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Physicochemical and Mechanical Characterization of Phosphate Sludge for Sustainable Concrete-Based Composites

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Abstract

The negative impact of industrial waste on the environment can be minimized by its recovery into technical applications. This study aims to characterize the phosphate sludge taken from the Metlaoui mine in Tunisia and to examine the influence of different industrial by-products, including gypsum, waste paper and wood, on their properties. The study of these effects helps to understand why construction material manufacturers consider these waste-derived components as valuable raw materials. To evaluate the chemical, mineralogical, and mechanical composition of materials, X-ray diffraction (XRD), X-ray fluorescence (XRF) and mechanical strength tests were performed. The study also examines the role of additional waste in the phase crystallization mechanism, including the formation of new phases and the suppression of others. The results indicate that the mechanical properties of the sludge are improving each time there is an integration of additional waste. Therefore, the resulting composite material has potential for application in construction as a durable and environmentally responsible material.

Keywords: Phosphate Sludge, Industrial Wastes, Fluorescent X-rays, XRD,Compression test.

Abbreviations

E	Entrance mud
S	Outlet mud

1. Introduction

For industry, managing industrial waste is a challenge, it also applies to the construction sector [1]. The latter seeks to have less impact on the environment. For this, it uses innovative materials. Among these wastes, phosphate sludge from the extraction and processing of phosphate ores is a resource with a low utilization rate that has significant potential for the production of composite materials [2]. However, although it is available and has interesting physico-chemical properties, its use in composites has not been studied. The objective of this study is to fill this gap by describing phosphate sludge and assessing its integration with various industrial wastes such as gypsum, paper, and wood, to develop a material with outstanding performance and the ability to meet the specific requirements of the construction sector.

This research examines how phosphate sludge can be transformed into a solid composite material by adding specific components. Specifically, it focuses on improving its mechanical and chemical performance [3]. By studying the mechanisms of crystallization and the interactions between the components, we note that the different phases directly influence the strength and durability of the material. This study is based on advanced techniques such as diffraction (XRD) and fluorescence (XRF), which allow the mineralogical and chemical characterization of the materials studied. Strength tests are also performed. This is used to check if the material can be used for construction.

The addition of phosphate sludge to a composite material is a scientific approach and part of a broader strategy for the responsible management of industrial waste. At the moment, phosphate sludge is stored in large tanks. This poses risks to the environment: contamination of groundwater and weakening of soil in case of heavy rains. Recycling [4] this waste has two advantages: it neutralizes impacts and reduces the consumption of virgin raw materials in construction. This helps to move towards a circular economy. By mixing industrial residues such as gypsum, wood, and paper with phosphate sludge, a new material with improved properties is obtained. Gypsum [5] insulates from noise and cold and makes the structure more solid. Waste wood, a bio-based material [6], is increasingly used in construction. They improve absorption and acoustic resistance. As with waste paper, its

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use reduces carbon traces and optimizes the durability of the composite.

Previous research has focused on phosphate sludge as an additive in cement production[7]. This approach is different and innovative. It consists of creating a robust and high-performance composite. This opens up new possibilities for the use of this material in different applications, such as structural strengthening, thermal, and acoustic performance improvement.

This study shows that mixing phosphate sludge with other industrial waste is good. This allows for better management of mining industrial waste and its transformation into long-lasting building materials. This research is important for science, but also for society. Although resources must be used more efficiently, this helps protect the environment and change production habits for a more environmentally friendly industry.

To convert phosphate sludge into composite materials, a detailed process must be followed. Before using, you should clean them and make sure they are not too wet. This allows better mixing with cement. The sludge is then dried and ground to be mixed with hydraulic or organic binders. Sometimes heat or chemicals are used to make it stick better. To do it right, you have to measure the mud and aggregates well.

2. Materials and Methods

2.1. *Materials*

In this section, the used methodology to obtain materials is presented.

2.1.1. Phosphate Sludge and its preparation for the experiences

The Phosphate Company (CPG) process is essential for producing phosphate sludge[8]. To obtain it, the rock is extracted, crushed, washed, and floated. This allows the phosphate to be recovered and the impurities to be removed. But when washing, there is a large amount of sludge to be treated. Therefore, solutions must be found to manage waste without polluting and recover water in the best possible way. Sludge is water, fine particles, and soluble matter. It is treated or stored in tanks. To collect water, CPG uses flocculation and densification techniques. This results in two types of sludge:

- Input sludge (E): recovered from settling ponds.
- Sludge (S): removed after flocculant removal, facilitated by drainage channels in sedimentation valleys.

To improve sludge clustering and water recovery[9], coagulation-flocculation techniques have been studied with bioflocculants such as Slim Floc 150[10], aluminum sulfate, and cactus powder. After separation, the sludge is dehydrated (Fig. 1-a), dried at 100°C for 24 to 48 hours (Fig. 1-b), and then crushed (Fig. 1-c) into fine powder (Fig. 1-d). This allows them to be physico-chemically characterized and used in concrete. In addition, phosphogypsum, a by-product, is reused to produce phosphoric acid[11], which promotes industrial sustainability.

2.1.2. Strength test

A 40 mm stop cubic shape is selected for the compression test in order to accommodate the model TBTWC-1000A compression machine, which has a deformation rate of 2 mm/min. At room temperature, the measurements were taken. After the drying process, the cubic concrete samples exhibited noticeable imperfections. In both cases, the mud inlet (Fig. 2-a) and mud exit (Fig. 2-b) sections experienced structural damage, leading to visible crushing. The incorporation of gypsum and other waste materials showed minimal influence on the mechanical integrity of the concrete during analysis. However, the overall structural integrity of the composite material was significantly affected, as illustrated in Fig. 2-c, suggesting a notable impact on its mechanical performance.

Figure 1. The raw material preparation process

Figure 2. The samples for the compressive strength test

The machine's core software has automatically generated all of the characters based on force, deformation, and extension. The following formula was used to determine the compressive strength (σ).

$$\sigma = \frac{F}{A} \tag{1}$$

where A is the concrete sample's cross-section, measured in millimeters squared (mm^2), F is the force the concrete sample supported during the compression test, measured in newtons (N), and σ is the compressive strength in pascals (Pa) or megapascals (MPa).

2.1.3. Gypsum

The gypsum from this region is mainly composed of calcium sulfate hemihydrate [12]. This gypsum was analyzed using the XRD technique and processed with X' Pert high ScorePlus software [13]. The utilized gypsum must adhere to the specifications outlined in EN 13279-1 [14].

2.1.4. Additives and Wastes

Recycled materials have gained popularity in building construction as a means of lowering trash accumulation and conserving natural resources. There are two common categories of waste:

Shredded egg trays and other paper waste from recycled paper goods are analyzed using the X-ray diffraction (XRD) technique (Fig. 3-a). The main components of this waste are organic materials like cellulose and inorganic minerals like kaolinite and calcite [15]. Wood chips, sawdust, bark, and scraps are the types of wood waste produced by wood processing businesses such sawmills, carpentry shops, and furniture factories (Fig. 3-b) [16].

The egg carton and the carpentry business provided the paper and wood waste used in this sculpture, respectively. Building materials can be made by mixing these common waste items with gypsum and phosphate sludge.

Figure 3. Wastes used: (a) Paper waste, (b) Wood waste

After analysis, it was found that in order to recycle the dried phosphate sludge, fresh materials were needed. Other waste types must be added in order to produce a new, practical building material. 90% phosphate waste, 5% gypsum, and varying proportions of paper and wood waste make up the ideal mixture. To obtain the best dosages for our test samples, these ingredients were not picked at random but rather deliberately, taking into account the effects of each waste during the building phase. The amount of distilled water utilized in the mixture is increased by the remaining 5% of waste, as shown in the table below.

Table 1. Distribution of wood and paper waste as a percentage.

Samples	Wood waste(%)	Paper waste(%)	Distilled water
E1/S1	1	1	+3%
E2/S2	2	1	+2%
E3/S3	1	3	+1%
E4/S4	2	3	0

2.2. Sample preparation

For the purpose of chemical analysis, four different types of samples were prepared: a dried sludge sample from the decanter inlet (E), a second sample consisting of the same sludge mixed with gypsum, wood waste, and paper waste, in order to examine the influence of these elements on the chemical components present in the sludge, a third sample of dried sludge from the decanter outlet (S), and a fourth sample that was mixed with the other elements. The specifics of the samples used, including their composition, are presented in Table 1. The flocculent is what distinguishes the inlet end of the decanter from the outlet slurry. The presence of a flocculant has an impact on the concentration of sludge.

This was calculated as soon as the sludge was removed, based on the concentration formula:

$$C = \frac{m}{V_{eau}} \ avec \ V_{eau} = 1l \tag{2}$$

The anionic Slim Floc flocculent, which is used in drinking water purification, industrial process water management, wastewater treatment, and eutrophication control, is employed in this study [17]. Inlet and output sludge concentrations used are 80 g/l and 235 g/l, respectively.

3. Experimental methods

3.1. Chemical analysis

To evaluate the composition of building materials, chemical analyses are crucial [18]. This guarantees that the materials fulfill the necessary safety and quality requirements.

The X-ray fluorescence method (XRF) is one of the chemical tests [19]. In the current work, this methodology is applied to ascertain the constituent elements and their concentrations. Ensuring quality and adherence to construction standards is crucial.

3.2. X-ray diffraction

To analyze the crystallinity of all samples, an automated multi-function X-ray diffractometer [20] (PANalytical X'Pert Pro) equipped with a Cu Kα radiation source was used. Around 25 °C was the room temperature when the observations were taken. The range of angle 2θ was 20–45. The phases present, their textures, and the residual stresses can be ascertained by interpreting the measurements using databases (ICDD PDF-4+ and COD) and software (DIFFRAC EVA, TOPAS, High Score, etc.) [21]. By measuring the diffracted beam's intensity in relation to the incident beam's 2Θ deviation angle, diffractograms can be created.

3.3. Compression test

For the compression test, a cubic or cylindrical specimen is required. In our case, a 40 mmx40 mm x40 mm) cubic sample is used in the TBTWC-compression machine mode

A mixture of 183g of mixing mud and 36 ml of distilled water is used to create the sample. Following that,

this mixture is left at ambient temperature (25°C) for 28 days (Fig.5). After drying, the sample was scraped to create the required cubic shape, measuring 40*40*40 mm.

4. Results and discussion

4.1. Chemical characterization

The results of the XRF analysis in this study are consistent with previous research on the composition of phosphate sludge and its use as building materials. Studies on the recycling of industrial waste have shown similar oxide compositions, especially in terms of SiO_2 concentrations, CaO and P_2O_5. A study on the use of phosphate sludge in cementitious materials revealed SiO levels ranging from 28% to 35%, which is consistent with the values observed in our samples (31.43% to 32.64%). In addition, the amount of calcium in our study is similar to the results found for cement-based applications. In these cases, calcium plays an important role in chemical reactions and the solidity of materials.

Figure 4. The TBTWC-compression machine model1000A

Figure 5. Specimen process of preparation

Table 2. Samples chemical characteristics

Samples	MgO	Al_2O_3	SiO_2	K_2O	CaO
E1	0.26	0.02	31.42	0.52	22.67
E2	0.28	0.02	28.71	0.47	23.30
S1	0.25	0.06	32.64	0.54	25.41
S2	0.24	0.02	26.25	0.42	25.11

Adding phosphate sludge to cement improves the hydration reaction and properties of concrete. Tests show that phosphate sludge makes the structure stronger, increases chemical stability, and can replace virgin limestone. This reduces carbon emissions during cement manufacturing.

Industries can use phosphate sludge-based concrete composites. In construction, the combination of phosphate sludge with industrial waste can be integrated into light aggregates, concrete, and cement. This makes the structure more resistant and improves its durability. It is resistant to chemicals and heat, making it ideal for ceramic and refractory materials. In environmental engineering and waste management, it can be used to stabilize landfill sites and waste containment solutions.

4.2. Mineralogical Analysis

Mineralogical analysis further supports the compatibility of phosphate sludge with cement formulations. Previous research has identified C_2S and C_3S phases as essential components for mechanical performance, with values typically ranging between 3% and 6%, which matches the proportions found in our samples. The presence of calcite (7%–23%) in ourstudy is also consistent with literature emphasizing its role in improving durability and chemical stability.

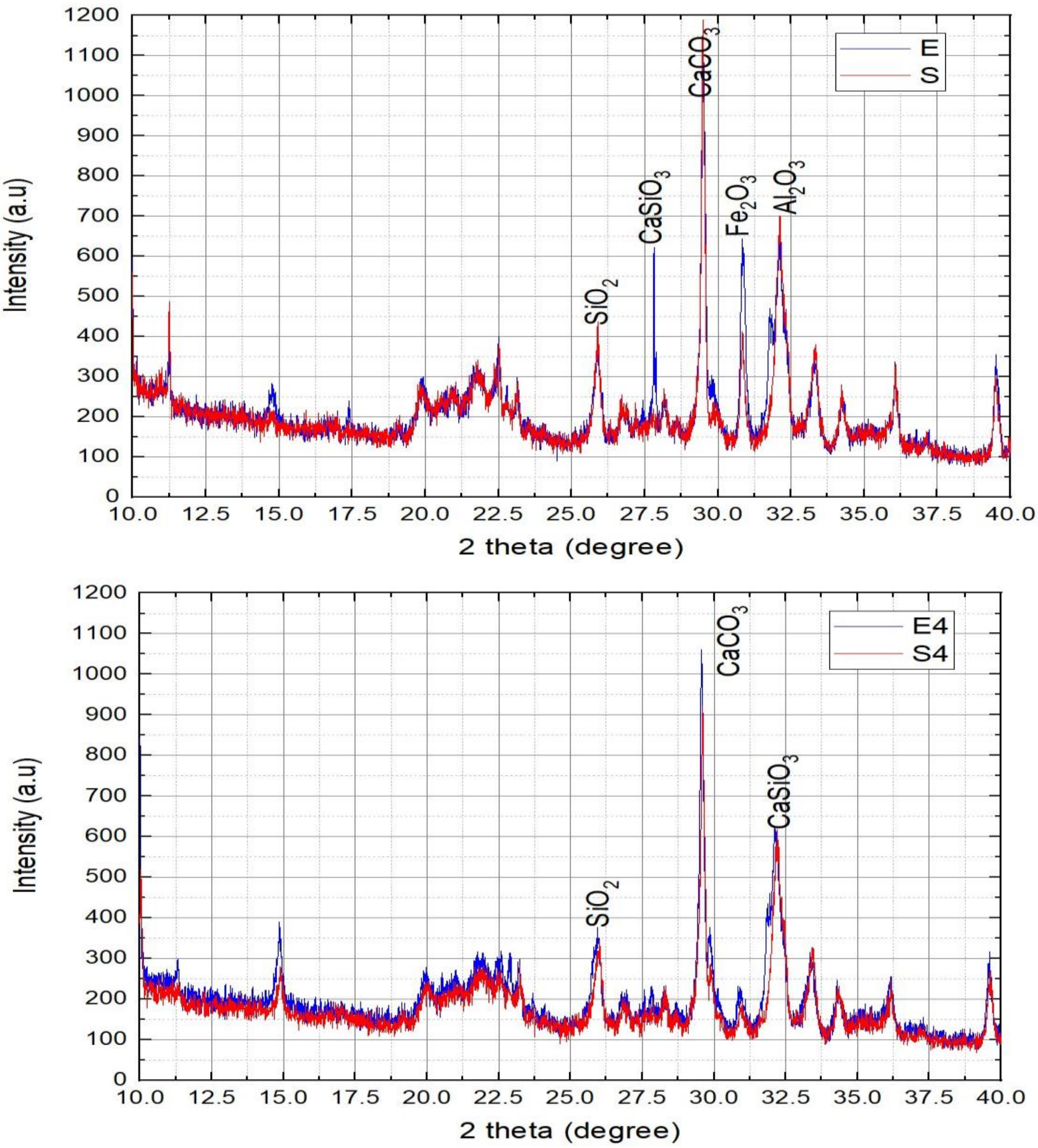

Figure 6. X-ray diffraction spectrum of the E sample, E4 sample, S sample, and S4 sample.

4.3. Density

Since our samples are made of concrete, it was decided to determine their density before performing the compression test. This is because density is a crucial characteristic of concrete, and it is correlated with both strength and durability: the denser the concrete, the more resilient it is to mechanical forces like compression, tension, and bending. Table 1 indicates that there are multiple dosages, thus there will be multiple samples. The following density formula (3) was then applied, and the outcomes are displayed in Figure 7.

$$concrete\ density = \frac{m_c}{v_c}\ by\ g/cm^3 \qquad (3)$$

Integrating phosphate sludge into industrial applications presents several challenges, primarily due to the variability in its chemical composition, which influences its reactivity and compatibility with cement formulations. The presence of elements such as SiO_2, CaO, and P_2O_5 offers significant potential, but its use requires careful management of impurities and metallic oxides like Fe_2O_3 and MgO, which can affect chemical stability. Additionally, the density and cohesion of phosphate sludge are key factors in its incorporation into composites, as they directly impact compressive strength and material durability. Therefore, adjustments must be made to ensure optimal mechanical performance and minimize constraints related to unwanted chemical reactions.

4.4. Mechanical properties

In order to determine the sample's compressive strength, first the maximum force that our concretes could support is determined and it is verified by utilizing the compression machine's results.

The Young's modulus is concurrently examined, also referred to as the modulus of elasticity. For an isotropic and homogeneous material, such as the one in our example, the modulus of elasticity (E) is often computed using the Hooke law.

The basic formula is:

$$E = \frac{\sigma}{\epsilon} \qquad (4)$$

Where E is the modulus of elasticity (in Pascal), σ It is the constraint (in pascals), $\epsilon = \frac{\Delta L}{l}$ is the unit deformation (without unit)

Regarding mechanical properties, our compressive strength results (ranging from 2.34 MPa to 4.62 MPa) are lower than conventional cement values, which typically exceed 25 MPa. However, studies on waste incorporation into concrete have reported similar reductions in strength, particularly when organic waste is introduced. For example, research on paper and wood waste integration found that excessive organic content led to a decrease in compressive strength, mirroring our observations.

The fig.10 illustrates the difference in elastic strength and shows how the compressive strength can be calculated using the curve.

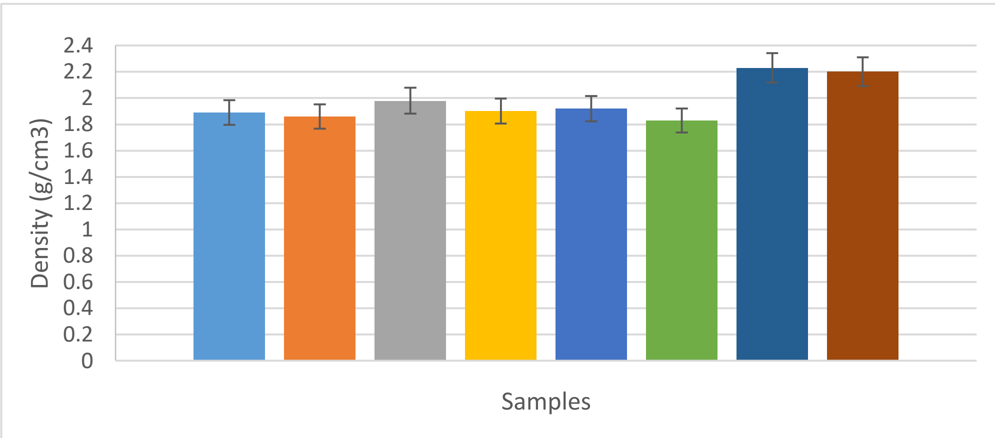

Figure 7. The density of each sample for the strength test

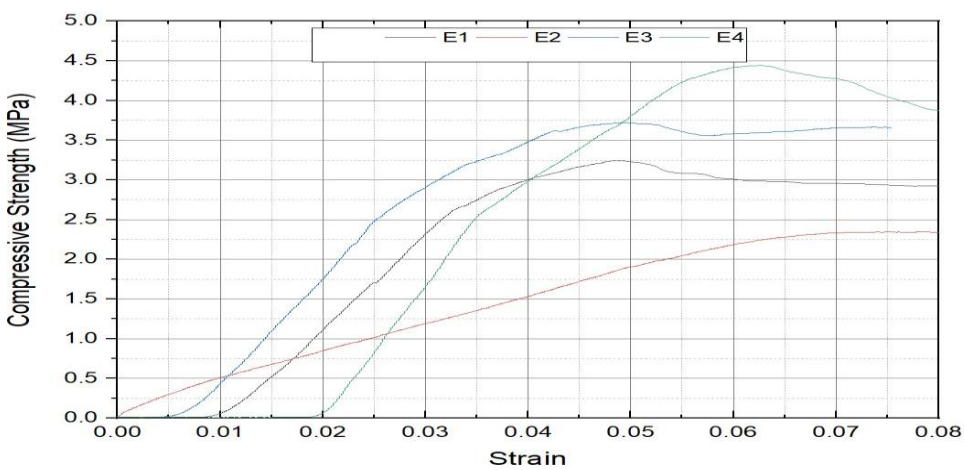

Figure 8. The compressive strength curve results in sample E1, sample E2, sample E3, and sample E4

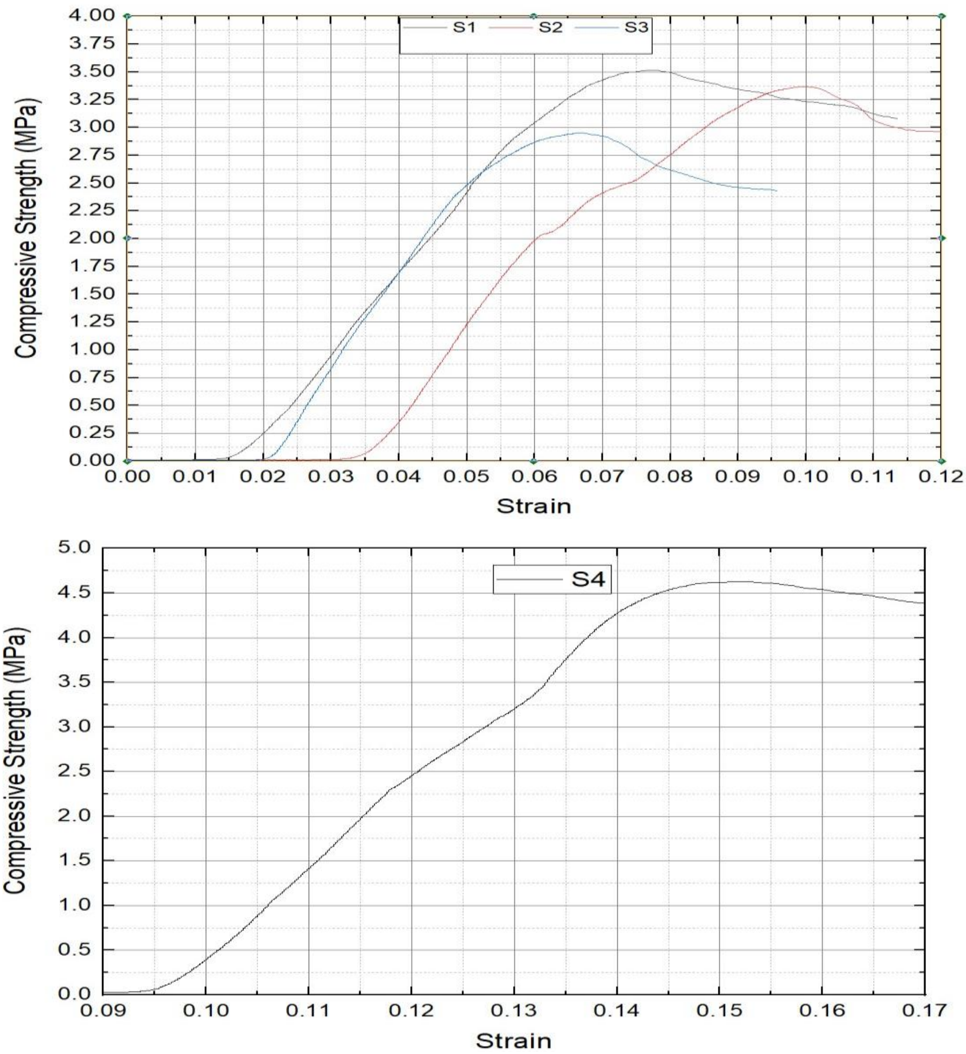

Figure 9. The compressive strength curve results in sample S1, sample S2, sample S3, and sample S4.

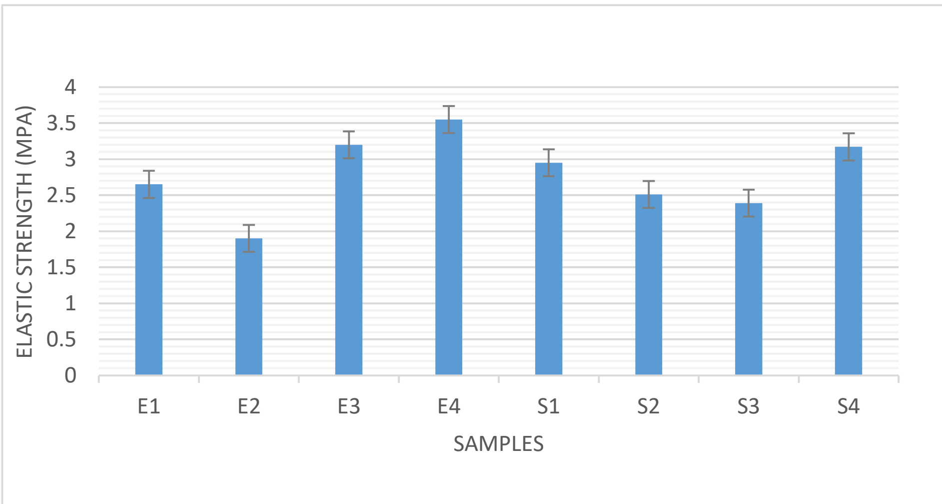

Figure 10. Elastic Strength of all compositions

Four primary forms of cracking can be distinguished for the study of cracking, based on the literature: shrinkage cracking, oblique cracking, shear cracking, and vertical cracking. Given that the highest forces supported by samples E1, E3, S1, and S4 are 5.18 KN, 5.95 KN, 5.62 KN, and 7.4 KN, respectively, it is concluding that the vertical cracks in these samples are primarily the result of excessive loads. The maximum force for the first three is then observed to be quite close to one another, but for S4, the load is not the sole determinant, suggesting that either a material problem or a design defect is responsible for this crack. Samples E2, E4, S2, and S3 all have shear cracks. Samples E2 and E4 recorded maximum forces of 3.75 KN and 7.11 KN, respectively. Samples S2 and S3 had maximum force measurements of 5.38 KN and 4.72 KN, respectively. Poor or incorrect design can lead to concentrated stress points, which can cause cracks. On the other hand, E1 and E4 also showed signs of oblique cracks. Inappropriate structural design that is, the disregard of important components is the origin of these fissures. In samples E3 and E4, it is also seen the presence of shrinkage cracks. This is because the mixture has a high water/mixture ratio, or a high water content, which reflects chemical reactions between the elements.

Cracking behavior analysis aligns with literature on structural failure mechanisms. Vertical cracks due to excessive loads, shear cracks from stress concentration, and shrinkage cracks from high water content have all been documented in previous studies on concrete durability. The identification of these failure modes in our samples confirms the relevance of our findings to broader research on waste.

Overall, our results demonstrate strong consistency with existing studies on phosphate sludge recycling and waste incorporation in cementitious materials. While mechanical strength[22] remains lower than conventional cement, the chemical and mineralogical compatibility suggests promising applications in sustainable construction. Further optimization of waste proportions and processing techniques could enhance performance, making industrial waste a viable alternative in eco-friendly material development.

Table 3. Cement and Concrete Uniform Specifications

Type of analysis	Typical results					
Chemical analysis						
Oxide composition	MgO	Al_2O_3	SiO_2	K_2O	CaO	Fe_2O_3
	0.39	0.31	13.82	0.32	63	2.57
Mineralogical analysis						
Crystalline phases	Identification of portlandite, ettringite, and other cement hydration products					
Compressive strength	25(in MPa)					

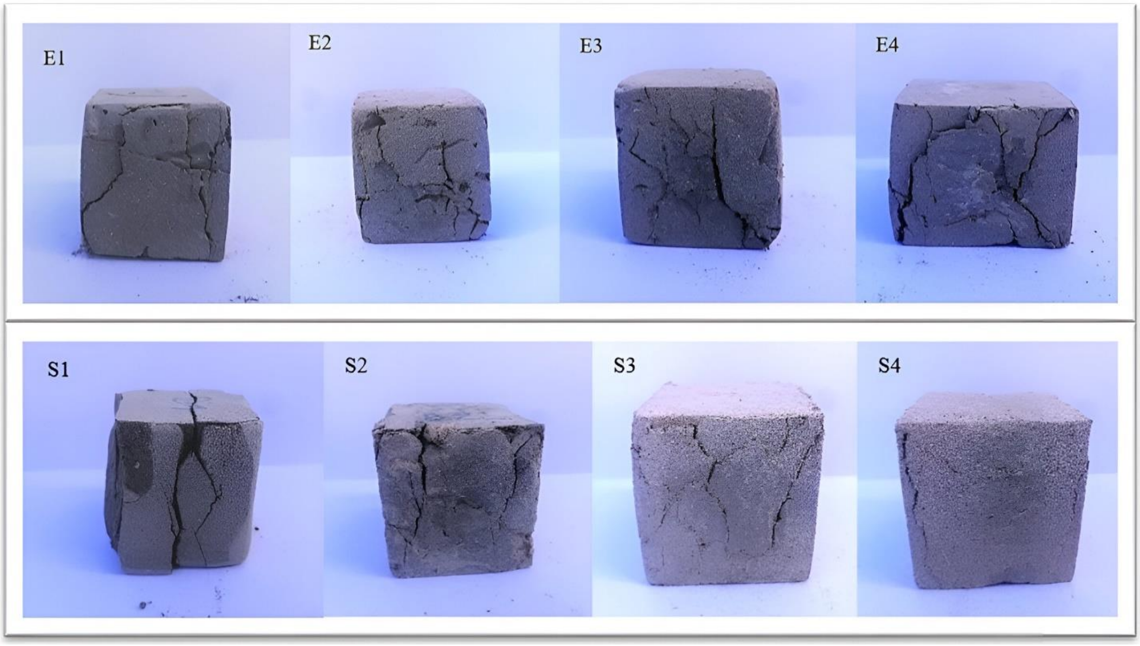

Figure 11. The compressive strength behaviors result of sample E1/S1, sample E2/S2, sample E3/S3 and sample E4/S4.

5. Conclusion

This study shows that phosphate sludge can be used as a valuable resource for building materials. Indeed, they contain essential oxides such as SiO and CaO that improve the mechanical, chemical, and aesthetic properties of materials. Flocculation has a low impact on sludge chemistry, but it has a strong impact on their mechanical and physical properties. This requires fine adjustments when adding materials such as wood and waste paper. To improve compressive strength, these components must be optimized. This ensures the structural reliability of the material.

The results show that it is important to recycle waste containing phosphorus to protect the environment, especially in the Metlaoui free trade zones. Indeed, if not recycled properly, it can be dangerous for the environment. Gypsum can help produce calcium and sulfate ions. Tests have confirmed this. To make high-quality materials, you have to find the right balance between these elements. This helps improve how they hold together, their resistance, and how they can be used.

Going forward, the study will aim to improve recovery by looking for other ways to strengthen the formula, not just with gypsum. The data show that the mixture has a low compressive strength. Therefore, a robust reinforcement strategy is required to use it in industry. This research also shows that innovative materials made from industrial waste have economic and social benefits. They provide a sustainable way of recycling in many industries.

Moving forward, the study will focus on developing a refined recovery technique to optimize the formulation by exploring alternative reinforcing agents beyond gypsum. Given the compressive strength data, it is evident that a robust reinforcement strategy is required to ensure the material's viability for industrial applications. Additionally, this research underscores the socioeconomic benefits of innovative environmental materials derived from industrial waste, offering a sustainable recycling pathway for various industries.

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