



## LEATHER TANNING SLUDGE IN CEMENT MORTAR: SUSTAINABLE VALORISATION

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### ABSTRACT

**Purpose:** This study aims to investigate the valorisation of sludge from Leather Tannery Wastewater Treatment Plants (LTWWTP) by incorporating it into cement mortars.

**Theoretical framework:** The substantial quantity of waste generated by industries coupled with the scarcity of natural resources calls for the analysis, development, and implementation of treatment methods to recover and reuse materials, thereby minimising the environmental impact of waste disposal. The leather tanning industry, a significant polluter due to its use of hazardous and heavy materials, requires sustainable solutions for waste management. One promising approach is the incorporation of waste into cementitious matrices.

**Method:** Three scenarios were examined: low-moisture sludge, ash obtained via incineration, and wet oxidation. Test specimens were prepared with varying proportions of cement replacement (0, 3, 5, 7, and 10%), and their flexural and compressive strengths were evaluated. Additionally, chemical analyses were conducted to assess solubility.

**Results and Conclusion:** The results indicate that sludge can be used in small proportions without compromising the mechanical strength of the mortar. Substituting 10% cement with ash using the INC-2 incineration method improved mortar quality, exhibiting a higher compressive strength of 41 MPa and minimal water absorption (3.28%).

**Research implications:** Drawing from these promising findings, it can be confidently asserted that utilising WWTP sludge in mortar is a practical, eco-conscious solution for civil construction. Furthermore, it is a pivotal step towards addressing the challenge of hazardous sludge disposal.

**Originality/value:** The findings from this research will contribute to sustainable waste management practices and pave the way for responsible and eco-friendly utilisation of industrial waste in cementitious materials, thus minimising environmental burdens.

**Keywords:** Cement Mortar, Circular Economy, Recycling, Sludge, Tanner Industry.

## LAMAS DE CURTIMENTA EM CIMENTO ARGAMASSA: VALORIZAÇÃO SUSTENTÁVEL

### RESUMO

**Objetivo:** Este estudo visa investigar a valorização da lama proveniente de Estações de Tratamento de Efluentes de Curtumes (ETEC) por incorporação em argamassas de cimento.

**Referencial Teórico:** O volume significativo de resíduos gerados pela indústria, juntamente com a escassez de recursos naturais, requer a análise, desenvolvimento e implementação de métodos de tratamento adequados à recuperação e reutilização de materiais, minimizando o impacto ambiental da sua deposição. A indústria de

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curtumes, um poluidor considerável devido ao uso de materiais perigosos e metais tóxicos, necessita de soluções sustentáveis para a gestão de resíduos. Uma abordagem promissora é a incorporação desses resíduos em matrizes cimentícias.

**Método:** Três cenários foram testados: lamas com baixa umidade, cinzas obtidas por incineração e por oxidação húmida. Amostras foram preparadas com diferentes proporções de substituição de cimento (0, 3, 5, 7 e 10%), e a resistência à flexão e compressão foram avaliadas. Além disso, análises químicas foram realizadas para avaliar a solubilidade.

**Resultados e Conclusão:** Os resultados mostram que a lama pode ser utilizada em pequenas proporções sem comprometer a resistência mecânica da argamassa. Notavelmente, a substituição de 10% de cimento por cinzas utilizando o método de incineração INC-2 resultou em uma melhoria na qualidade da argamassa, apresentando uma resistência à compressão de 41 MPa e absorção de água mínima (3,28%).

**Implicações da Investigação:** Face aos resultados promissores, pode concluir-se que o uso de lama de ETEC em argamassas se apresenta como uma solução prática e ecologicamente consciente para a construção civil. Além disso, representa um passo fundamental para enfrentar o desafio da deposição de lamas perigosas.

**Originalidade/Valor:** Os resultados desta investigação são um contributo na implementação de práticas sustentáveis de gestão de resíduos e, representam um caminho para a utilização responsável e ecologicamente correta de resíduos industriais em materiais cimentícios, minimizando o impacto ambiental.

**Palavras-chave:** Argamassa de Cimento, Economia Circular, Reciclagem, Lama, Indústria de Curtume.

## LODO CURTIDO DE CUERO EN MORTERO DE CEMENTO: VALORIZACIÓN SOSTENIBLE

### RESUMEN

**Objetivo:** Investigar la recuperación de lodos de plantas de tratamiento de curtiduría (ETEC) mediante su incorporación en mortero de cemento.

**Referente teórico:** El importante volumen de residuos generados por la industria, junto con la escasez de recursos naturales, requiere el análisis, desarrollo e implementación de métodos de tratamiento adecuados para la recuperación y reutilización de materiales, minimizando el impacto ambiental de su depósito. La industria del curtido, que es un contaminante importante debido al uso de materiales peligrosos y metales tóxicos, necesita soluciones sostenibles para la gestión de los desechos. Un enfoque prometedor es incorporar estos residuos en las matrices de cemento.

**Método:** Se probaron tres escenarios: lodos con baja humedad, cenizas obtenidas por incineración y por oxidación húmeda. Las muestras se prepararon con diferentes proporciones de sustitución de cemento (0, 3, 5, 7 y 10%), y se evaluó la resistencia a la flexión y compresión. Además, se realizaron análisis químicos para evaluar la solubilidad.

**Resultados y Conclusión:** Los resultados muestran que el lodo puede ser utilizado en pequeñas proporciones sin comprometer la resistencia mecánica del mortero. En particular, la sustitución de cenizas por cemento al 10% utilizando el método de incineración INC-2 resultó en una mejora en la calidad del mortero, presentando una resistencia a la compresión de 41 MPa y mínima absorción de agua (3,28%).

**Implicaciones de la investigación:** A la vista de los resultados prometedores, se puede concluir que el uso de barro ETEC en mortero se presenta como una solución práctica y ecológicamente consciente para la construcción civil. También es un paso fundamental para hacer frente al desafío de eliminar los lodos peligrosos.

**Originalidad/Valor:** Los resultados de esta investigación son una contribución a la implementación de prácticas sostenibles de gestión de residuos y, representan un camino hacia el uso responsable y ambientalmente racional de los residuos industriales en materiales de cemento, minimizando el impacto ambiental.

**Palabras clave:** Mortero de Cemento, Economía Circular, Reciclaje, Barro, Industria de Curtiduría.



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## 1 INTRODUCTION

The growing awareness of socio-environmental issues, stricter legal requirements, large quantities of waste generated by industries, and limited availability of energy and raw materials have prompted researchers to explore various treatments and applications for industrial waste (Santos *et al.*, 2023; Morais, 2005).

Industrial waste can be classified into two phases: liquid and solid. Different treatment methods can be employed in the liquid phase, including preliminary processes, such as removing coarse solids through fixed or rotating screens, as well as primary, secondary (biological), and tertiary treatments. These processes lead to forming a solid phase, commonly known as sludge, which is the main focus of this study. Sludge is characterised by its high content of organic matter and water, and the source of the solid materials primarily influences its composition, the quantity of sludge generated, and the specific treatment processes involved (Carneiro, 2020).

The tanning industry utilises various polluting chemical substances at different stages of its production process (from receiving the hides to the tanning process), thereby affecting the environment (Simões *et al.*, 2019). The tanning of animal hides to transform them into leather involves the addition of chemical substances to enhance their durability and improve their physical characteristics. Tanning agents, whether vegetable-based (tannins) or mineral-based (such as metallic chromium), are added, with the latter being the most commonly used agent in the production of marketable leather (wet blue leather). The waste generated from this process is considered hazardous and has a high potential for contamination, necessitating proper disposal measures to prevent environmental harm (Oliveira *et al.*, 2008; Teixeira *et al.*, 2015).

The treatment of wastewater generated from tanning processes in wastewater treatment plants (WWTPs) is of utmost importance because of the generation of large quantities of toxic waste. Legislative bodies have established legal limits for specific parameters, such as COD, BOD, total dissolved solids, and pH, for this type of industry to prevent the discharge of harmful substances into the environment (Sreeram and Ramasami, 2003).

The construction industry needs to work on waste management and the pursuit of sustainable practices. One alternative is the incorporation of these wastes into a cementitious



matrix, as this industry tends to grow and requires highly mouldable and preparable materials (FETICOP, 2023). According to information from the Portuguese Federation of Construction and Public Works Industry, cement consumption in the national market has steadily increased since 2011, reaching its highest value by 2022 (FETICOP, 2023).

## 2 OBJECTIVES

This study aims to present an innovative approach for the valorisation of sludge derived from WWTPs in the tanning industry by incorporating it into cement mortar and evaluating its integrity and durability.

## 3 METHODOLOGY

Tannery wastewater treatment plant (WWTP) sludge was collected from a factory in the Alcanena region of Portugal. The waste had a pH of 5.33 and exhibited a high concentration of various metals and minerals and a chemical oxygen demand (COD) of 251 g kg<sup>-1</sup>.

Incorporating sludge into cement can positively and negatively affect the various parameters. For instance, the durability of the final product may be affected by the presence of high levels of organic matter and heavy metals. Therefore, it is essential to characterise the materials being studied thoroughly (Grabowski, 2020).

The tests performed for characterisation included moisture content, total solids, fixed solids, organic matter content, organic carbon content, loss to fire, and atomic absorption spectrophotometry (AAS) to confirm the concentration of an element in a sample.

To ensure that the composition of the sample and the presence of organic matter do not alter the properties of the cement produced, it is necessary to degrade the organic matter before incorporating it into concrete (Sampaio, 2017).

According to Swarnalatha et al. (2008), to degrade the organic matter, the material was subjected to burning, involving five processes: sludge oven drying, sludge sun drying, two methodologies of incineration, and wet oxidation (Swarnalatha *et al.*, 2008).

Concrete specimens (CS) were manufactured using prismatic moulds with dimensions of 40x40x160 mm, with each specimen weighing approximately 600 g. Six CS were produced for each concentration of sludge used, with three used for testing on the 7th day of concrete curing and the remaining three used on the 28th day of curing.



The CS was produced by mixing sand, cement, distilled water, and sludge, where a part of the cement mass was replaced to form a cement paste. The pastes consisted of replacements of 3%, 5%, 7%, or 10% cement with sludge.

The NBR 5738 (ABNT) standards are followed to produce the CS, which involves applying the paste into the moulds and then using a horizontal compactor (ABNT, 2016). After production, they were kept on the bench in the geotechnical laboratory of the Polytechnic Institute of Bragança for 48 h, after which they were removed from the moulds and transferred to a humid chamber maintained at 20-25°C with 90% humidity.

Finally, flexural and compressive strength tests, as well as chemical analyses for solubility, were performed.

## 4 RESULTS AND DISCUSSION

Table 1 shows the metal composition of the sludge ( $mg\ kg^{-1}$  of dry matter) provided by AQUANENA company. Subsequently, several tests were conducted using atomic absorption spectrophotometry to verify the concentrations of specific elements, and the results were confirmed. In other words, the test outcomes yielded values that closely resembled those initially provided.

The sample was dissolved entirely to determine the elemental concentration using AAS. This was accomplished by digesting the sample using Parr Pumps. The digestion procedure involved combining approximately 200 mg of the sample with 2 or 4 mL of 65% nitric acid ( $HNO_3$ ), heating it in a microwave at different time intervals (30 or 60 s) and power levels (600 or 800 W).

**Table 1**

*Composition (metal) of sludge from WWTP.*

Element	Concentration ( $mg\ kg^{-1}$ of dry matter)
Lead	17.2
Manganese	890
Phosphorus	6210
Strontium	133
Arsenic	6.14
Zinc	86.3
Cobalt	5.08
Iron	8880
Nickel	13.8
Sodium	5760
Magnesium	8100



Aluminium	14500
Titanium	298
Sulfur	8670
Potassium	12600
Calcium	127000

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Source: AQUANENA internal report

To determine the moisture content, approximately 3 g of the sample was dried in porcelain containers in an oven for 24 h at a temperature of 105°C, and it can be calculated according to equation 1:

$$H = \frac{(A-B)}{A} * 100 \quad (1)$$

where:

*H* percentage of moisture

*A* weight of the wet sample minus the container, mg

*B* weight of the dry sample minus the container, mg

To define total and fixed solids, it is necessary to understand that solids are a state of matter characterised by their rigidity, ability to maintain their shape, and the presence of equilibrium with the liquid phase resulting from their fusion. Any substance that exhibits these characteristics, even after undergoing various processes, such as drying and calcination, can be classified as a solid (Sabesp, 1999).

Total solids can be classified by all the matter that remains in the container after the total drying of a given mass at 105°C, and is calculated using equation 2:

$$TS = \frac{(A-B)}{(C-B)} * 100 \quad (2)$$

where:

*TS* percentage of total solids

*A* mass of the dry material plus the crucible, g

*B* crucible weight, g

*C* mass of the material at the beginning plus the crucible, g



Fixed solids are the total matter remaining in the vessel after a given mass is calcined at approximately 550°C.

$$FS = \frac{(D-B)}{(E-B)} * 100 \quad (3)$$

where:

*FS* percentage of fixed solids

*D* mass of the material after calcination plus the crucible, g

*B* crucible weight, g

*E* mass of the material at the beginning plus the crucible, g

An adapted version of the method developed by Sampaio (2017) was used to analyze the organic matter content. Approximately 100 mg of dried and ground sludge was transferred to a 500 mL Erlenmeyer flask, to which a potassium dichromate solution ( $K_2Cr_2O_7$ ) of 1N and 20 mL of concentrated sulfuric acid ( $H_2SO_4$ ) were added. The mixture was then vigorously shaken for one minute to prevent any residue from adhering to the bottom of the flask, and left to rest for 30 minutes. After this period, 200 mL of distilled water, 10 mL of concentrated orthophosphoric acid, and 1% ferroin indicator were added, and the solution was titrated with 0.5N ammonium ferrous sulfate until a red-brown color was obtained.

Sampaio initially reported that the colour obtained during titration should be green. However, in this experiment, the colour changed from green to red-brown, and according to the literature, the colour change can vary depending on the sample, ranging from blue to green (as observed by Sampaio) or green to red-brown (Sampaio, 2017; APHA, 2022).

The organic matter content is given by (4):

$$OM = 1.725 * \frac{\left(10 - \left(V_2 * \frac{10}{V_1}\right)\right)}{m} \quad (4)$$

where:

*OM* percentage of organic matter content

$V_1$  volume of ammonium ferrous sulfate spent in the blank titration, mL

$V_2$  volume of ammoniacal ferrous sulfate used in the sample titration, mL



$m$  mass of sludge sample, g

For organic carbon content measurement (TOC) equipment available at the CIMO - Mountain Research Center laboratory of the Polytechnic Institute of Bragança was employed. The TOC technique employs total oxidation and acidification of the sample at 720°C to distinguish between organic and inorganic carbon.

The loss to fire test determines the percentage of mass lost or decomposed by a sample when subjected to a pre-established thermal heating cycle, typically involving high temperatures, and conducted in an oven or muffle (equation 5). This characterisation is essential as per NBR 12653, where the material is classified as class N pozzolanic when LF is less than or equal to 20%, and as classes C and E when the result is less than or equal to 6% (ABNT, 2016).

$$LF = \frac{(m_1 - m_2)}{m} * 100 \quad (5)$$

where:

$LF$  the percentage of loss to fire

$m_1$  mass of the (crucible + sample), before calcination, g

$m_2$  mass of the (crucible + sample), after calcination, g

$m$  mass of the sample, g

Table 2 presents the results of all the sludge characterisation tests.

**Table 2**

*Sludge characterisation*

Parameter	Value
Moisture content (%)	73.3
Total Solids (%)	26.7
Fixed Solids (%)	13.5
Organic matter (%)	29.9
Organic carbon (mg L <sup>-1</sup> )	21.4
Loss to fire (%)	89.1

Preparing test specimens using only sludge served as a standard for other analyses because the sludge only underwent water removal without any other physical or chemical changes (Carneiro, 2020). Five types of drying were chosen in the sludge preparation stage for



application to the partial replacement of cement. The first method used was sludge oven drying (SOD), in which the samples were dried in porcelain crucibles using a Scientific Laboratory Oven Series 9000, model 972, at 105°C for 24 h. This process creates a thin layer that increases the contact surface with the oven's heat, thereby enhancing the efficiency of the drying process.

Sludge sun drying has not yet been conducted, primarily because of the unfavourable climatic conditions in the region. The planned methodology involved placing the samples in a container and covering them with aluminium to prevent external humidity. The samples will then be exposed to the sun for approximately 7 to 15 days, with additional care taken to retrieve them during the night to avoid exposure to dew or rain.

Incineration was selected as the technique expected to yield the best results, and two burning techniques were employed to eliminate organic matter from the sludge effectively. The first technique (INC-1) involved a two-step process: first, the sludge was pre-dried at 105°C for 24 h and then fired in a muffle furnace at 300°C for 0.5 hours, followed by a final firing at 900°C for 3 hours (Grabowski, 2020). The second technique (INC-2), proposed by Swarnalatha *et al.* (2008) adopts a temperature progression scale from 100°C to 800°C over a period of 8 h. This approach aimed to optimise the incineration process by gradually increasing the temperature to ensure the complete decomposition of organic matter.

For wet oxidation (SWO), typical operating conditions include temperatures ranging from 150 to 330°C and pressures from 1 to 22 MPa, using pure or atmospheric oxygen for 15 to 120 minutes. However, due to laboratory limitations, the samples in this study were prepared at a pressure of 2.02 bar (0.22 MPa) and a temperature of 121°C for 18 minutes using an autoclave. The samples were then oven-dried at 105°C for 24 h.

Mortar specimens (MS) were prepared using prismatic moulds of 40×40×160 mm, with each specimen weighing approximately 600 g (Figure 1).

### Figure 1

*Mould design for producing mortar specimens.*





For each concentration of sludge used, two MS were produced, with one sample used for testing after 7 days of curing, and the other for testing after 28 days of curing. The composition of standard MS was 54% sand, 14% distilled water, and 32% cement. The only difference for the production of MS-containing sludge was in the cement concentration, where the replacement was done with previously treated sludge at percentages of 3%, 5%, 7%, and 10%, totalling ten mortar specimens.

In a stainless-steel tank attached to a mechanical mixer, all components were added and mixed at low agitation (140 rpm around the shaft) for approximately 30 s, after the agitation speed was increased to a high speed (285 rpm) for an additional 30 s. Following this period, the stainless-steel tank was removed from the mixer, and using a spatula all materials present on the wall of the tank were transferred to the mixture.

To produce MS, the NBR 5738 standards were meticulously followed. The process involved applying cement paste to the moulds and then compacting it using a horizontal compactor. After production, the MS were left under the bench for a curing period of 48 hours (Figure 2). Subsequently, they were carefully removed from the moulds, transferred to a humidity chamber with a controlled humidity of 90%, and maintained at a temperature of 20-25°C.

**Figure 2**

*Mortar specimens after 48 hours on the bench.*



Three tests were conducted on the MS produced on days 7th and 28th of the concrete curing. These tests included flexural strength, compressive strength, and solubilisation tests.



In the compression test, a force of 4 cm was applied from the end of the specimens until the peak of the applied force was reached. The equipment used for the tests is shown in Fig. 3.

Figures 4 and 5 illustrate the final results of this test for each specimen for the 7th and 28th day of curing, respectively.

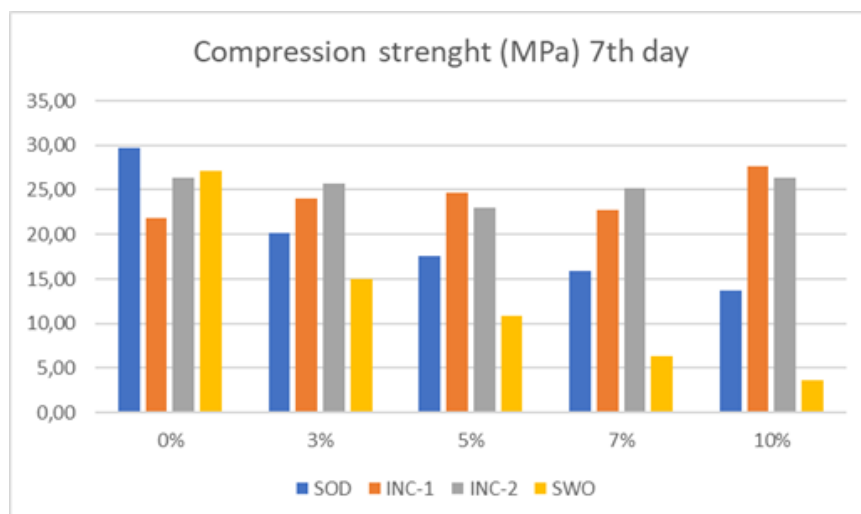
**Figure 3**

*Compressive strength testing equipment.*



**Figure 4**

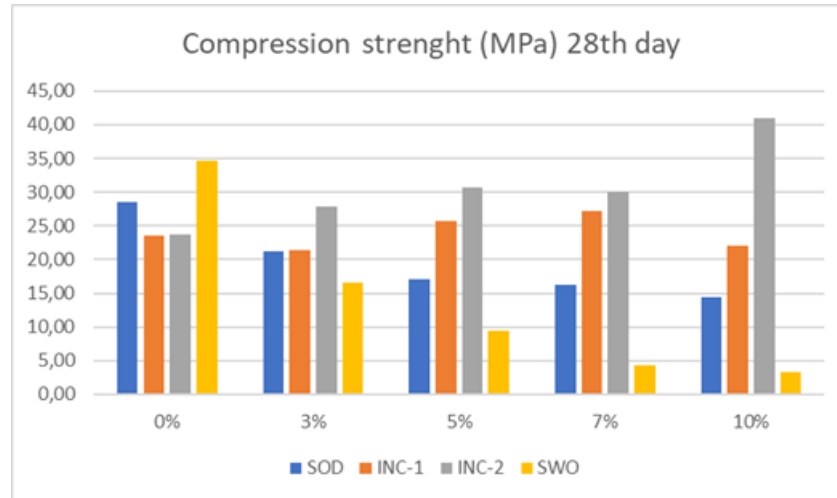
*Final resistance to compressive strength at 7-day.*





**Figure 5**

*Final resistance to compressive strength at 28-day.*



The mortar is classified by the compression resistance that the specimen supports on the 28th day of curing according to the EN 998-1 standard, as shown in Table 3 (European Standard, 2017).

**Table 3**

*Requirements for compressive strength applied to mortars.*

Category	Value (N mm <sup>-2</sup> )
CS I	0.4 - 2.5
CS II	1.5 - 5.0
CS III	3.5 - 7.5
CS IV	≥ 6

Source: European Standard (2017), EN 998-1:2016

We can define the best classification for mortars based on the presented results. According to Figure 5, it is evident that only one of the specimens exhibited a higher mechanical strength compared to the standard, namely, the specimen with 10% INC-2. This observation can be attributed to the slow-burning process, which effectively degrades all the organic matter in the residue. Notably, the dry sludge specimens demonstrated similar values and behaviours regarding the resistance drop profile and strength values. In contrast, the SWO specimens displayed the least favourable outcomes.

In the flexural test, a force was horizontally applied to the centre of the specimen until it ruptured, as depicted in Figure 6. The equipment used to test flexural strength is shown in Figure 7. These tests aimed to detect the effects of incorporating previously treated sludge into concrete mortar.



**Figure 6**

*Mortar specimens after the flexural test.*



**Figure 7**

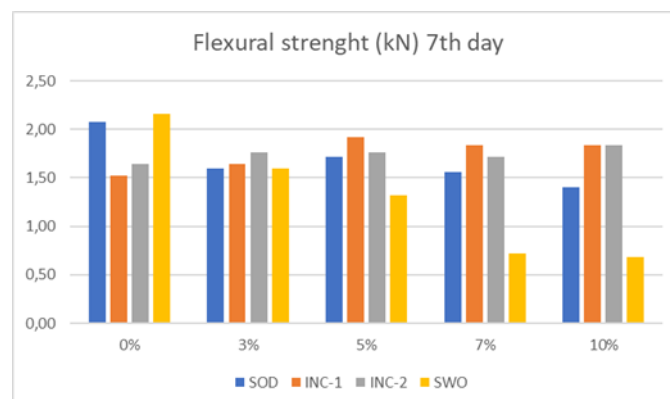
*Flexural strength testing equipment.*



Figures 8 and 9 show the final flexural strength of each specimen on the 7th and 28th day of curing, respectively.

**Figure 8**

*Flexural strength at 7-day.*



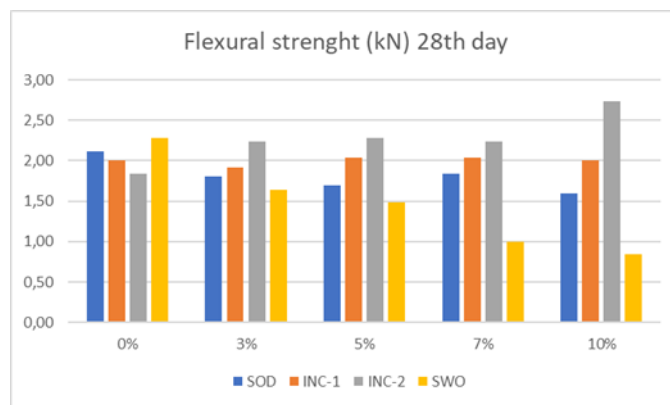


The flexural test showed a consistent behaviour similar to the compression test, where all sludge treatment methods (except SWO, which displayed different results) applied for cement replacement exhibited a positive correlation between the percentage of substitution and the corresponding increase in values.

According to Table 3, it is possible to categorise mortars. Mortars produced with 7% and 10% cement substitution by SWO particles belong to category CS III, whereas all treatments and replacement ratios fall under category CS IV.

**Figure 9**

*Flexural strength at 28-day.*



The water absorption test follows the LNEC E 394-1993 specification (LNEC, 1993), aiming to determine the percentage of water absorbed by a prismatic specimen after 48 hours of immersion in distilled water. The percentage of water absorption is given by equation (6):

$$A_i = \frac{m_2 - m_1}{m_1} * 100 \quad (6)$$

where:

$A_i$  percentage of water absorption

$m_1$  mass of the dry specimen, g

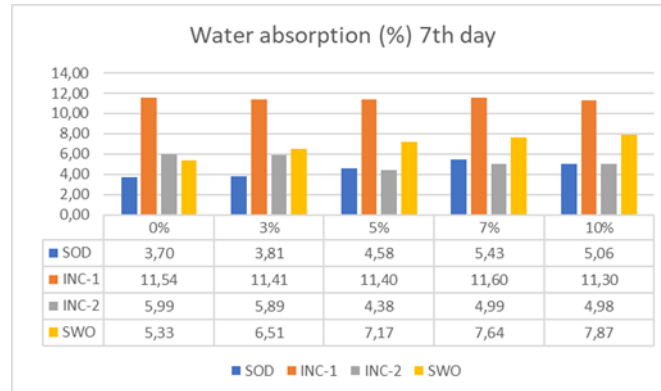
$m_2$  mass of the saturated specimen after 48 hours, g

The water absorption results for the 7th and 28th day of curing are presented in Figures 10 and 11, respectively.



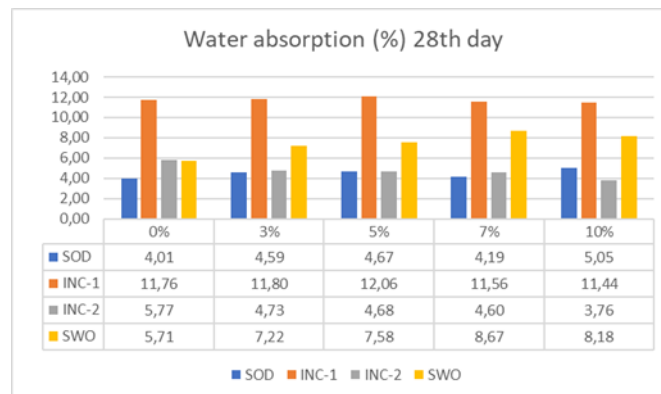
**Figure 10**

*Results of water absorption at 7-day.*



**Figure 11**

*Results of water absorption at 28-day.*



To confirm that the best result was obtained for the specimen with 10% INC-2 substitution with sludge, we can refer to the water absorption data presented in Figure 11, which shows that this specimen yielded the lowest result compared to the standard. Conversely, the tests conducted with SWO indicated higher water absorption values across the board, indicating poorer performance compared with the standard (0% substitution).

When comparing some of the results with the study "Eco-Friendly Cement Mortar with WWTP Sludge Recovery," conducted by Grabowski (2020), it was observed that three of the methods used by the author to reduce organic matter coincided with the principles adopted in this work, namely, SA (sludge ash), DS (oven dried sludge), and SWO (wet oxidised sludge), referred to as INC, SOD, and SWO, respectively.

The results obtained in this research demonstrated comparability and exhibited superiority compared to those reported in Fig. 12 (Grabowski, 2020). Although the previous research achieved its best result with a 7% substitution using the SA method, this study achieved

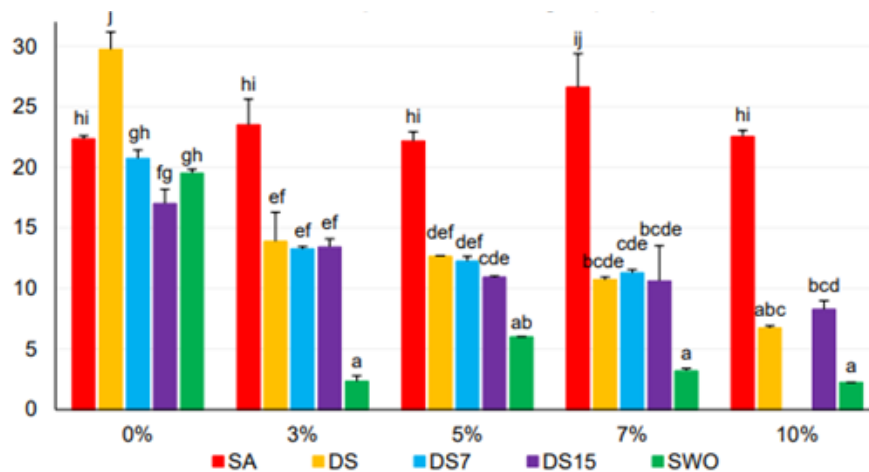


a 10% substitution using the same process, leading to a notable increase in the replacement of residues with cement and improved compression results. Additionally, the DS (SOD) method produced outcomes like those obtained in an earlier study. However, the SWO method showed less favourable results, making it the least successful approach in this study.

These results underscore the potential and effectiveness of the techniques applied in this study to enhance the properties of cement mortar by incorporating WWTP sludge from the tannery industry, surpassing the findings reported in a previous study involving WWTP sludge residue.

**Figure 12**

*Final resistance to compressive strength (MPa).*



Source: Grabowski (2020).

## 5 CONCLUSIONS

This study aimed to investigate the feasibility of incorporating WWTP sludge from the Tanner industry into mortar as a construction material and evaluate its mechanical and chemical properties. These results indicate that sludge can be used in small proportions without compromising the mechanical strength of the mortar. Substituting 10% cement with ash using the INC-2 incineration method improved mortar quality, exhibiting a higher compressive strength of 41 MPa and minimal water absorption (3.28%). Based on these encouraging findings, it can be concluded that using WWTP sludge in mortar is a viable and environmentally friendly option for civil construction.



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