

Article

Incorporation of Liquid WTP Sludge into Compacted Soil–Cement Mixtures

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Abstract: The sludge from water treatment plants (WTP) is a waste from the water process. This study evaluated the effect of incorporating water treatment plant (WTP) sludge, replacing the water used in compacted soil–cement mixtures. The materials were characterized by Scanning Electron Microscopy (SEM) associated with Energy Dispersive Spectroscopy (EDS) and Atomic Absorption Spectrometry (AAS). The soil, with the addition of liquid WTP sludge, presented an apparent dry specific weight (γ_d) of 1.77 $\text{gf}\cdot\text{cm}^{-3}$, the optimum moisture value in the compaction test of 15%, and the cement contents tested were 7, 11, and 14%. The specimens were molded using a WTP sludge–cement–soil mixture under the conditions mentioned above, and the simple compression results showed values within the range of 2.5 to 9.3 MPa, as specified by the Brazilian Technical Standard (NBR) 8491/2012. The hydraulic conductivity performed on the test specimen after 28 days of curing resulted in a coefficient (k) of $7.49 \times 10^{-9} \text{ cm}\cdot\text{s}^{-1}$, classified as little permeable. The result obtained from aluminum leaching was 0.12 $\text{mg}\cdot\text{L}^{-1}$, within the maximum limit allowed by NBR 10004/2004. Therefore, liquid WTP sludge has a significant capacity for incorporation into the compacted soil–cement mixture and the potential to manufacture ecological bricks, an alternative environmentally sustainable brick.

Keywords: brick; characterization; specimen; WTP sludge; technical standard



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1. Introduction

Water pollution is increasing along with water scarcity. It is estimated that globally, 2 billion people still lack access to clean water [1]. The United Nations has adopted 17 Sustainable Development Goals (SDGs), with targets set until 2030, including ensuring access to clean water and sanitation for all, outlined in SDG 6, as well as promoting sustainable infrastructure that requires fewer raw materials from natural sources, as stated in SDG 11.

To provide clean drinking water to the Brazilian population, it is necessary to treat and adjust the potability parameters required by health authorities at water treatment plants (WTPs). In conventional WTPs, typically, inorganic coagulant agents of metallic base are added, which contribute to the formation of flocs, aggregates of suspended impurities in the captured water. Aluminum polychloride (PAC) is an inorganic-based coagulant that is more efficient in removing suspended solids in water treatment [2]. These flocs settle due to differences in density, thus generating WTP sludge as a residue [3,4].

The generated sludge has approximately 96% water in its composition and is typically made up of silt, sand, clay, organic compounds, flocculated colloidal particles, coagulants, and auxiliary additives [5]. It is often disposed of in water bodies and sometimes directed

to landfills. When directly disposed of in water bodies, it degrades the environment due to heavy metals and other substances present in its composition, potentially causing fish mortality and siltation—the deposition of settleable solids [6]. Generally, the predominant elements in their combined forms present in WTP sludge are oxygen (O), aluminum (Al), silicon (Si), and iron (Fe), with a lesser predominance of metals such as calcium (Ca) [7–9]. One alternative to contain this contamination is solidification by mixing it with another material [7], such as in the manufacture of soil–cement ecological bricks.

The construction sector is the largest consumer of raw materials from natural resources, accounting for 40 to 50% of the total [10–12]. Therefore, it becomes essential to develop more sustainable practices, such as using process waste to manufacture new products, thus minimizing the use of renewable natural resources [13].

Studies with partial replacement of material such as cement with WTP sludge show promise with incorporation values of up to 20% [14].

Within this context, this study proposes the incorporation of liquid WTP sludge into the mixture of compacted soil and cement, replacing water, which is the essential natural resource for life.

2. Materials and Methods

2.1. Preparation of Materials for Mixing

A water treatment company located in the south of Brazil provided the waste WTP sludge used in the study, which uses polyaluminum chloride (PAC) as a coagulating agent. The soil was obtained from deposits in the same region. The stabilizing agent used was Portland cement CP II F32, which contains calcium silicate, aluminum, iron, calcium sulfate, carbonate filler, and pozzolan.

The materials were prepared for characterization. The WTP sludge was dehydrated, and the soil and cement were disaggregated. Disaggregation was carried out to achieve better homogenization among the materials in the soil–cement–sludge mixture and, consequently, better quality of the formed product.

2.2. Characterizations

2.2.1. Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS)

Scanning Electron Microscopy (SEM) was used for the morphological characterization of the dried solids of the WTP sludge, soil, cement, and test specimens (TSs). The analysis was performed using a Vega 3 LMU model scanning electron microscope manufactured by Tescan – Kohoutovice, Czech Republic). Initially, the sample received the deposition of a thin layer of gold (Au) and palladium (Pd) on its surface using a Quorum SC7620 model sputter coater. Additionally, the Energy Dispersive Spectroscopy (EDS) detector, the X-Act model manufactured by Oxford, was used for atom identification, expressed in their elemental forms. The SEM was coupled with EDS for this purpose.

2.2.2. Soil Characterization

The soil characterization is shown in Figure 1.

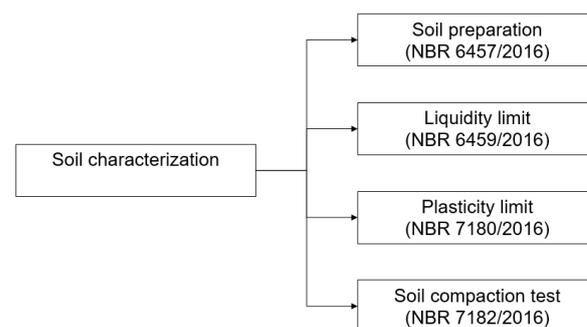


Figure 1. Steps of soil characterization.

For soil characterization, the procedures described in the respective Brazilian technical standards (NBR) were followed.

2.2.3. Soil Compaction Test with WTP Sludge

The Standard Compaction Test, or Proctor Test, was standardized by NBR 7182 (ABNT, 2016) [12].

From this test, a compaction curve was constructed using Microsoft Excel software (Office 365 Education) to determine the natural specific weight for the soil–WTP sludge mixture and obtain the optimal moisture content for maximum grain compaction.

2.3. Molding of Cylindrical Test Specimens

For molding the TS, a three-part cylindrical mold with dimensions of 5 cm in diameter and 10 cm in height was used. For the preparation of the mixture, soil (from soil preparation according to NBR 6457/2016) [15] and cement were initially mixed to achieve uniform homogenization. Subsequently, liquid WTP sludge was added while maintaining a constant moisture content to ensure a homogeneous mixture. Moisture directly influences the molding and extraction of the TS from the mold. Test specimens with moisture levels lower than estimated became more sensitive to demolding. The molding of the TS was divided into three compaction stages, and the TS was molded using a manual mechanical press.

2.4. Hydraulic Conductivity Test on Soil–WTP Sludge–Cement Mixture

Following the description of the soil permeability test outlined in NBR 14545/2021 [16], the experiment was conducted in a hollow cylinder belonging to the permeameter set, where the TS of the soil–WTP sludge–cement mixture was molded. After adjusting the TS and the set, the seal was checked, and the sample was saturated with distilled water in an upward manner.

For the soil–WTP sludge–cement mixture hardened at 28 days of age, the TSs were subjected to a hydraulic load to determine their hydraulic conductivity coefficient (k), following the procedure described in NBR 14545/2021 [16].

Analysis of Leachate

From the hydraulic conductivity test on the compacted soil–WTP sludge–cement sample, through the flow of distilled water in the specimen, leachate was obtained. This leachate material was subjected to aluminum content analysis since aluminum polychloride is the coagulating agent used in the WTP where the sludge was collected. The analysis was based on the parameter described in NBR 10004 [17], which establishes the maximum solubilization limit for aluminum, to validate whether incorporating sludge into the mixture would generate environmental impacts such as aluminum leaching.

2.5. Uniaxial Compression Test on Cylindrical Test Specimens

The test was conducted following the steps described in NBR 8492/2012 Soil–Cement Brick—Dimensional analysis, determination of compressive strength, and water absorption—Test method [4].

To analyze whether it meets the requirement that the tested sample must not have an average uniaxial compressive strength value lower than 2.0 MPa or an individual value lower than 1.7 MPa [18], a curing time of 28 days was chosen. Then, the TSs were immersed and subsequently subjected to the uniaxial compression test as required by the standard. The press used was a Shimadzu brand (Japan), model AGI Autograph AGS, with a load cell of 10 kN. However, using a press with a load cell of 1000 kN was also necessary for the TSs that were not ruptured in the 10 kN press.

3. Results and Discussion

3.1. Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS)

The morphology of the WTP sludge, soil, and cement is depicted in Figure 2.

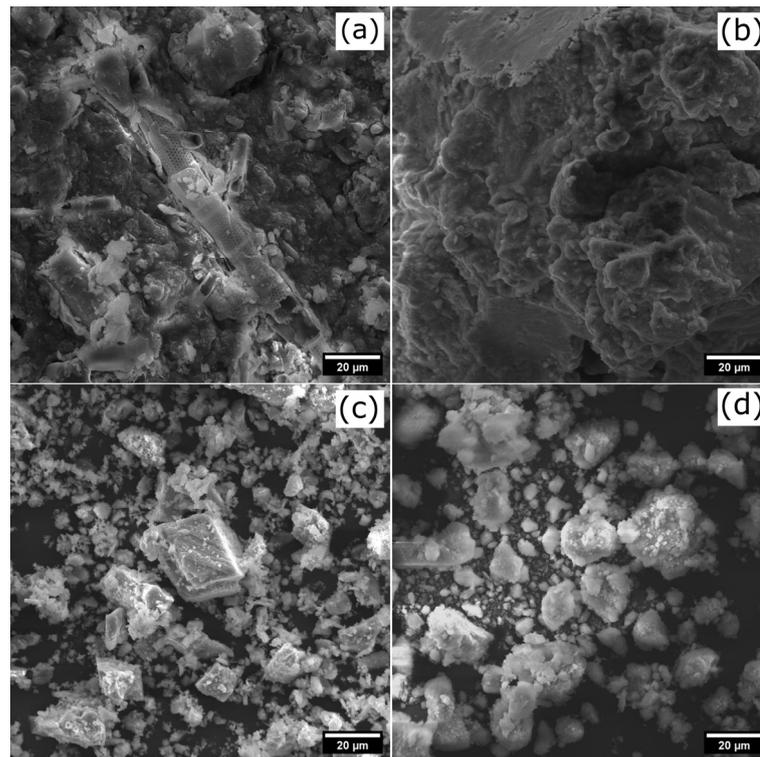


Figure 2. SEM WTP sludge (a), soil (b), cement (c), and specimen (d).

In relation to the structural mapping of the samples, in Figure 2a, a heterogeneous aspect with different structures, an irregular amorphous and random pattern of compounds of different shapes distributed in the same sample, is observed, as well as an elongated structure in the center of the region. In Figure 2b, particles with slightly more regular shapes are observed, albeit with different sizes distributed throughout the sample, whereas in Figure 2c, a larger particle is noted in the central region compared to the others distributed, and in Figure 2d, particles closer together with small empty spaces between them are observed.

Following the SEM analysis, the sludge sample was mapped, and each elemental atom was identified by EDS. The results were obtained for the same analyzed sample, with readings taken from two surface points using the equipment.

Each color represents the atomic predominance of the elements depicted in Figure 3. In Figure 3a, the atomic segregation by color allows for mapping the distribution of atoms in the sample. Silicon atoms concentrate in larger areas, while aluminum is unevenly dispersed throughout the sample. The predominance of oxygen occurs due to the presence of oxides and other molecular combinations. In Figure 3b, clustering with larger areas for carbon atoms can be observed, likely due to the content of organics present in the sample. Silicon atoms are localized in concentrated groups in different regions of the sample, while oxygen, carbon, and aluminum atoms have shown uneven dispersion, with their presence in almost every part of the sample. In Figure 3c, a predominance of calcium and silicon atoms was noted. In Figure 3d, a more filled region is observed, with small spaces between the materials.

Table 1 expresses the average values in percentage of the elements identified by EDS in the respective samples.

In the research by Anjum et al. (2017) [19], the percentage of mass of combined atoms in the form of oxides present in the sludge was quantified as 30.86% aluminum, 28.70% silicon, and 10.72% iron. Rodrigues and Holanda conducted a study that shows similar results, with the combined atomic elucidation in the form of oxides with aluminum at 31.18%, silicon at 29.59%, and iron at 21.10% [6]. Meanwhile, the WTP sludge characterized

by Nguyen et al. (2023) [5] contained in its composition 44.16% oxygen, 16.82% silicon, and 30.26% aluminum.

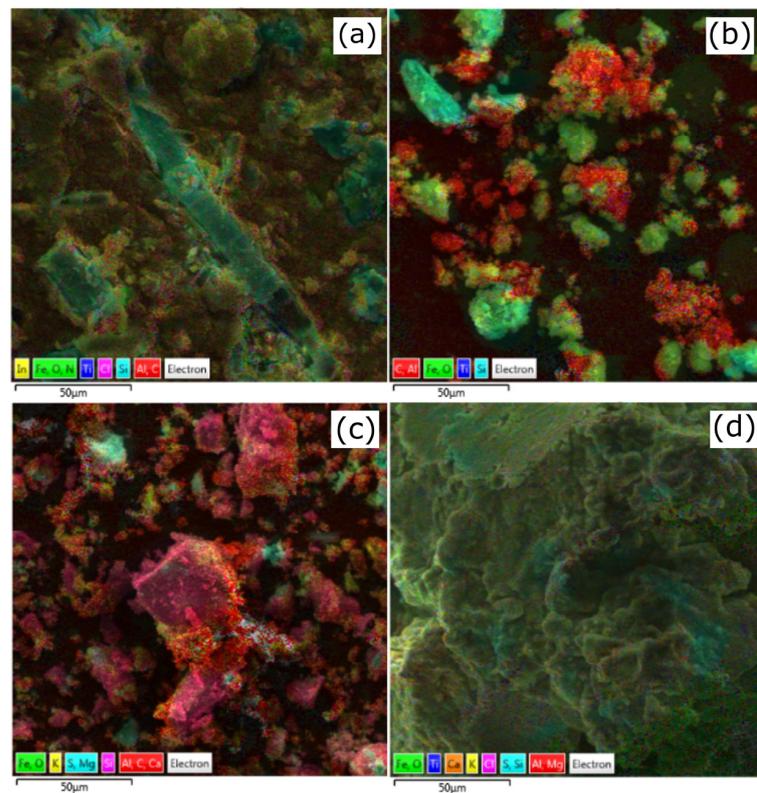


Figure 3. EDS of the samples from WTP sludge (a), soil (b), cement (c), and test specimen (d).

Table 1. EDS characterization data.

Atoms	WTP Sludge (%)	Soil (%)	Cement (%)	Test Specimen (%)
O	53.4	35	72.6	68.8
Si	19.6	3.2	5.8	9.9
Al	18.4	4.0	1.3	1.1
Fe	5.2	1.8	0.9	2.0
Ca	0.1	-	35.2	3.7
C	-	64.8	-	-

The variability in the chemical predominance of WTP sludge composition is due to several factors that interfere with its formation, from the quality of the captured water to larger volumes of treated water where greater amounts of coagulant are used.

The carbon content in the soil of 64.8% reflected the presence of organic matter in the soil sample, which, when preparing the sample for SEM and EDS, may concentrate in a specific part of the sample.

3.2. Soil Characterization

Initially, soil characterization tests were performed to classify the materials. Soil sample preparation followed the procedures described in NBR 6457/2016, “Soil Samples—Preparation for Compaction Tests and Characterization Tests.” [15]. For the determination of the liquid limit, the procedures established in NBR 6459/2016, “Soil—Determination of Liquid Limit”, were adopted [10]. The determination of the soil’s plastic limit was conducted according to the test procedure contained in NBR 7180/2016, “Soil—Plastic Limit” [11]. For the soil compaction test with WTP sludge, the procedures described in NBR 7182/2016, “Soil—Compaction Tests”, were followed [12]. As for the organic matter content,

this analysis was performed according to the procedures stipulated in NBR 13600/2022, “Soil—Determination of Organic Matter Content by Burning at 440 °C” [6]. The results expressed in Table 2 were obtained.

Table 2. Results for soil consistency and organic content.

Sample	
Liquid limit (%)	27
Plastic limit (%)	23
Plasticity index (%)	4
Organic matter content in soil (%)	5.45

The soil characterization results using the sludge in the mixture were close to those obtained in the study [20], where the same soil with water obtained a plasticity index of 6% (a liquidity limit of 28%) and organic matter content of 2.60%. In terms of consistency, the variation presented is considered small because of the variability arising from the testing procedure and the nature of the material evaluated.

The general requirements contained in NBR 10833/2012, “Manufacture of Soil–Cement Bricks and Blocks Using Manual or Hydraulic Press—Procedure”, determine the essential characteristics for the soil used in the production of soil–cement bricks [21], with a liquid limit $\leq 45\%$ and a plasticity index $\leq 18\%$. Evaluating the results obtained in the moistening of this soil with water and with sludge, this soil–WTP sludge–cement mixture met the requirements.

3.3. Soil Compaction Test

The dry bulk specific weight (γ_d) and the optimum moisture content (w_{opt}) were obtained through the soil compaction test with the addition of liquid WTP sludge, replacing water. The compaction curve was plotted as illustrated in Figure 4.

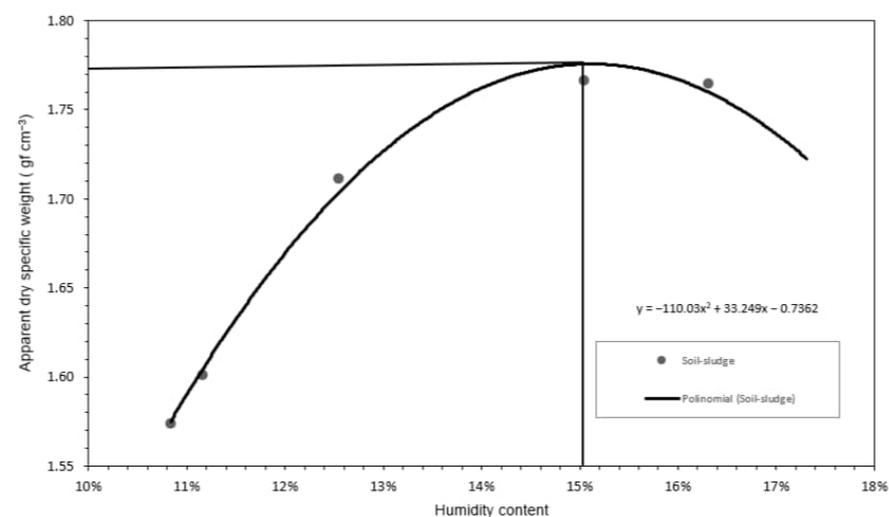


Figure 4. Soil with liquid sludge WTP compaction curve.

The results from the compaction test showed an optimum moisture content for maximum soil grain compaction of 15% and a dry bulk specific weight (γ_d) of 1.77 g cm⁻³ by switching water for WTP sludge. The use of sludge replacing water in the compaction tests caused a slight increase in the optimum moisture content and a decrease in the maximum apparent dry specific weight, when compared to the values obtained for the same soil in the study [20], which resulted in a dry apparent specific weight of 1.82 gf/cm³ and an optimum moisture content of 14%.

According to γ_d , it was possible to measure the quantities of materials related to each value of dry bulk specific weight, analyzing the weights specified below. Table 3 reports the respective values obtained for each mixture proportion utilized on the tested specimens.

Table 3. Soil–sludge–cement mixture proportions.

γ_d —Cement Content %		Mixture Proportions
γ_{d1} —1.65 gf cm ⁻³		Soil (g)—Sludge (g)—Cement (g)
γ_{d1} —7%		331.42—47.42—24.94
γ_{d1} —11%		317.17—47.42—39.20
γ_{d1} —14%		306.48—47.42—49.89
γ_{d2} —1.70 gf cm ⁻³		Soil (g)—Sludge (g)—Cement (g)
γ_{d2} —7%		341.47—48.86—25.70
γ_{d2} —11%		326.78—48.86—40.38
γ_{d2} —14%		315.76—48.86—51.40
γ_{d3} —1.75 gf cm ⁻³		Soil (g)—Sludge (g)—Cement (g)
γ_{d3} —7%		351.51—50.29—26.45
γ_{d3} —11%		317.17—50.29—41.57
γ_{d3} —14%		306.48—50.29—52.91

3.4. Cylindrical Test Specimens with the Addition of Liquid WTP Sludge in the Mixture

The TSs were molded based on the values of dry bulk specific weight contained in Table 4.

Table 4. Initial data for the preparation of cylindrical test specimens.

Data	
γ_{d1} (gf cm ⁻³)	1.65
γ_{d2} (gf cm ⁻³)	1.70
γ_{d3} (gf cm ⁻³)	1.75
h obtained (%)	15

Three mixtures were selected for the experiment, with the liquid WTP sludge at the optimum moisture content (h) represented in relation to the calculated proportions. Figure 5 illustrates a TS of a compacted soil–WTP sludge–cement mixture.

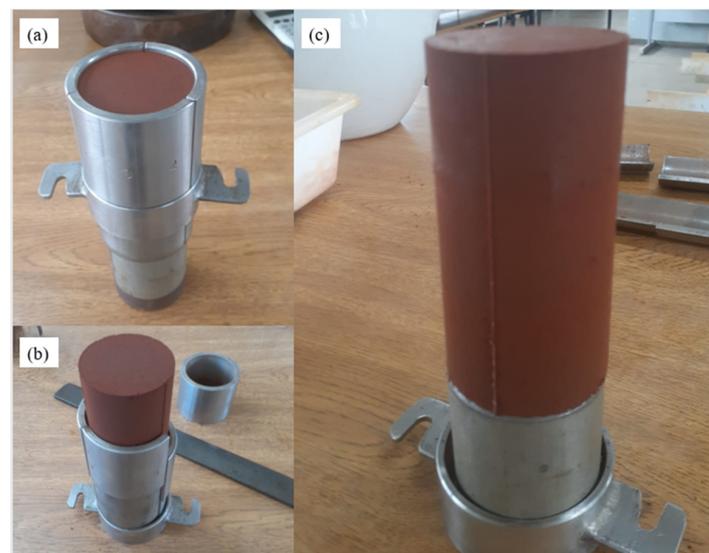


Figure 5. Test specimen molded under ideal conditions. Test specimen molded by compression in the mold (a), extraction of the test specimen from the mold (b), perfectly extracted TS (c).

3.5. Uniaxial Compression Test

Following the procedures of standard 8491 [17], the uniaxial compression test was conducted using equipment with a load cell of 10 kN and 1000 kN. Table 5 expresses the results for the effects of incorporating liquid WTP sludge into the mixture.

Table 5. Average values obtained from the axial uniaxial compression test on test specimens.

Bulk Specific Gravity—Cement Content—Number of Test Specimens	Uniaxial Compressive Strength (MPa)
γ_{d1-7-3}	2.60
$\gamma_{d1-11-3}$	3.65
$\gamma_{d1-14-3}$	4.85
γ_{d2-7-3}	3.68
$\gamma_{d2-11-3}$	3.15
$\gamma_{d2-14-3}$	5.40
γ_{d3-7-3}	4.61
$\gamma_{d3-11-3}$	8.75
$\gamma_{d3-14-3}$	8.68

According to NBR 8492 [4], we can observe that the tested sample must not have average compressive strength values lower than 2.0 MPa, with a minimum age of seven days. Therefore, any mixture among those tested meets the minimum requirement. Uniaxial compression strength results for mixtures using sludge were two to three times higher than those found by [20] in soil–cement mixtures with water, cured for 14 days. However, it is worth highlighting that this result was expected, considering that the curing time in the present research was 28 days and the fact that the samples had a greater quantity of liquid in the mixture due to the optimum moisture content resulting from the compaction test, generating greater water availability for cement hydration reactions.

In the experiment conducted by [9], they varied the incorporation of dehydrated WTP sludge in the compacted soil–cement mixture. For 1% incorporation in the TS, the compressive strength was 6.02 MPa; for 3%, it was 5.71 MPa; for 7%, it was 5.17 MPa; for 15%, it was 4.75 MPa; and for 20% incorporation of sludge, it was 3.95 MPa. Thus, they concluded that under the tested conditions, the ideal value for dry bulk specific weight (γ_d) was 1.75 g cm^{-3} as a parameter for the mixture. Comparing the results obtained in this research, with the γ_{d3} mixture parameter of 1.75 g cm^{-3} , the values obtained were 4.61 MPa for 7% of added cement and 8.68 MPa for 14% of added cement.

With the data obtained from this test on the specimens, it is observed that the compression results for all selected mixes did not fall below the minimum limit required for the material's application in structures, thus validating this sustainable alternative of incorporating liquid WTP sludge into compacted soil–cement mixtures.

Disposal of process residues, such as tannery sludge, in a cement mixture, showed that an influence on the mechanical resistance of the material is related to the number of days of curing, obtaining 45 MPa with a 7-day-old test specimen, and predictions of 71.3 MPa for a specimen with 28 days of curing [22].

Regarding the immobilization of WTP sludge constituents in cement, in [14], the authors obtained good results with the incorporation of a partial replacement of 20% in cement.

With the addition of variables of up to 20% of WTP sludge in cement, the mechanical resistance values reached from 25.4 MPa to 39.4%. They concluded that longer curing time influences the mechanical resistance of the material; however, traces above 20% of WTP sludge negatively influence mechanical resistance values [23].

3.6. Hydraulic Conductivity Test on Soil–WTP Sludge–Cement Mixture

The soil permeability allows the flow of water through it and is measured by the coefficient of hydraulic conductivity (k). The soil's particle size distribution directly affects

the coefficient of hydraulic conductivity, with larger grains resulting in a higher permeability coefficient, common in sandy soils, while smaller particles lead to a lower coefficient, typical of clayey soils, as depicted in Figure 6 [7,19,24].

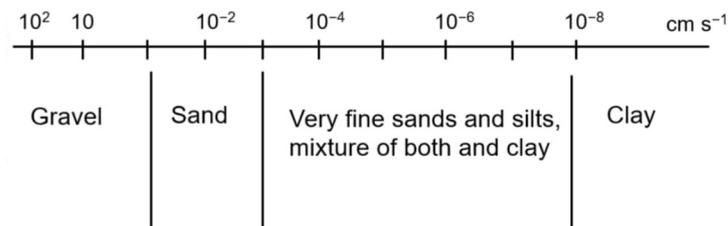


Figure 6. Scale for typical hydraulic conductivity values in soil.

The permeability coefficient (k) of the hardened mixture, composed of soil, WTP sludge, and cement, at 28 days old and with molding properties γ_d 2-7 (dry bulk density of 1.70 g cm^{-3} and 7% cement content), was $7.49 \times 10^{-9} \text{ cm s}^{-1}$. However, ref. [13] obtained a hydraulic conductivity (k) value of $4.0 \times 10^{-8} \text{ cm s}^{-1}$ for the soil and $8.0 \times 10^{-8} \text{ cm s}^{-1}$ for the mixture with WTP sludge and soil. They concluded that the addition of WTP sludge to the soil mixture causes a decrease in the k coefficient.

Morselli et al. (2022) [25], when conducting the test on compacted material of 100% dehydrated WTP sludge, obtained a k value equal to $1.64 \times 10^{-4} \text{ cm s}^{-1}$.

Compared to the studies [25,26], the hydraulic conductivity test on the compacted mixture of soil and cement hydrated with liquid WTP sludge at 28 days old and 5 days of saturation resulted in $7.49 \times 10^{-9} \text{ cm s}^{-1}$, indicating low permeability characteristics for the mixture.

Thus, it is validated that the effect of incorporating WTP sludge into the mixture does not accelerate water infiltration when saturation occurs in the resulting product.

Analysis of Leachate Obtained from the Permeability Test of the Mixture

The leachate collection was derived from soil percolation [1] during the permeability test and then realized by aluminum content analysis by atomic absorption spectroscopy (AAS). The result was compared with the maximum limit parameter in the extract contained in Annex G of NBR 10004 [17]. About the model proposed, estimated in the standard as 0.20 mg L^{-1} , being the maximum acceptable limit, the leachate content showed 0.12 mg L^{-1} , staying below the maximum allowed upper limit. Roque et al. (2022) [26], conducting a study on adding WTP sludge to mixtures with soil for geotechnical purposes, obtained a leached aluminum content value of 0.25 mg L^{-1} .

Based on our results, taking into consideration a comparison with other studies and the standard, the incorporation of WTP sludge into the mixture does not result in contaminations carried in aqueous media by the predominant aluminum metal in its combined structural form with the environment.

4. Conclusions

- Incorporating WTP sludge into a compacted soil–cement mixture has proven to be a suitable disposal method for this waste, replacing water in the mix.
- The chemical characterization of WTP sludge about the materials identified atomic coincidences in the structural constituents of the materials.
- All selected mixes exhibited excellent strength to the applied load, on simple compressive strength, with the highest load supported being 8.63 MPa for mix γ_d 3 (1.75 g cm^{-3}).
- The determination of the permeability coefficient (k) showed that the mixture has low permeability properties, resulting in difficulties in water infiltration over time.
- The leached aluminum content appears promising in environmental terms, as its concentration was found to be below the maximum limit allowed by Brazilian technical standards.

Therefore, the incorporation of liquid WTP sludge as a substitute for water in compacted soil–cement mixtures has been validated through analyses and tests for application in eco-friendly brick production. It has proven to be an excellent environmental alternative for disposing of this waste, mitigating the impact caused by its disposal in the environment. The effects on the mixture were all significant, as the results of the analyses and tests met the required standards.

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Data Availability Statement: The data will be made available upon request.

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