

Investigate and Characterize of Textile Industry Sludge for the use of Construction Material in case of Hawassa industrial park, Ethiopia.

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Abstract

The swift expansion of Ethiopia's textile industries is a significant driver of economic progress. Nevertheless, the sludge produced by their wastewater treatment facilities is frequently deemed hazardous due to contamination with heavy metals from dyes and chemicals. In this study; a physical and chemical analysis of sludge was conducted using gravimetric method, while test for compressive strength, soundness and setting time were performed with blending ratios of 5%, and 10%. The analysis of sludge revealed a moisture content of 27%, volatile organic matter of 17%, and an inorganic ash content of 53%. The effects of replacing cement with textile sludge evaluated in terms of compressive strength and water absorption after 2, 7, and 28 days, using a design mix ratio of cement to sand of 1:3 and 225 ml. of water to form the cement paste. The analysis indicate ensuring sludge loading remains below 10% when combined with OPC cement guarantees desired properties, but raising the sludge content diminishes compressive strength. Extending curing periods results in enhanced compressive strength, as seen in a significant rise from day two to twenty-eight. Blending cement with sludge decreases raw material expenses and cement prices, offsetting the 10% rise in construction material costs while maintaining quality. This fosters economic growth in brick manufacturing industries by reducing energy usage, cutting expenditures, and reducing brick weight, thereby reducing transportation costs and mitigating environmental pollution associated with cement manufacturing.

Keywords: Compressive strength, cement mix, water-cement ratio.

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1 Introduction

Numerous countries have embraced sustainable development initiatives aimed at integrating materials for partially substituting cement (Tay, 1991; Izadifard, 2021). Challenges related to raw material costs and environmental pollution have spurred research into cement substitutes or alternatives in construction. With growing concerns about resource scarcity, the adoption of alternative materials that partially replace cement is gaining momentum. Managing industrial sludge waste presents a significant challenge in Ethiopia, particularly with the rapid expansion of industries, notably the textile sector (Beshah et al., 2021). Rotary kilns in cement plants can incinerate various materials due to their exposure to high temperatures and the clinker's ability to absorb contaminants in an alkaline environment (Jiang, X., Li, Y., & Yan, J., 2019). Despite this, the cement industry is a major source of pollution, accounting for 5–6% of CO₂ emissions from human activities and contributing to approximately 4% of global warming (Mohamad et al., 2022).

Limestone, constituting 50% of carbon emissions from cement production, entails costs and environmental impacts in mining, transportation, and processing. A sustainable approach involves using these raw materials as partial cement substitutes, reducing reliance on limestone. Textile industry sludge, a hazardous waste in Ethiopia, lacks proper disposal methods and toxicity assessment. Cement production's reliance on bulk materials like limestone, pumice, clay, gypsum, sand, and coal leads to resource depletion. Exploring alternatives like sludge for cement substitution in construction addresses environmental and resource sustainability in the cement sector while mitigating disposal challenges associated with hazardous waste (Greco-Coppi et al., 2023).

The textile industry is a significant global polluter, with its dye-laden wastewater discharging 10–25% into the environment during the dyeing process (Periyasamy et al., 2018). Managing sludge from textile processes has become a pressing issue. This sludge, a by-product of physical, biological, and chemical processes in wastewater treatment, is complex, containing organic and inorganic substances, pathogenic microorganisms, and heavy metals. In developing countries like Ethiopia (typically Hawassa and Bole Lemi Industrial park) experiencing rapid textile industry growth, sludge production escalates (Teferi, M., 2020; Jamshidi et al., 2011). Compliance with relevant legislation is crucial for proper sludge management, ensuring resource

recycling without compromising public health or environmental integrity (EU Directive 91/271/EEC; Oodegard et al., 2002).

The primary methods for disposing of sludge include landfilling, agricultural application to enhance soil fertility, sludge drying, and utilizing it for energy production through incineration and as an alternative to clinker in cement blending (Fytli et al., 2006). Sludge drying facilities reduce its volume and yield a biologically stable, odorless product. This treated sludge is then employed as a partial substitute for raw materials in cement manufacturing, ensuring the quality of concrete remains uncompromised (Husillos-Rodriguez et al., 2013; Stasta et al., 2006; Zabaniotou and Theofilou, 2008).

The pollution of air and water due to various waste sources has surged. Effective sludge management, following the 'four R's' - Reduce, Reuse, Recycle, and Renewable energy, is vital. Textile industry sludge disposal presents a notable challenge. Yet, with adequate treatment, it can serve as an alternative energy source and raw material in cement and construction sectors. Repurposing industrial sludge as construction material addresses the issue of heavy metal content, rendering it unsuitable for agriculture, providing a practical solution (Baeza-Brotons et al., 2014).

This study aims to investigate and characterize textile sludge, while also estimating the potential percentage of sludge that can replace cement in construction applications in case of Hawassa industrial park. In developing nations like Ethiopia, where housing affordability is a pressing issue and cement costs are rising, alternative materials like textile sludge offer promising solutions. By reducing reliance on cement and utilizing abundant industrial by-products, such as textile sludge, construction becomes more cost-effective and environmentally sustainable. These alternative materials possess favorable mechanical properties and economic value while minimizing environmental impact.

2. Methodology

2.1. Location and Materials

This research undertake within the Hawassa industrial park in southern Ethiopia, situated 320 km from Addis Ababa. The primary data source and materials used are; Sludge from the park of waste water treatment plant, ordinary Portland cement, and standard sand. Data collection involves the preparation and testing of samples obtained from park, adhering to Ethiopian

standards ES 1176-7. The three plastic bag samples 15 kg weighted are undergone milling and sieving process to homogenize and the physical and chemical analysis were representative. This approach ensures a rigorous examination of the sludge's characteristics, cement, and sand in accordance with established standards.

Cement: Ordinary Portland cement (OPC) produced in Mughar Cement factory's was used in this study.

Water: is used for mixing of concrete specimens is a tap water which is available in the laboratory.

Standard sand: Standard sand, utilized in concrete and mold testing, ensures consistency and accuracy in laboratory procedures and quality control standards.

2.2. Preparation of samples

The sample preparation involved mixing materials in a 1:3 ratio of sludge, cement, and standard sand, and 225ml of water. Following mixing, the resulting paste was poured into properly secured 4mm³ iron molds. To ensure thorough compaction, the cubes were vibrated using a jointing vibrator. Afterward, the mortar samples were left to set overnight within the molds. Upon removal from the molds, their weight was recorded. These specimens were then submerged in a curing tank for compressive strength testing at intervals of 2, 7, and 28 days. Post-curing, the specimens were examined to assess the mechanical properties of the mortar, accounting for variations in sludge percentage within the mortar mixture (Table 1). The mortar samples were manufactured and cured using clean drinking water sourced from public providers, adhering to the CES 58 standards for drinking water and household use.

Table1: Mix proportion for preparing specimen

| Sludge (%) | No of cubes | Cement (gm.) | Sludge gm. | Water (ml) | Standard sand gm. |
|------------|-------------|--------------|------------|------------|-------------------|
| 0% | 3 | 450 | 0 | 225 | 1350 |
| 5% | 3 | 427.5 | 22.5 | 225 | 1350 |
| 10% | 3 | 405 | 45 | 225 | 1350 |
| 15% | 3 | 382.5 | 67.5 | 225 | 1350 |
| 20% | 3 | 360 | 90 | 225 | 1350 |

2.2.1. Curing of prism specimens

The mortar was shape subsequent to its mixing. The molds and specimens kept in a laboratory environment for 24 hours before being relocate to the curing tank. Evaluation took place after 2, 7, and 28 days of the curing process. The testing at the two-day mark aimed to ascertain the early strength. The Portland cement sludge specimens underwent curing by complete immersion in the curing medium. Bowl baths, each with a capacity of 50 liters of water, were utilize for curing the mortar samples, ensuring appropriate placement and curing without spillage.

2.2.2 Chemical composition of sludge

The chemical composition of the sludge was analyzed using gravimetric methods at the Mughher cement factory to identify the oxides of Al_2O_3 , Fe_2O_3 , Al_2O_3 , MgO , CaO , and SO_3 . A 0.5g sample of the sludge (previously finely ground and air-dried at 105°C for an hour) was placed in a platinum crucible and mixed with 0.5g of sodium-potassium carbonate (fusion mixture). The crucible, with its lid on, was then placed in a muffle furnace, heated gradually, and finally ignited at $925 \pm 25^\circ\text{C}$ to complete the fusion process. After cooling, the crucible was transferred to a 500ml beaker containing 100ml of water, to which 25ml of 1:1 HCl was slowly added while the beaker was heated on a sand bath. The crucible was washed with hot water, and any remaining lumps in the solution were crushed using a glass rod before adding an excess of 5ml of 1:1 HCl.

The solution resulting from the fusion was evaporated to dryness, and then 20ml of 5% HCl was added and shaken in, followed by 100ml of hot distilled water. This mixture was heated on a hot plate until boiling while being continuously stirred with a glass rod. The solution was filtered through ash-less filter paper No.40 (What-man) or pulp, and the residue was washed five times with hot water. This rigorous procedure ensured the accurate determination of the chemical composition of the sludge using gravimetric methods at the Mughher cement factory.

The precipitate was utilized for SiO_2 analysis, whereas the liquid obtained after filtration was employed for the analysis of Al_2O_3 , Fe_2O_3 , SO_3 , MgO , CaO , and SO_3 through the Gravimetric method. To ascertain the loss on ignition (LOI), 1g of the substance tarred platinum crucible and ignited in a muffle furnace at a temperature of $750 \pm 50^\circ\text{C}$ for one hour. After cooling to room temperature in desiccators, the crucible was reweighed following the procedure outlined in ES 1176-2 (equ 1).

$$\% \text{ LOI} = \frac{A-B}{A} * 100 \dots \dots \dots \text{equ 1}$$

Table 2:

| Material content | Composition (%) | Accepted standard (%) |
|---|--------------------------|--------------------------|
| Silicon Oxide (SiO ₂) | 22 | ≥64.0 |
| Aluminium Oxide (Al ₂ O ₃) | 14.39 | 12.00-17.00 |
| Iron Oxide (Fe ₂ O ₃) | 2.45 | 2.30-3.50 |
| Calcium Oxide (CaO) | 5.29 | 1.50-2.20 |
| Magnesium Oxide (MgO) | 1.16 | 0.30-1.20 |
| Sulphur Oxide (SO ₃) | 1.1% | ≤0.3 |
| Moisture before oven dried | 26.6 | ≤10 |
| Moisture after oven dried | 2.16 | ≤2.0 |
| Loss on Ignition (LOI) | 45.82 | 4.00-7.00 |
| Blaine Cm ² /gm. | 4994 cm ² /gm | 4112 cm ² /gm |

Sieve analysis:- The fine sludge sample was weighed and passed through a mesh with a size of 45 micrometers. The weight of the sludge remaining on the sieve was measured, and the percentage of sludge passing through was calculated.

2.2.3 Setting time and soundness of materials

Setting time and soundness tests were conducted using normal consistency cement paste. Initial and final setting times were measured using Vicat and Le Chatelier apparatus, following the guidelines of ES 1176-3. Three replacement paste mixes were prepared, containing 0%, 5%, 10%, and 15% sludge, respectively, for the setting time and soundness tests. This approach allowed for the examination of how different levels of sludge replacement affect the setting time and soundness characteristics of the cement paste.

$$\text{Consistency} = \frac{\text{water consumed}}{\text{weight of cement sample}} * 100 \dots \text{equation 2}$$

Table 3: Results of OPC and sludge blend

| S.no | Cement sludge mix (%) | Initial time (m.) | Final time (m.) | Soundness(expansion, ml) |
|------|-----------------------|-------------------|-----------------|--------------------------|
| 1 | 0 | 176 | 213 | 0.3 |
| 2 | 5 | 181 | 217 | 0.8 |
| 3 | 10 | 198 | 238 | 1.3 |

Compressive strength test: The specimens were removed from the curing basin, surface dried, weighed, and then positioned at the center of the hydraulic automatic compression machine for testing.

2.3 Proximate Analysis

A sludge sample weighing 100 grams (denoted as Wa) was measured using a digital weighing balance. The sample was subsequently transferred onto a metal container, spread evenly, and placed inside an electric oven for one hour at a temperature of 105 degrees Celsius. After the

hour elapsed, the sample was taken out of the oven and left to cool to room temperature. The weight of the sample after oven drying (denoted as W_c) was then measured again. The moisture content was determined following the guidelines outlined in ASTM D 2974-87.

$$\text{Moisture content} = \frac{W_a - W_c}{W_c} * 100 \dots \text{equation 3}$$

The sludge samples' initial weight was determined and placed into a crucible, followed by oven drying and ignition in a muffle furnace set at 1050 degrees Celsius for 30 minutes. The subsequent weight measurement yielded the final weight. From these values, the percentage of volatile matter was computed.

$$\text{Loss of ignition (Volatile Matter(\%))} = \frac{W_{vo} - W_v}{W_{s0}} * 100 - \dots \text{equation 5}$$

Where: W_{vo} denotes the initial weight of the sample plus the crucible,
 W_{s0} signifies the initial weight of the sample, and
 W_v represents the resulting weight of the crucible plus the sample sludge waste.

Data processing and analysis:- The experiment was conducted in a completely randomized design samples were analyzed. The results obtained for different tests are analyzed using excel and a nova statistical software. The root mean square error (RMSE) is a metric used to evaluate the accuracy of a statistical model's predictions indicate 2.6 is quite acceptable less than 5%. It quantifies the average difference between predicted values and actual observed values in dataset.

| Response: Compressive strength | | | | | |
|---|----------------|----|----------------|------------|----------|
| ANOVA for Selected Factorial Model | | | | | |
| Analysis of variance table [Partial sum of squares] | | | | | |
| Source | Sum of Squares | DF | Mean Square | F Value | Prob > F |
| Model | 3802.04 | 5 | 760.41 | 8.842E+005 | < 0.0001 |
| A | 3650.54 | 2 | 1825.27 | 2.122E+006 | < 0.0001 |
| B | 148.84 | 1 | 148.84 | 1.731E+005 | < 0.0001 |
| AB | 2.66 | 2 | 1.33 | 1546.51 | < 0.0001 |
| Pure Error | 0.026 | 30 | 8.600E-004 | | |
| Cor Total | 3802.07 | 35 | | | |
| Std. Dev. | 0.029 | | R-Squared | 1.0000 | |
| Mean | 27.23 | | Adj R-Squared | 1.0000 | |
| C.V. | 0.11 | | Pred R-Squared | 1.0000 | |
| PRESS | 0.037 | | Adeq Precision | 2430.633 | |

| | Term | DF | Sum of Squares | Mean Square | F Value | Prob > F | % Contribution |
|--|-------------|----|----------------|-------------|------------|----------|----------------|
| | Intercept | | | | | | |
| | A | 2 | 3650.54 | 1825.27 | 2.122E+006 | < 0.0001 | 96.01 |
| | B | 1 | 148.84 | 148.84 | 1.731E+005 | < 0.0001 | 3.91 |
| | AB | 2 | 2.66 | 1.33 | 1546.51 | < 0.0001 | 0.070 |
| | Lack Of Fit | 0 | 0.000 | | | | 0.000 |
| | Pure Error | 30 | 0.026 | 8.600E-004 | | | 6.786E-004 |
| | Residuals | 30 | 0.026 | 8.600E-004 | | | |

Figure 1. Data processing and statistical analysis

3. Result and Discussion

Analyzing and characterizing textile industry sludge for potential use in construction materials entails a thorough process aimed at verifying its appropriateness, safety, and effectiveness. This method involves several sequential steps, starting with the collection of samples, followed by evaluations of its physical characteristics like particle size, density, and porosity, as well as its chemical composition including elemental makeup, organic content, and pH levels. Subsequent stages involve assessments employing techniques such as X-ray diffraction (XRD) and scanning electron microscopy (SEM), along with mechanical tests to gauge properties like compression and flexural strength. The sludge's suitability for construction purposes, taking into account its safety, performance, and environmental ramifications. These procedures draw upon various studies and sources, including those by Adamson and Subra (2018), Hossain et al. (2019), Lee et al. (2020), Liu et al. (2017), Yang et al. (2021), Zhang et al. (2019), Zheng et al. (2018), and Zhu et al. (2020).

3.1 Sludge characteristics

Physicochemical analysis of sludge entails evaluating its physical and chemical attributes, including moisture content, particle size distribution, density, pH, organic matter content, elemental composition, and the presence of contaminants like heavy metals. This assessment is crucial for assessing its compatibility for use in concrete and mortar production.

After oven drying, the sludge sample exhibited a moisture content of approximately 2.16%, with low silicon content and high organic content. The surface area of the sample measured 4994 cm²/gm, compared to the reference sample's 4112 cm²/gm. The loss on ignition (LOI) indicated a

value of 45.82%, exceeding the standard value due to the sludge's elevated organic matter and fine content.

Excessive moisture and unwanted impurities, such as carbon, can significantly impact the quality of mortar and concrete produced using sludge as an additive to cement. Gravimetric analysis results revealed that the sulfate composition in the oven-dried sludge sample was 1.1%, within the range specified by the ES 1176-2 standard, which states that the maximum sulfate content of raw material used as an additive should not exceed 3.5%. This indicates that mortar or concrete utilized in construction, blended with sludge at the optimal level with OPC, would not be susceptible to sulfate attack from its source.

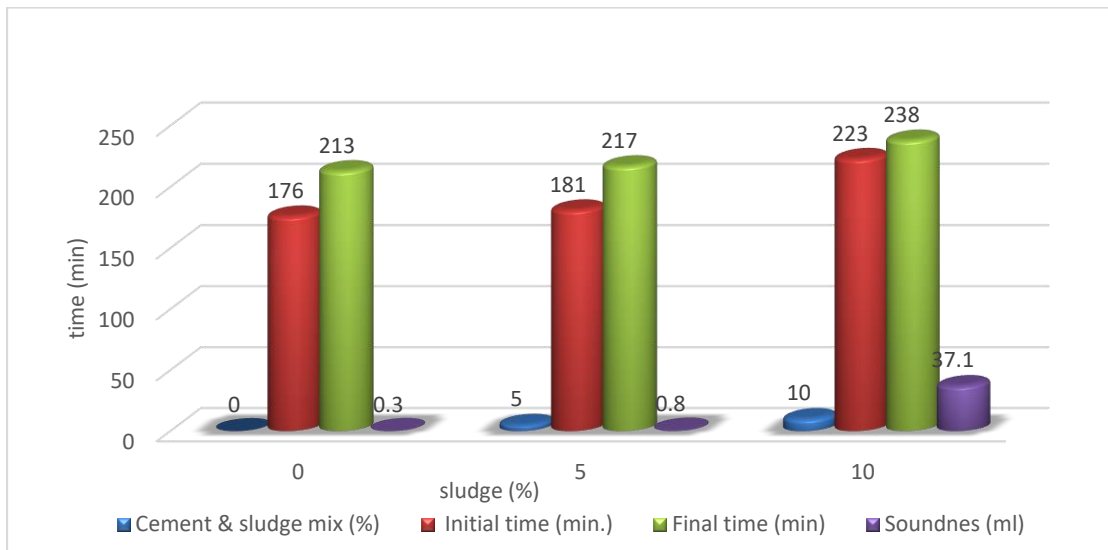


Figure 2. Properties of Consistency, Setting time and Soundness Sludge blended with cement

Variations in normal consistency, setting times, and soundness are observed with different sludge percentages. When the sludge content varied from 0 to 10%, the normal consistency of the paste increased by 10%, attributed to a higher presence of cementitious binder in the mixture. Consequently, the overall volume increased, necessitating more water to maintain consistent paste consistency across different sludge percentages.

The setting time findings revealed that both initial and final setting times increased as the sludge content rose. With sludge content increasing from 0 to 10%, the initial setting time saw a 12.5% increase, while the final setting time rose by 11.7%. This can be attributed to the higher sludge content reducing both the cement content and surface area in the mixture, thus slowing down the

hydration process. The 10% replacement showed a 36min. delay (standard ranges 75-260 min.) in setting time compared to ordinary Portland cement paste. As a consequence, slower hydration leads to a lower rate of heat development (Schindler, A. K., & Folliard, K. J., 2005), which is particularly important for applications such as mortar or mass concrete in construction, where sludge can be predominantly utilized, among other general purposes.

Table 4: Moisture contents of materials

| No | Materials | Weight of before dry (gm) | Weight of after dry (gm) | Mc (%) |
|----|-----------------|---------------------------|--------------------------|--------|
| 3 | Portland cement | 100 | 99.8 | 0.2 |
| 4 | Sludge | 100 | 97.84 | 2.16 |

The moisture content in mortar should fall within the range of 0 to 10%, indicating compliance with the standard (Ezziane, et al., 2007).

The sludge sample contains zinc as the primary heavy metal, with other metals present in decreasing order: zinc, copper, chromium, nickel, lead, arsenic, cobalt, mercury, and cadmium. The concentrations of each metal in the sludge were measured in milligrams per kilogram (mg/kg) as follows: zinc (272), copper (50), chromium (27), nickel (11), lead (10), arsenic (5), cobalt (3.3), mercury (0.4), and cadmium (0.26). The concentrations of all heavy metals in the textile sludge were found to be significantly below the standard values set by the USEPA for land application and disposal restrictions of sludge.

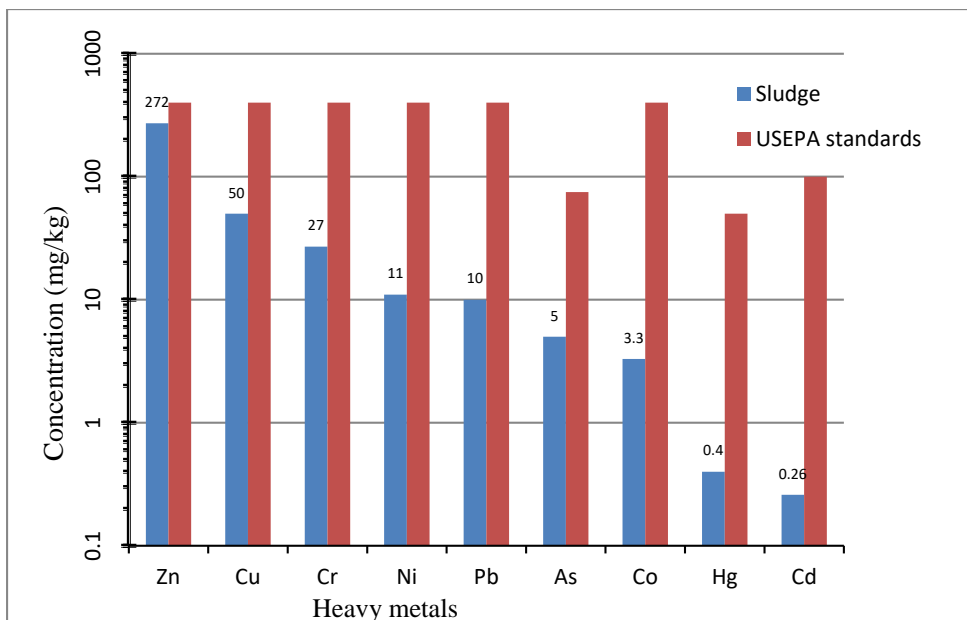


Fig. 5 Heavy metal concentration of sludge

3.2 Compressive strength

The compressive strength of the sludge combined with OPC cement, reaching up to 10%, adheres to the standard specifications, although there is a decline in the percentage increase of compressive strength. Each group of triplicate specimens was divided into two equal sections, and compressive strength evaluations were performed at 2, 7, and 28-day intervals.

Table 5. The compressive strength corresponding to various sludge ratios.

| No | reference sample | | | 5 % sample | | | 10 % sample | | |
|----------|------------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|-------------|
| | 2 days | 7 days | 28 days | 2 days | 7 days | 28 days | 2days | 7days | 28 days |
| 1 | 23.42 | 40.51 | 60.32 | 16.88 | 29.38 | 41.47 | 12.41 | 26.12 | 37.13 |
| 2 | 23.45 | 40.49 | 60.28 | 16.86 | 29.39 | 41.49 | 12.37 | 26.15 | 37.08 |
| 3 | 23.5 | 40.51 | 60.31 | 16.87 | 29.42 | 41.48 | 12.44 | 26.06 | 37.13 |
| 4 | 23.52 | 40.52 | 60.3 | 16.93 | 29.43 | 41.51 | 12.43 | 26.09 | 37.11 |
| 5 | 23.54 | 40.53 | 60.29 | 16.91 | 29.38 | 41.52 | 12.36 | 26.07 | 37.07 |
| 6 | 23.57 | 40.45 | 60.31 | 16.95 | 29.4 | 41.53 | 12.39 | 26.11 | 37.08 |
| Ave | 23.5 | 40.5 | 60.3 | 16.9 | 29.4 | 41.5 | 12.4 | 26.1 | 37.1 |

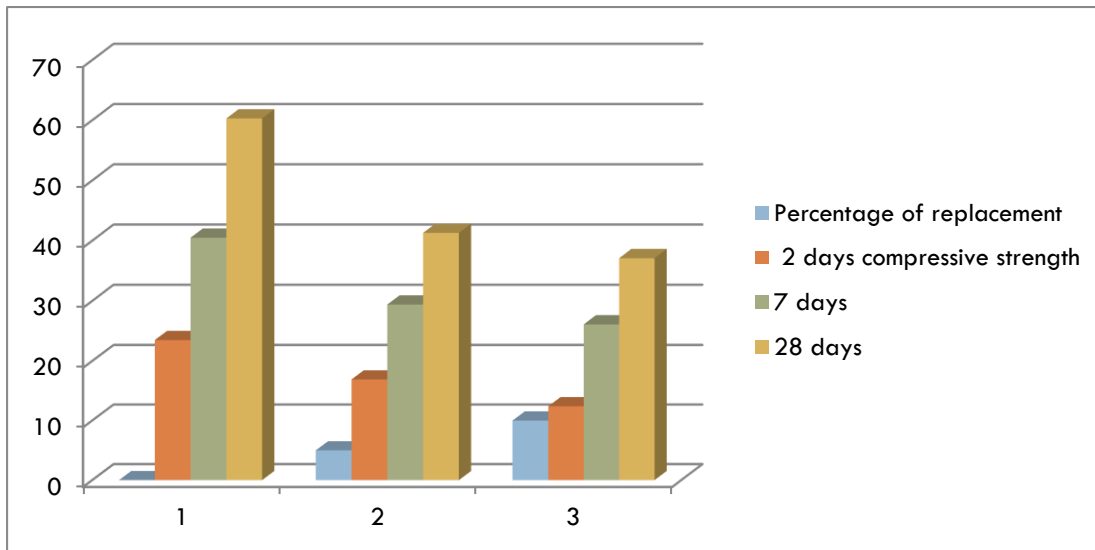


Figure 3. Average compressive strength of specimen samples

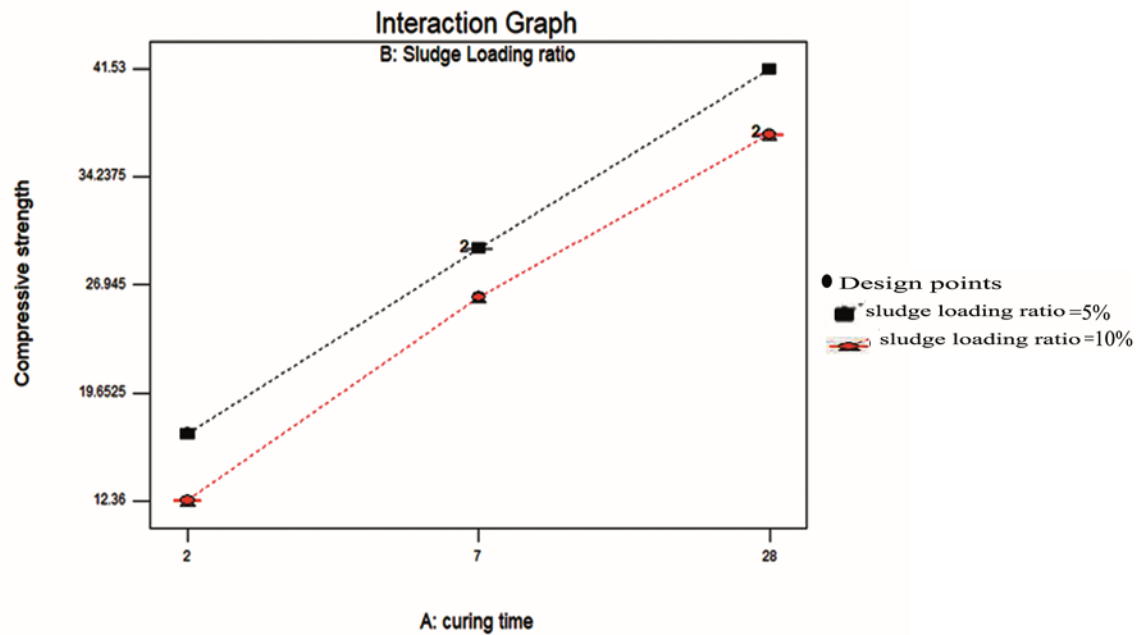


Figure 4. Compressive Strength (MPa) versus curing days (5% black and 10% red broken slop is a sludge ratio).

From the table 5 and Figure 4 above, the sludge loading ratio has greater impact on the compressive strength of as compared with the curing time with contribution effect of 96 %. And the result showed that insignificant effect of interaction between sludge loading ratio and curing time. And the result showed that insignificant effect of interaction between sludge loading ratio and curing time.

The considerable impact of curing time on concrete and mortar. This refers to the duration during which concrete or mortar remains moist and at a controlled temperature post-placement to ensure proper hydration of cementitious materials. Extended curing periods result in enhanced compressive strength, evidenced by a significant increase from day two to day twenty-eight. Strength development is closely linked to durability, as hydration reactions persist within the cementitious matrix, forming hydrated cementitious products. Furthermore, adequate curing mitigates shrinkage, reduces cracking, and improves resistance to environmental factors like freeze-thaw cycles and chemical attack.

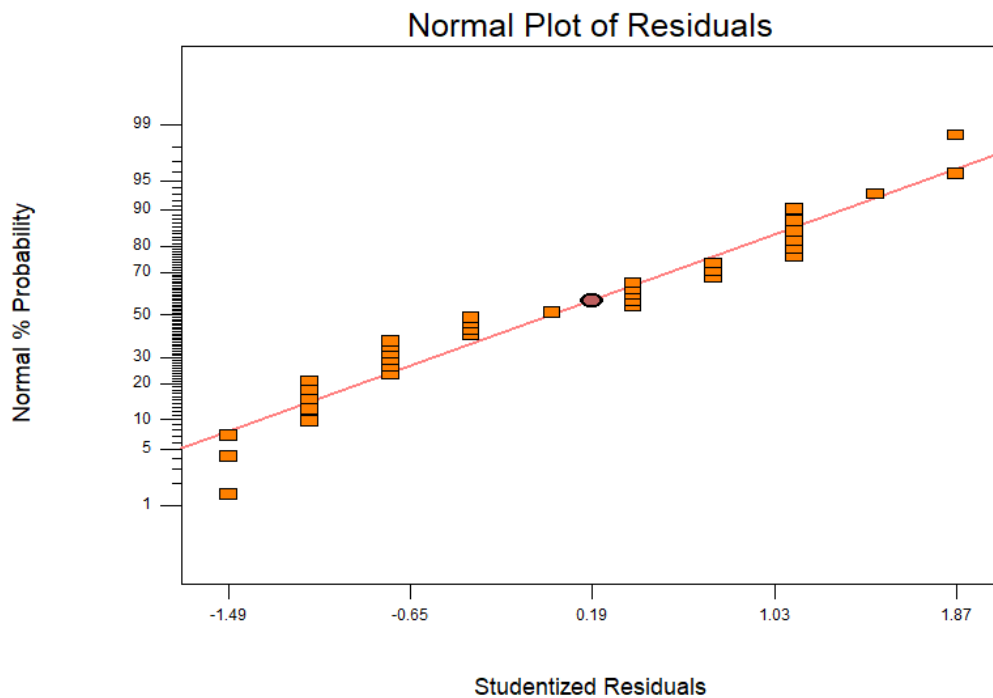


Figure 5 Compressive Strength (MPa) and curing time (days) (add

According to Figure 5 normal probability plot, the residuals adhere to a normal distribution. Residuals denote the variance between actual experimental findings and predicted outcomes by the software. The plotted points for the experimental data form a straight line, signifying that the linear model satisfies both analysis of variance (ANOVA) requirements and a normal error distribution.

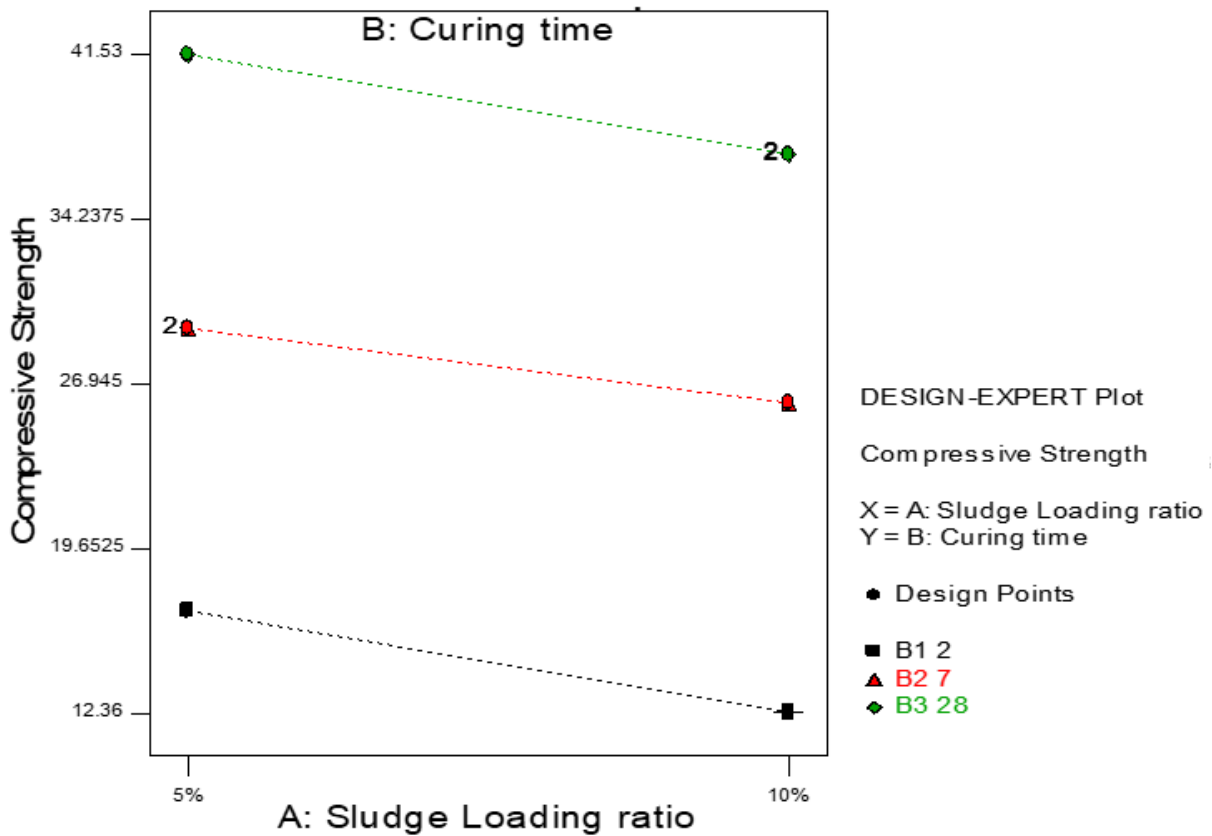


Figure 6 Compressive Strength (MPa) and curing time (days)

Figure 6 indicates that higher sludge loading ratios have a detrimental impact on compressive strength, resulting in a decrease as the ratio increases. The loading ratio influences strength, workability, durability, and environmental aspects. The effect of sludge loading in concrete and mortar is variable, depending on factors like sludge type, composition, and proportion. In general, maintaining the sludge loading below 10% ensures desired properties, increasing the sludge content compromise compressive strength. Careful consideration and control are essential to optimize the benefits of sludge loading while minimizing adverse effects on concrete and mortar performance.

4. Conclusion

The production of energy-efficient concrete and mortar involved integrating textile sludge at varying ratios and temperatures. Analysis of the sludge's characteristics indicated that its heavy metal content was significantly below the regulatory limit set by USEPA. The test results showed that the compressive strength of samples with various sludge proportions during early curing stages (2 and 7 days) exceeded the average (10 and 16 MPa) of the reference control sample for cement II. In this study, maintaining sludge loading below 10% with OPC cement ensures desired properties, while increasing sludge content compromises compressive strength.

Prolonged curing periods lead to improved compressive strength, as evidenced by a notable increase from day two to day twenty-eight. Blending cement with sludge reduces raw material costs and cement prices, addressing the escalating costs of construction materials by 10% without compromising quality. This contributes to economic prosperity for brick manufacturing industries by reducing energy consumption, cutting costs, and decreasing brick weight, thereby lowering transportation expenses.

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