application of a residue, the *Workability Test* is the analysis to study how the material can be applied according to its resistance to compression. The test starts from the preparation of different dies, knowing the diameter of the cross-section, and then the height of the trial. Figure 4.10 shows the die used, which not only contains the material to constipate, but also is covered on the top by wipes, to stabilize the moisture in the sample. The procedure is the following:

Figure 4.10: Image of the die containing the material to be constipated

1. Seal the die with some tape
2. Apply a small amount of soapy detergent to improve the extraction after finishing the constipation
3. Charge the die with the specific material until reaching a height equal to two times the die's diameter length
4. Cover the top of the material with wipes
5. Wait the time decided for constipation (by 48, 72, 120, 168 hours)
6. Extract the material from the die
7. Put the trial into the press (showed in Figure 4.11)
8. Perform the analysis by taking note of:
   - Settlement
   - Load charged onto the sample
9. Load the weight until the material starts to fail, and it doesn't resist the load applied (as Figure 4.12)
10. Don't stop burdening the sample and take note of the post-peak.

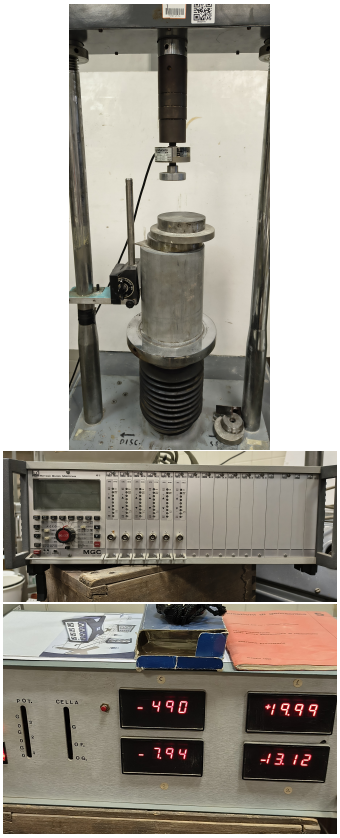

Figure 4.11: Setup for workability test

Figure 4.12: Trial rupture on the press

To know the exact moment the trial must be defined as fractured, some thorough pictures are given in the British Standard *EN BS 13286-41* of 2021 [26] where,

for different situations, the test can be stated as completed. It is also appropriate to calculate the **Volumetric Mass**, which is an important indication of the applicability of that material. The procedure to evaluate it is defined with Equation 4.6:

$$\rho_V = \frac{W_0}{V_0}[\frac{kg}{m^3}] \tag{4.6}$$

At the numerator, there is the mass before compression, and the other value is the initial volume measured with its H_0 and area A. Since the die has a cylindrical shape, the cross-section is measured in Equation 4.7:

$$A = \pi \cdot \frac{D_0^2}{4}[mm^2] \tag{4.7}$$

Consequently, the volume is:

$$V_0 = H_0 \cdot A \cdot 10^{-9}[m^3] \tag{4.8}$$

In Subsection 5.1.5, there are the charts related to the *Workability Analysis* in which the evolution of the *Vertical Stress* σ_z according to the *Vertical Deformation* is studied.

4.2 Mineralogical Characterization

To better understand what is contained in the structure of any compound, *Mineralogical characterization* is the next step to evaluate the concentration of any element inside the samples. The evaluation in the study is divided into two analyses:

- XRF: X-Ray Fluorescence
- XRD: X-Ray Diffraction

In the next Subsections 4.2.1 and 4.2.2 descriptions and explanations will be given of each analysis.

4.2.1 XRF Analysis

As well as the book "*Nanomaterial and Polymer Membranes*" quotes "X-ray fluorescence (XRF) is the emission of characteristic, secondary or fluorescent X-rays from a sample excited by bombarding it with high-energy X-rays or gamma rays. X-rays are expressed in terms of their energy (keV) or wavelength in nanometers (nm). XRF is a consequence of changes in the electrons in the shells of the atom's orbital when a high-energy incident, called a primary X-ray, collides with an atom

and disturbs its stability. [...] As a result, an electron from a higher energy level falls to fill this space. The difference in energy produced when the electron moves between these levels is considered secondary X-rays, which are characteristic of the atom." [27] By calculating the different energies required to excite and bring back the electron in its shell, it characterizes each atom. This evaluation allows to distinguish elements by composition and concentration. To perform the analysis **SciAps® X555** [28], the product has been used to evaluate the elemental analysis of samples SS and SE. The procedure to prepare the material and perform the analysis is the following:

1. Starting from the dried material (prepared as described in Subsection 4.1.2), by using a spatula, put it in a small soil cup
2. Fill until the bottom is covered and a layer of about 5 mm is made.
3. Cover the trough with plastic film
4. Insert one ring to keep the plastic film together twith the soil cup
5. Fix a second ring on the top to stretch the film uniformly to avoid the formation of rough surfaces

Figure 4.13: Setup of the samples ready to evaluate XRF

It is important to get a smooth surface on the top because, since the fluorescence is an emission of rays and waves, the film might reflect them in a certain direction. The consequence could be an altered analysis, and some issues can arise. After the sample was prepared (as shown in Figure 4.13), the procedure to actually analyze the material is the following:

1. Connect the machine to a PC
2. Calibration phase of the equipment:

i. Take the plate tied to the pistol and insert it in its housing over the camera

ii. Cover the system with a specific tool to filter X-rays and to avoid harmful situations

iii. Initialize, by using the display and menu shown from the pc, the calibration

iv. Wait until a message of "finish" isn't shown on the monitor

3. After calibration, the cup can be placed on the camera, and the X-ray filter tool can also be inserted

4. Choose the method for analyzing

5. Run the *Average* test, by analyzing three times in three different positions (every time take off the filter tool, move the cup onto the wanted points, put on the tool again), and then the software will compute an average of the three evaluations

6. All the analyses (the single and average ones) will be saved in the memory, and an export of CSV/PDF files can be done, since the machinery is connected to a PC

After the calibration, there is the possibility to use different types of analysis, according to the aim of which elements must be detected

- *Soil method* - it gives an elemental analysis from a general point of view, and it detects light compounds until magnesium (sodium and lighter elements can't be evaluated)

- *Mining method* - it analyses more thoroughly, and it allows detecting more hazardous and more toxic heavy metals even at trace concentrations.

Before commenting on the values, it is appropriate to check if the concentrations obtained, which are the minimum and maximum for each element, comply with the equipment limit. The compliance assessment is important to verify whether the analysis diverges or not. Just to compare the two methods, in Section 5.2 a detailed study of the results is introduced.

4.2.2 XRD Analysis

After elemental evaluation, another important step of the *Mineralogical characterization* is the *XRD Analysis*. The model used for the analysis is the **Rigaku ® Smartlab SE** [29] manufactured by the Rigaku ® Corporation. The machine is equipped with software that must be connected to a PC to manage the analysis

and save the results. The XRD technique produces diffraction patterns through X-ray interactions with crystalline structures in samples to identify mineral phases. Every phase is detected by counting the interactions, and the result is a peak with a certain intensity. **High intensity** means accurate information when, at a specific scan rotation angle, there is a high counter number. To enhance the peak and the resolution, there is a specific parameter that allows to improve the mineral phase. It is the **P/B ratio**, and it represents the comparison between the peak and background signal. If the ratio is high, many peaks, at different intensities, are shown, and the single phase is more accurate. It is useful because it is able to investigate compounds that have low concentrations. The instrumentation is composed as Figure 4.14 shows. The monochromator is the most important component because it is able to focus on a single wavelength and maximize the P/B ratio. The monochromatization method, used by the machine, employs the Bragg reflection theory to evaluate the peak intensity. The other important part is

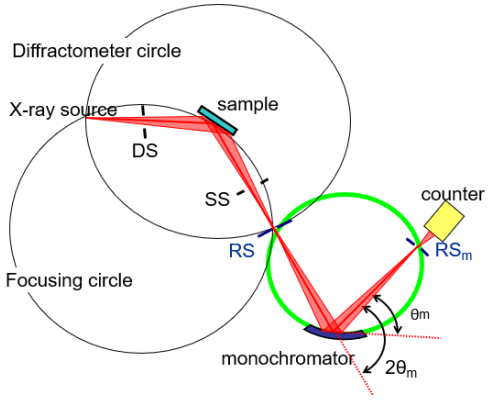

Figure 4.14: XRD instrumentation setup

the DS-SS-RS system, in which the X-ray beam is adjusted in order to maximize the ray absorption. First, the DS size determines the divergence angle that leads to the irradiated area on the sample surface. To adjust the irradiated area, it regulates the offset between two plates, in order to let a certain fraction of X-rays pass. Divergence Slit is related to the intensity and peak resolution. The scattering slit works in the same way as DS, in fact, it is of the same size too. The aim of SS is to prevent scattering. Receiving slit records the receiving width; therefore, intensity and angle resolution depend on its size. The focusing method used during XRD evaluation is the 2θ method, which is useful for thin films as the experiment wanted. The procedure to prepare the trial is:

1. In the instrument toolbox, two plates are needed (one plate equipped with a groove to contain the material, and the other is a transparent plate to allow the diffraction of X-rays)

2. By helping with a surgical knife, fill the hole made by the two plates
3. Insert the trial in its housing
4. Close the machine, calibrate it, and run the measurement

The software shows the step-by-step procedure to run the analysis, and during the calibration, it suggests all the adjustments needed for the specific material. In Subsection 4.2.2 all the results are discussed.

Chapter 5

Results

5.1 Geotechnical Characterization results

5.1.1 Moisture

In Table 5.1, the moisture evaluation is summarized. Surprisingly, SS has more moisture than SE, despite they should be made of the same material. However, SE was at a point where many tractors passed by, and they moved a lot of debris to enlarge the storage area. The continuous perturbation might have altered the distribution and the moisture held by the cuttings. Moreover, SS was placed in a sheltered spot, which allows it to be more unaltered and to stabilize the moisture content. The cold temperature has helped to maintain the material with a stable humidity, hence it was closer to a sludge.

Table 5.1: Moisture content assessment

Sample	Weight pre-drying (g)	Weight post-drying (g)	Water lost (g)	Moisture content (%)
SS	3888,92	2879,6	1009,32	35
SE	1494,6	1150,16	344,44	30

5.1.2 Grain Size

Figure 5.1 shows the distribution of grains in both SE and SS. Comparing the lines, it is possible to denote the difference in grain size contained in each sample, highlighting the most dominant size class. In the SS sample, the sand content is low, while the curve drops in the silt class. Sludge Silos result in a silt, since the drop is almost entirely in the silt class. The SE sample is similar, but the curve starts to drop actually in the sand class. Conversely to SS, Sludge External is closer to being a silty sand since at 0.06 mm of retention size 70% of the total

material passes through, which cannot be neglected. Analyzing the average grain size, D_{50} value must be evaluated for both samples:

- SS D_{50} is equal to 20 μm
- SE D_{50} is equal to 32 μm

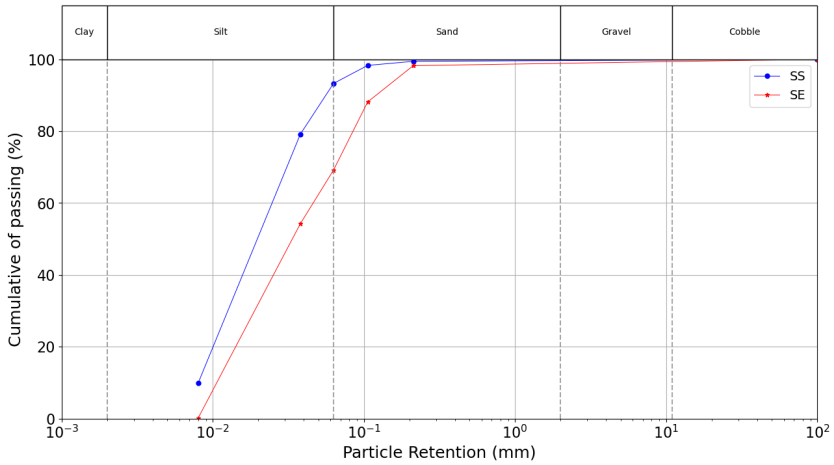

Figure 5.1: Grain Size Distribution of SS and SE

5.1.3 Consistency Evaluation

After the procedure described in Subsection 4.1.4, the results of **Liquidity tests** are reported in Table 5.2.

Table 5.2: Measurements to evaluate the Liquid Limit Test

Sample	Blows	Tare (g)	Net (wetted, g)	Total (dried, g)	Net (dried , g)	Water lost (g)	Moisture Content(%)
SE	6	0.77	10.94	8.8	8.03	2.91	27
	9	0.75	14.29	11.33	10.58	3.71	26
	12	0.9	14.46	12	11.1	3.36	23
	17	1.35	15.97	13.73	12.38	3.59	22
	18	0.79	11.26	9.36	8.57	2.69	24
	22	0.75	21.09	17.12	16.37	4.72	22
	26	0.79	15.47	12.96	12.17	3.3	21
	30	0.71	15.98	13.45	12.74	3.24	20
SS	7	2.15	18.33	14.98	12.83	5.5	30
	8	2.19	13.12	11.69	9.5	3.62	28
	11	2.14	11.81	10.85	8.71	3.1	26
	18	2.12	12.17	11.24	9.12	3.05	25
	23	0.76	11.73	9.72	8.96	2.77	24
	29	0.77	12.84	10.71	9.94	2.9	23

Table 5.3: Measurements to evaluate the Plastic Limit Test

Sample	Tare (g)	Net (wetted, g)	Total (dried, g)	Net (dried, g)	Water lost (g)	Moisture content (%)	w_P
SS	0.81	7.79	7.1	6.29	1.5	19	20
	0.78	6.52	5.85	5.07	1.45	22	
	0.77	8.63	7.57	6.8	1.83	21	
	0.8	5.46	5.12	4.32	1.14	21	
	0.77	8.5	7.59	6.82	1.68	20	
	0.7	5.86	5.55	4.85	1.01	17	
SE	0.82	5.95	5.39	4.57	1.38	23	21
	0.78	6.27	6.03	5.25	1.02	16	
	0.77	8.12	7.63	6.86	1.26	16	
	0.82	6.82	6.25	5.43	1.39	20	
	0.78	6.27	7.44	6.66	-0.39	-6	
	0.74	9.95	7.64	6.9	3.05	31	

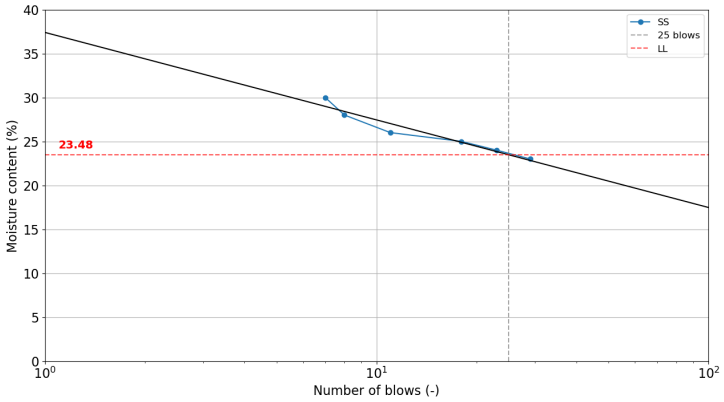

Figure 5.2: Distribution of the scatter plot and the resulting linear fit of sample SS

By comparing the blows and the water content, in Figures 5.2 and 5.3, the evaluation of the Liquid Limit Test is provided, as well as the linear fit of the interpolated points. The fitting equation, in Figure 5.2, of SS sample is highlighted in Equation 5.1.

$$y = -9.96 \cdot log_{10}(x) + 37.41 \tag{5.1}$$

Equation 5.2 is the linear fitting for the SE sample. Since the distribution has the semilogarithmical x-axes, both equations show a log_{10}. Moreover, both show a decreasing trend of moisture, which is what was expected during the preparation.

$$y = -9.5 \cdot log_{10}(x) + 34.44 \tag{5.2}$$

Both equations resulted from the *Polyfit* method on Python, and, providing the values of blows and moisture content, the algorithm found the best interpolation. The algorithm allows to know the values of *Slope* and *Intercept* too, which are those shown in Equations 5.1 and 5.2. The slope in Equation 5.1 is higher, which

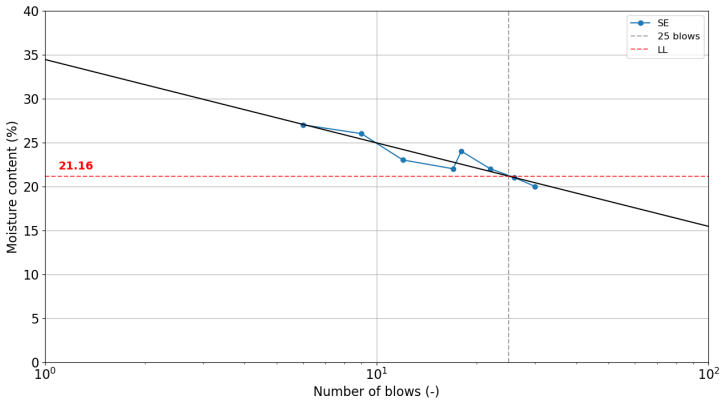

Figure 5.3: Distribution of the scatter plot and the resulting linear fit of sample SE

means the material tends to lose more water, despite the difference in moisture content in the first and then in the last point evaluated (see Table 5.2) is the same. Another characteristic that arises from the two fits, is that in Figure 5.2, there was an unusual value of moisture. Maybe, for that test piece, the blows were over the actual number, and the procedure was not correct. To know the actual effectiveness to predict the value, in Table 5.4, R^2 coefficients are displayed, and as expected, the linear fit for the SS sample is better. The SE sample is still good, but not enough to be sure of the result. For the *Plastic Limit Test* results, shown in Table 5.3, some values are highlighted in red. During the experiment, some mistakes seem to have happened. The weight evaluation, after drying them in the oven, a test piece was found to weigh more than the weight reported previously before the drying process. Hence, for the plastic limit w_L that test piece was not taken in the calculations. In Figures 5.2 and 5.3 the w_L is highlighted in red but Table 5.4

Table 5.4: Summary of R-squared and consistency evaluation for SS and SE samples

Sample	R^2	w_L **at 25 blows**	**Plasticity Index**
SS	0.93	23.48	3.48
SE	0.88	21.16	0.16

summarizes the analysis for each sample. It is an important value for estimating the **Plasticity Index** and, thus, the actual behaviour of the material. In Table 5.4, plasticity indices are displayed too, because they are important to evaluate the coordinates for entering Casagrande's chart, shown in Figure 4.6. The calculations were performed by Python code, and the prompt is given in the appendix section

at Listing A.2 As reported in Figure 5.4, the two materials lie in different regions

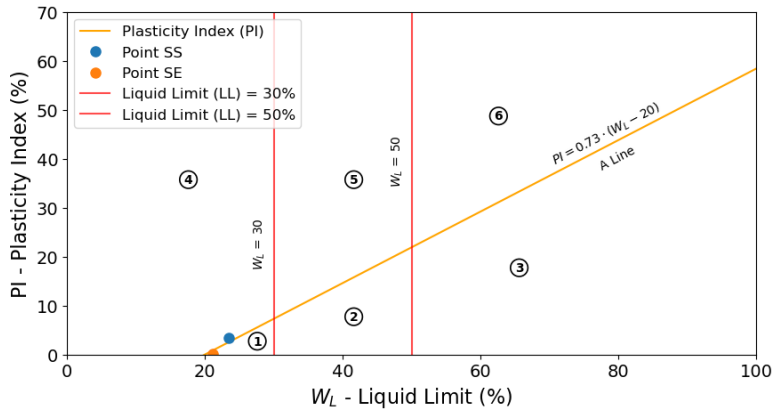

Figure 5.4: Casagrande's Plasticity chart with SS and SE consistency evaluation

of Casagrande's chart. SS lies in the region of *Inorganic clay of low compressibility*, whilst SE is in the region of *Inorganic silt of low compressibility*. It is an interesting insight since both materials have similar aspects, but the experiment demonstrates two different behaviours and so different applications in the market.

5.1.4 Load Test Evaluation

Comparison at the unaltered state of SS and SE

Figures 5.5 5.6 5.7 5.8 5.9 5.10 are referred, respectively, to SS and SE samples at the unaltered state without any cement additives. Analyzing the Time vs. Settlement graphs, they indicate a surprising stiffness during the unloading phase, as the test piece doesn't swell and maintains an internal rigidity that can be a sign of good stability and strength. Another important feature is the comparison of Figures 5.6 and 5.9, which are surprisingly different. In Figure 5.6, the curve shows a typical soil trend with a $\Delta\epsilon_z$ of about 0.065 during the Load phase and 0.015 during the Unloading phase.

Regarding SE, Figure 5.9 shows a steep drop at the beginning, and then it follows a linear trend. The possible reason is that SE, at unaltered state, was not sufficiently compressed in the mould, hence, it reaches stabilization after 20 kPa.

The final evaluation is a comparison between the Stress and the modulus of Oedometer E_d, that allows to understand the level of pre-consolidation held by each sample. Analyzing Figure 5.7 shows a peak at the beginning at σ_z= 120 kPa with a related E_d= 100 MPa and then drops down until increasing again at about σ_z=400 kPa. The trend described means SS, at an unaltered state, reaches the pre-consolidation immediately, but analyzing the trend during the unloading phase,

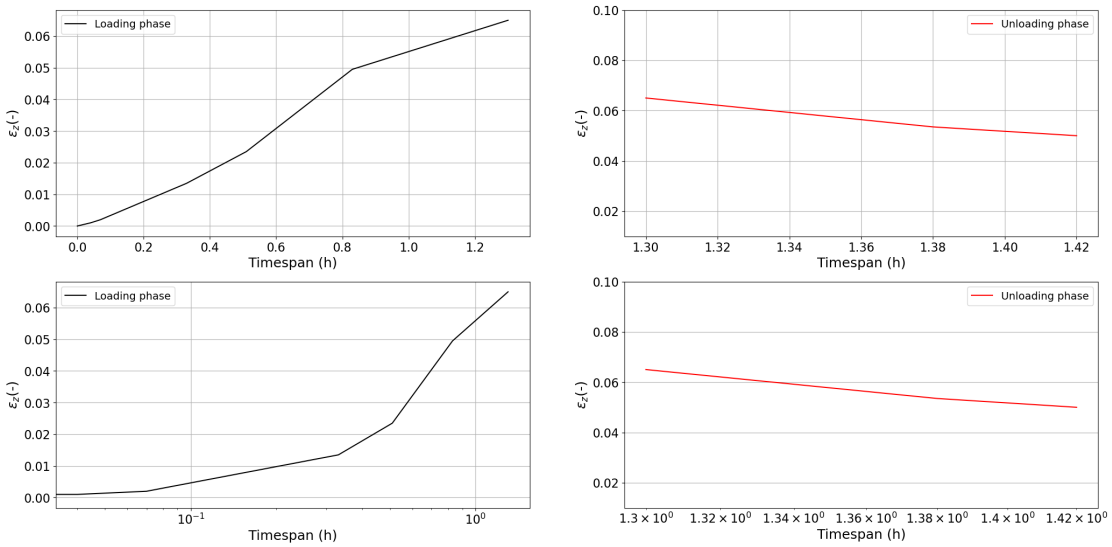

Figure 5.5: SS (unaltered state): Time & Log(Time) vs Settlement during Loading and Unloading phase

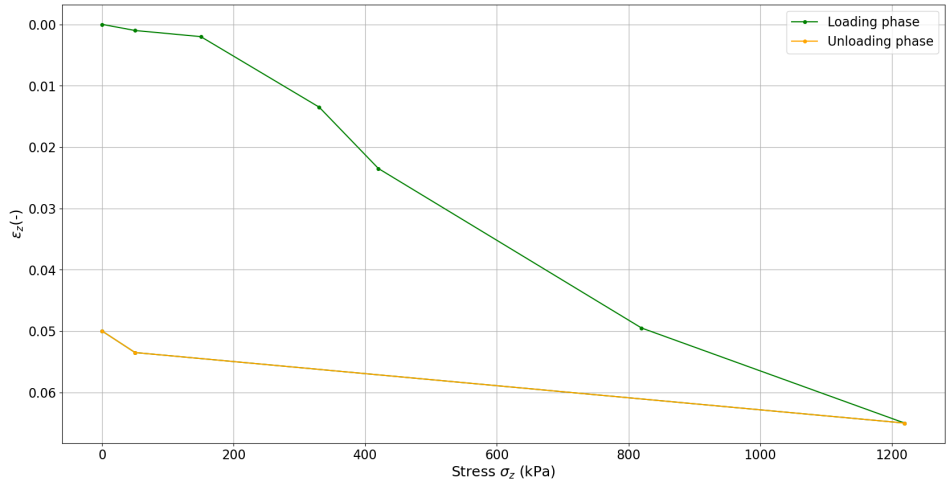

Figure 5.6: SS (unaltered state): Stress vs Settlement during the entire Load Test

the peak of E_d doesn't change. Conversely, SE reaches the peak at the same point as SS, but during the unload, the peak rises to almost 120 MPa. It is an interesting feature because it means it is able to reach higher values of pre-consolidation stress. The possible reason for the rise of E_d in SE only is related to the time at which it passes after the unloading phase started. In fact, SS started the unloading phase after 1 hour, whilst SE's unloading phase was performed after 48 hours, allowing

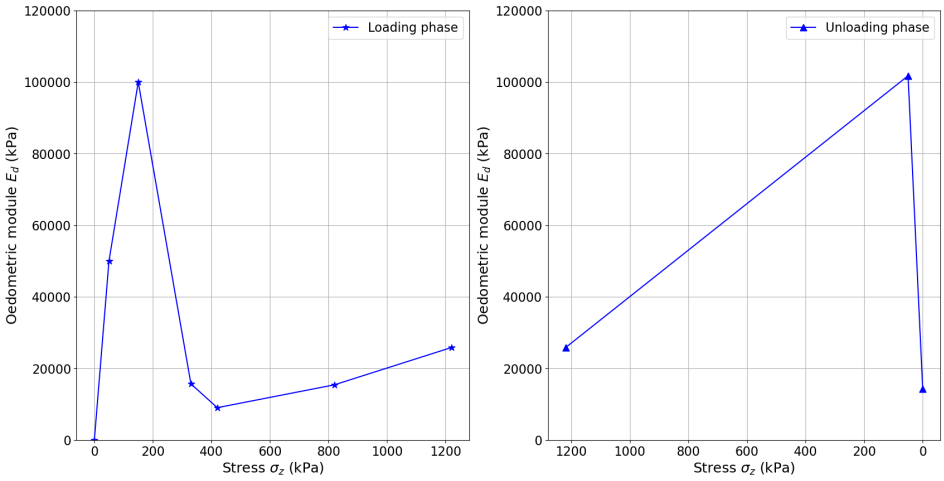

Figure 5.7: SS (unaltered state): Stress vs Oedometric Module during Loading and Unloading phase

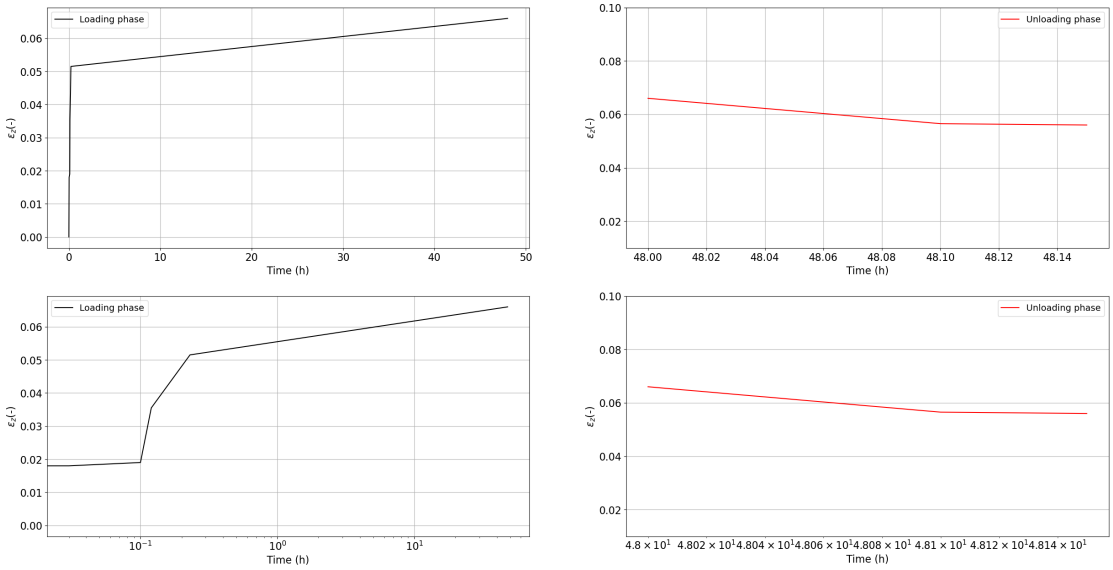

Figure 5.8: SE (unaltered state): Time & Log(Time) vs Settlement during Loading and Unloading phase

the sample to stabilize and increase its stiffness.

Comparison between SS and SE adjusting the composition of both with 10% cement

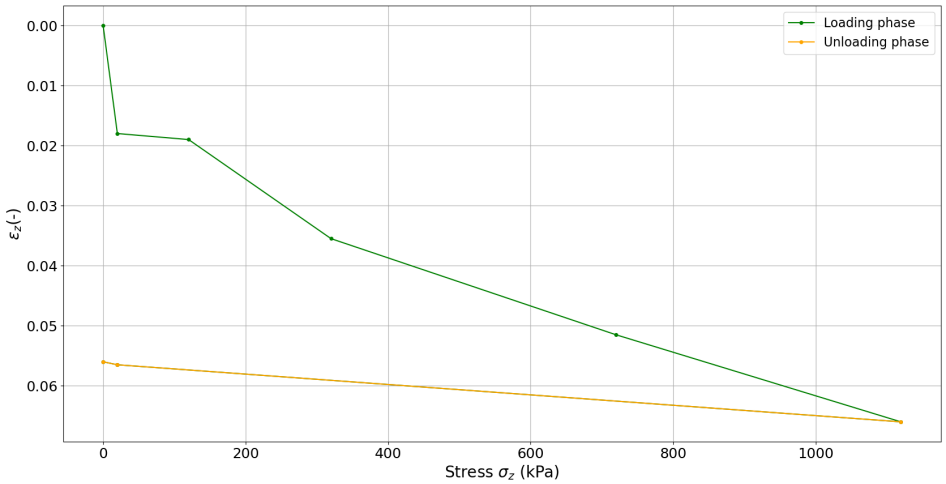

Figure 5.9: SE (unaltered state): Stress vs Settlement during the entire Load Test

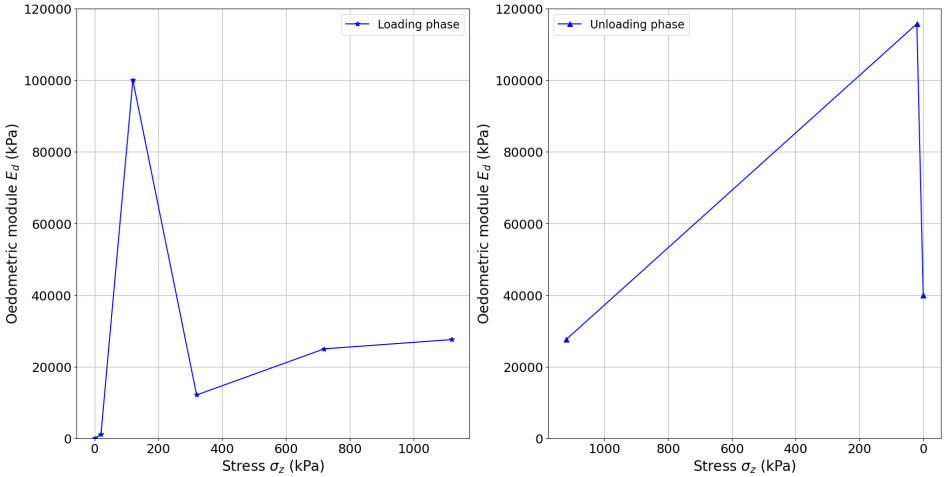

Figure 5.10: SE (unaltered state): Stress vs Oedometric Module during Loading and Unloading phase

After the cement addition, the results of the Confined In-Grip Load Test of SS are shown in Figures 5.11 5.12 5.13 and SE at Figures 5.14 5.15 5.16. The first interesting feature is the settlement variation during the unloading phase. Comparing the Time (or Log(Time)) vs. Settlement graphs with SS and SE at unaltered state, the samples addicitivated with cement tend to remain with the same deformation as before unloading. By comparing, for instance, SS, the $\Delta\epsilon_z$ in Figure 5.11 is neither equal to 0.01. On the other hand, in Figure 5.5 $\Delta\epsilon_z$ is almost 0.02, which is always a great result and it is another demonstration that Portland

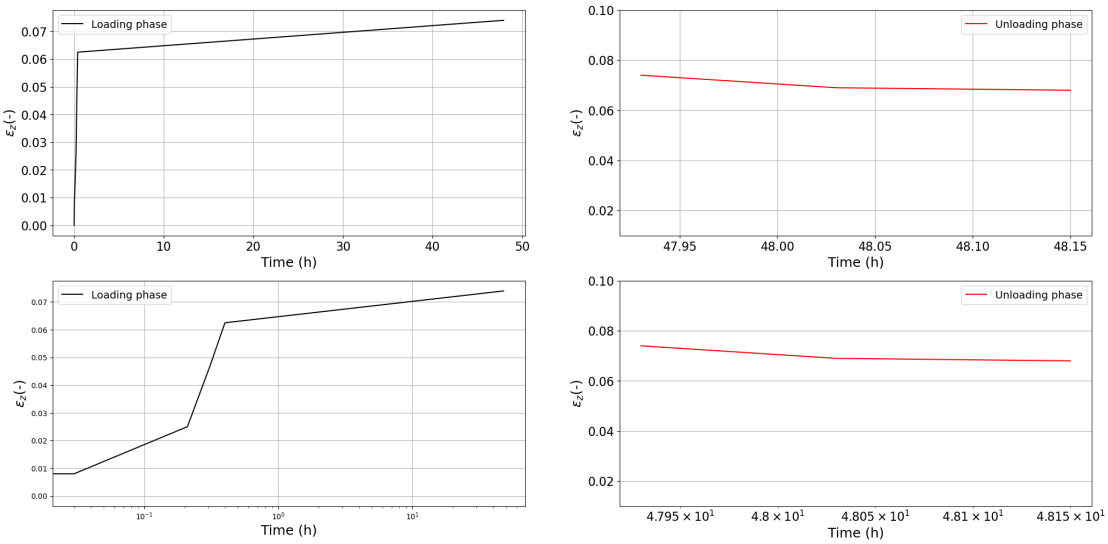

Figure 5.11: SS (unaltered state) +10% additive: Time & Log(Time) vs Settlement during Loading and Unloading phase

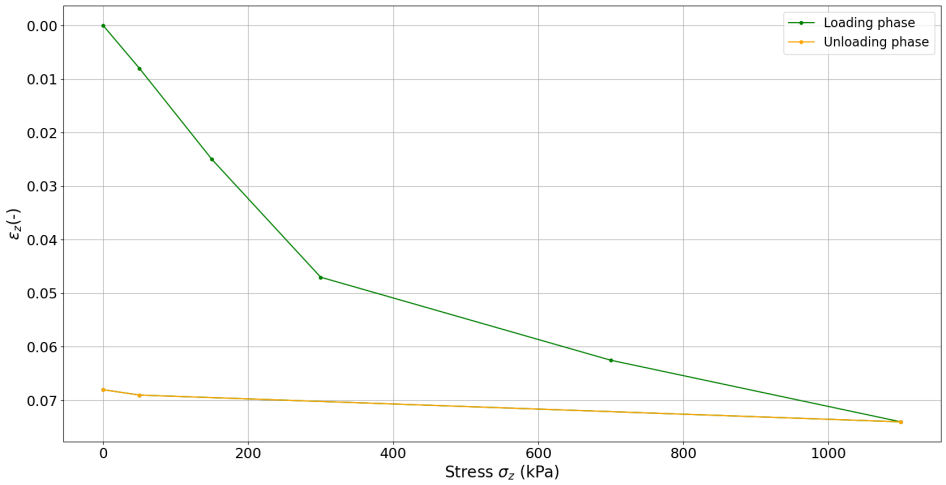

Figure 5.12: SS (unaltered state) +10% additive: Stress vs Settlement during the entire Load Test

cement is able to improve the stability of the samples. Same characteristics are demonstrated comparing Figures 5.8 and 5.14.

Concerning σ_z vs ϵ_z, both Figures 5.12 and 5.15 do not follow a typical soil trend. In Figure 5.12, a steep drop is visible at the beginning, and after about 240 kPa, the trend flattens because the material is gaining stiffness. Therefore, the slope during the unload phase is flatter, a sign that the material is improving

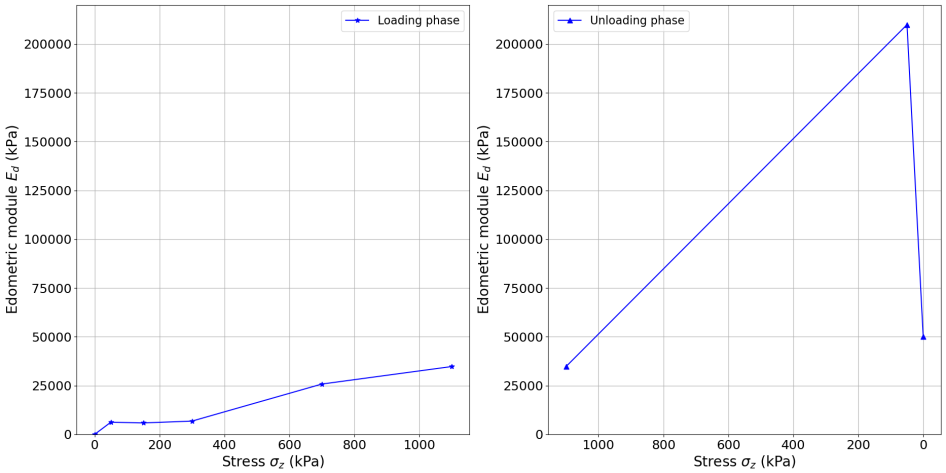

Figure 5.13: SS (unaltered state) +10% additive: Stress vs Oedometric Module during Loading and Unloading phase

its strength. It is also demonstrated that, analyzing Figure 5.13, which is the best comparison to estimate the actual pre-consolidation, the highest stiffness is achieved. During the load phase, the trend rises, but a peak is not achieved. Here, the cement works as an enhancer of mechanical strength; hence, the compound is not closer to the pre-consolidation stress. Although the loading phase does not give an appreciable E_d module value, the unloading phase highlights a maximum of 210 MPa at σ_z= 50 kPa.

Regarding Figure 5.15, the trend is close to a soil; however, the settlement is lower than SS +10%. A little difference, compared to Figure 5.12, is the unloading phase in which in SE +10%, the $\Delta\epsilon_z$ is higher than SS +10%, meaning a higher tendency to swell and lose its consolidation. Regarding the analysis of E_d in Figure 5.16, a first pre-consolidation was reached E_d=67 MPa at σ_z=150 kPa. Then, it drops and starts to rise again. The highest peak is achieved in the unloading phase, which corresponds to E_d=200 MPa at σ_z=20 kPa (a load of 0.4 kg).

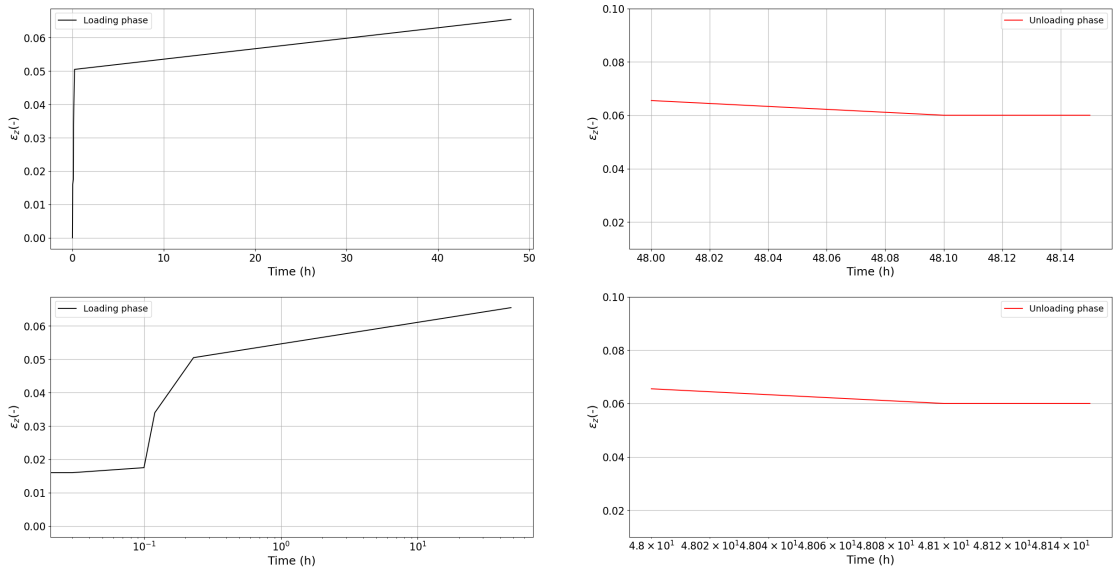

Figure 5.14: SE (unaltered state) +10% additive: Time & Log(Time) vs Settlement during Loading and Unloading phase

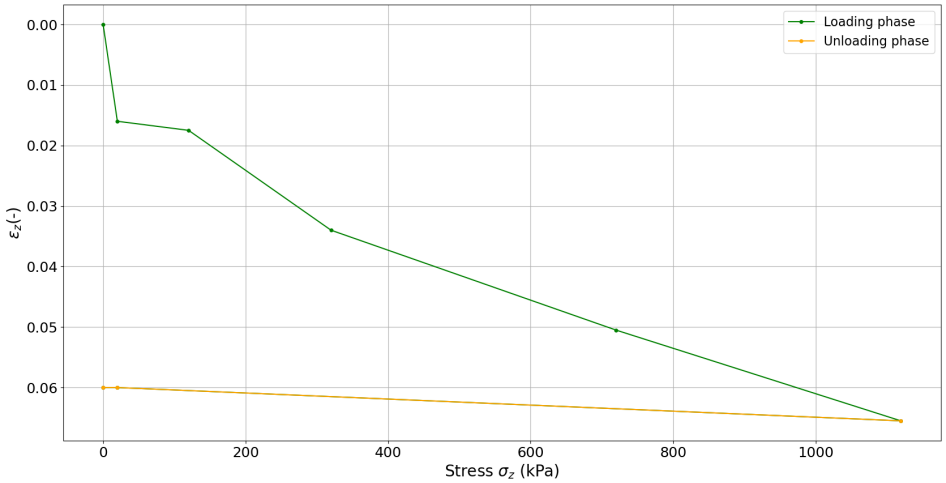

Figure 5.15: SE (unaltered state) +10% additive: Stress vs Settlement during the entire Load Test

Comparison on what happens to SS whether it is dried overnight or cured for 1 hour

To compare different scenarios, the thesis study opted to see what happens when SS reacts:

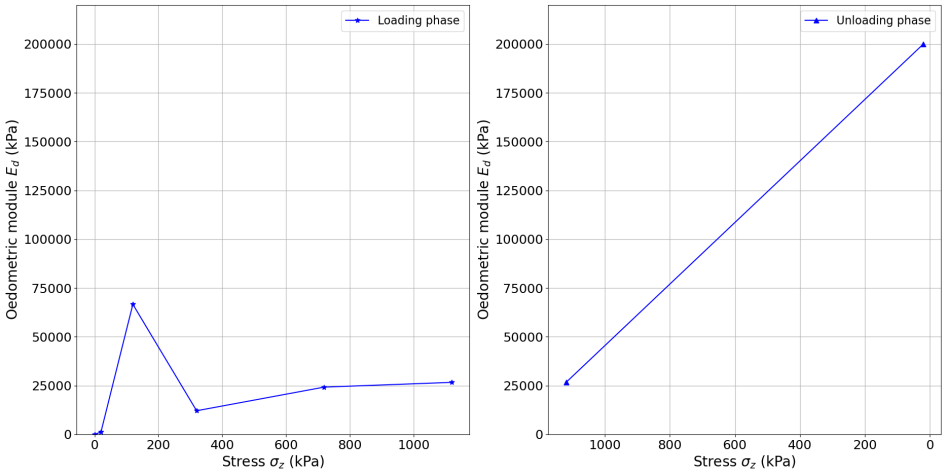

Figure 5.16: SE (unaltered state) +10% additive: Stress vs Oedometric Module during Loading and Unloading phase

- By drying the sample overnight and trying to test it with and without 10% additive
- By stabilizing it for 1 hour in the oedometer ring, either in an unaltered state and with 10% additive

Analyzing what happens among the tests without addictivation, Figure 5.17 achieves a $\epsilon_z \cong 0.05$ whilst in Figure 5.23 $\epsilon_z \cong 0.05$ $\epsilon_z \cong 0.11$ which means it tends to settle more than the dried one. It needs to be observed that the sample left to dry overnight has a moisture of 29.2% (the table with all the data is left to check in Table A.4 in Appendix A), the calculation is the same reported in Equation 4.1

In contrast to what was evaluated in Figure 5.5, the settlement is smoother in both SS dried one night and SS cured for one hour. However, in the unloading phase ϵ_z remained more stable in Figures 5.17 and 5.24. Regarding the settlement study, by varying σ_z, Figure 5.17 shows a pronounced $\Delta\epsilon_z$ during the unloading phase, which is the highest among the figures analyzed so far.

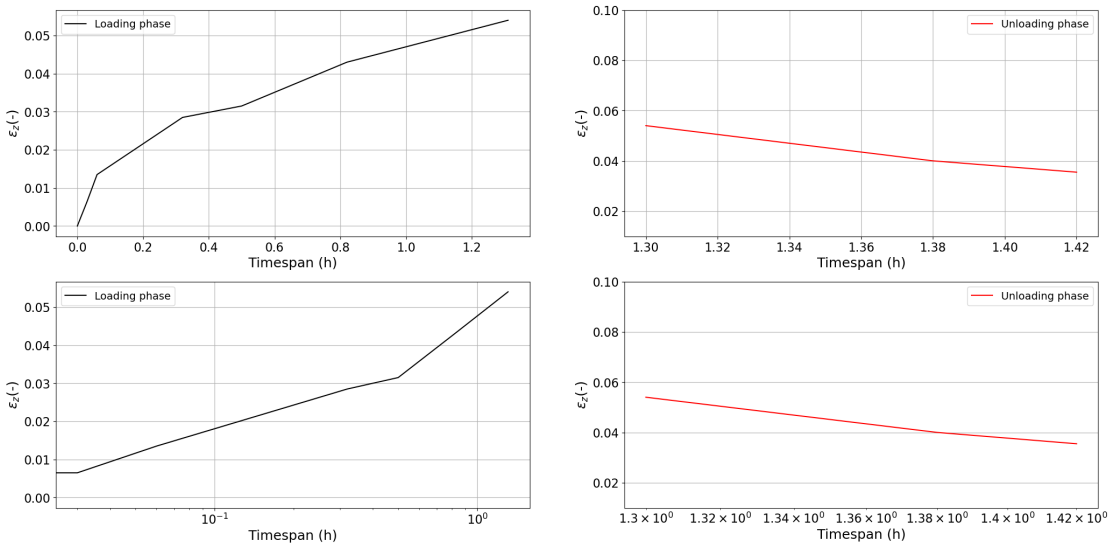

Figure 5.17: SS (dried overnight): Time & Log(Time) vs Settlement during Loading and Unloading phase

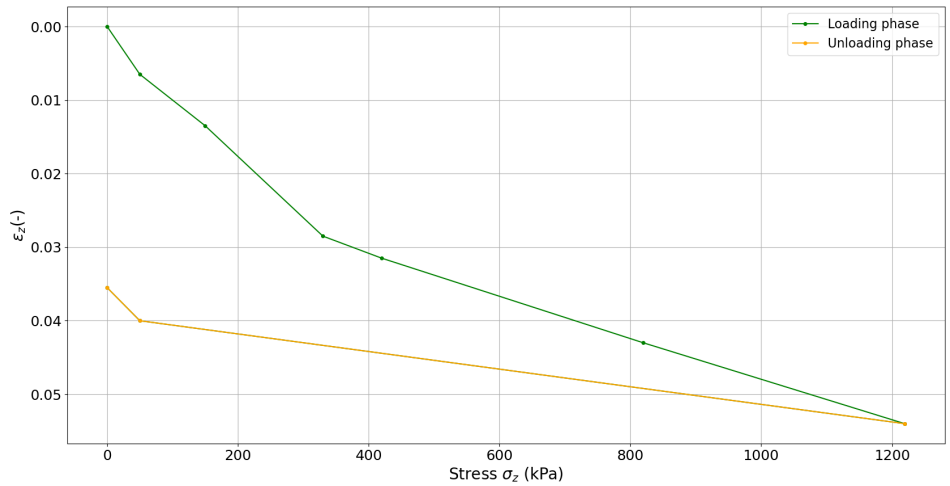

Figure 5.18: SS (dried overnight): Stress vs Settlement during the entire Load Test

However, both SS dried and cured have a hyperbolic trend during the loading phase, which suggests the diversion from a typical natural soil.

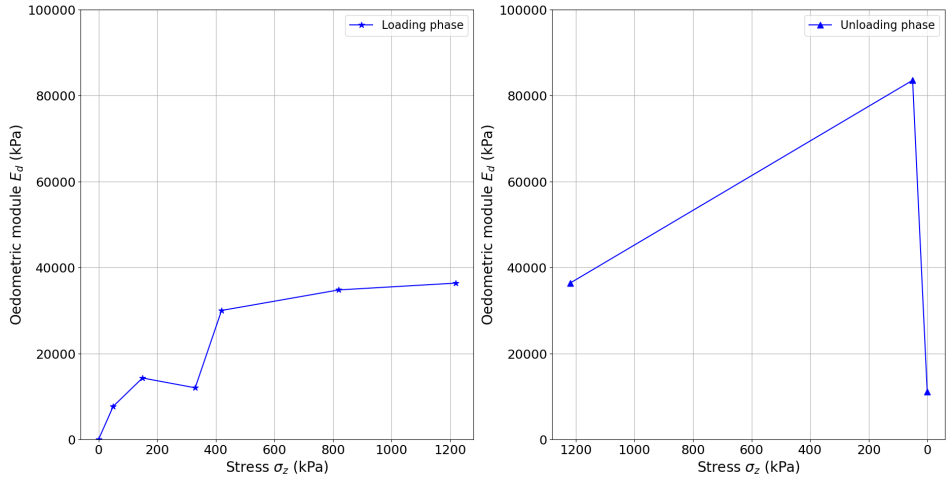

Figure 5.19: SS (dried overnight): Stress vs Oedometric Module during Loading and Unloading phase

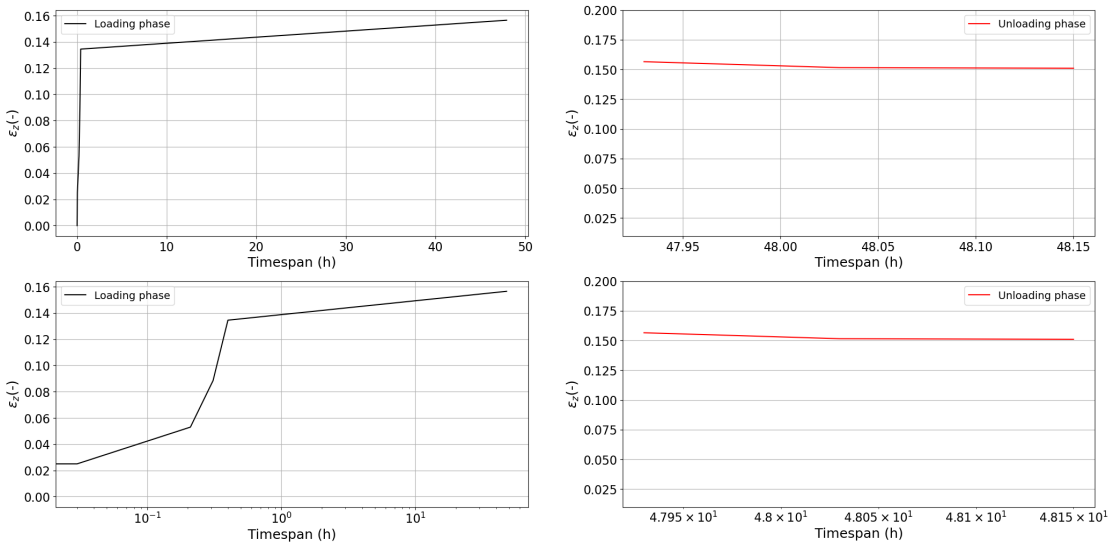

Figure 5.20: SS (dried overnight) +10% additive: Time & Log(Time) vs Settlement during Loading and Unloading phase

Evaluating E_d, by varying σ_z, two different behaviours has been discovered. In Figure 5.19, the curve flattens after about σ_z=400 kPa, meaning the pre-consolidation was far from the maximum load of 1500 kPa. Therefore, during unloading, a peak is achieved at σ_z=20 kPa corresponding to E_d=83 MPa. In Figure 5.25, E_d skyrocketed to 70 MPa, which means that the pre-consolidation was close, but it was not reached. The critical point was achieved when the load

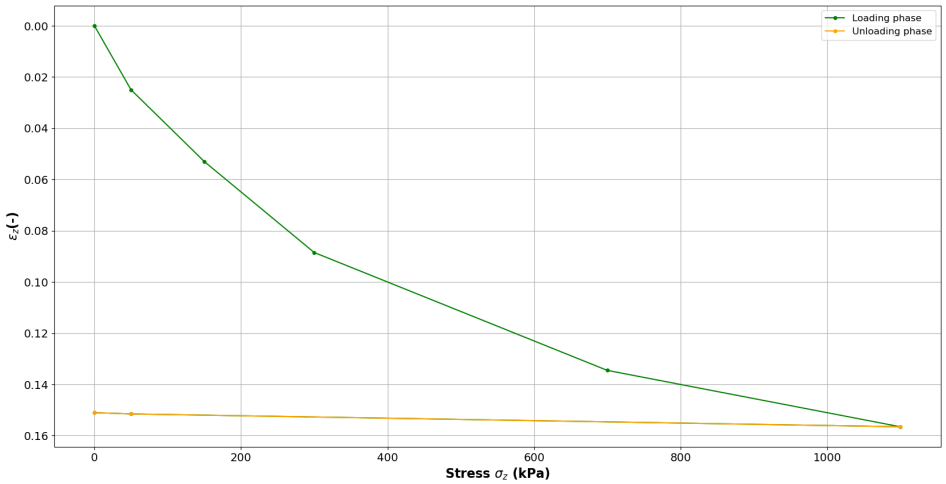

Figure 5.21: SS (dried overnight) +10% additive: Stress vs Settlement during the entire Load Test

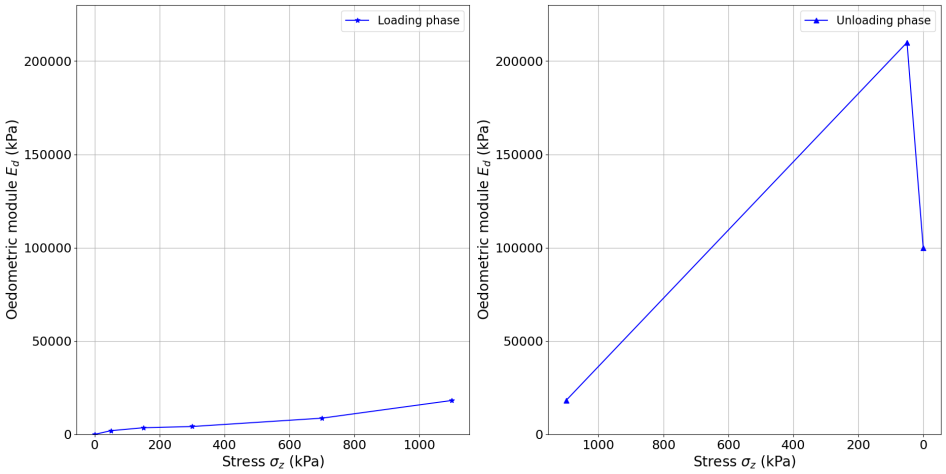

Figure 5.22: SS (dried overnight) +10% additive: Stress vs Oedometric Module during Loading and Unloading phase

was dropping, and a peak of E_d=267 MPa at σ_z=1019 kPa was achieved. It is a particular behaviour because in Figure 5.25 is the first time in the entire In-Grip Load Test analysis that the peak E_d was not reached at the lowest level of load during the unloading phase.

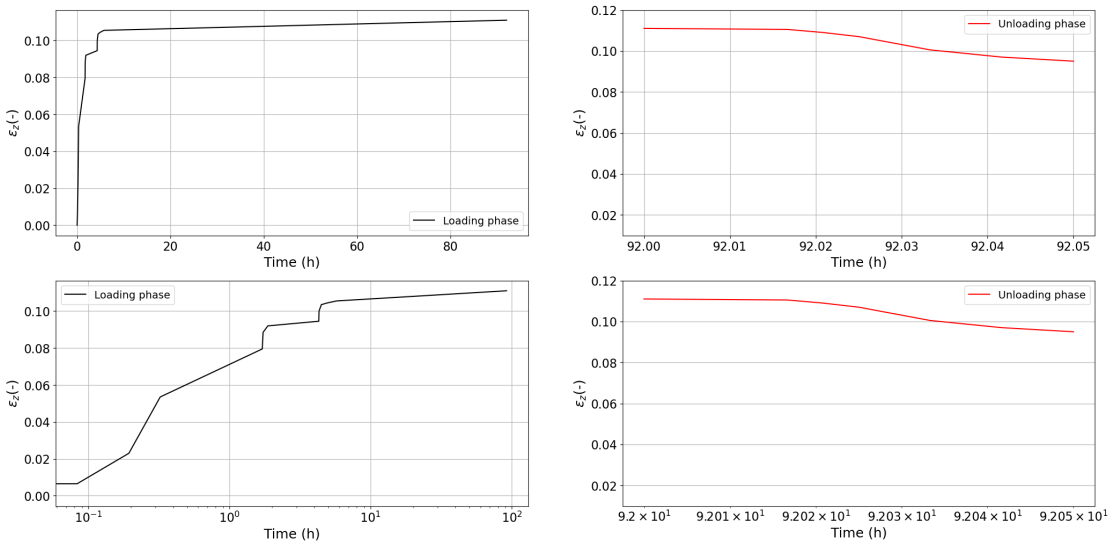

Figure 5.23: SS (unaltered state) stabilized for 1 hour: Time & Log(Time) vs Settlement during Loading and Unloading phase

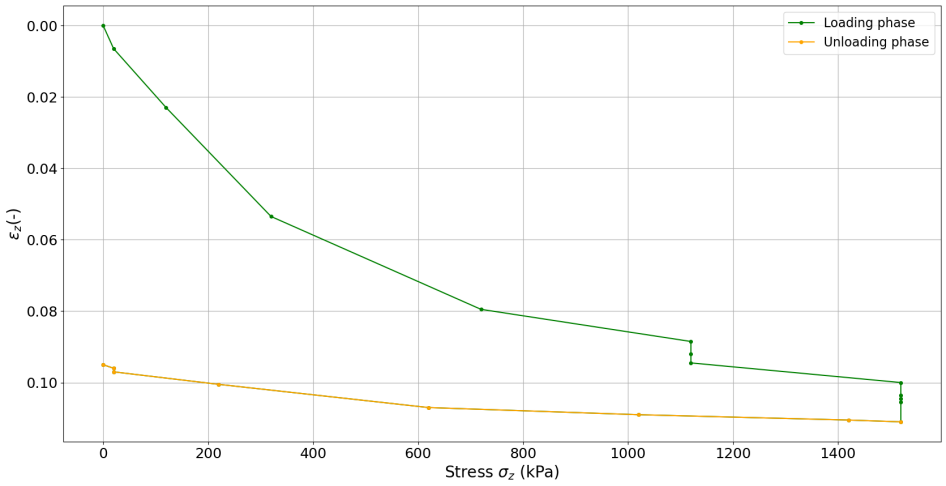

Figure 5.24: SS (unaltered state) stabilized for 1 hour: Stress vs Settlement during the entire Load Test

The analysis of the distribution regarding Figures 5.20 and 5.26 shown different behaviours. In Figure 5.20 ϵ_z sharply increased and then the settlement stabilized again.

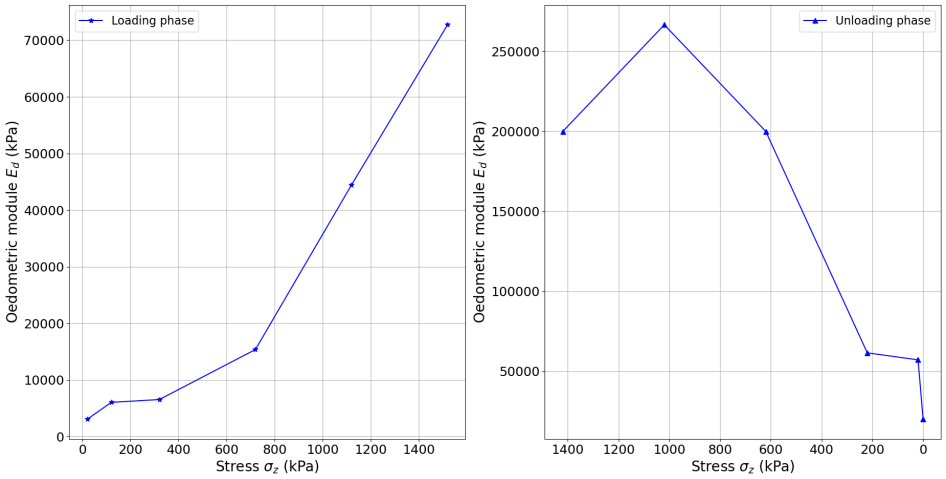

Figure 5.25: SS (unaltered state) stabilized for 1 hour: Stress vs Oedometric Module during Loading and Unloading phase

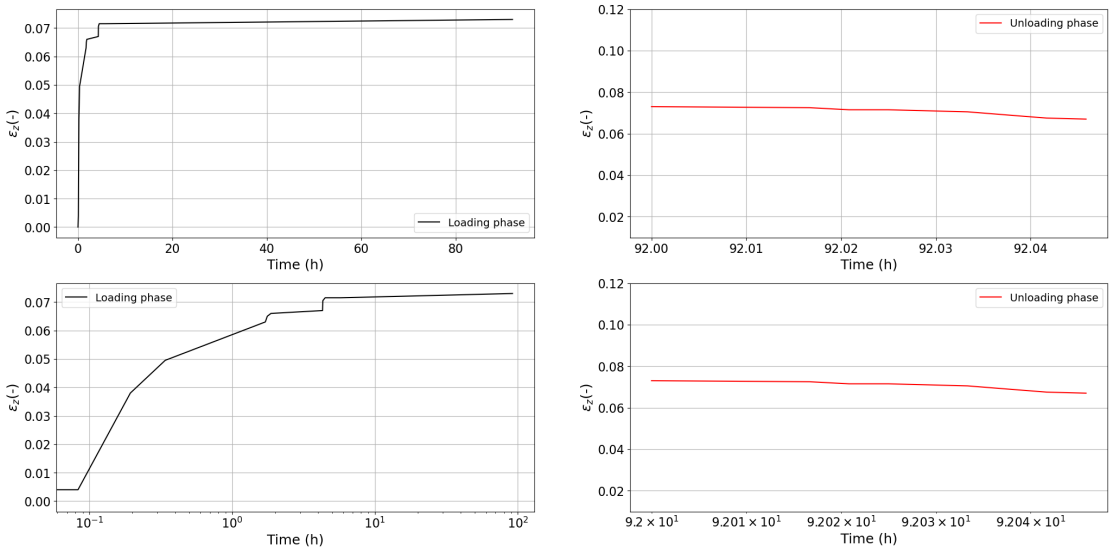

Figure 5.26: SS (unaltered state) +10% additive and stabilized for 1 hour: Time & Log(Time) vs Settlement during Loading and Unloading phase

After the loading phase, ϵ_z remained quite stable, meaning that the material kept the structure, and it did not swell. In Figure 5.26, the trend is different. The material maintained a regular rise, and then at the highest load, the settlement was stabilized, meaning that SS cured for one hour, with 10% additive, could keep its structure and it maintained its shape.

The stress-settlement evaluation demonstrated the diversion from the natural

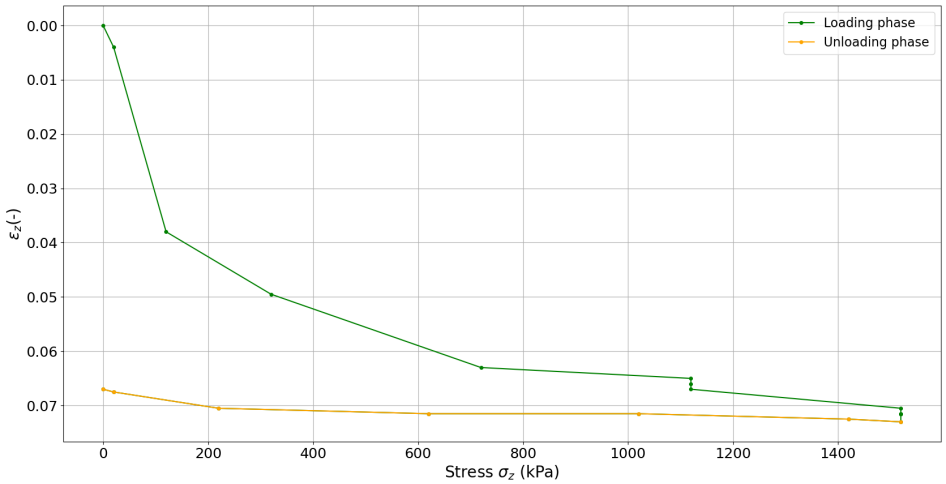

Figure 5.27: SS (unaltered state) +10% additive and stabilized for 1 hour: Stress vs Settlement during the entire Load Test

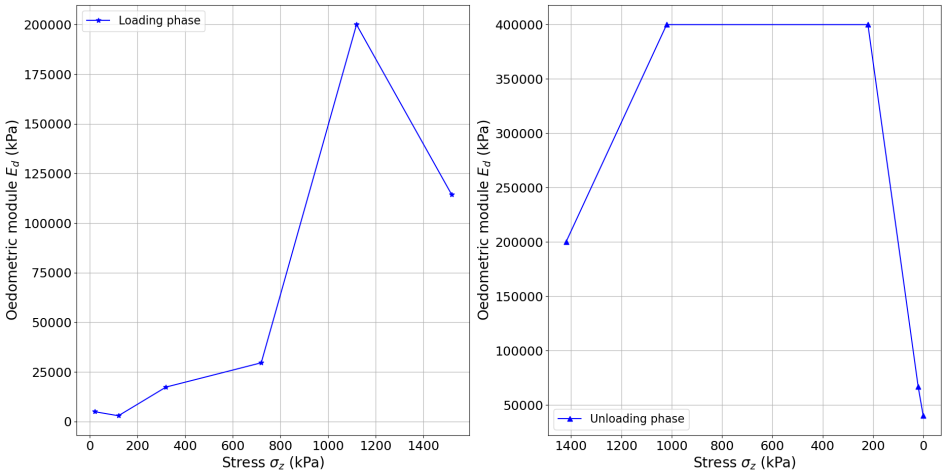

Figure 5.28: SS (unaltered state) +10% additive and stabilized for 1 hour: Stress vs Oedometric Module during Loading and Unloading phase

soil for both Figures 5.21 and 5.27. As precisely, Figure 5.27 has a steeper curve during the loading phase, meaning it was not properly consolidated even after curing for one hour. Furthermore, in the last part of the load, the curve is almost flat, meaning that it was increasing its stiffness, and pre-consolidation was close.

Regarding Figure 5.18, it was not steep; however, it means the sample was far from achieving consolidation because it is noticeable the difference in ϵ_z among both graphs. In Figure 5.21, the level of settlement is higher than SS cured for one hour, and it could justify why in Figure 5.22 the pre-consolidation was not even

close since the curve is almost flat. Between dried overnight and cured SS, the highest value of E_d was achieved in Figure 5.28, which is 400 MPa. In the same figure, the sample showed a trend similar to some of the previous test pieces, where during the load phase, it reached the pre-consolidation point at $\sigma_z \cong 1150$ kPa (a corresponding E_d=200 MPa). Afterwards, in the unloading phase, an unusual feature was discovered because at different stress levels the material achieved the same oedometer module of 400 MPa.

5.1.5 Workability Test

The final evaluation regarding the geotechnical characterization is the **Workability Test**. The aim is to understand how the material behaves at different curing times. Traditionally, 48 hours is the first curing time at which the samples are analyzed. After that, the curing can last for 72, 120, ecc, even for weeks. In Table 5.5 the relative volumetric mass for each test piece is summarized.

Table 5.5: Summary of Volumetric Mass

Sample	**Curing Time (hours)**	**Volumetric Mass (kg/m^3)**
SS 1	120	1526.24
SS 2	72	1698.11
SE 1	72	1693.39
SS +10% 1	48	1735.32
SS +10% 2	48	1694.65
SS +10% 3	120	1681.87
SS +10% 4	120	1661.24
SE +10% 1	168	1161.60
SE +10% 2	168	1176.04
SE +10% 3	120	1630.97

Figure 5.29: SS with zero curing time

Hence, the curing process changes the mineralogical composition, and the compound could benefit in terms of mechanical and structural strength. In Figure 5.29, the SS sample extruded immediately after being compacted in the die is shown. Although it looks stable and it doesn't collapse, the actual situation is a material that has no strength, and it fails easily with low loads. However, leaving it outside, it has demonstrated a great hardening quality which could be interesting for some applications. This feature allows to perform a curing process to see if:

- The material can harden by itself even without an additive
- The cement addition enhances the hardening process
- Adding Portland cement allows for improving the mechanical strength, checking how it changes with different curing times

SS Unaltered State

In Figure 5.30, the distribution is quite regular, and the cracking occurred at about 101 kPa ($\sim$ 20 kg). The corresponding volumetric mass is the lowest of all the test pieces (check Table 5.5), which means higher porosity, and, regarding applicability, it can be employed as a thermal insulator. However, it needs to specify that when the test piece was extruded, some holes were present. As said previously, due to insufficient capacity to properly constipate, the material could have some issues.

The criticality arose from the type of die used. It was taller than wide, and the result is an insufficient ability to homogenize the material with a proper tool. Thus, after taking off the die, some cement was used to fill the most critical holes. It has been employed as a dry material, hence it did not interact or react with the SS

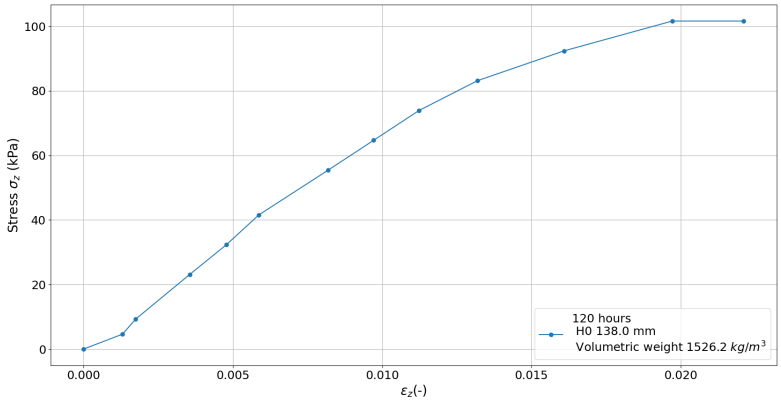

Figure 5.30: Workability Chart: SS 1 cured for 120 hours

material. On the other hand, Figure 5.31 shows a different compressive strength. As mentioned at the beginning of the subsection, a low curing time demonstrated that SS collapses earlier. The σ_{fail} is equal to 60 kPa, which corresponds to 13 kg. Comparing the two situations, SS with 120 hours is better since it is lighter and σ_{fail} is greater, achieving 100 kPa.

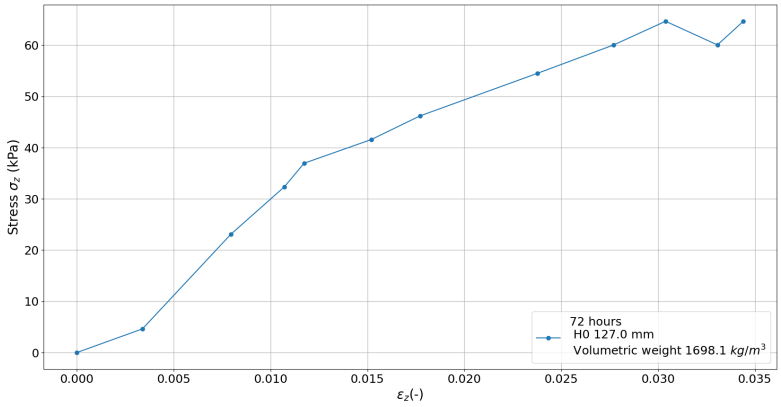

Figure 5.31: Workability Chart: SS 2 cured for 72 hours

SE Unaltered state

About SE at the unaltered state, only one sample was prepared, and it was not full of holes. However, after extruding the cylinder, the test piece was very compacted and homogeneous without any deformations. Figure 5.32 evaluates the evolution of the vertical stress σ_z by studying the variation of the settlement. A first small crack can be seen at σ_z=148 kPa, but it has been tough, and it was able

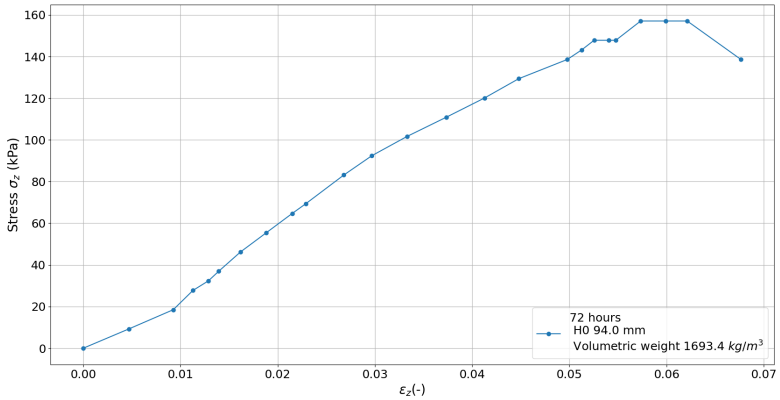

Figure 5.32: Workability Chart: SE 1 cured for 72 hours

to fail at 158 kPa ($\sim$ 34 kg). It is better to overload the test piece, since sometimes the sample has sufficient stiffness to handle more load.

Compared to SS, it is able to achieve a higher level of stress. Probably, the inner structure was more stable, and it owns lesser fractures. The test has thus demonstrated that with medium curing, SE is able to achieve a higher toughness level than SS.

SS +10% addictive

The following figures analyze what happened in SS with 10% addition. Comparing Figures 5.33 and 5.34, which are samples cured for 48 hours, the trend is already different. Checking the stress load at ϵ_z=0.01 in Figure 5.33 is 175 kPa, whilst in Figure 5.34 is equal to 75 kPa.

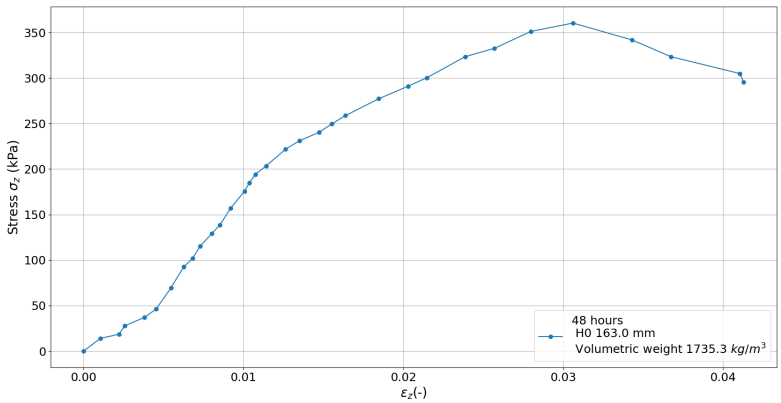

Figure 5.33: Workability Chart: SS 1 + 10% additive cured for 48 hours

Though $\Delta\sigma_z$=100 kPa is $\sim$ 1 kg, the impact is relevant since the difference is one third of the total load required to break and fail both test pieces. Analyzing both samples' volumetric masses, they might justify why SS in Figure 5.33 achieves a higher level of toughness along the entire test, and it continues to show great mechanical strength in the post-peak section. Table 5.5 shows the specific weight values of *SS +10% 1* and *SS +10% 2*, which are respectively 1735.32 kg/m^3 and 1694.65 kg/m^3, hence it justifies why it works better. Furthermore, the other reason why *SS +10% 1* shows better performance is its initial height (H0), which is equal to 163 mm compared to 105 mm by *SS +10% 3*.

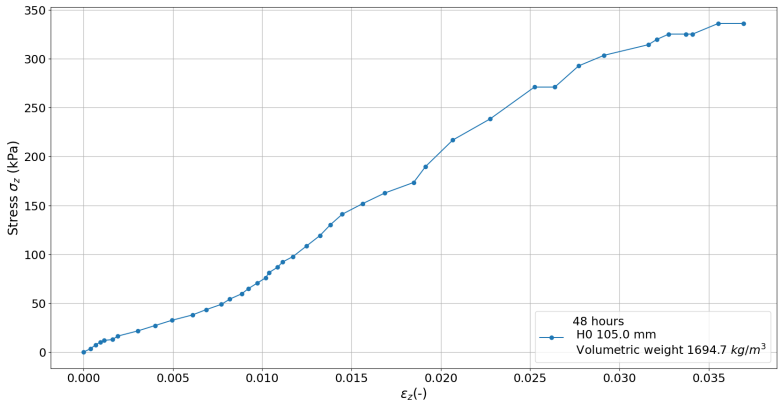

Figure 5.34: Workability Chart: SS 2 + 10% additive cured for 48 hours

Discussing the trends regarding Figures 5.35 and 5.36, sample *SS +10% 3* had an outstanding behaviour. It was able to achieve a vertical stress of 877 kPa ($\sim$ 190 kg). It could be interpreted in two ways:

- **Opportunity** $\rightarrow$ the sample has been prepared very well, and by curing for 120 hours, the cement has worked efficiently to enhance the mechanical strength

- **Issue** $\rightarrow$ during preparation, a mistake, probably, has occurred, since *SS +10% 4* has achieved a maximum σ_z=420 kPa ($\sim$ 78 kg).

Further investigations are required because ϵ_z was different too. Analyzing the total settlement, in Figure 5.35, *SS +10% 3* achieved 0.02, while *SS +10% 4* reached over 0.04. Furthermore, it is noticeable a not regular trend in Figure 5.36 because the inner structure was not sufficiently compacted and many fractures, or holes, were created.

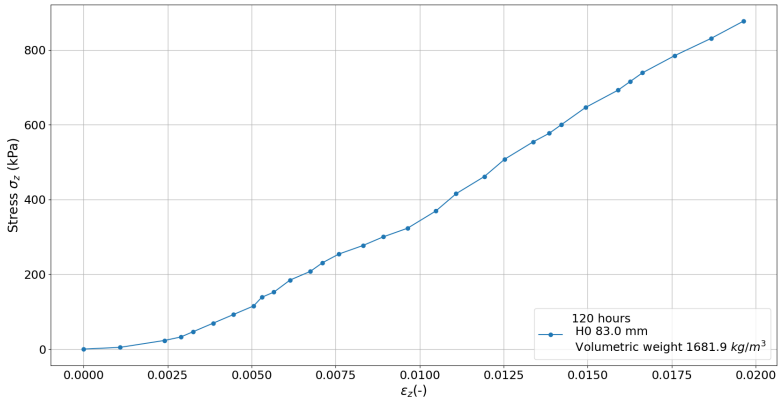

Figure 5.35: Workability Chart: SS 3 + 10% additive cured for 120 hours

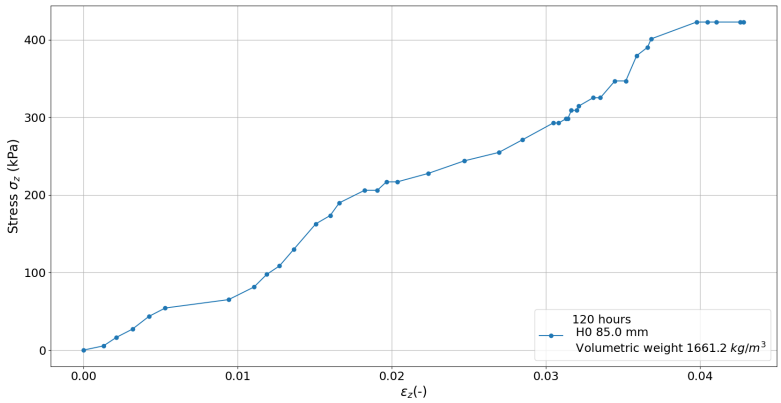

Figure 5.36: Workability Chart: SS 4 + 10% additive cured for 120 hours

SE +10% addictive

The mixture SE +10% has demonstrated different behaviours. Analyzing the test pieces cured for 168 hours, their structures had low-density, low-compacted, and high-cracking. It can be demonstrated by checking the specific weight of both SE +10% 1 and SE +10% 2, which are significantly lower than any other sample tested. However, even with the issue, the test was performed to see what the behaviour would be if the material were laid at this condition. Comparing Figures 5.37 and 5.38, H0 didn't matter in the toughness. *SE +10% 2* has had a better performance, demonstrating a regular stiffness till it has reached 160 kPa ($\sim$ 35 kg). After that, the sample lost any performance. On the other hand, *SE +10% 1* achieved a top stress of 115 kPa ($\sim$ 25 kg), which is a step down. But it was able to maintain the same top σ_z longer than any other material.

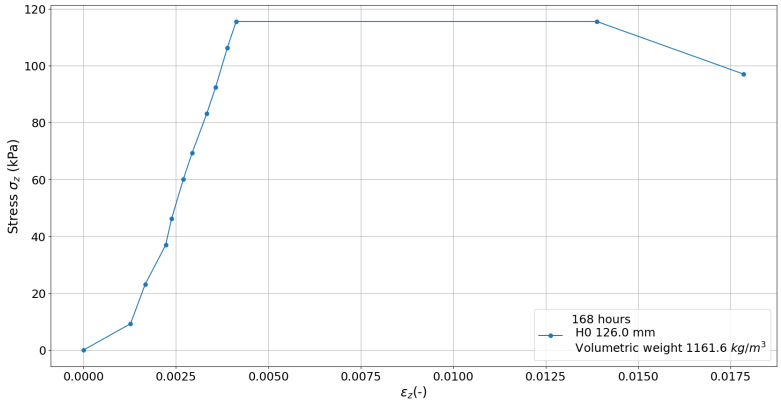

Figure 5.37: Workability Chart: SE 1 + 10% additive cured for 168 hours

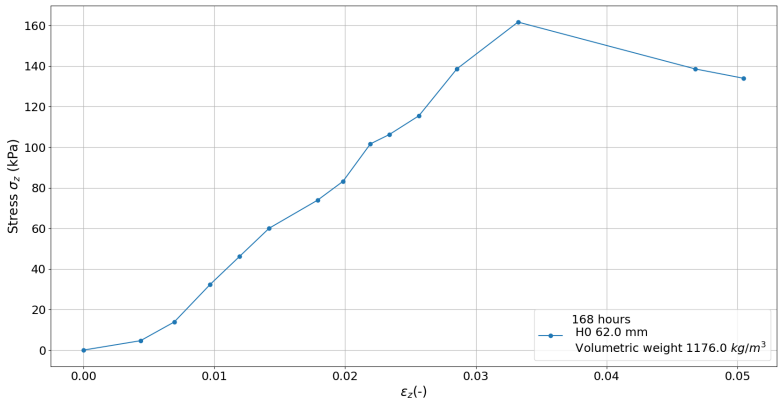

Figure 5.38: Workability Chart: SE 2 + 10% additive cured for 168 hours

The last sample is *SE +10% 3*, which was analyzed after 120 hours of curing. The test piece was quite well compacted and with, apparently, no cracking. It was not a perfect cylinder shape, but it was enough to be analyzed. Figure 5.39 shows a regular trend along the entire test. The top σ_z achieved was 438 kPa ($\sim$ 97 kg). The post-peak section is interesting because, as *SE +10% 1* has behaved, it was able to maintain a consistent stress for longer settlement, until the material was definitely cracked. Another important insight is the difference between *SE +10% 3* and *SS +10% 4*. Both samples have been cured for 120 hours. Though, as mentioned earlier, Figure 5.36 shows an irregular trend instead of Figure 5.39.

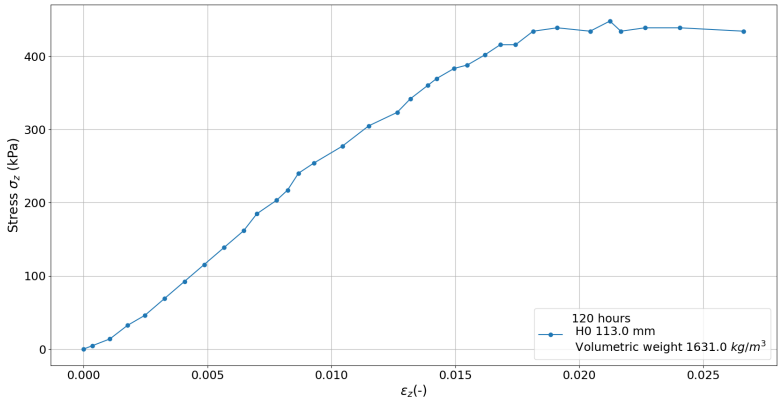

Figure 5.39: Workability Chart: SE 3 + 10% additive cured for 120 hours

5.2 Mineralogical Results

5.2.1 XRF

The procedure described in Subsection 4.2.1 has given the results summarized in Table 5.6 which is related to the *Soil Method*. It must be specified that all concentration values are related to the single element. Despite the tool detecting elements with atomic number greater than Z=12, gaseous compounds like O_2, C, and N are not reported. Those elements are estimated to be the most abundant; therefore, the missing percentage should be given by them. Checking the different concentrations, *Fe* is the top metal by presence. Its value stands at 3.6658 %wt in SS and 3.9192 %wt in SE.

Due to the type of method used, light elements such as *Al* and *Si* are not calibrated, and the machine can't provide an exact value of their concentration. Therefore, *Soil* method, as mentioned in Subsection 4.2.1, detects mostly heavy metals.

To understand the light metal content, Table 5.7 shows the concentration after using the mining method.

Analyzing the scale of concentrations among elements, Table 5.7 has many values similar to what XRF has analyzed with the other method. However, the mining method evaluation highlighted the actual *Si* and *Al* content that can't be neglected, and it is important highlighting them because of the next XRD evaluation. The most relevant metals detected are: *Si, Ca, Fe, Al, P, S*. The largest value is given by *Si*, which is equal to 19 %wt in SS and 20%wt in SE. Some of the heavy metals detected in both Tables 5.6 and 5.7 might come from the abrasion between the stone and the diamond cutting tools (as mentioned by Spriano [7] in Chapter 1). However, it is important to compare the concentrations

Table 5.6: XRF concentration measurements for soil method

Element	**SS (%wt)**	**SE (%wt)**
Al	No data	No data
Si	No data	No data
Ni	0.01160	0.00900
Cu	0.00980	0.00980
Zn	0.00400	0.00580
Se	Out of range	Out of range
Zr	0.01120	0.01700
Nb	0.00130	0.00140
Mo	Out of range	Out of range
Ag	Not compliant / Under LOD	Out of range
Cd	Out of range	Out of range
Sn	0.00240	0.00250
Ta	No data	No data
W	No data	No data
Pb	0.00220	0.00220
Bi	No data	No data
Sb	Out of range	Out of range
Au	No data	No data
P	Out of range	Out of range
S	Out of range	Out of range
Y	0.00190	0.00210
Nd	Out of range	Out of range
La	0.00600	0.00560
Ce	0.00780	0.00850
Pr	Out of range	Out of range
Sm	Out of range	Out of range
Eu	Out of range	Out of range
Gd	Out of range	Out of range
Co	Out of range	Out of range
Fe	3.66580	3.91920
Mn	0.05170	0.06210
Cr	0.00180	0.00310
V	0.01480	0.01190
Ti	0.40080	0.45720
Ca	Not compliant / Under LOD	Not compliant / Under LOD

Table 5.7: XRF concentration measurements for mining method

Element	SS (%wt)	SE (%wt)
Al	2.03100	2.36920
Si	19.08490	20.24900
Ni	0.00820	0.00970
Cu	0.00920	0.00990
Zn	0.00400	0.00680
Zr	0.01790	0.02240
Nb	0.00220	0.00320
Mo	0.00110	0.00200
Ta	0.00410	0.00460
Pb	0.00460	0.00410
P	1.39840	1.63820
S	0.25750	0.29950
Fe	3.58090	3.90890
Mn	0.05340	0.06480
Cr	0.00180	0.00160
V	0.01030	0.01370
Ti	0.35960	0.42880
Ca	6.01270	4.02290

obtained by XRF with Table 2.1, which is related to the legal threshold of the *Environmental code*. Before starting the comparison, it is also important to correlate the results with the report from the certificate laboratory Merieux Nutrisciences Corporation that has characterized the waste of Tomaino Graniti s.r.l. Those analyses were performed following two standards: UNI EN 13657:2004 [30], and UNI EN ISO 11885:2009 [31]. For Chromium VI the method used is the EPA 3060A 1996 + EPA 7196A 1992, which are equal to BS EN 15192:2021 [32]. Comparing the two procedures, the certificate laboratory used another approach, namely, it didn't analyze through XRF but a dilution in aqua regia solution, and afterwards, ICP-OES was employed. The biggest differences between them are:

- *Sample state during analysis* → in XRF is at solid state, in the other, the material is diluted in a solution
- *Calibration* → different element calibrations are imposed in the two instrumentations

In Table 5.8, the results of the sludge characterization on a dry basis by the laboratory are shown. Table 5.9 shows the compliance among the results obtained

Table 5.8: Results of the waste characterization by the certificate laboratory in 2025

Parameters	**Concentration Detected [mg/kg]**	
	Trial 1	**Trial 2**
Sb	< 10	< 10
As	< 5	5.59
Cd	< 10	< 10
Co	43.5	13.2
Total Cr	< 10	14.9
Cr VI	< 1	< 1
Hg	< 5	< 5
Mo	< 5	< 5
Ni	< 10	< 10
Pb	< 10	15.2
Cu	63.2	19.9
Se	< 5	< 5
V	50.7	117
Zn	27.8	55.7

by the XRF analysis, performed in the thesis study, and the certified laboratory.

Table 5.9: Comparison among XRF analysis and Certified Laboratory procedure regarding compliance Table 1 in Annex 5 Title V Part V

Element	**Green and Public Site**		**Industrial Site**	
	XRF analysis	Certified Laboratory	XRF analysis	Certified Laboratory
Sb	Out of range	Compliant	Out of Range	Compliant
As	Not Identified	Compliant	Not Identified	Compliant
Cd	Not Identified	Unknown	Not Identified	Compliant
Co	Not Identified	Only Trial 2 is compliant	Not Identified	Compliant
Ni	Compliant	Compliant	Compliant	Compliant
Pb	Compliant	Compliant	Compliant	Compliant
Cu	Compliant (soil/mining)	Compliant	Compliant (soil/mining)	Compliant
Zn	Compliant	Compliant	Compliant	Compliant
Hg	Not Identified	Compliant	Not Identified	Compliant
Total Cr	Compliant	Compliant	Compliant	Compliant
Cr VI	Not Identified	Compliant	Not Identified	Compliant

It is noticeable that the lab used a more sophisticated procedure, which allowed to investigate a wider number of heavy metals. However, XRF analysis is compliant for all the elements for which the national legislation has fixed thresholds. Whilst for some metals, such as Co, the concentration isn't compliant in one of the two trials. Since in Table 5.8 the entry of Cadmium isn't a specific number but, as it happened in Table 5.6, is under the **Limit of Detection** imposed in the metal calibration. Another important comparison to discuss is between Table 5.11 and Table 2.2. The certified laboratory followed the standard APAT CNR IRSA 3010 B Man 29 2003 + APAT CNR IRSA 3020 Man 29 2003. They can be found in the book Analytical Methods for Water of 2003 [33]. However, the elements are compliant with the legal thresholds.

Table 5.10: Results of the wastewater characterization by the certificate laboratory in 2023

Parameters	**Concentration Detected [mg/l]**
Al	1.5
As	< 0.003
B	0.28
Cd	< 0.001
Total Cr	< 0.0025
Cr VI	< 0.05
Fe	0.45
Mn	0.03
Ni	0.007
Pb	< 0.002
Cu	0.006
Se	0.02
Zn	0.015
Total P	0.12

Table 5.11: Compliance assessment of industrial wastewater parameters

Parameter	**Status**
Al	Compliant
As	Compliant
Ba	—
B	Compliant
Cd	Compliant
Total Cr	Compliant
Cr VI	Compliant
Fe	Compliant
Mn	Compliant
Hg	—
Ni	Compliant
Pb	Compliant
Cu	Compliant
Se	Compliant
Sn	—
Zn	Compliant

5.2.2 XRD

The analysis provides a spectrum related to the compound diffraction at different diffraction angles. For the SS sample, Figure 5.40 shows the resulting evaluation, while for SE the peaks are highlighted in Figure 5.41. The graph has on the x-axis the orientation of the X-ray beam and on the y-axis the intensity of the resulting diffraction. Surprisingly, the peak distribution is different between SS and SE; hence, by using the software associated with the XRD machine, a research of match among the sample's spectra and the database's spectra was performed exploiting a software tool called '*Main Phase Identification*'. In Table 5.12, the possible minerals are listed. It is a powerful tool since it gives many parameters for each identified compound. In the table, the most important features for this study to investigate are:

- **Concentration** → the software computes, on %wt basis the relative compound concentration in the sample
- **RIR** → it is a specific scale parameter that corrects the concentration associated with the spectrum intensity

The analysis has demonstrated the high content of **Quartz** and **Anorthite, Na-bearing** in both samples. Therefore, it means that both samples are mostly made of diorite.

Table 5.12: Summary of minerals and crystals detected by XRD analysis

Sample	Crystal Determined	Concentration wt%	RIR value (-)
SS	Quartz	32.64	3.035
	Anorthite, Na-bearing	48.79	0.629
	Strontium Silicon Titanium Oxide	2.37	51.028
	Chlorite-Serpentine	0.32	22.335
	Sanidine	15.89	0.765
SE	$\alpha - SiO_2$, Quartz	44	3.026
	Anorthite, Na-bearing	49	0.629
	Strontium Silicon Titanium Oxide	5	50.747
	Chlorite-serpentine (NR)	0.39	22.392
	Bismuth Molybdenum Oxide	1.41	16.836
	Samarium Tungsten Molybdenum Oxide	0.28	32.193

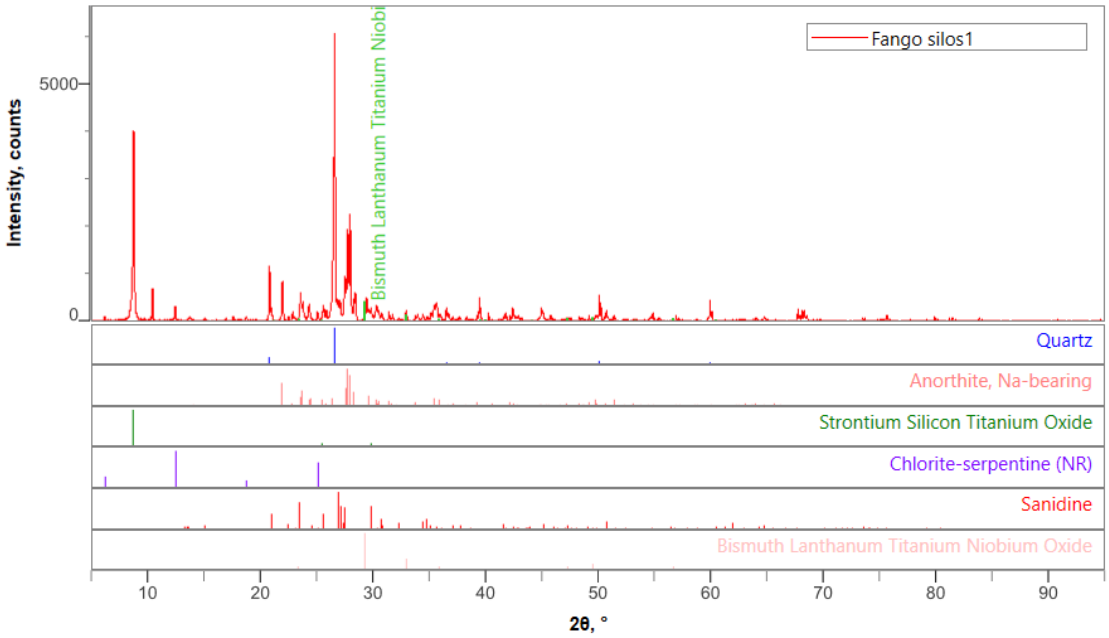

Figure 5.40: Comparison of SS XRD spectrum result and compound matched to it from database

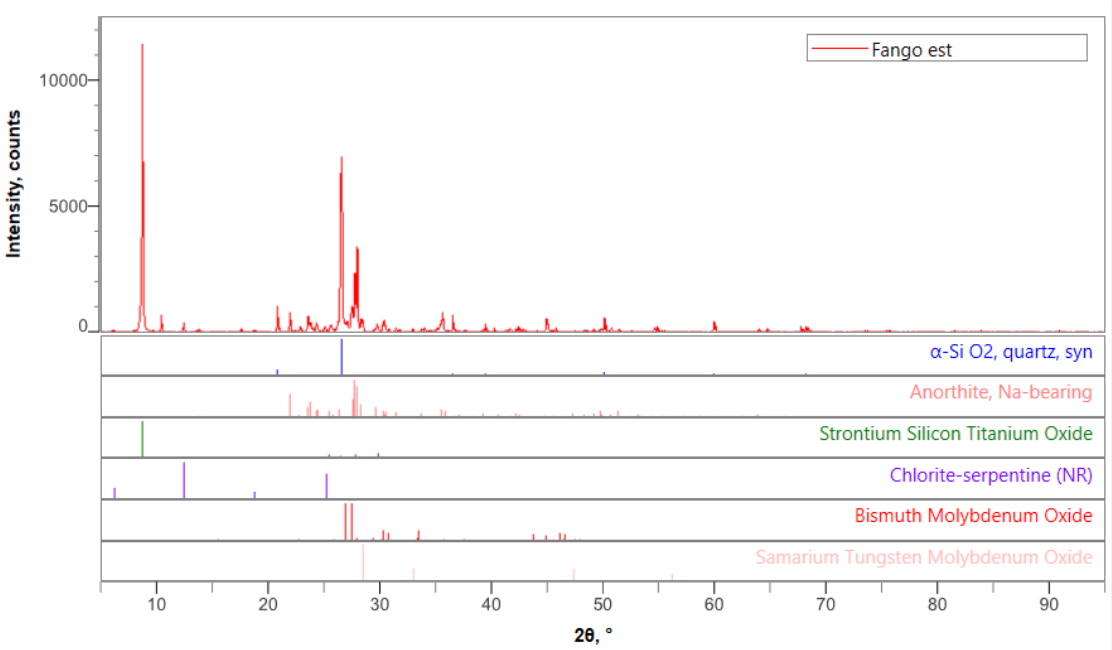

Figure 5.41: Comparison of SE XRD spectrum result and compound matched to it from database

Chapter 6

Discussion of the results

The first observation is that the sludge from the bottom of the silos was not inside the tank because to avoid clogging issues during maintenance, all the material was discharged in the same point of the silos. Actually, it arose an issue since the cold temperature during night and the location of the plant, which is at 345 m on height [34], and the rigid temperatures could affect the material. After a day it has become fluid and workable and for the geotechnical analyses the material might be altered changing, perhaps, the intrinsic structure of the sludge. As we can see, in Table 5.1 the sludge coming from the bottom of the silos is wetter than the other. The reason why it could have lost some water, since the material is almost similar, is linked by the different place the two materials were taken. The sludge under the silos stays without being moved, hence, it might lose water by only evaporation. In contrary, The external slurry was used for enlarging the area of machine manoeuvring. Consequently, the analysis might deal with an unstable and altered material, since it is often crushed and perturbed. As first hypothesis, both materials were considered in the same way. Analyzing the external aspect, the texture, of both materials, is a fine powder, similar to silt/clay, with grayish colour and to the touch is putty, especially SS. Unfortunately, by roughly analyzing SE, there were some shards of other stone that are mixed together. Hence in the next tests, in particular those for mechanical properties, some variations could be figured out. In Figure 5.1, for grains <0.038 mm, the accurate evaluation of the particles should be performed by sedimentation analysis, a point at 0.008 mm has been fixed as particle retention average provided by Prat Dumas paper filters [35] used during the bottom fraction's filtration. Analyzing the distribution of Figure 5.1 the terrain is in between of a well-assorted and uniform soil, because the curve lays, on most of the part, in a single class. SE could be classified as a sandy silt since 70% is int he range of silt class. On the other hand, 90% of SS lays between 0.002-0.063 mm of particle retention, hence it is a clayey silt. In the same graph, SS curve stops at about 10%, which means that one tenth of the mass could be passed through the filter. Probably, during the analysis, some particles

were lost due to saturation of the filter. Since the solution was muddy, especially the last fraction, it happened that the filter was just oversaturated and, despite the vacuum system was working, it was not able to dry. As consequence, there were complication to take off the paper disk with the material and some particles were lost. In the paper analyzed in Section 3.2, a slump test was performed since grain size distribution and clay content heavily influences the achievable plasticity parameters [19]. Although the material was analyzed as a natural soil, it was decided to favor the material for dry uses without studying, therefore, slump test was decided to not be evaluated. The w_L evaluation must be revised because, as mentioned before, the material is not natural. A practical note to highlight during the procedure to prepare the samples on Casagrande's spoon is the criticality to place the material on the instrument. When the material started to be dry, the groove formation began to be more complicated. For this reason, the test stopped at 30 blows or less because the unusual and unnatural structure of the waste has not allowed to go forward. As mentioned, in Figure 5.4 SS and SE lay in different places. However, low levels of w_L represent low plasticity which is an important feature for road subgrade which means, under load, the slurry has small deformation [3]. Regarding the earlier statement, a stress resistance and settlement comparison was evaluated between samples in Subsections 5.1.4 and 5.1.5. Checking $\Delta\epsilon_z$ in all test pieces, it is very similar among unaltered state samples. However, some samples had a steep drop in the first part of the test; maybe, the method used to prepare the sample in the mould was wrong. Only SS (unaltered state and enhanced with 10% cement) stabilized for one hour was prepared with standard UNI EN ISO 17892-5:2017 [36]. All the other samples were prepared by putting the material in the mould and its undisturbed status was maybe lost. Therefore, the trend was approximately correct in all graphs and interesting values were discovered. For instance, adding 10% of cement results in a greater mechanical improvement reaching a modulus E_d=200 MPa in almost every enhanced samples. Without Portland cement, the most interesting material was SS at unaltered state which achieve the peak at 100 MPa. It is a value greater than any modulus reported in Table 3.3 and SS could have a valuable reuse in tunnel muck. Other consideration is the achievement of pre-consolidation which was not equal for all test pieces. SS, especially with cement, had not always reached the peak during the load phase but its modulus stabilized at a certain E_d value. However, a common behaviour among SS and SE, in all their forms, was achieving the module peak during the unloading phase. That is very significant and intriguing, since it means the waste gives its best performance when the load is put off. What happens physically is the ability by both SS and SE to strengthen and keep their consolidation during unloading. Despite it was expected that the material could lose its stiffness and it could bulge to its original shape, unexpectedly it was able to pre-consolidate and gain much higher performance than before. Therefore, a deeper investigation regarding the

quick load/unload with partial water saturation of the samples must be performed because it allows to better understand the actual consolidation of the material. Workability evaluation gave the opportunity to understand the curing and the mechanical resistance associated to the type of waste and the curing time of study. Since the time was insufficient and the number of dies was limited, the maximum curing time achievable was 168 hours. Although the issue, some interesting facts were achieved during the test. At first sight, the mechanical resistance is greater when the curing time is larger. Just comparing SS at an unaltered state after 72 and 120 curing hours, the cracking stress was achieved, respectively, at 60 and 101 kPa. Greater values were achieved by adding the cement to the composition. However, also in this case, the curing time plays a key role in the mechanical strength. Concerning the volumetric weight, it is a great value to compare the different materials' strength. Among the same sample, it was demonstrated that different specific weight values mean different behaviours and compaction levels. Conversely to what expecting, following Figures 5.31 and 5.30 higher value of ρ_V means lower σ_z achieved before cracking. As mentioned in Subsection 5.1.5, those values are related to the possible applicability, since light material might be applied, for instance, for thermal insulation. For construction purposes it might not be compatible because, in that sector, higher volumetric weights are required. Finally, SE seems to have greater mechanical properties, since, at equal curing time, the cracking occurred at higher σ_z applied. Unfortunately, the same comparison cannot be discussed in case of cement addition, since the compaction level in SE +10%, demonstrated proportionally by ρ_V, is much lower than SS +10%. Nevertheless, even the addition of 10% by weight of cement enhances the resistance by a factor of two or three times the unaltered state material. It is important to quote the possibility of pozzolanic potential by the granite slurry that can enhance the reactivity and the durability. Medina [37] studied the reactivity of granite waste at different curing and thermal conditions. Although calcium and its hydroxyl form ($Ca(OH)_2$) are responsible of the pozzolanic activity, granite is poor of those compounds. It was found that lime content is about 22% and to define a material as pozzolan, 25% of lime content is required. However, Medina [37] demonstrated that at very high curing time (on month scale) granite starts to gain some hydration activity, even though it is the same order of magnitude of fly ash. Therefore, Medina [37] has stated that at high temperatures, granite waste induces some pozzolanic activity with the formation of scantly crystalline metakaolinite andothercrystalline phases. For what concern the mineralogical analysis, XRF and XRD give a theoretical composition. Nevertheless, they are correlated since high level of *Si* and *Al* is given by the presence of Quartz, Anorthite-Na bearing and Sanidine (in SS). The other tracked compounds might be associated to the composition of the chosen diamond wire or diamond sew by the plant. The trace of *Chlorite-Serpentine* is related to the natural formation of the diorite which under specific conditions there could be the inclusion of that

mineral [38]. Also the high level of *Fe* means the diamond wire left some traces by friction. As said in Chapter 1, some metals, coming from the cutting stone processes, could be present in the sludge. Metal powder or super-abrasive grains are responsible of contamination in the sludge by heavy metals such as *Fe, Mn* [7]. Regarding the possibility of hazardous consequences related to them, a certified laboratory evaluated the concentration of any harmful compounds that is the legal procedure during waste characterization and management in any process plant. The document, which Tomaino Graniti kindly provided, demonstrated that heavy metals in the slurry are below the legal compliance. Finally, the last observation is the behaviour related to the water influence. Previously, a comparison of the mechanical properties between SS at unaltered state and dried overnight was discussed. A test was conducted to understand what could be the response of the sludge before and after setting in the water. Before hardening, both materials tend to returns on their mud state resulting to creep. However, after the sludge has set, it began to take water by capillarity. Since the grain size distribution demonstrated that SS and SE (at unaltered state) are predominately silt, the force to suck water was high and in a couple of minutes the test piece was already saturated. Lasting for more than the saturation, the water suction didn't stop and the sample lose any capability to stay compacted. Testing the behaviour in the short time is not comparable for diorite slurry. As discussed earlier in this section, it could be useful to perform the same test after some months in order to allow the diorite slurry to start a pozzolanic activity and analyze whether it is able to stay hard and does not return to the mud state. Though, an important insight to highlight is the great ability to harden in just a few hours. This feature, coupled with the low plasticity, could be an opportunity for the application in some embankment works. Although the critical result an interesting insight is given during the preparation of both SS and SE with cement addition. After mixing together the sludge and the cement, the material resulted to be drier than the unaltered state. Even before take off the test piece from dies the water flown outside was lower than the unaltered state samples. Hence, it might be hypothesized that, since Portland cement needs water to harden, the amount contained in the sludge, can be sufficient to interact and start the hardening process. Further researches are required to understand with specific Water-Cement ratio the possible improvement on mechanical strength.

Chapter 7

Conclusions

Despite the artificial production of the material, it has shown many features of a natural soil as demonstrated in the evaluations summarized in Chapter 5. Even though the material is the result of the cutting stone process with diamond tools, the performances obtained went beyond any expected results.

Adjusting the composition of both materials with 10% cement, the mechanical properties vary differently, but further investigations are required by studying the response in different Water-Cement ratios. However, the level of stiffness recorded is appreciable in both materials.

The experiment has demonstrated that diorite slurry cannot stay hard in the presence of water because granite has no pozzolanic activity. However, it is possible to further investigate the possibility of mixing it with limestone at high temperature, in order to add a fraction of calcium which allows to achieve the 25% of lime content [37]. For some construction purposes, as the tunnel muck described in Section 3.2, granite sludge cannot be recommended at unaltered state; however, the addition of cement improves significantly the mechanical properties.

The most interesting feature is the low plasticity demonstrated, which means that it could be applied in sectors where the material must have low deformation. An example of possible application, regarding this feature, is into road subgrade in which there are many problems of using expensive compounds [3]. Given the fast hardening and the last characteristic mentioned, granite slurry fulfills many requirements and many opportunities in civil construction sector.

According to the results obtained by the thesis study mechanical strength, mineralogical compliance and geotechnical surveys have demonstrated the great potential diorite cutting waste might have as byproduct. Although, sometimes the companies does not find attractive the idea of changing the traditional procedure.

To actually use a waste as raw material, many production plants follows two important key points: the market condition about that compound and the adherence to legislative obligations.

As discussed in Section 3.3 granite slurry has the potential to replace a fraction

of the classical fine aggregates in concrete. However, sometimes the production plant does not prefer a slurry since the grains are smaller than sandstone and some issues related to water absorption and hydration process might arise. Furthermore, since the grain size is mostly on the scale of silt, water drainage could be compromised and, from a civil works point of view, it is an aspect to consider and manage. Hence, market demand is difficult to find when, at the moment, the traditional approach is cost-effective.

The other important aspect of employing recovered waste as secondary raw material is the national legislation requiring its use. The concept of circular economy is not already spread worldwide from a legislative point of view. However, in Italy with the *Ministerial Decree June* 28^{th}*, 2024, No 127* highlights the importance and the methods to actually analyse and reuse the waste (in that case the inert waste).

The granite waste can be associated to this category since it is an inorganic compound and it does not react. In the same decree, Table 5 in Annex ii lists some important standards for inert waste regarding: performance conformity and technical suitability in case of recovering. It is an important step forward to recognize some disposable compounds as new secondary raw materials, moreover, it changes the class from waste to byproduct.

Appendix A

Geotechnical calculation

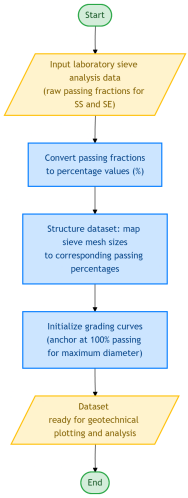

Figure A.1: Python workflow to plot the grain size distribution

Table A.1: Weigh evaluation of grain size distribution

Sample	Mesh size	Tare (g)	Total (g)	Net (g)	Net total (g)	Passing (-)	Passing (%)
Sludge Silos (initial withdrawal 622.4 g)	0.212	6.202	9.9	3.70		0.9941	99.41
	0.106	6.202	13.2	7.00		0.9828	98.28
	0.063	6.202	37.5	31.30	561.43	0.9325	93.25
	0.038	6.202	94.1	87.90		0.7913	79.13
	0.01	-	-	431.54		0.0980	9.80
Sludge External (inital withdrawal 282.6 g)	0.212	6.202	11.3	5.10		0.9820	98.20
	0.106	6.202	34.7	28.50		0.8811	88.11
	0.063	6.202	59.8	53.60	282.82	0.6915	69.15
	0.038	6.202	48.4	42.20		0.5421	54.21
	<0,038	-	-	153.43		0.0008	0.08

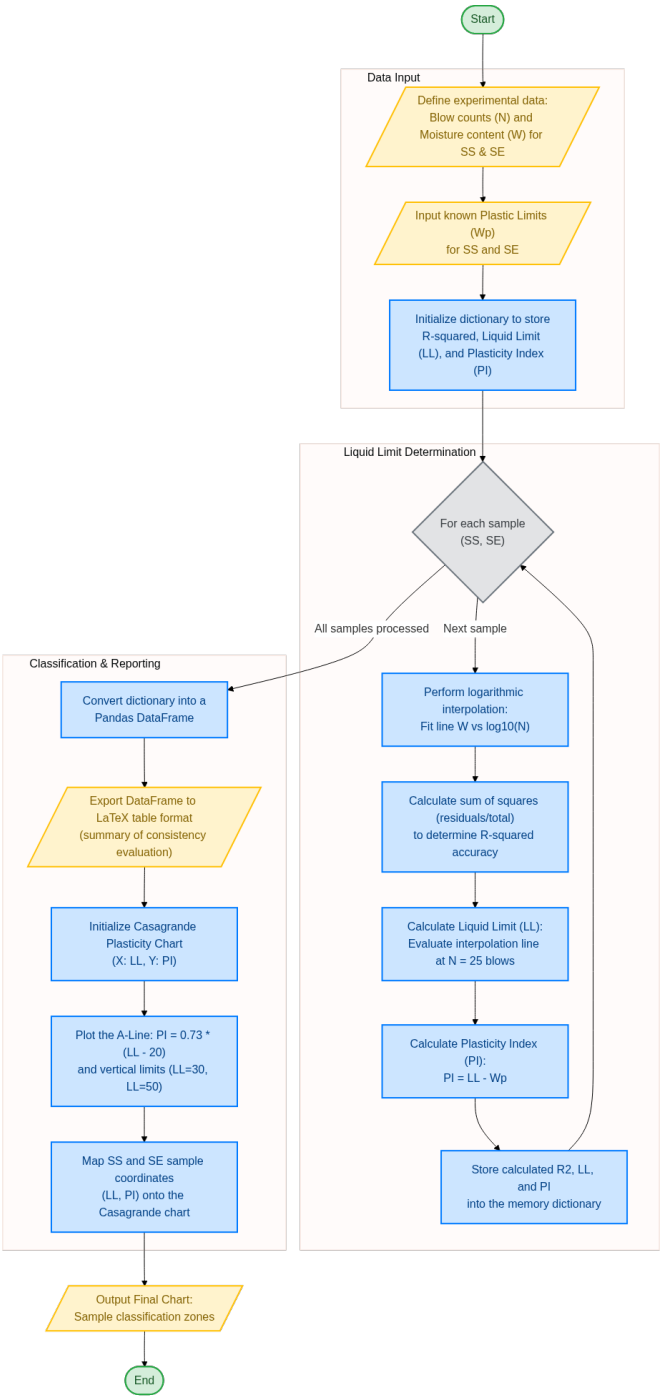

Figure A.2: Python workflow to plot scatter plot of the Liquid Limit Test and the Linear fit

Table A.2: Summary of values from the Laterally Confined In-Grip Load Test

Sample	Minuts (min)	Seconds (s)	Load (kg)	Branch's Distance (mm)
Sludge Silos (unaltered state)	0	0	0	8.69
	2	34	1	8.67
	4	18	3	8.65
	19	52	6.6	8.42
	30	30	8.4	8.22
	49	40	16.4	7.7
	78	0	24.4	7.39
	83	30	1	7.62
	85	0	0	7.69
Sludge Silos (dried overnight)	0	0	0	12.98
	1	59	1	12.85
	3	43	3	12.71
	19	17	6.6	12.41
	29	55	8.4	12.35
	49	5	16.4	12.12
	78	30	24.4	11.9
	83	0	1	12.18
	85	0	0	12.27
Sludge Silos (dried overnight) +10% of cement	0	0	0	9.7
	1	59	1	9.2
	12	43	3	8.64
	18	21	6	7.93
	24	6	14	7.01
	2876	0	22	6.57
	2882	30	1	6.67
	2889	0	0	6.68
Sludge Silos (unaltered state) +10% cement	0	0	0	14.05
	1	59	1	13.89
	12	43	3	13.55
	18	21	6	13.11
	24	6	14	12.8
	2876	0	22	12.57
	2882	30	1	12.67
	2889	0	0	12.69
Sludge External (unaltered state)	0	0	0	10.2
	1	59	0.4	9.84
	5	43	2.4	9.82
	7	25	6.4	9.49
	13	52	14.4	9.17
	2880	0	22.4	8.88
	2886	30	0.4	9.07
	2889	0	0	9.08
Sludge External (unaltered state) +10% cement	0	0	0	13.82
	1	59	0.4	13.5
	5	43	2.4	13.47
	7	25	6.4	13.14
	13	52	14.4	12.81
	2880	0	22.4	12.51
	2886	30	0.4	12.62
	2889	0	0	12.62

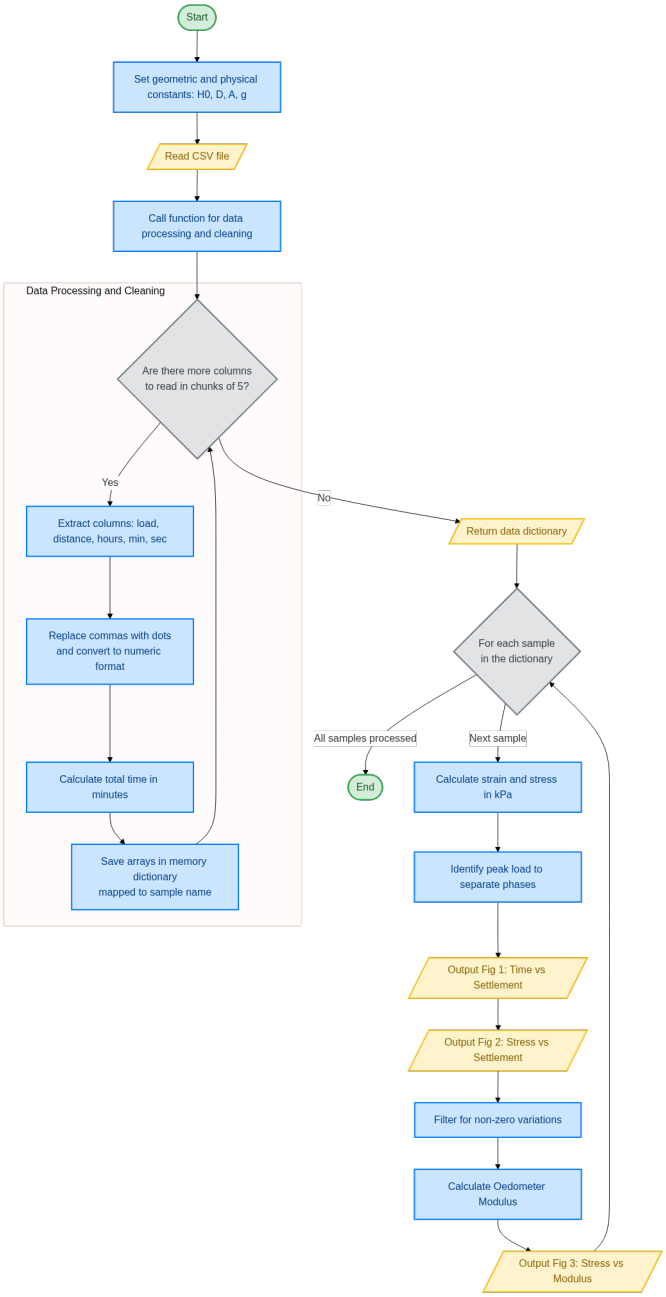

Figure A.3: Python workflow to plot the Laterally Confined In-Grip Load Test charts

Table A.3: Summary of values from the Laterally Confined In-Grip Load Test after curing 1 hour

Sample	Minuts (min)	Seconds (s)	Load (kg)	Branch's Distance (mm)
Slusge Silos (unaltered state) stabilized by 1 hour	0	0	0	9.81
	0	0	0.4	9.68
	0	38	2.4	9.35
	0	24	6.4	8.74
	60	32	14.4	8.22
	60	57	22.4	8.04
	60	19	22.4	7.97
	240	54	22.4	7.92
	240	51	30.4	7.81
	240	0	30.4	7.74
	240	0	30.4	7.72
	300	0	30.4	7.7
	5520	0	30.4	7.59
	5520	0	28.4	7.6
	5520	15	20.4	7.63
	5520	30	12.4	7.67
	5520	0	4.4	7.8
	5520	30	0.4	7.87
	5520	45	0.4	7.89
	5520	0	0	7.91
Sludge Silos (unaltered state) +10% and stabilized by 1 hour	0	0	0	9.93
	0	0	0.4	9.85
	0	38	2.4	9.17
	0	24	6.4	8.94
	60	32	14.4	8.67
	60	50	22.4	8.63
	60	19	22.4	8.61
	240	54	22.4	8.59
	240	18	30.4	8.52
	240	0	30.4	8.5
	240	0	30.4	8.5
	300	0	30.4	8.5
	5520	0	30.4	8.47
	5520	0	28.4	8.48
	5520	15	20.4	8.5
	5520	30	12.4	8.5
	5520	0	4.4	8.52
	5520	30	0.4	8.58
	5520	45	0	8.59

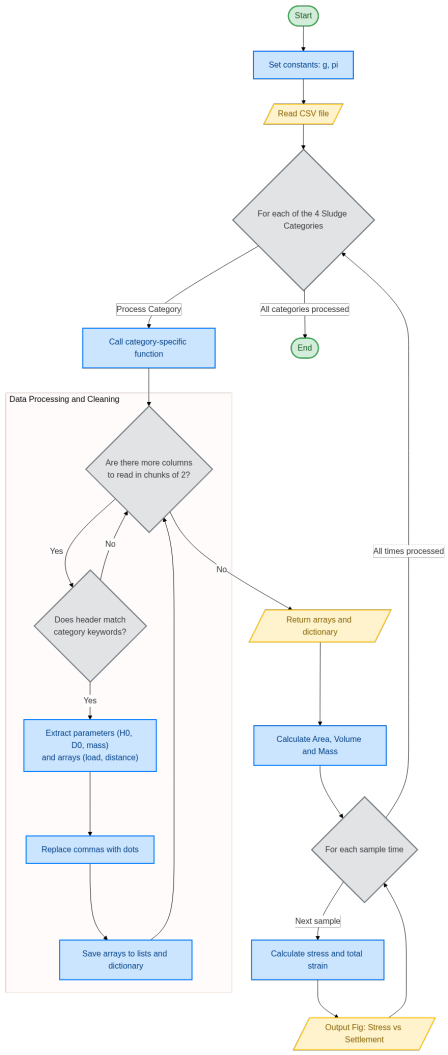

Figure A.4: Python workflow to plot the evolution of Vertical Stress regarding the Vertical deformation to study the workability

Table A.4: Moisture content of SS dried overnight

Sample	Weight pre-drying (g)	Weight post-drying (g)	Water lost (g)	Moisture content (%)
SS	17.75	13.74	4.01	29.2
SE	1494,6	1150,16	344,44	30

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