# ECO-FRIENDLY CEMENT MORTAR WITH WWTP SLUDGE RECOVERY

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## DEDICATION

*I dedicate this work to my mother,*

*Maria Aparecida dos Santos,*

*who always supports and encourages my dreams.*

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**ABSTRACT**

The increase in sludge generation in wastewater treatment plants (WWTP) makes sludge management a difficult task so that it is necessary to create alternatives for the final sludge disposal. The mineralogical characteristics of the sludge allow it to be applied to construction materials. The construction industry is the one that most exploits and consumes natural resources and uses energy intensively, thus generating quite considerable environmental impacts. Creating sludge utilisation techniques in the construction industry provides a safe and stable destination for sludge and contributes to reducing the environmental impacts that the construction industry already has. Thus, the general objective of this project was to evaluate the technical feasibility and economic valuation of the use of WWTP sludge for partial replacement of cement in the production of mortar. For the study, sludge was collected from the WWTP in Bragança, and it was characterised by pH, humidity, total solids, and organic matter content. Different methods (5) were used to prepare the sludge for the mortar. Mortars were produced with 0 (standard, reference mortar), 3, 5, 7 and 10% replacement of cement by treated sludge. With the manufactured mortar, specimens were moulded to test the resistance to flexion and compression. Mortar with 7% sludge ash showed greater resistance than standard mortar, only mortars with wet oxidised sludge showed a resistance rating lower than standard mortar. Thus, the use of dry sludge mortar and ash presented satisfactory results. Comparisons of energy consumption have shown that using sludge in mortars represents significant energy savings.

**KEYWORDS**: Eco-friendly, concrete, mortar, WWTP, sludge, mechanical resistance, energy consumption.

**RESUMO**

O aumento da geração de lodo nas estações de tratamento de águas residuais (ETAR) torna o gerenciamento do lodo em uma tarefa difícil, de modo a ser necessário criar alternativas para a disposição final da lama. As características mineralógicas da lama permitem que ela seja aplicada em materiais de construção. A indústria da construção é a que mais explora e consome recursos naturais e utiliza energia de forma intensiva, gerando assim impactos ambientais bastante consideráveis. Criar técnicas de aproveitamento do lodo na indústria da construção confere um destino seguro e estável para o lodo e contribui na diminuição dos impactos ambientais que a indústria da construção já tem. Assim o objetivo geral desse trabalho foi avaliar a viabilidade técnica e valorização econômica da utilização de lamas de ETAR para substituição parcial de cimento na produção de argamassa. Para o estudo foi recolhido lama da ETAR de Bragança, e caracterizada em relação ao pH, umidade, sólidos totais e teor de matéria orgânica. Foram realizados métodos diferentes (5) para preparar a lama para a argamassa. Foram produzidas argamassas com 0 (argamassa padrão, de referência), 3, 5, 7 e 10% de substituição do cimento pela lama tratada. Com a argamassa fabricada, foram moldados corpos de prova para o teste de resistência à flexão e à compressão. A argamassa com 7% de cinzas de lama mostrou maior resistência que a argamassa padrão, somente as argamassas com lama oxidada via húmida apresentou classificação de resistência inferior a argamassa padrão. Desse modo o uso de lama seca e cinzas em argamassa apresentaram resultados satisfatórios. Comparações de consumo energético mostraram que usar a lama em argamassas representa economia de energia significativa.

**PALAVRAS CHAVE:** Ecológico, betão, argamassa, ETAR, lama, resistência mecânica, consumo energético.

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## INDEX OF ABBREVIATIONS

DS – oven dried sludge
DS7 – sun-dried sludge for 7 days
DS15 – sun-dried sludge for 15 days
SA – sludge ash
SWO – wet oxidised sludge
TS – Total Solids
WTP – Water Treatment Plants
WWTP - Wastewater Treatment Plant

## INDEX

# 1 INTRODUCTION

In 2018 it is estimated that cement production was 3,99 billion tonnes worldwide. The nation that produces the most cement is China, which in 2019 produced 54.5% of the world's cement, while the European Union produced 4.4% [1].

In the production of cement, limestone rock is baked at 1450 ºC with clay, sand and iron ore, this forms the clinker [2]. EN 197-1: 2012 classifies cement according to the composition of clinker in the mixture, Portland cement (CEM I according to the standard) must contain a concentration greater than 95% of clinker [3].

To produce one ton of cement, 60 to 130 kg of fuel oil or its equivalent are required, depending on the type of cement and the process used, and about 101 kWh of electricity [4, 5]. There are impacts generated in practically all the productive phases of cement, such as extraction, production and even final disposal [2]. $CO_2$ emissions from the cement industry represent more than 5% of world emissions and are expected to reach 30% in 2050 [6].

The mixture of cement with water forms the cement paste which when mixed with small aggregates (sand) forms the mortar. Mortar has several attributions in civil construction according to its technical characteristics. Mortar can be applied in masonry laying to elevate walls and walls of bricks or blocks, to coat walls, walls and ceilings for later receipt of finishes such as paint or coatings, among other applications [7]. The technical characteristics for each application are defined by the standard EN 998-1: 2017 [8].

To reduce the environmental impact of mortar and concrete (mixing mortar with coarse aggregates) due to the cement manufacturing process, the incorporation of residues in these civil construction products should be encouraged [2].

In 2013, 83% of the Portuguese population already had access to urban wastewater drainage and 82% to urban wastewater treatment [9]. In 2020, Portugal has 189 wastewater treatment plants (WWTP) across the country [10]. Year after year, water consumption shows a growth trend, and in line with this, the amount of sludge generated at WWTP increases.

One way to manage the volume of sludge produced sustainably is to combine the mineralogical composition of the sludge which is essentially hydroxides and oxides of silica, aluminium and ferric with the production/application in construction materials, in this way, there is the valorisation of the sludge and the transformation into a safe and stable product [11, 12].

To add value and present an alternative for the final disposal of the sludge generated at the Bragança WWTP, mortars were developments from the partial replacement of cement with sludge. Besides, this application reduces the impacts generated by cement production.

# 2 OBJECTIVES

## 2.1 GENERAL OBJECTIVE

Evaluate the technical feasibility and economic valuation of the use of WWTP sludge for partial replacement of cement in the production of mortar.

## 2.2 SPECIFIC OBJECTIVES

- Characterise the sludge collected at the Bragança WWTP.
- Develop methodologies for obtaining sludge with reduced organic matter content.
- Conduct mechanical tests on the manufactured mortar.
- Analyse of technical feasibility and economic valuation on the methodologies developed.

# 3 STATE OF THE ART

## 3.1 WASTEWATER

One of the most important natural resources on the planet is water due to the role it plays in maintaining the balance of ecosystems and biological cycles, thus preserving its quality is essential [13].

Wastewater, popularly called sewage, is the volume of water that has had its natural characteristics altered by domestic, commercial, or industrial use. Water in this state contains contaminating agents that are potentially harmful to human health and nature. Thus, the treatment of water before returning it to the environment is essential for the protection of public health and the preservation of the environment [14, 15, 16].

### 3.1.1 Bragança wastewater treatment plant

The treatment of urban wastewater in Portugal is regulated by Directive 91/271/EEC, subsequently amended by Directive 98/15/EC and Regulation (EC) nº 1882/2003 [17].

In the district of Bragança urban wastewater is treated by the company Águas de Trás-os-Montes and Alto Douro, which is part of the Águas do Norte group. According to the flowchart present at the treatment plant, represented in Annex 1, the raw effluent upon arriving at the plant passes through the inlet (physical treatment), then receives biological treatment and is then able to return to the environment in safe environmental conditions. During the stages, there is the generation of residues, such as coarse solids, sands, fats, and sludge (pre-treatment, primary and secondary). The effluent treated at that station is discharged into the Fervença river.

In wastewater treatment, we have 3 main lines: liquid line, solid line (sludge line) and deodorisation line (air deodorisation line) [15].

#### 3.1.1.1 Liquid line

In the liquid line, preliminary, primary, secondary (or biological) and tertiary treatment occurs [14, 15, 16].

In the preliminary treatment, the harrow removes coarse solids and the grit/degreaser removes sand and fats. This treatment avoids the formation of foams and clogging in the following processes. The removed solids are sent to incineration or landfill [14, 15, 16].

The primary treatment carried out in the primary sedimentation tank removes sediment components from the suspended solids and part of the organic matter. These sediments form the primary sludge. Despite being a physical process, coagulants/flocculants can be added to obtain pollutant flakes of larger dimensions, thus more easily settling. The sludge from this stage has a high content of organic matter [15].

In secondary (or biological) treatment, the effluent undergoes two processes: anoxia tank and aeration tank. In the anoxia tank, nitrogen is removed biologically to prevent eutrophication of the receiving river by increasing this nutrient [15, 16]. In aeration, oxygen is available for the biological process (aerobic microorganisms) to occur and for there to be no deposition of organic matter at the bottom of the tank [14, 15]. In these processes, conditions specific to bacterial activity are established so that organic matter and nutrients present are consumed [15].

After that the effluent goes to the secondary decanter that clarifies the effluent by removing the mineralised and flocculated biomass in the previous tank [16]. The tertiary treatment corresponds to the disinfection, that can be carried out by adding chlorine, ozone or by ultraviolet radiation [15].

#### 3.1.1.2 Solid line

The solid line consists of a by-product called sludge, which contains a high content of organic matter and water, and its composition depends mainly on the origin of the solids, the quantities generated and the type of process that the sludge was subjected to previously [15].

The volume of sludge produced is reduced by decreasing the water content in the thickeners. There is the concentration of the total solids in the sludge and the water that comes out returns to the process in the liquid line [14, 15].

The next process is the digestion of the sludge in the digesters. In this stage, the sludge under anaerobic conditions goes through three phases [14].

In the first phase, hydrophilic and fermentative bacteria break down organic matter into smaller molecules. Hydrophilic bacteria break down proteins, amino acids, polysaccharides and fats while fermentative bacteria break down the products generated by hydrophilic into carbon dioxide, gaseous hydrogen and acetic acid [14].

In the second stage, acetogenic bacteria do the same process as fermentative bacteria. In the third phase, methanogenic bacteria transform carbon dioxide, hydrogen gas and acetic acid into methane - biogas. The bacteria present in the digester act symbiotically so that there is no excessive accumulation of toxic substances [14].

#### 3.1.1.3 Deodorisation line

Deodorising the line represents the quality of life for those who live in the vicinity of a WWTP and comfort for close tourists. There are several methods for reducing the odours formed, which can be of three types: physical, chemical, or biological. The most used methods are using activated carbon or chemical scrubbers (most commonly chlorine solution such as sodium hypochlorite and sodium permanganate) [15].

#### 3.1.1.4 WWTP sludge

The sludge resulting from the digester is biomass and is a by-product of the WWTP, being classified as waste with the LER code 19 08 05 - Sludges from treatment of urban wastewater. They are generally not sanitised and are collected, transported and sent for temporary storage, or direct recovery by composting, before application to agricultural soils or they can still be deposited in a landfill [18].

According to the Águas de Portugal Group in 2018, around 39 thousand tonnes of sludge was produced in Portuguese territory, the trend of increasing annual sludge production can be seen in Figure 1 [18].

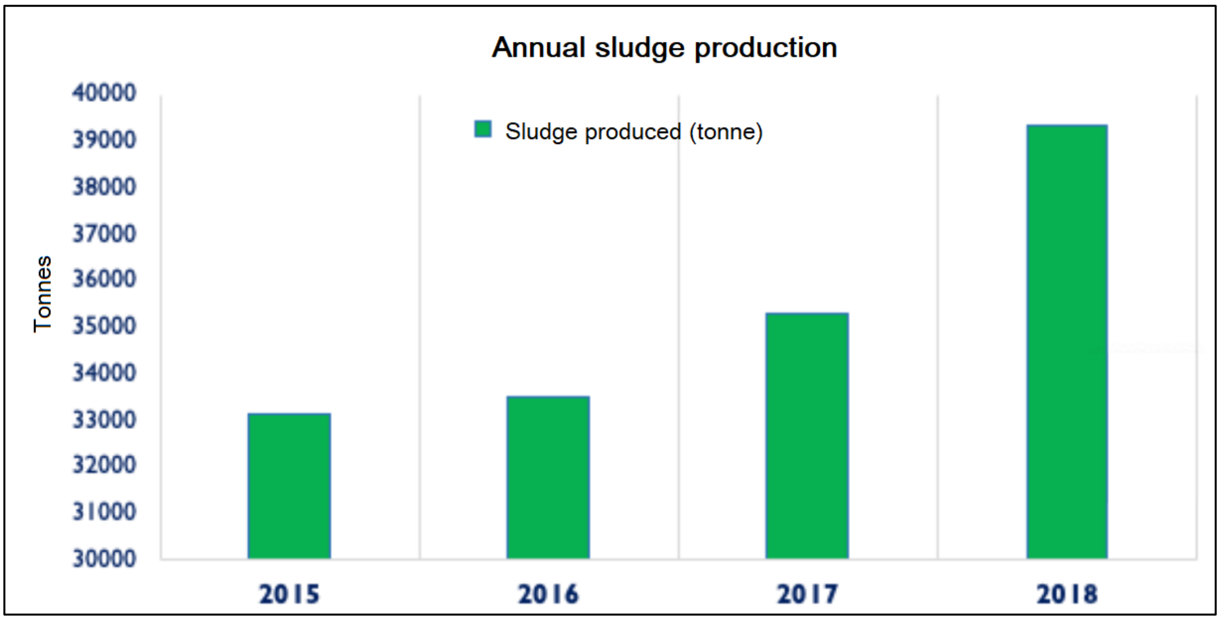


**Figure 1: Annual sludge production from WWTP [18]**

With the significant increase in the volume of sludge generated in the WWTP, the management of sewage sludge that relates to sustainable development is essential [12].

The mineral composition of the sludge is essentially hydroxides and oxides of silica, aluminium and ferric [11]. This characteristic of the sludge allows its application in construction materials, such as in the manufacture of bricks, tiles, ceramics, cement, light aggregates and in the partial replacement of cement or sand in concrete pastes and the preparation of mortars [15, 12, 11, 19].

The valorisation of the sludge for transforming it into safe and stable products mitigates some of the expensive and energy-consuming stages for something that will be discarded [12].

## 3.2 CIVIL CONSTRUCTION

According to the International Council for Research and Innovation in Building and Construction – CIB, the construction industry is the one that most exploits and consumes natural resources and uses energy intensively, thus generating quite considerable environmental impacts [20, 21].

According to the Federação Portuguesa da Indústria da Construção e Obras Públicas – FEPICOP for 2020, the sector is expected to grow by 5.5% for the year as a whole [22].

Cement is the most widely used building material today and concrete is the most consumed material worldwide [23, 24]. In 2018, the cement industry sold more than 3.963 million tons of cement, equivalent to around 276 million euros, in the same year of ready-mixed concrete 9.999 million tons, about 249 million euros [25].

### 3.2.1 Cement

Portland cement is a fine powder with agglomerating properties, which hardens under the action of water and after that remains stable even in the presence of water, for this reason, it is considered a hydraulic binder. To obtain Portland cement, limestone, clay, sand and iron ore extraction are inevitable [2].

The production of Portland cement has a percentage of 90% limestone and the production process begins with the extraction of the rock. After being extracted, the rock goes through the crushing process to improve the drying efficiency that happens next. In drying, the limestone remains with a residual moisture of 2%, so it is crushed to a fine powder. In the next step, there is the addition of clay, sand and iron ore, the clay added will represent a percentage of 10% by weight of the cement mixture, the sand and iron ore serve to correct the content of silica and iron oxide, respectively [2, 26, 27].

After obtaining a homogeneous mixture of a fine powder, the volume passes to the clinker process. The mixture is introduced in rotary kilns at 1450 ºC, to form the clinker, tricalcium silicate, dicalcium silicate, tricalcium aluminate and tetracalcium aluminoferrate. After this process the cement is ready, however, additives can be added that will give it certain properties for more specific applications, such as the addition of plaster that controls the setting, slags that increase durability and resistance and pozzolanic materials that provide greater impermeability to the concrete [2, 26, 27].

Cement manufacturing is a process that involves high economic and environmental costs. Of the total amount of $CO_2$ released into the atmosphere during cement production, 30% corresponds to the burning of fuels for clinker and 70% to the decarburization of limestone [23, 28]. There are impacts generated in practically all production stages of cement, such as extraction, production and even final disposal [2]. $CO_2$ emissions from the cement industry represent more than 5% of world emissions and are expected to reach 30% in 2050 [6].

To reduce the extraction of raw materials, the incorporation of residues from other industrial processes contributes to the reduction of the environmental impacts generated in the production of cement [2].

### 3.2.2 Mortar

The mixture of cement and water results in cement paste and the mixture of cement paste with sand forms the mortar, so the mortar is the result of the homogeneous mixture of a binder with fine aggregates and water. Mixing the mortar with coarse aggregates (gravel, stone) forms the concrete [7].

The bonding agent can be cement, plaster, or lime, and this is called mortar. When there are two bonding agents, such as cement and lime or plaster and lime, the mortar is said to be mixed and if there is only one binder it is said to be simple. To have specific characteristics or properties, additives or adjuvants can be added so that the mortar reaches these specifications [7].

Mortar can be classified about the nature of the binder (binding agent), the consistency, plasticity and density of the mortar and the form of preparation or supply, as described in Table 1.

Mortars can also be classified according to their functions, such as for masonry construction, for wall and ceiling cladding, floor cladding, for ceramic cladding or the recovery of structures, as shown in Table 2.

The standard EN 998: 1-2017 classifies mortars and defines the requirements for evaluating the quality of mortars [8]. The EN 13139: 2005 specifies the properties of the aggregates involved in the application of different types of mortar and the standard EN 197-1: 2012 presents the technical requirements and compliance criteria for each type of cement sold [3, 29].

**Table 1: Mortar classification [7]**

| Classification criteria | Type |
|---|---|
| The nature of the binder | Aerial mortar<br>Hydraulic mortar |
| Type of binder | Lime mortar<br>Cement mortar<br>Cement and lime mortar<br>Plaster mortar<br>Lime and plaster mortar |
| Number of binders | Simple mortar<br>Mixed mortar |
| Mortar consistency | Dry mortar<br>Plastic mortar<br>Fluid mortar |
| As for the plasticity of the mortar (% of fine aggregates in the mortar) | Poor or thin mortar (< 15)<br>Medium or full mortar (between 15 and 25)<br>Rich or fatty mortar (> 25) |
| Mortar mass density ($\rho$ = g.cm$^{-3}$) | Light mortar ($\rho$ < 1,4)<br>Normal mortar (2,30 ≤ $\rho$ ≤ 1,4)<br>Heavy mortar ($\rho$ > 2,3) |
| As to the way of prepared or supply | Mortar prepared on site<br>Semi-ready mix for mortar<br>Industrialised mortar<br>Mortar dosed in central |

**Table 2: Classification of mortars according to function [7]**

| Function | Type |
|---|---|
| For masonry construction | Laying mortar (masonry elevation)<br>Fixing mortar - sealing masonry |
| For wall and ceiling cladding | Mortar of roughcast<br>Plastering mortar<br>Plastering mortar<br>Single-layer mortar<br>Monolayer decorative coating mortar |
| For floor covering | Subfloor mortar<br>High-resistance mortar for flooring |
| For ceramic tiles (walls / floors) | Mortar for laying ceramic tiles - adhesive<br>Grouting mortar |
| For recovery of structures | Repair mortar |

### 3.2.3 Application of waste in construction materials

The incorporation of waste in civil construction describes an alternative to reduce the volume of waste deposited in landfills and reduce the volume of mined materials, minimising the environmental impact that the construction industry has on the environment [6].

There are potential end uses of by-products such as building fillings, concrete aggregates, and pavements. In the environmental scenario, the replacement or complementation of a binder in a concrete product is one of the most interesting applications, such as the replacement of cement in concrete [6].

Blast furnace slag is a by-product of the steelmaking industry. In the cooling process, the slag creates a glassy product that, after being crushed, can be incorporated into the cement mixture, forming the slag cement. The incorporation of the slag increases the strength of the mixture [30].

The application of slag cement in airport runways has shown that a smaller thickness is necessary to obtain the same effect as a concrete paste with Portland cement. This is reflected in the decrease in the emission of greenhouse gases associated with the production of airport runways [30].

The particles that come out of the flue gas during the combustion of coal are called fly ash. Air pollution control devices that capture these particles [6].

In 2012, the United States produced 52.1 million tons of fly ash, of which 11.8 million tons were used to produce concrete/concrete products/grout [6]. The replacement of 10% to 20% of cement by fly ash in the concrete paste results in an increase in the resistance to compression and corrosion of the concrete produced [31].

Due to the similarities in chemical composition between water station sludge and clay, there is an incentive to replace the clay with sludge in the manufacture of bricks and ceramic materials (tiles and blocks). These materials promote the solidification and immobilisation of the potentially toxic elements present in the sludge. However, bricks manufactured with the addition of sludge have less resistance to compression and greater water absorption [6].

The incorporation of sludge in construction products affects the characteristics of the products manufactured due to the presence of organic substances and heavy metals. When partially replacing the fine aggregate in the concrete slurry, the sludge can cause deterioration of the concrete properties [12].

In ceramic materials, the addition of dry sludge gives porosity and uneven surface, which affects the resistance to water absorption and resistance to ice [12].

Pozzolanic additives can be added to cement to improve strength. The pozzolanic activity consists of binding lime in the presence of water, which results in the formation of water-insoluble calcium silicates. Thus, although the addition of sludge negatively influences the fluidity of the mixture, by absorbing water, and extends the hardening time, it can contribute to increasing the compressive strength of the mass [12].

Aligning sustainable construction practices with waste management makes the implementation of WWTP sludge in construction products an alternative to minimise the impacts generated by cement industry and at the same time provides a safe and stable sludge destination [6, 12].

## 3.3 DEGRADATION OF ORGANIC MATTER

It is necessary to eliminate volatile compounds and degrade the organic matter in the sludge before they are incorporated into construction materials. This is because organic matter can degrade or decompose causing changes in the manufactured product, thus reducing its durability. The mineralogical alteration of the sludge is desirable since the rearrangement of the material can increase the pozzolanic activity of the manufactured product [23].

Thermal processes have the advantage of reducing volume and weight, destroying toxic organic compounds, including pathogens, minimising odours, and recovering energy through steam turbines. Thus, the main objective of the thermal processing of the WWTP sludge is to use its energy content, with the calorific value of the WWTP sludge being between 12,000 to 20,000 $kJ.kg^{-1}$ while coal has a calorific value between 14,600 to 26,700 $kJ.kg^{-1}$ [32, 33].

Rabie et al. (2019) studied the influence of the incorporation of wet sludge and dry sludge on the physical and mechanical properties of concrete mixtures. The scientists tested mixtures with concentrations of 5, 10 and 15% of sludge on the weight of cement. The sludge was dried at 200 °C for 2 hours [34].

The authors observed that the addition of 5% of dry sludge decreased 1% of the compressive strength of the concrete while with wet sludge the decrease was 7% after 28 days of curing. In the other concentrations, they obtained similar behaviour, the concrete with dry sludge obtained greater resistance to compression than the concrete with wet sludge. With 10 and 15% dry sludge the loss of resistance was 8 and 22% respectively, so the sample with 5% dry sludge was the one that came closest to the standard [34].

Costa (2011) simulated a sludge pond for drying the sludge with the sun and after 15 days of exposure to the sludge, it had a humidity of 60%. The author applied the sludge in a concrete mixture with traces of 1:2:3 of cement, sand, and gravel respectively. The dry sludge was added in partial replacement of sand. Compressive strength tests after 28 days of curing showed that all specimens with added sludge resulted in less than standard strength (without added sludge). Higher sludge concentration, the lower the compressive strength. The concentration of 5% was the closest to the standard, 29.03 and 33.43 fck respectively [35].

### 3.3.1 Incineration

Incineration involves the complete oxidation of the volatile matter and the production of an inert residue - ash. Industrially, incineration can be carried out in fluidised bed reactors that have more attractive operating costs and capital than the conventional multiple fireplace type [32, 33]. In the European Union, about 22% of the sewage sludge is incinerated [36].

After incineration, about 30% of the weight of solids remains ashes, occupying a volume of only 10% compared to the initial volume. The resulting ash has a high content of heavy metals, so the correct final disposal can become a problem. The incorporation of ash in construction materials is an alternative that results in stable and safe products [32, 33].

The burning of sludge as an alternative fuel and the incorporation of ash in the final product appears to be a more promising alternative compared to independent incinerators [32].

The burning of the sludge in the study by Sampaio (2017) was carried out in a muffle furnace, first at 800 °C for 1 hour and then at 1000 °C for 4 hours. Specimens were produced with ash concentrations of 0, 5, 10 and 20% to replace cement. The sample with 5% showed better results than the standard, 38.3 against 37 MPa in the compressive strength test on the 28th day of cure. The other concentrations showed lower values than the standard, 27.2 and 23.7MPa for ash concentrations of 10 and 20% respectively [23].

Nakic (2018) burned dry sludge (with humidity less than 10%) at 900 °C for 2.5 hours using an electric laboratory muffle. Specimens were produced by replacing 10% of the cement with ash. After 28 days of curing, the ash sample showed a slightly higher compressive strength than the reference sample [28].

### 3.3.2 Wet oxidation

Wet oxidation is a hydrothermal treatment (thermal hydrolysis) where there is rapid solubilisation of organic residues and liquid oxidation of dissolved organics or oxidizable inorganic compounds. Typical operating conditions for wet oxidation are temperatures from 150 to 330 °C and pressures from 1 to 22MPa using pure or atmospheric oxygen for 15 to 120 minutes [32, 37, 38].

In wet oxidation, there is the production of low impact exhaust gases, mineralised solid fraction and high resistance liquid fraction [38].

Khan et al. (1999) tested the influence of temperature and retention time on the wet oxidation of WWTP sludge. The tests were performed in an autoclave reactor, with oxygen injection. They observed that at 300 °C and with a residence time of 30 minutes, 75% of the organic matter is oxidized and at this same temperature, approximately 90% of the total volatile solids were destroyed [39].

Malhotra and Garg (2019), evaluated the performance of wet oxidation in the degradation of sewage sludge. The study was carried out in a stirred high-pressure batch reactor made of stainless steel. Upon reaching the desired temperature the content was stirred at 1000 rpm. The batch time (from 2 to 5 hours) and the system temperature (from 140 to 180 °C) were varied. The authors observed that the maximum solubilisation of total organic carbon was at 160 °C in the presence of oxygen, with a pH of 6.3 and a residence time of 3.5 hours [40].

# 4 METHODOLOGY

This study was carried out at the Chemical Process (D-002) and Geotechnics Laboratories, at the Instituto Politécnico de Bragança, from September 2019 to July 2020.

The sludge used was collected on October 15, 2019, at the Wastewater Treatment Plant in Bragança, of the company Águas de Trás-os-Montes and Alto Douro, part of the Águas do Norte Group.

## 4.1 SLUDGE CHARACTERISATION

The incorporation of sludge in mortar can affect the durability of the manufactured product due to the presence of organic substances and heavy metals, so the sludge characterisation under analysis is essential [12]. Sludge characterisation includes pH, humidity, total solids, carbon (%) and organic matter (%).

### 4.1.1 pH

The pH determination was based on Embrapa's (1997) soil pH analysis methodology [41].

To determine the pH value, 100 mL of sludge was dissolved in 100 mL of distilled water under magnetic stirring for 15 minutes. After resting for 10 minutes the pH of the supernatant liquid near the phase change interface, was read with a calibrated pH meter [41].

### 4.1.2 Moisture content

The determination of humidity, that is, the percentage of water contained in the sludge mixture, was carried out by drying 3 g of sludge in a dry porcelain crucible, in an oven at 105 °C for 24 hours. The mass was gauged the procedure repeated at intervals of 1 hour time until constant weight of dry sludge sample (4% or 50 mg change, the first to occur) [23, 42]. The test was performed in quintuplicate and the humidity calculation according equation 1:

$$M = \frac{A - B}{100} \qquad \text{Eq. 1}$$

Where:
M moisture content, %
A weight of wet material plus plate, mg
B weight of dry material plus plate, mg

### 4.1.3 Total solids content

The determination of total solids (TS) was carried out simultaneously with the determination of humidity, only the mathematical equation used is different (equation 2) 2540 G [42]:

$$TS = \frac{(C - D) \times 100}{E - \mathrm{D}} \qquad \text{Eq. 2}$$

Where:
TS Total solids, %
C weight of dry material plus plate, mg
D dish weight, mg
E weight of the sample at the beginning plus the dish, mg

### 4.1.4 Organic matter content

To determine the organic matter content, the same method used by the research by Sampaio (2017) was applied [23].

Approximately 250 mg of dried and ground sludge was transferred to a 500 mL erlenmeyer and mixed with 10 mL of 1N potassium dichromate solution and with 20 mL of concentrated sulfuric acid. The mixture was stirred for one minute at light rotations and then left for 30 minutes. After this period, 200 mL of distilled water, 10 mL of concentrated orthophosphoric acid and 1% ferroin indicator were added, which was then titrated with 0.5N ammoniacal ferrous sulphate solution until green colour was reached. The blank was performed, that is, the same procedure described above, but without the addition of the sludge sample [23].

The calculation of the organic matter content is given by the equation 3:

$$OM = 1.725 \times \frac{\left(10 - \left(V_2 \times 10 \times {V_1}^{-1}\right)\right) \times 0.4}{m} \qquad \text{Eq. 3}$$

Where:

OM Organic matter, %

$V_1$ volume of ammoniacal ferrous sulphate spent on white titration, mL

$V_2$ volume of ferrous ammonia sulphate spent on the titration of the sludge sample, mL

m mass of sludge sample analysed, g

## 4.2 SLUDGE PREPARATION

To make the specimens with the replacement of cement by sludge, the sludge was processed. Different methodologies have been proposed to study the influence that sludge processing has on the mechanical strength that the specimen acquires.

### 4.2.1 Sludge oven drying

The making of specimens with only dry sludge, serves as a standard for the other analyses since the sludge had only water removal, without other physical or chemical changes.

The material was dried in a porcelain crucible and spread to form a thin layer increasing the contact surface with the heat of the oven which contributes to the efficiency of the drying process.

The temperature and drying time were determined based on the work of Nakic (2018), Sampaio (2017), Silva (2016) and Garcia (2011). The sludge was dried in a

Scientific Series 9000 Laboratory Oven, model 972, at 105 °C for 24 hours [23, 28, 43, 44].

### 4.2.2 Sludge sun drying

A sludge sample was left in the sun to dry naturally, as in Costa's (2011) work, however for a period of 7 and 15 days [35]. To increase drying efficiency, a thin layer of sludge was used in an aluminium container, this system was exposed to the sun every day of the experiment period and was covered at night to avoid dew or rain. The residual unit was determined after the drying period in the sun (105 °C for 24 hours).

### 4.2.3 Incineration

The dried sludge (105 °C for 24 hours) was burned for 0.5 hour at 300 °C and for 3 hours at 900 °C in a muffle furnace. The residence times and temperatures were defined based on the works of Sampaio (2017), Garcia (2015), Nakic (2018), Hagemann et al. (2019) and Melo et al. (2008) [23, 28, 43, 45, 46].

### 4.2.4 Wet oxidation

As presented in topic 3.3.2 of this work, typical conditions for wet oxidation operation are temperatures between 150 and 330 °C and pressures from 1 to 22 MPa using pure or atmospheric oxygen, with residence time ranging from 15 to 120 minutes [32, 37, 38]. However, in the technical impossibility of testing under such conditions, the sludge was subjected for 18 minutes to a pressure of 2.2 Bar (0.22 MPa) and a temperature of 121 °C. The scheme was elaborated in the laboratory autoclave, of the Uniclave 88 brand, manual model. After the autoclaving of the sludge, it was oven dried for 24 hours at 105 °C.

## 4.3 MORTAR SPECIMENS

The mould used to prepare the specimens was prismatic 4x4x16 cm. Each piece weighs approximately 600 g and two specimens were made per concentration of sludge used. One sample for the tests on the 7th day of mortar curing and the other piece for the 28th day. The pieces were produced with tap water and sand with a grain size of 0.4 mm. The sand was dried at 105 °C for 24 hours before being used in the mortar.

All parts produced have the same concentration of water and sand, 14% and 54% respectively. The standard sample then has 32% cement, but in the remaining parts of cement mass it was replaced by treated sludge at concentrations of 3, 5, 7 and 10% of the cement by sludge.

The production of the specimens followed the NBR 5738 of Associação Brasileira de Normas Técnicas – ABNT, with the application of mortar in the moulds in the horizontal compactor with 70 strokes per layer. After the production of the pieces, they were under the bench of the Geotechnical Laboratory for 48 hours, then they were stored in the humid chamber under 20-25 °C with 90% humidity.

Figure 2 shows the mixer apparatus used to the mixture of pastes, the horizontal compactor, and the prismatic moulds.

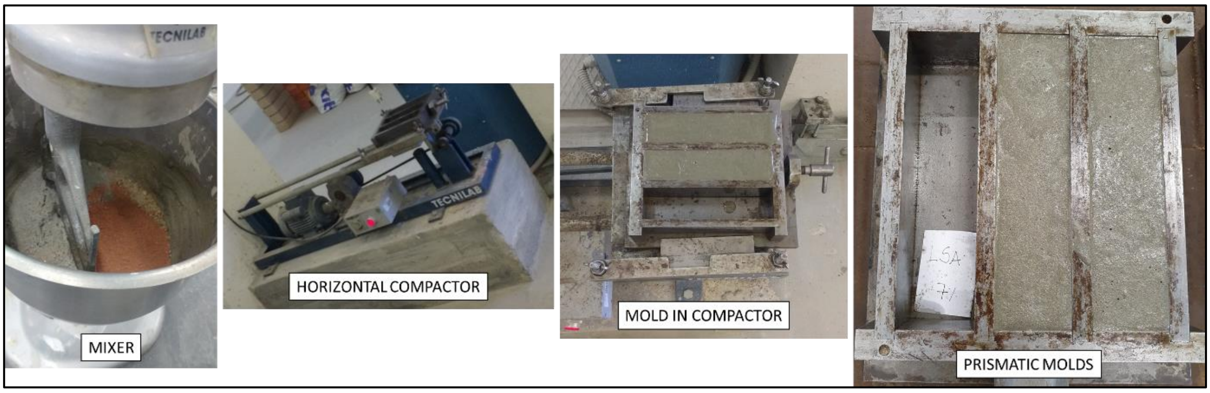


**Figure 2: Preparation of the specimens**

## 4.4 MECHANICAL TESTS

Two mechanical tests were carried out onto the specimens, flexural strength and compressive strength tests. The tests were done on the 7th and 28th day of mortar curing, in the Geotechnical Laboratory in Tecnilab equipment.

In the flexion test, a force is applied in the horizontal centre of the specimen until its rupture, as shown in Figure 3. Figure 4 shows the equipment used.

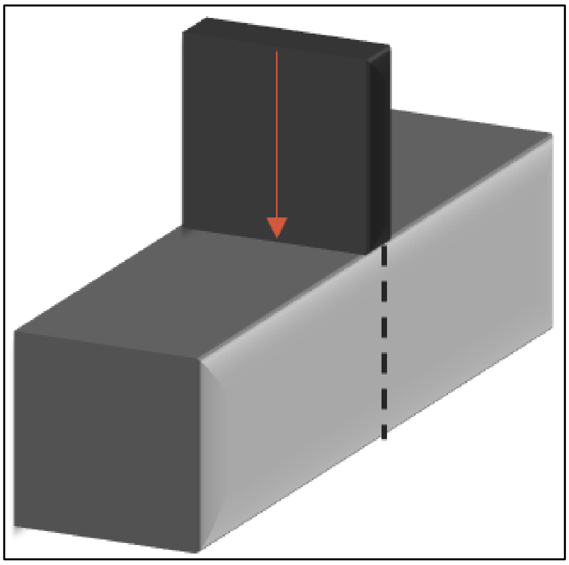

**Figure 3: Scheme of the application of force in the flexion test**


**Figure 4: Equipment for testing flexural strength**

In the compression test a force is applied 4 cm from the end of the specimens until the peak of the applied force is formed, as shown in Figure 5. The compressive strength test measures the maximum amount of compressive load that the specimens support after a healing period (7 to 28 days) before fracture. Figure 6 shows the equipment used in the compressive strength test.

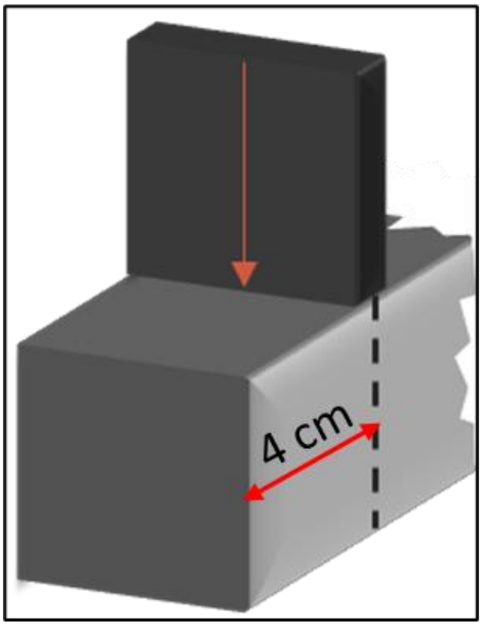


**Figure 5: Scheme of the application of force in the compression test**

**Figure 6: Compressive strength testing equipment**

As two points of mechanical resistance to axial compression are obtained, statistical analysis of the data was performed. Data were subjected to one-way analysis of variance (ANOVA simple) and the data were also subjected to Tukey's multiple range test to compare the averages. Statistical analyses were performed using STATGRAPHICS Centurion software.

According to the EN 998-1 standard, the mortar is classified by the compression resistance that the specimen supports on the 28th day of curing, according to Table 3 [8].

**Table 3: Requirements for compressive strength applied to mortars [8].**

| Categories | Values |
|---|---|
| CS I | 0.4 a 2.5 MPa |
| CS II | 1.5 a 5.0 MPa |
| CS III | 3.5 a 7.5 MPa |
| CS IV | ≥ 6 MPa |

## 4.5 ECONOMIC VIABILITY

The economic viability was evaluated according to the energy costs of each methodology in the preparation of the sludge, and how much the replacement of cement by sludge represents energy savings/expenditure in the energy balance of the mortar.

Equation 4 shows the calculation of savings or replacement costs.

$$F = G - (G \times h) + (I \times j) \qquad \text{Eq. 4}$$

Where:

F Economy or cost, $kWh.t^{-1}$ cement

G Energy expenditure of tonne cement production, $kWh.t^{-1}$ cement

h Cement concentration, fraction

I Energy expenditure in the treatment of tonnes of particles, $kWh.t^{-1}$ particles

j concentration of sludge particles

Energy costs for sludge treatments, as well as for cement production, were obtained through studies by other authors. Table 4 shows these values.

**Table 4: Energy demand for preparation of sludge**

| Process | Electricity demand |
|---|---|
| Cement production | 102 $kWh.t^{-1}$ cement [5] |
| Drum or fluidised bed dryers | 0.07 $kWh.kg^{-1}{}_{H2O}$ [47] |
| Drying operation | 39 $kWh.t^{-1}$ dry sludge [48] |
| Dry sludge and incineration | 275 $kWh.t^{-1}$ dry sludge<br>- 1024.5 $kWh.t^{-1}$ dry sludge (recovery) [48] |
| Co-incineration | - 250 $kWh.t^{-1}$ of dry sludge (recovery) [49] |

# 5 RESULTS AND DISCUSSION

The sludge was characterised by pH, moisture content, total solids, and organic matter.

Samples containing sludge ashes (SA), oven dried sludge (DS), wet oxidised sludge (SWO), sun-dried sludge for 7 days (DS7) and sun-dried sludge for 15 days (DS15) were prepared and tested.

## 5.1 SLUDGE CHARACTERISATION

The pH test showed that the sludge has a pH of 7.8. Table 5 shows the data obtained in the experimental tests, the values indicated with * were disregarded from the calculation of the average and the average deviation. The analysis of the data from the sludge characterisation tests showed that the moisture content is 83.2% with an average deviation of 0.2, the TS content is 16.8% with an average deviation of 0.2 and the organic matter content is 20.7% with an a average deviation of 4.4.

**Table 5: Sludge characterisation (humidity, total solids, and organic matter)**

| nº | Humidity (%) | Total solids (%) | Organic matter (%) |
|---|---|---|---|
| 1 | 83.4 | 16.6 | 25.3 |
| 2 | 97.7* | 2.3* | 27.1 |
| 3 | 83.4 | 16.6 | 19.3 |
| 4 | 83.1 | 16.9 | 12.8 |
| 5 | 82.9 | 17.1 | 19.1 |
| Average ± deviation | 83.2 ± 0.2 | 16.8 ± 0.2 | 20.7 ± 4.4 |

*Disregarded data for the average and average deviation.

According to Świerczek et al. (2018), the organic matter content found is low and indicates that the processed sludge has been stabilised [12]. This is consistent with the neutral pH value found as well as with the flowchart of the WWTP in Bragança, as the sludge understudy was collected after the filter presses, at the end of the entire process. The moisture content of the sludge is similar to the values normally found in the literature, such as in the work of Sampaio (2017) of 74.9% and the work of Ferreira

(2010) of 82.8%, 74.5% and 72.3 % for sludge from Lever, Ferro and Ferreira Water Treatment Plants (WTP) respectively [23, 50].

A pH value between 7 and 8, indicates that it has not been chemically stabilised with liming products such as calcium carbonate, for example, as in these cases the pH would rise to 12 [51].

The TS is in agreement with the values found by Gonçalves (2017) when studying the variation of some parameters of some WWTP in a period of 3 years, which was 19.7% in 2014, 15.9% in 2015 and 26.1% in 2016, all the values determined in January [52]. The author also says that the TS and humidity are related to the efficiency of the dehydration process to which the sludge was submitted [52]. In this way, it can be said that the low value of organic matter is related to the good efficiency of the digesters.

## 5.2 SLUDGE PREPARATION

### 5.2.1 Sludge oven drying

Figure 7 shows the appearance of a sludge sample after drying in the oven. From the DS preparation methodology, it is assumed that all water has been removed, so the DS humidity is 0%.

**Figure 7: Oven dried sludge sample**

### 5.2.2 Sludge sun drying

Figure 8 shows the system formed for drying the sludge. The sludge on the first day suffered a great reduction in volume due to water loss. The sample from the 15th day of drying contains a higher mass content than in the previous passages because it was necessary to add the dry sludge from more than one aluminium container.

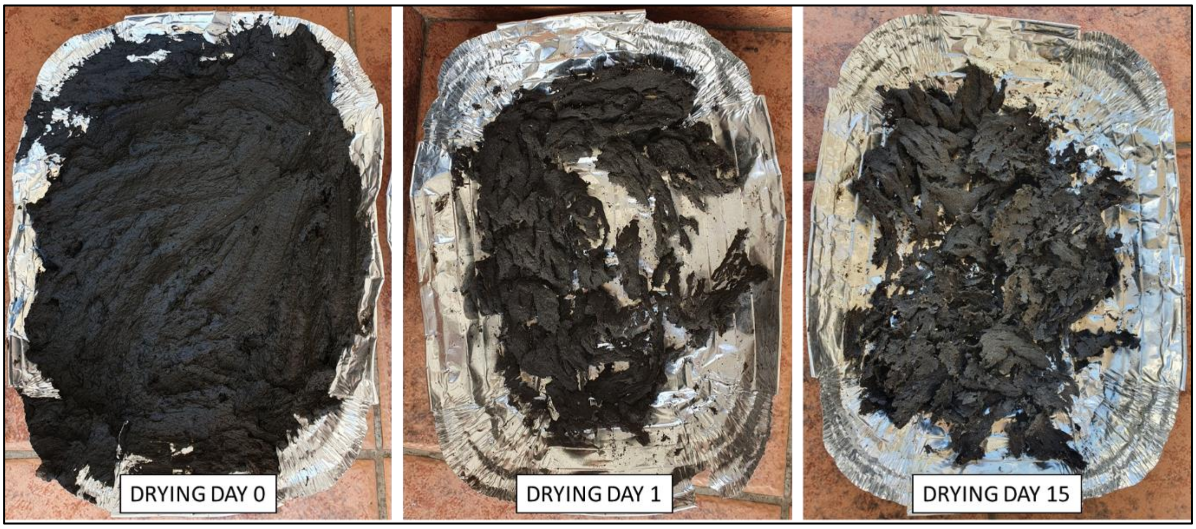

**Figure 8: Sun dried sludge samples**

The residual humidity of the sun-dried sludge was determined, for DS7 5.5% and for DS15 4.0%, as shown in Table 6.

**Table 6: Residual moisture from sun-dried sludge**

| nº | DS7 | DS15 |
|---|---|---|
| 1 | 5.6% | 3.9% |
| 2 | 5.5% | 3.9% |
| 3 | 5.6% | 4.0% |
| 4 | 5.4% | 4.0% |
| Average ± deviation | 5.5% ± 0.1% | 4.0% ± 0.0% |

The discussion shows that the system set up for drying the sludge proved to be efficient, drying from 83.2% humidity to 5.5% in 7 days of exposure. It is worth mentioning that the drying of the sludge, in this case, is influenced by the environment, such as the ambient temperature. Figures 9 and 10 were formulated based on meteorological data from the Instituto Português do Mar e da Atmosfera – IPMA [53]. Figure 9 shows the daily temperature during drying of the DS7 and figure 10 of the

DS15. It rained only on the first and third drying days of the DS7, 3.5 and 5.5 mm, respectively; however, the sludge was collected so as not to receive rain.

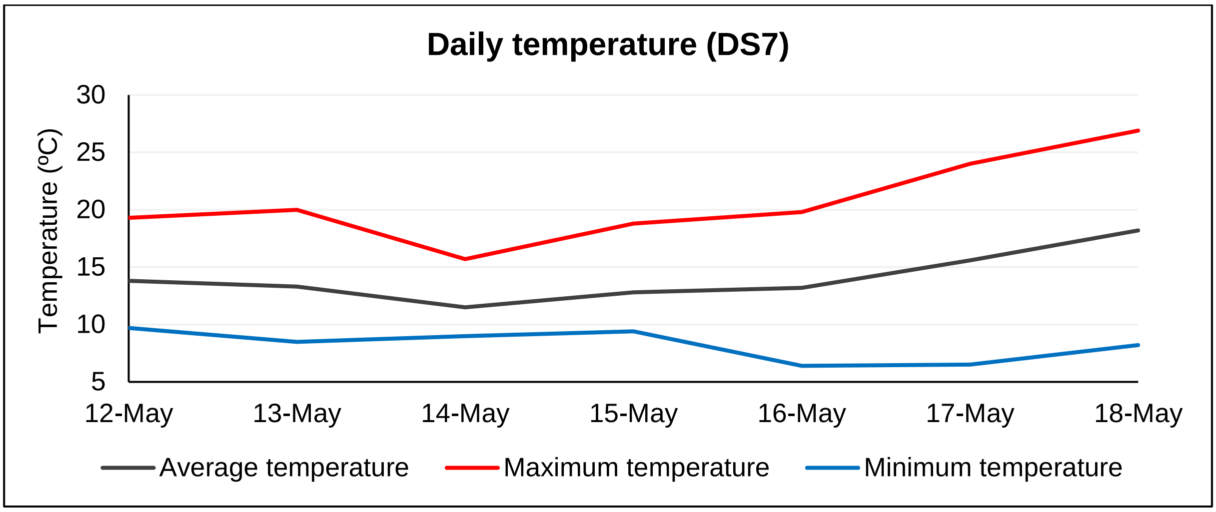


**Figure 9: Daily temperature during drying of DS7** [53]

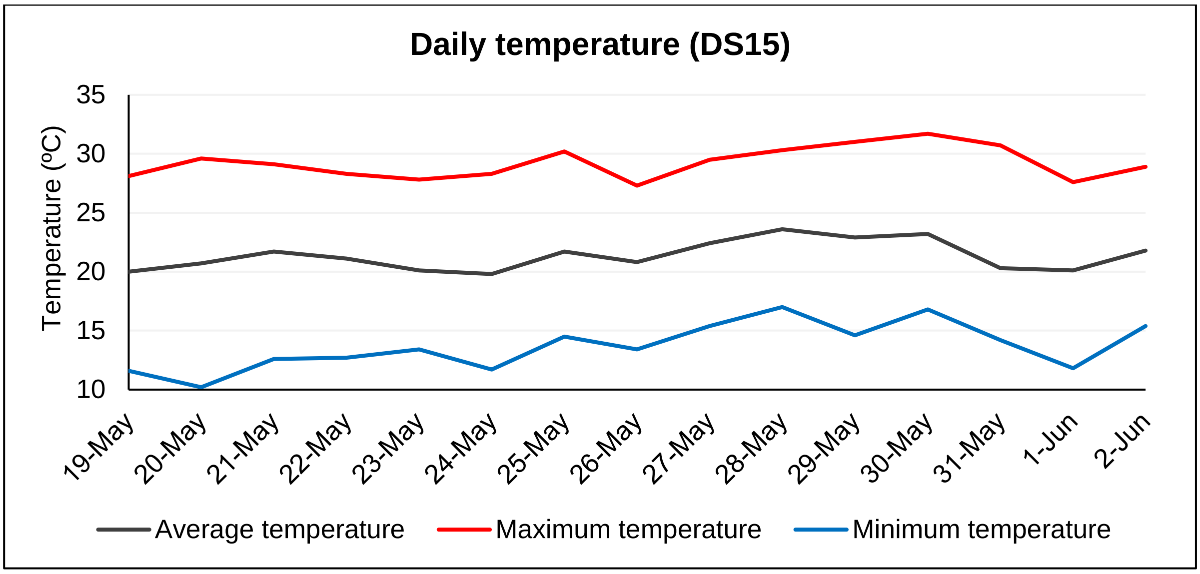


**Figure 10: Daily temperature during drying of the DS15** [53]

### 5.2.3 Incineration

The sludge before and after burning is shown in Figure 11. The burning process changes the colour of the sludge and reduces the volume because it reduces the organic matter in carbon dioxide and water.

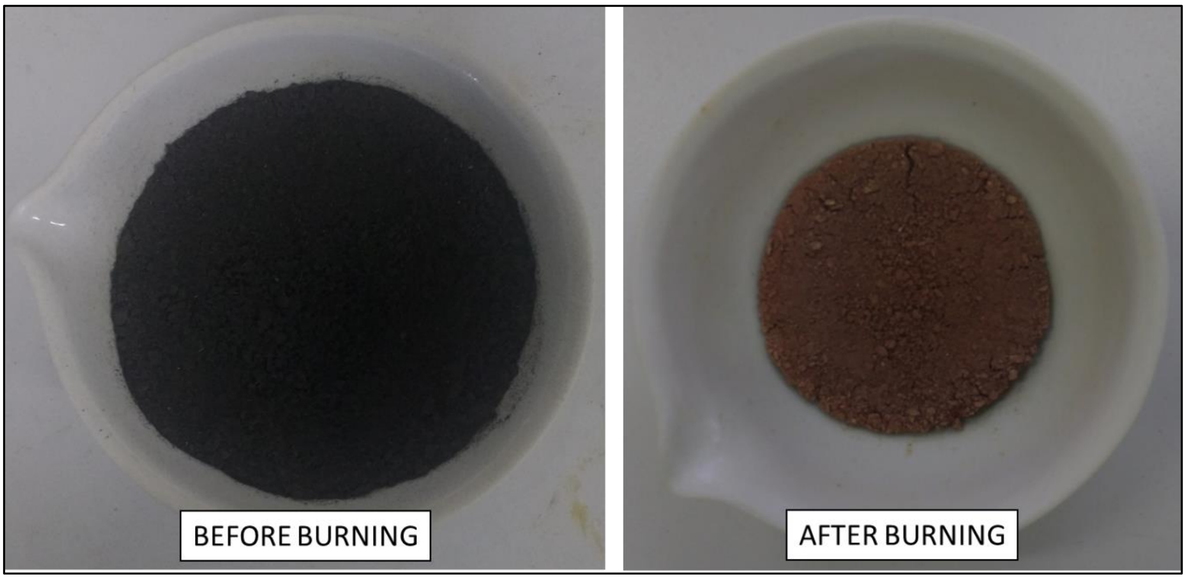


**Figure 11: Sample of dry sludge and sludge ash**

### 5.2.4 Wet oxidation

The appearance of the sludge after the autoclave process and after drying in an oven is presented in Figure 12.

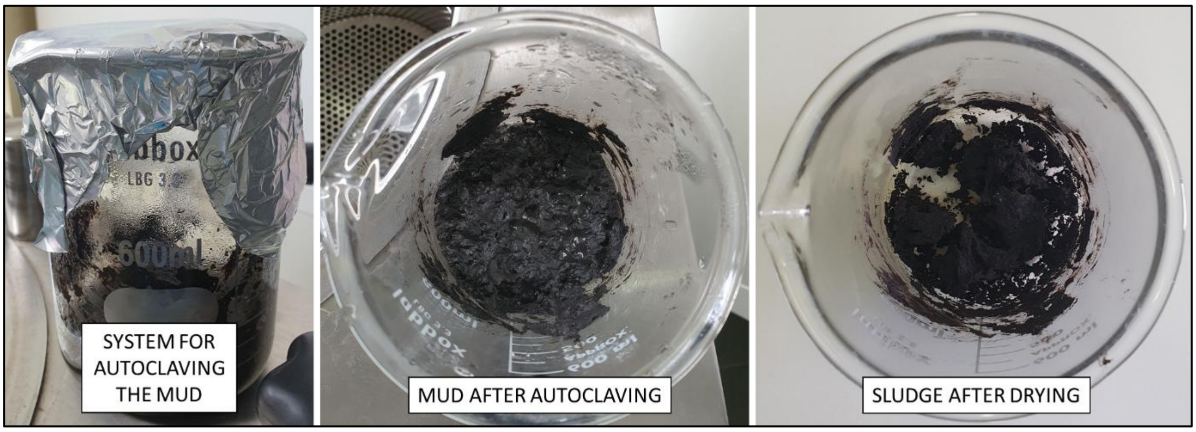


**Figure 12: Sludge sample during the autoclave process**

Autoclaving the sludge was an attempt to oxidise organic matter, however in milder conditions, using the equipment available. To evaluate the effectiveness of the process, the organic matter at the end of the autoclaving was determined, being 16.50% ± 1.32% the organic matter. There was a 27% reduction in organic matter.

## 5.3 MORTAR

The workability of mortars is the ease with which they can be mixed, transported, applied, consolidated, and finished, in a homogeneous state [7]. It was observed that the workability and viscosity decrease with the incorporation of the sludge material, regardless of the process that it went through, as well as in the works by Chang et al. (2010) [54]. According to Lynn 2015, this may be due to the porosity and absorption characteristics of the sludge particles [36].

The reduction in workability in a mortar with SA was not as significant as in mortar with DS7. Annexes 3 to 7 show how the samples looked after drying and, it is possible to see that the upper surface has become rougher with certain particles of treated sludge. A greater roughness of the final surface corresponds to a mortar of less workability. These parameters were not measured, there was only visual perception.

## 5.4 MECHANICAL TESTS

Due to the access restrictions to the laboratories of the Instituto Politécnico de Bragança due to the Covid-19 pandemic, it was not possible to collect all data on the correct date. Only 3 sets of tests were damaged, which should have been the 28th day of DS cure test, which was read on the 70th day, and in the SWO tests, which were tested on the 12th and 30th day of cure.

The amount of DS7 was not enough to manufacture specimens with 10% replacement of cement by sludge, so the sludge with this treatment was only applied in the manufacture of mortar pieces with 0, 3, 5 and 7%.

The tests intend to identify the changes that the incorporation of the treated sludge particles generate in the concrete mortar. The data obtained by the mechanical tests are shown in annexe 2. Figures 13 and 14 represents the final flexural compression and represents the average of the final axial compression strength of the specimens, respectively.

Analysis of variance was performed by simple ANOVA, considering each mortar preparation procedure and the concentration a different treatment for making the mortar, thus 48 observation points were analysed at 24 levels. The homogeneous groups created according to Tukey's test are arranged by the lower-case letters in Figure 14.

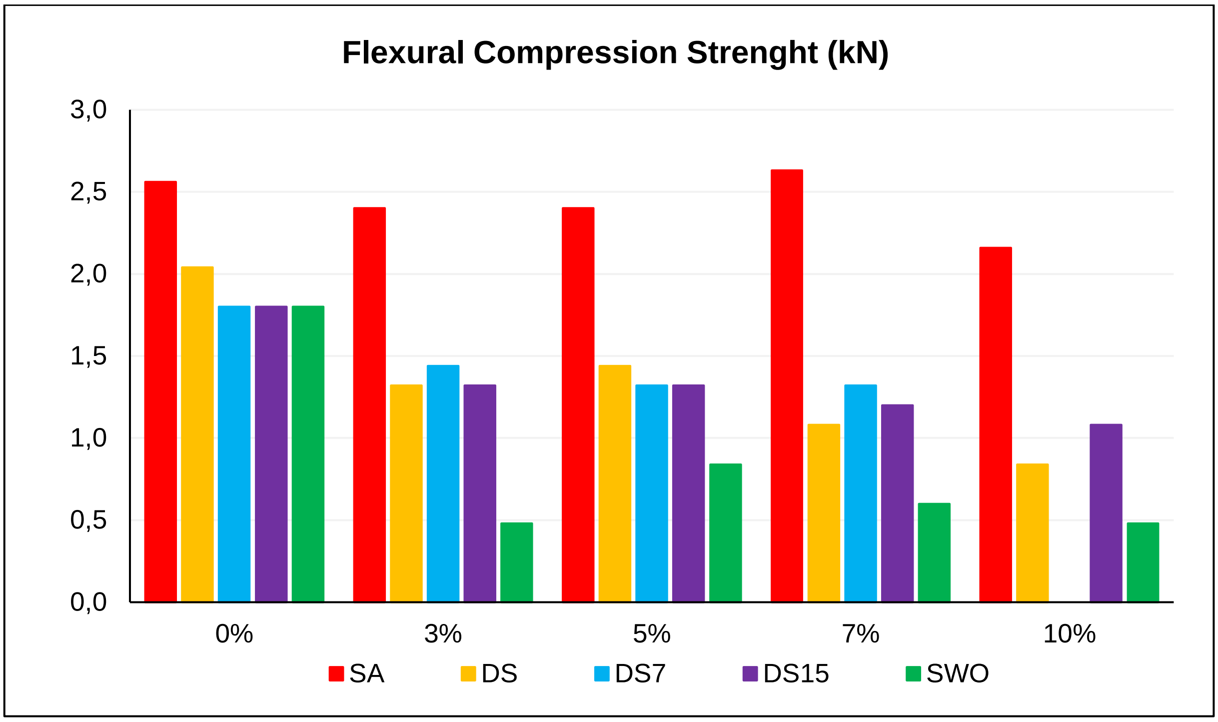


**Figure 13: Final flexural compression strength for the analysed specimens**

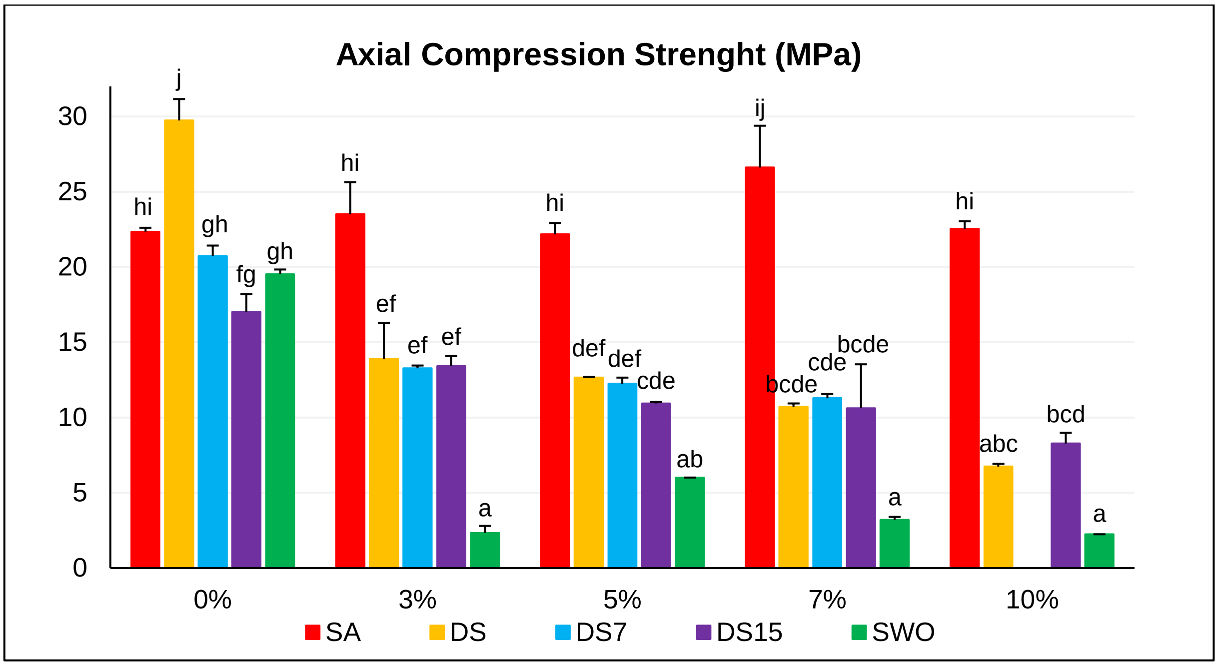


**Figure 14: Final resistance to axial compression for the analysed specimens**

According to the results, only one of the specimens showed greater mechanical resistance compared to the standard, the specimen with 7% SA. The dry sludge specimens showed very similar values and behaviours, as the resistance drop profile and the resistance values, while the SWO specimens presented the worst results.

Fonseca (2018) also made mortar specimens with dry sludge, but the author's results were different. For the 5% concentration, there was an improvement in resistance to both flexion and compression, compared to the standard. The specimen with 10% concentration was very close to the standard in the flexural strength test, but in the compressive strength test, it showed a result 27% lower than the standard. She also replaced cement to dry sludge in making mortars [26].

In Costa's (2014) work, the flexural and compressive strength of all samples obtained sludge ashes addition showed higher mechanical resistance than standard mortar. The author also replaced part of the cement mass for sludge ashes, however at concentrations of 0, 5, 10, 15 and 20% [27].

Although the increase in the sludge particles concentration decreases the flexural strength of the specimens, the concentration that shows the best results was 5% for dry sludge (DS15 and DS) or SWO. In the work of Fonseca (2018) the best results of resistance to flexion were obtained with a concentration of 5%, too [26]. In contrast to SA, the ideal concentration was 7%. In Costa's (2014) work, the greatest flexural strength was obtained with 20% ash, however tests with higher concentrations were not carried out to conclude whether this is the ideal maximum concentration [27].

Table 7 shows the mean mechanical resistance of the standards and the standard deviation. This variation may be due to changes in the cement due to the form of bag storage after opened, as well as irregularities in the quality of the sand used.

**Table 7: Mechanical resistance of the standards**

| Mechanical test | Average ± deviation |
|---|---|
| Flexural Compression Resistance (kN) | 2.00 ± 0.33 |
| Axial Compression Resistance (MPa) | 21.87 ± 4.59 |

Two additional statistical analyses were performed on the axial compression strength data. In one of the analyses the sludge concentration was disregarded, and the data were only processed by the mechanical resistance acquired according to the treatment of the sludge. In another scenario, the analysis was conducted disregarding the preparation method so that the data were treated by the mechanical strength and the slurry concentration in the mortar. The homogeneous groups formed from Tukey's test are shown in Table 8.

**Table 8: Average axial compressive strength by treatment and concentration**

| Treatment | Average (MPa) | Concentration (%) | Average (MPa) |
|---|---|---|---|
| SWO | 6.66 a | 10 | 9.95 a |
| DS15 | 12.05 ab | 7 | 12.49 a |
| DS7 | 14.38 b | 5 | 12.80 a |
| DS | 14.75 b | 3 | 13.28 ab |
| SA | 23.43 c | 0 | 21.87 b |

5.4.1 Dry sludge specimens

The compressive strength for specimens with different drying techniques showed similar results, as represented in Figures 13 and 14. To quantify the dispersion of the values, the standard deviation in relation to the average value was calculated. Table 9 shows the standard deviation obtained for each concentration.

**Table 9: Resistance to axial compression by drying techniques**

| | 0% | 3% | 5% | 7% | 10% |
|---|---|---|---|---|---|
| DS | 30.74 | 12.19 | 12.62 | 10.56 | 6.87 |
| | 28.73 | 15.58 | 12.69 | 10.87 | 6.61 |
| DS7 | 21.22 | 13.40 | 11.95 | 11.48 | |
| | 20.24 | 13.14 | 12.53 | 11.08 | |
| DS15 | 16.17 | 13.90 | 11.00 | 8.56 | 7.76 |
| | 17.84 | 12.95 | 10.86 | 12.68 | 8.78 |
| Standard deviation | 5.92 | 1.15 | 0.83 | 1.35 | 0.98 |

The deviation is even smaller when done in relation to the average axial compression strength of each method. In this scenario, the standard deviation is 0.32, 0.90, 0.36, and 1.08 for concentrations of 3, 5, 7 and 10% dry sludge.

Combine the residual moisture data from the sludge drying methods, the mechanical resistance of the mortar and the homogeneous groups obtained shows that drying the sludge in the sun does not confer chemical alterations to the material, since what changes, from one formulation to another is the moisture content of the particles. This discussion shows that, in the possibility of drying the sludge in the sun, there is no variation in the quality of the mortar obtained in relation to the same mortar produced with oven dried sludge, as long as the system formed contributes to the efficiency of the drying process and that the climatic conditions are favourable.

All mortars with dry sludge particles have a compressive strength greater than 6 MPa, therefore they are all of the category CS IV [8]. In this case the mortar can be applied in general purpose rendering/plastering mortar, in coloured rendering mortar and in one coat rendering mortar for external use, but in these applications it is also necessary to determine other parameters such as dry bulk density, adhesion, capillary water adsorption and others [8].

Through annexes 4, 6 and 7 and the residual humidity data, we can verify that the humidity of the incorporated particles influences the workability of the mortar. Specimens with DS7 have a more uneven surface and this is accentuated by increasing the concentration of sludge in the mortar. Likewise, specimens with DS15 have a less irregular surface than those previously mentioned, but it is more irregular than parts with DS.

### 5.4.2 Specimens with SWO

According to Table 3, mortars produced with substitution of 3, 7 and 10% cement by SWO particles belong to category CS II, whereas mortar with 5% belongs to category CS III and IV [8]. Despite this, all mortars produced with SWO particles were below the standard mortar, which belongs to the category CS IV [8]. Depending on performance in other parameters such as adhesion after weathering cycles, water penetration on relevant substrates after weathering cycles, the mortar of category CS II can be applied in all types of application described by EN 998-1 [8]. The mortar of category SC III can be applied in general purpose rendering/plastering mortar, in lightweight rendering/plastering mortar, in coloured rendering mortar and in one coat rendering mortar for external use [8].

Analysing the performance in relation to mechanical resistance and comparing it with mortars with sludge particles that were just dried, it is possible to infer that during the autoclaving process the sludge undergoes chemical changes that result in mortars with less resistance to compression.

Another aspect of specimens with SWO was porosity. In annexe 5 it is possible see an aerated surface, different from the other image attachments of the pieces. In addition, the specimens were more fragile, crumbling easily, which is consistent with the resistance results obtained.

### 5.4.3 Specimens with SA

For SA particles, all mortars produced are classified as CS IV [8]. In addition, these mortars achieved better performance in compressive strength, presenting resistance similar to a C20 concrete piece for concentrations of 3, 5 and 10% of burnt sludge. For the 7% concentration, the resistance shown is the same as for a C25 concrete piece [55].

Burn sludge at 900 °C, according to Lynn et al. (2015) increases the amorphous content of the final solid material, which increases pozzolanic activity. The increase in pozzolanic activity is related to the formation of oxides during burning, and the main ones in SA are silicon oxide, aluminium oxide and calcium oxide [36].

Due to the higher concentration of oxides in the reaction medium, the final structure has greater resistance, due to the pozzolanic reaction, that is, the replacement of calcium hydroxide by C-S-H [56].

As with the other methodologies, with SA the mortar also lost some of its workability with the incorporation of the particles, however, it was in a much smaller proportion than that visualised for the particles of the other treatment methods. The bibliographic survey by Lynn et al. (2015) also indicated a workability reduction, the authors calculated a reduction rate of 6% for every 10% of SA on mortars [36].

According to the research conducted by Paschoalino et al. (2006) cement does not harden by drying, but by chemical hydration reaction [57]. Several factors influence the mechanical strength of mortars, the quality of the clinker, the water/cement ratio, the content and quality of the aggregates and the dosage of hydroxide calcium [57].

Hagemann et al. (2019) evaluated the strength activity index to study the pozzolanic activity of the WWTP ash in the replacement of cement in mortars [45]. The authors observed that there is a maximum percentage of the incorporation of the sludge ash from WWTP, which starts from the compressive strength decreases [45].

Aishwarya and Suresh (2018) replaced part of the cement and fine aggregate with rice husk ash and waste foundry sand in the production of concrete. In their results, the mechanical resistance in the replacement with 10% rice husk ash revealed a peak of greater resistance in compared to the standard and concentrations of 5, 15, 20 and 25% [58]. In Fonseca's (2018) work, the peak mechanical strength occurred at 5% of cement replacement by sludge ashes in mortars [26]. In the work of Kaish et al.

(2018) the peak was 15% [31]. In Naamane's et al. (2016) work, the peak was 5% for mortars with ashes prepared at 700 and 800°C [59].

Taking into account the experiences of the authors mentioned above, the form of cement hardening highlighted by Paschoalino et al. (2006), and the conclusions of studies carried out by Hagemann et al. (2019), the peak mechanical resistance at 7% of cement replacement by SA may represent the ideal replacement concentration, the idea of error would only be discarded in the existence of replicates of these specimens [31, 45, 58, 59, 57].

## 5.5 ECONOMIC ANALYSIS

The average global demand for electric energy for the cement industry was 102 $kWh.t^{-1}$ cement in 2017 and has remained stable since 2012 [5]. According to Cochez and Nijs (2010), the efficiency and type of the cement production process causes the electricity demand to vary from 90 to 150 $kWh.t^{-1}$ cement [4]. The thermal energy demand for cement production is around 3.38 $GJ.t^{-1}$ clinker [4].

According to the literature review by Shnell et al. (2020) drum or fluidised bed dryers that transfer heat to the sludge by conduction and convection, which can be by hot gas or steam, consume about 0.07 $kWh.kg^{-1}_{H_2O}$ [47]. As for each ton of dry sludge, it is necessary to evaporate 777 $kg_{H_2O}$ (dry from 83.2% to 5.5% humidity), the electric energy demand in this process is 54.4 $kWh.t^{-1}$ dry sludge.

According to table 8, the concentrations (from 3 to 10%) in general do not represent a great influence on the mechanical resistance of the mortar so that they remained in the same homogeneous group, however, the substitution of 3% showed better results also belonging to the standard group. Thus, only these two concentrations of substitution were applied in the economic analysis calculations.

In these scenarios, the electricity consumption for the portion corresponding to cement in the mortar is 100.6 $kWh.t^{-1}$ cement for the concentration of 3% and 97.2 $kWh.t^{-1}$ cement for 10%. Considering the value of 102 $kWh.t^{-1}$ cement for the production of cement and considering equation 4, energy savings would be 8.6 $kWh.t^{-1}$ cement to replace 10% of dry sludge in a dryer or 2.6 $kWh.t^{-1}$ cement to replace 3%.

Still in this sense, if the sludge is dried with the sun, where considering that there is no energy expenditure, the saving would be 3.1 $kWh.t^{-1}$ cement for the concentration of 3% and 10.2 $kWh.t^{-1}$ cement for 10%.

According to the data collected by Xu et al. (2014), the drying operation consumes 39 $kWh.t^{-1}$ dry sludge and 1.53 $kJ.t^{-1}$ dry sludge and incineration consumes 275 $kWh.t^{-1}$ dry sludge but the operation has energy recovery of 1024.5 $kWh.t^{-1}$ dry sludge [48]. In the drying process, with data from Xu et al. (2014), there is a consumption of 100.1 $kWh.t^{-1}$ cement (3%) and 95.7 $kWh.t^{-1}$ cement (10%) for the proportion of cement with sludge in mortars. Therefore, the electric savings are 1.9 $kWh.t^{-1}$ cement (3%) and 6.3 $kWh.t^{-1}$ cement (10%) due to the incorporation of dry sludge in mortars. In the incineration process, electrical consumption is 76.5 $kWh.t^{-1}$ cement (3%) and 16.9 $kWh.t^{-1}$ cement (10%) for the cement fraction in mortars with sludge ashes. The reduction in consumption represents savings of 25.5 $kWh.t^{-1}$ cement (3%) and 85.2 $kWh.t^{-1}$ cement (10%) in the production of mortars.

In a study by Lundin et al. (2004) the co-incineration of WWTP sludge and waste generates 2300 kWh of district heating and 250 $kWh.t^{-1}$ dry sludge of electricity through the combined production of heat and energy [49]. In this scenario, electrical consumption is 91.4 $kWh.t^{-1}$ cement (3%) and 66.8 $kWh.t^{-1}$ cement (10%) for the proportion of cement in mortars with sludge ashes. The use of ash represents savings of 10.6 $kWh.t^{-1}$ cement (3%) and 35.2 $kWh.t^{-1}$ cement (10%).

Table 8 shows the energy savings calculated for each of the processes considered.

**Table 10: Energy savings according to the reference values**

| | | Energy saving ($kWh.t^{-1}$ cement) | |
|---|---|---|---|
| **Process** | **Electricity demand** | 3% | 10% |
| Drum or fluidised bed dryers | 0.07 $kWh.kg^{-1}_{H2O}$ [47] | 2.6 | 8.6 |
| Drying operation | 39 $kWh.t^{-1}$ dry sludge [48] | 1.9 | 6.3 |
| Sun-drying | 0 | 3.1 | 10.2 |
| Dry sludge and incineration | -749.5 $kWh.t^{-1}$ dry sludge [48] | 25.5 | 85.2 |
| Co-incineration | -250 $kWh.t^{-1}$ dry sludge [49] | 10.6 | 35.2 |

# 6 CONCLUSIONS AND FUTURE RESEARCH

## 6.1 CONCLUSIONS

The mechanical quality of mortar specimens with the incorporation of WWTP sludge was tested, thus studying the technical feasibility of the practices. From the comparison of energy production costs, between the cement production and the methodologies for preparing sludge for subsequent application in mortars, the economic valuation of practices was also evaluated.

From the discussions of energy expenditure, the characteristics of the sludge collected at the Bragança WWTP presents great potential for application in construction materials. The low organic matter contributes to the energy saving of sludge preparation processes in incineration.

The use of sludge ashes in small concentrations does not alter the mechanical strength of the mortar, in the concentration of 7% substitution of cement for ashes the mechanical quality increases in comparison with the standard mortar.

Between sun and oven drying there is no change in the quality of the mortar produced, but the moisture in the sludge does, in relation to the spreading and the roughness of the surface. On the other hand, the incorporation of the sludge reduces the mechanical strength of the mortar in relation to the standard mortar, however even in concentrations of up to 10% this loss of strength does not make the mortar to be classified at a lower resistance than the standard mortar.

In applications where the workability of the mortar does not need to be high, the incorporation of sludge represents a good outlet for stable and safe disposal for WWTP sludge and is an economical practice.

The autoclaving of the sludge is not an interesting option since it did not show the degradation of all organic matter and made the mechanical resistance lower than when the sludge was just dry, in addition to leaving the mortar aerated and crumbling.

The practices of burning and drying the sludge obtained results that justify the procedures and the subsequent application in the mortar while, autoclaving the sludge proved not to be interesting for this purpose of the civil application.

## 6.2 FUTURE RESEARCH

According to the standard EN 998-1: 2017, the quality of the mortar is evaluated in 12 test parameters. In this work, only one of the parameters was tested, so to test the other parameters verifies the quality and safety of the mortar with WWTP sludge. Besides, the parameters define the ideal use of the mortar, for example for the use of plaster, or general use, for the coloured mortar bear, among others. Tests can be done in a significant number of repetitions.

To analyse the feasibility of correcting the loss of roughness and workability with the use of additives, such as plasticisers, in a mortar with sludge with residual moisture around 5.5%, as in the case of sludge dried in the sun.

To quantify of the change in mortar density by incorporating the sludge.

To evaluate of the immobilisation of heavy metals presents in the sludge from the application in mortar or concrete through leaching and solubility.

To study the implications for the mortar quality due to the seasonality of the sludge characteristics.

To investigate the sludge application in concrete formulations.

To understand the properties of sludge that influence the mechanical behaviour of mortar or concrete:

- To analyse the elementary composition of the sludge through TXRF (Total Reflection X-ray Fluorescence), or LIBS (Laser-Induced Breakdown Spectroscopy).
- To analyse the crystalline structure of the sludge and the products manufactured through XRD (X-Ray Diffraction).
- To analyse the surface aspects and the composition of the sludge and products manufactured through EDX (X-ray Dispersive Energy Spectroscopy) and SEM (Scanning Electron Microscopy)
- To understand the behaviour of sludge organic matter degradation through TGA (Thermogravimetric analysis).

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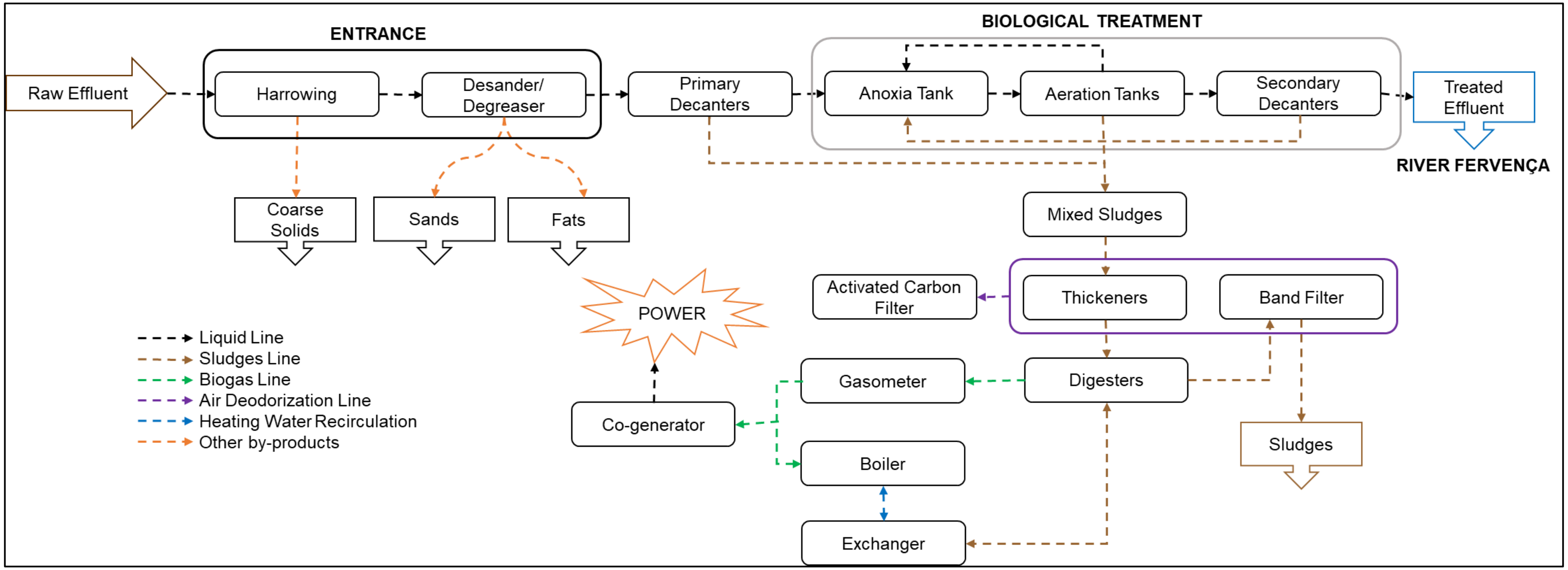
# ANNEXES

**Annexe 1: Flowchart of the Wastewater Treatment Plant at Bragança**



**Annexe 2: Mechanical testing of specimens**

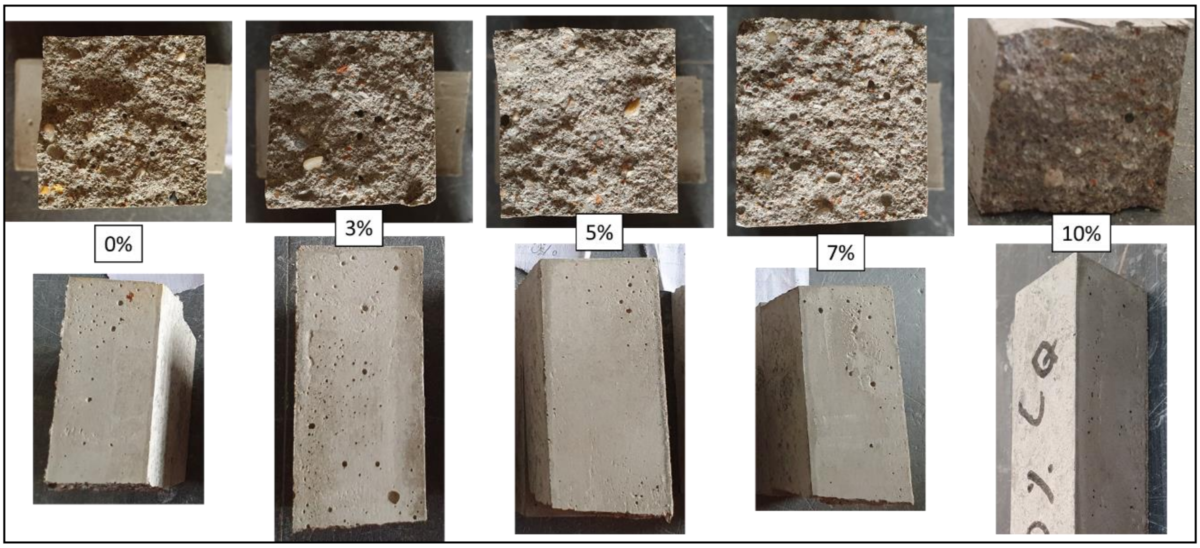
| Day | Sludge concentration: | | 0% | 3% | 5% | 7% | 10% |
|---|---|---|---|---|---|---|---|
| **Specimens with ashes** | | | | | | | |
| 7 | Axial compression | Load (kN) | 33.03 | 28.44 | 26.70 | 35.96 | 33.73 |
| | | | 32.44 | 29.47 | 29.15 | 35.20 | 32.78 |
| | | Pressure (MPa) | 20.65 | 17.78 | 16.69 | 22.47 | 21.08 |
| | | | 20.28 | 18.42 | 18.22 | 22.00 | 20.49 |
| | Flexural compression | Strength (kN) | 1.92 | 1.68 | 1.68 | 2.28 | 1.92 |
| 28 | Axial compression | Load (kN) | 36.05 | 35.18 | 34.62 | 45.71 | 36.63 |
| | | | 35.46 | 40.02 | 36.31 | 39.43 | 35.50 |
| | | Pressure (MPa) | 22.53 | 21.99 | 21.63 | 28.57 | 22.89 |
| | | | 22.16 | 25.01 | 22.70 | 24.64 | 22.19 |
| | Flexural compression | Strength (kN) | 2.56 | 2.40 | 2.40 | 2.63 | 2.16 |
| **Specimens with oven dried sludge** | | | | | | | |
| Day | Sludge concentration: | | 0% | 3% | 5% | 7% | 10% |
| 7 | Axial compression | Load (kN) | 37.25 | 19.20 | 15.15 | 10.00 | 7.12 |
| | | | 37.27 | 18.85 | 13.20 | 10.25 | 7.44 |
| | | Pressure (MPa) | 23.28 | 12.00 | 9.47 | 6.25 | 4.45 |
| | | | 23.30 | 11.78 | 8.25 | 6.41 | 4.65 |
| | Flexural compression | Strength (kN) | 2.16 | 1.32 | 1.20 | 0.84 | 0.60 |
| 70 | Axial compression | Load (kN) | 49.18 | 19.50 | 20.19 | 16.90 | 11.00 |
| | | | 45.97 | 24.93 | 20.30 | 17.40 | 10.58 |
| | | Pressure (MPa) | 30.74 | 12.19 | 12.62 | 10.56 | 6.87 |
| | | | 28.73 | 15.58 | 12.69 | 10.87 | 6.61 |
| | Flexural compression | Strength (kN) | 2.04 | 1.32 | 1.44 | 1.08 | 0.84 |
| **Specimens with wet oxidised sludge** | | | | | | | |
| Day | Sludge concentration: | | 0% | 3% | 5% | 7% | 10% |
| 12 | Axial compression | Load (kN) | 25.50 | 4.53 | 9.29 | 4.38 | 3.45 |
| | | | 28.73 | 3.40 | 7.57 | 3.68 | 3.55 |
| | | Pressure (MPa) | 15.94 | 2.83 | 5.81 | 2.73 | 2.15 |
| | | | 17.95 | 2.12 | 4.73 | 2.30 | 2.22 |
| | Flexural compression | Strength (kN) | 1.68 | 0.36 | 0.72 | 0.48 | 0.24 |
| 30 | Axial compression | Load (kN) | 30.86 | 3.18 | 9.62 | 4.91 | 3.57 |
| | | | 31.59 | 4.26 | 9.59 | 5.34 | 3.58 |
| | | Pressure (MPa) | 19.29 | 1.99 | 6.01 | 3.07 | 2.23 |
| | | | 19.74 | 2.66 | 6.00 | 3.34 | 2.24 |
| | Flexural compression | Strength (kN) | 1.80 | 0.48 | 0.84 | 0.60 | 0.48 |

**Annexe 2: Mechanical testing of specimens (cont.)**

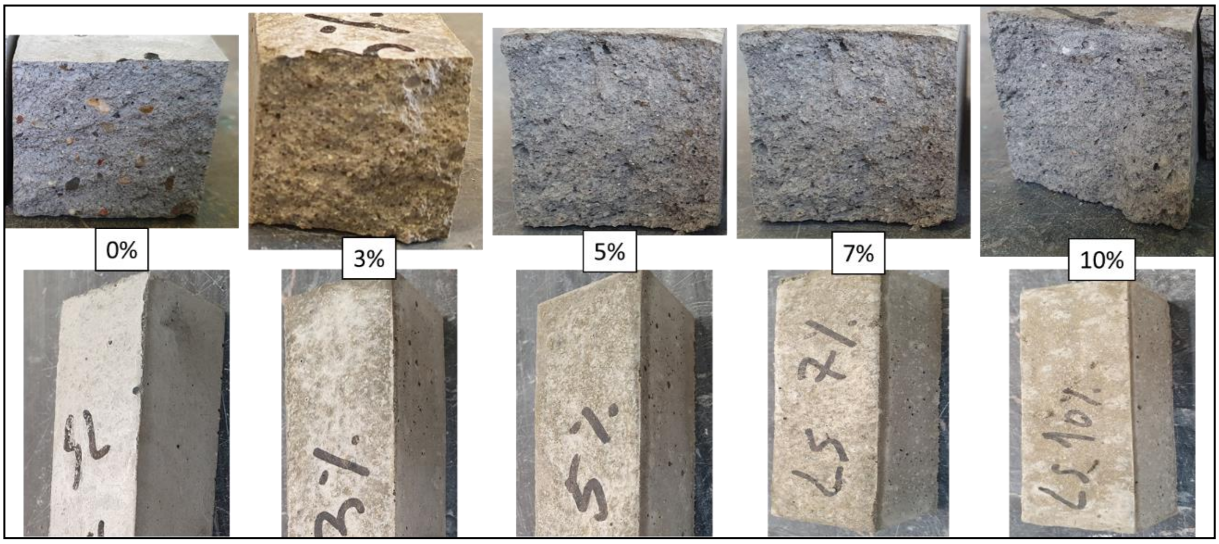
| Specimens with sun-dried sludge for 7 days | | | | | | |
|---|---|---|---|---|---|---|
| Day | Sludge concentration: | | 0% | 3% | 5% | 7% |
| 7 | Axial compression | Load (kN) | 20.42 | 11.78 | 8.98 | 8.19 |
| | | | 22.88 | 11.60 | 11.74 | 12.70 |
| | | Pressure (MPa) | 12.76 | 7.36 | 5.61 | 5.12 |
| | | | 14.30 | 7.25 | 7.34 | 7.94 |
| | Flexural compression | Strength (kN) | 1.20 | 0.96 | 0.84 | 1.08 |
| 28 | Axial compression | Load (kN) | 33.96 | 21.45 | 19.14 | 18.37 |
| | | | 32.39 | 21.03 | 20.04 | 17.75 |
| | | Pressure (MPa) | 21.22 | 13.40 | 11.95 | 11.48 |
| | | | 20.24 | 13.14 | 12.53 | 11.08 |
| | Flexural compression | Strength (kN) | 1.80 | 1.44 | 1.32 | 1.32 |

| Specimens with sun-dried sludge for 15 days | | | | | | | |
|---|---|---|---|---|---|---|---|
| Day | Sludge concentration: | | 0% | 3% | 5% | 7% | 10% |
| 7 | Axial compression | Load (kN) | 23.46 | 15.55 | 14.69 | 11.84 | 8.99 |
| | | | 21.52 | 10.83 | 15.45 | 12.62 | 6.90 |
| | | Pressure (MPa) | 14.66 | 9.72 | 9.18 | 7.40 | 5.62 |
| | | | 13.45 | 6.77 | 9.66 | 7.88 | 4.31 |
| | Flexural compression | Strength (kN) | 1.56 | 1.08 | 0.84 | 1.08 | 0.72 |
| 28 | Axial compression | Load (kN) | 25.86 | 22.24 | 17.60 | 13.69 | 12.42 |
| | | | 28.55 | 20.72 | 17.37 | 20.28 | 14.05 |
| | | Pressure (MPa) | 16.17 | 13.90 | 11.00 | 8.56 | 7.76 |
| | | | 17.84 | 12.95 | 10.86 | 12.68 | 8.78 |
| | Flexural compression | Strength (kN) | 1.80 | 1.32 | 1.32 | 1.20 | 1.08 |

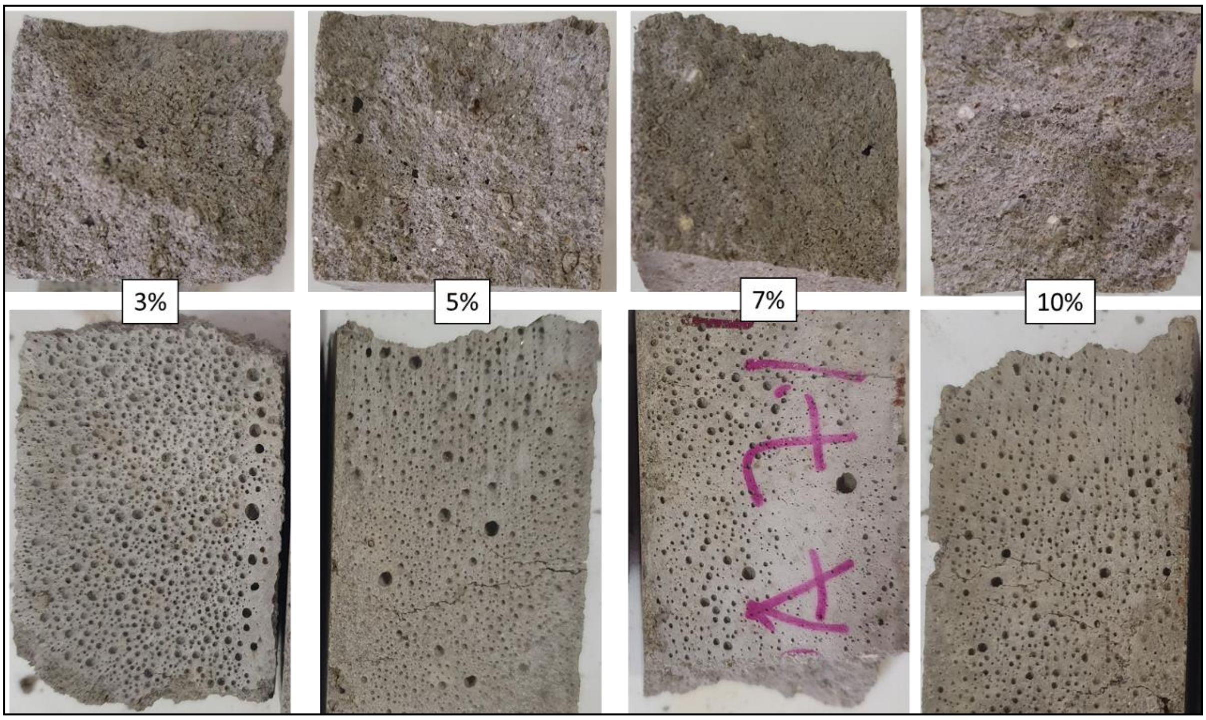
**Annexe 3: Images of specimens with sludge ash**



### Annexe 4: Images of specimens with oven dried sludge




### Annexe 5: Images of specimens with wet oxidised sludge



**Annexe 6: Images of specimens with sludge dried in the sun for 7 days**


**Annexe 7: Images of specimens with sludge dried in the sun for 15 days**