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Copper Mining Solid Waste as Replacement Aggregate of River Sand in Cement Mortar to Investigate the Effect on Fresh, Durability and Microstructural Properties: A Walkthrough to Sustainability

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Abstract: The present study investigates the feasibility and efficiency of river sand replacement with copper mine tailing waste as fine aggregate in cement mortar. The scope of the present study includes durability, microstructure, and morphology of mortar mixes created by different percentages replacing copper tailing. The study ranges from 0% to 50% by volume replacement levels of sand by CT with an interval of 10% in 1:3 (rich Mortar) mortars. The quantity of cement remains constant in all the mixes, and the amounts of fine aggregate and tailings are adjusted properly. Compressive, flexural, and tensile bond strength are different mechanical properties of all the mix that will be evaluated at 7 and 28 days. The effects of the replacements are to be analysed on the formation of structure, C-S-H gel, and composition with regard to the studies through morphology and microstructure tests conducted on the mortar specimens. The study shows positive results by doing up to 20% replacement with CT. After 3CT2, the results show a decrease in the results obtained from various durability tests. It will be carried out using codes, standard specimen casting, and testing methods. The results of this research will be very valuable in terms of the knowledge to be provided on copper tailing potential as a viable replacement in cement mortar and contribute much to developing far more environmentally friendly materials and construction processes.

Introduction

Mortar is the second most utilized material in the construction sector after concrete. The application of mortar as a binder has been known since prehistoric times. Initially, mortar mix was made from mud and gypsum and was used to connect the mud blocks or curved blocks of stone (Moropoulou et al., 2005). During 3000 BC, asphalt was used to produce mortar, which was basically overtaken by slaked lime. These mortars were commonly utilized in Roman as well as Greek architecture. The Romans later invented pozzolana lime mortar through the addition of pozzolana material in lime-based mortar. Cement mortar first gained popularity in the 1870s when the United States implemented Portland cement.

The mortar made from cement is still commonly used because of its homogenous quality and ability to develop strength rapidly as compared to lime or pozzolana lime mortar. (Ballester et al. 2007). A traditional mortar combination consists of three components: a substance known as a binder (cement), finely ground materials (river sand or manufactured sand) and water, the cement acting as a bonding unit and the water contributing to accurate mixing; fine aggregate is an intrinsic part of a mortar mix. It offers essential durability properties as well as mechanical properties to the complete cement mortar mix. Using high-quality fine aggregate is important for producing superior mortars (Turgut and Murat Algin, 2007; Martínez et al., 2013; Farinha et al., 2015).

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During the mining of Copper, the residual waste materials left over after copper extraction is known as copper tailing, which offers a possible advantage when used as a replacement in cement mortar. Using copper tailing in cement mortar provides a wide range of advantages for the environment, economy and technology (Khyaliya et al., 2017; Schipper et al., 2018). From an environmental perspective, the utilization of copper tailings mainly addresses the huge problem of waste management associated with mining activities. Large quantities of tailings often require vast acreage for disposal, which threatens possible environmental hazards such as water pollution and soil degradation (Elshkaki et al., 2016). Use of copper tailings as construction materials reduces not only the amount of waste but also ensures less environmental risks. Copper mining waste materials have been earlier researched as potential alternatives for the conventional fine aggregates used in cement mortars. In some of these studies, copper mining waste materials were reported to be suitable for partly or wholly replacing normal fine aggregates. Other researchers recommended their use in cementitious mixtures (Balwan et al. 2024).

Ghazi et al. studied eight concrete mixes that contain copper mine tailings in place of cement at different replacement percentages, including 10%, 20%, 30%, 40%, 50%, and 70%. Various X-ray diffraction and fluorescence procedures by X-rays were used to assess the physical and chemical characteristics of the tailing. The workability, compressive strength, durability, resistance to chloride ion penetration, and the heavy metal leaching of the concrete samples were assessed using the techniques described in ASTM. Sample of concrete containing up to 70% copper mine tailings exhibits either equivalent or superior compressive strength to the control for 90 days. Tailings-containing samples indicated that the hydration process was more complete and resulted in crystallization of calcium silicate hydrate. This material plays a major role in the development of concrete strength. In addition, chloride ion penetration resistance was increased with time in samples containing 10-40% tailings thus, their durability was improved. Importantly, the leaching tests showed that the heavy metal leachability from all concrete samples was lower than the EPA's allowed limits, thus, tailings-incorporated concrete is environmentally safe (Ghazi et al. 2022).

Zhao et al. (2023) studied the application of iron and copper tailings in steam-cured mortar. The process of steam curing, which is widely used in producing precast concrete products to improve the rate of cement hydration at early ages for improving the strength gain of concrete, is used in this paper, which discusses the effects of various replacement levels of ICT on the mechanical properties, permeability, and durability of steam-cured mortar. The findings of the research showed that substitution of river sand by ICT in steam-cured mortar up to 20% improved both the mechanical characteristics of the material and permeability. At a substitution rate of 10%, mortar compressive strength at 1 day increased by 1.09% and, at 28 days, by 7.27% compared with the control sample (Zhao et al. 2023).

Copper tailings are low-activity solid wastes and must be mixed with ingredients like fly ash, slag, or metakaolin to find successful geopolymer systems applications. A limited number of studies have been conducted on using copper tailings either as aggregates or admixtures in cement-based products, whose results represent improvements in mechanical features and pore structure. However, until now, few investigations have been reported regarding the use of copper tailings in geopolymer-based materials, especially under alkaline conditions (Zhang et al. 2023).

Sepúlveda-Vásquez et al. identify mining waste, including copper tailings, as a potential SCM. Copper tailings are formed as by-products while copper is being purified; for every ton of purified copper, 200 tons of tailings are produced. Previous studies have shown that copper tailings can be used as a partial replacement for cement. Research results show that the addition of 10% of copper tailings to the cement leads to a gain in concrete compressive strength of about 16.2% at the age of 28 days.

CMT is high in silica (SiO_2) and iron oxide (Fe_2O_3). Thus, it can be considered as a good prospect for application as an SCM in cement-based products. Its pozzolanic properties reacting itself with calcium hydroxide in the presence of water to form cementitious products are able to enhance concrete and mortar in both strength and durability. Previous studies demonstrated that CMT enhanced the mechanical properties of cement-based materials in terms of compressive and flexural strength and improved their resistance to chemical attack and environmental degradation (Arunachalam et al., 2023).

Shuimu Hu and Wenmin Zhang studied the influence of various concentrations (i.e., 0%, 25%, 50%, and 100%) of CMW on the setting time, hydration characteristics, compressive strength, and microstructure of LCC mortars. The need to reduce the ecological impact of ordinary cement has formed the foundation for developing low-clinker cement based on a wider context of sustainable construction practices (Hu and Zhang, 2023).

Wang et al. underline the urgency of finding sustainable solutions for the tailings left during copper production CTs that are heavily environmentally at risk due to heavy metal contaminants and acid mine effluent. The current procedures for recycling them via reduction roasting and acid leaching have high intensities of energy use and pose significant environmental risks. A novel approach for the functionalization of CTs using tannic acid is proposed here. That is to say, it improved CT reactivity by 35.6% after 28 days, hydration in mortar, and compressive strength. Such improvements are characterized by eco-efficiency and cost-benefit, which attained CO_2 reduction and cost savings of 34.7% and 33.0%, respectively (Wang et al., 2023).

İlknur Bekem Kara investigated that copper tailing, a by-product of mining and human activities, has become a global environmental concern with the increasing demand for copper. In this work, mortars were prepared with various CT replacements of 5%, 10%, and 15% blended with 2% NS and 10% MS by weight, and the mechanical properties of the mortars were tested. Results show that in mainly crystalline CT, the sum of SiO_2, Al_2O_3, and Fe_2O_3 is 91.40%, while the ignition loss is 4.04%. Addition of 5% CT increases the compressive strength compared to the reference mixture (İlknur Bekem Kara, 2021).

Ince et al. investigated the inclusion of copper mine tailings, which raises a number of the broadest concerns related to sustainability. Cement production is a significant contributor, about 5%, to global CO_2 anthropogenic emissions. One pathway to reduce environmental impact is through the use of alternative resources mine tailings which can reduce demand on virgin raw materials and energy use (Wang et al., 2023).

Maharishi et al. investigated natural fine aggregates that have been replaced by copper slag in different volumes: 20, 40, 60, 80, and 100%. Strength increased in the mix that replaced copper slag with natural fine aggregates by up to 40% compared with the control mix. Specifically, the mix with 40% substitution of copper slag (CS-40) had the highest compressive strength after 28 days of curing, which lay approximately 13% more than the strength of the control mix (Maharishi et al., 2023).

Dandautiya & Singh mentioned in their research that concrete is a critical material in the construction industry and is only surpassed in use by water on a global scale. Nevertheless, one of the basic constituents of concrete, cement, has an extremely high energy requirement in its processing and is responsible for about 5-7% of all CO_2 emissions worldwide. In this context of environmental issues, the approach turns to by-product-based substitute materials like FA and CT, both coming out from industries as by-products (Dandautiya and Singh, 2019).

Onuaguluchi and Eren lead off with a description of the expanding problem of copper tailings, stating that for every ton of copper produced, a large volume of solid and liquid tailings is generated, which can create serious acid mine drainage and poisoning of community soil and water by heavy metals near copper mines. Among all the mixes that were tested, a 5% addition of the pre-wetted copper tailings provided the best results, indicating that this level of incorporation may prove optimal (Onuaguluchi and Eren, 2012).

Poloko et al. note that China continues to be the largest recipient of copper in the world, as illustrated in the article. The authors proceeded to discuss the decline in copper prices since 2011, which has forced mining companies to adopt economical and environmentally sustainable treatment procedures. Results of the XRF analysis indicated that the ore is primarily composed of quartz, SiO_2, as the major gangue material, at a percentage composition of 60.6%. Other major components are Al_2O_3, CaO, MgO, and TiO_2; Fe, S, Pb, and Co were also present in trace amounts (Poloko et al., 2019).

Materials and Methods

Raw Materials of Mortar

Portland Pozzolana Cement (IS-1489, 1991) employed in this study was as per the specifications stipulated in IS 1489 - Part 1, 1991. Details of physical properties of PPC are presented in Table no 1, and the figure taken through SEM is given in Figure 1.

Table 1 Portland Pozzolana Cement Physical Properties.

Physical Properties	Result
Specific Gravity	2.9
Bulk Density in kg/m^3	1100
Normal Consistency in %	31
Initial Setting Time in minutes	132
Final Setting Time in Minutes	228
Compressive Strength of Cement (MPa)	36.3

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procured from local vender in Pratap Nagar, Jaipur.

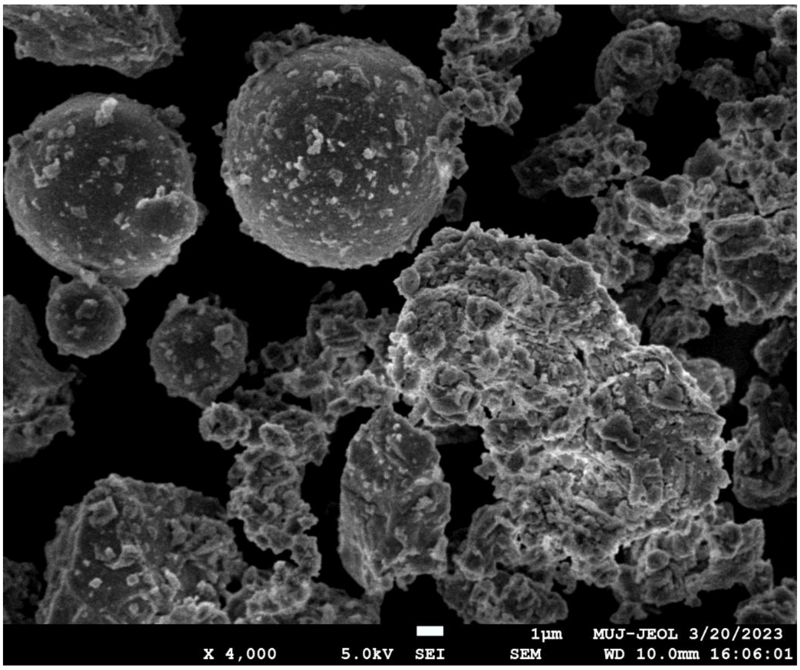

Figure 1. Scanning Electron Microscopy Image of Cement.

Table 2. Physical Properties Tested of Fine Aggregates.

Sl.NO.	Properties	River Sand	Copper Tilling
1	**Specific Gravity**	2.60	3.20
2	**Moisture Content %**	10	16
3	**Fineness Modulus**	1.94	1.64
4	**Bulk Density (kg/m^3)**	1628	1467

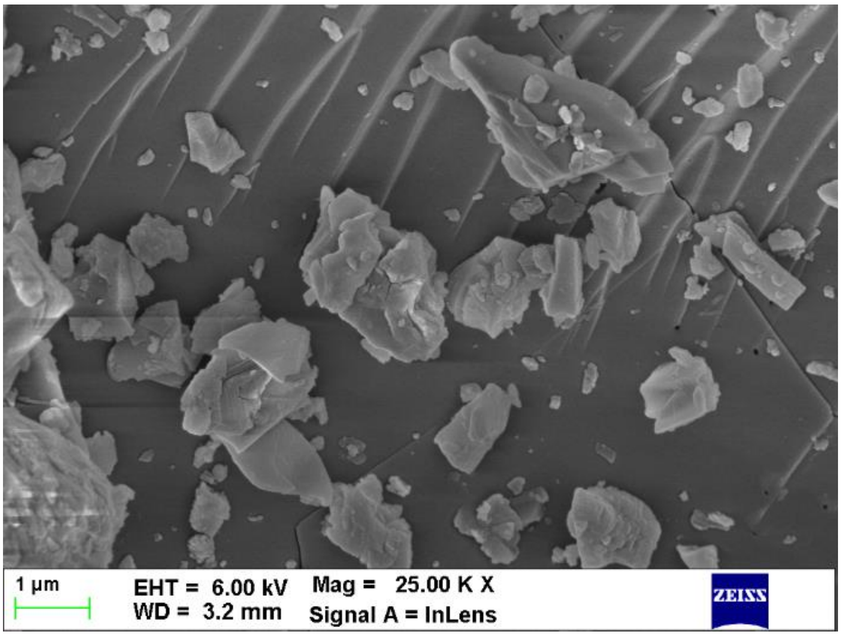

Figure 2. Scanning Electron Microscopy Image of River Sand.

The conventional natural river sand that was used was fine river sand (FS), which complies with IS: 1542-1992 (sand for plaster) (IS-1542, 1992) and IS: 2116-1980 (sand for masonry mortar) (IS-2116, 1980). The sand was Based on the grain-size distribution, the physical properties of fine sands and copper tailing are given in Table 2 below. Scanning electron microscopic images of fine sand are shown in Figures 2.

X-ray diffraction spectra for Fine Sand are shown in Figure 3, where the dominant mineral is silica at diffraction angles of 26.83°. It is observed that the percentage concentration of silica (SiO_2) is as per requirement for a mortar (Rathore et al., 2024).

Copper tailing (CT) was procured from a mining site located at Khetri, Jhunjhunu (Rajasthan) and used in mortar as a partial replacement for sand. Prior to its characterization and use, the CT was sun-dried to remove its inherent moisture. Based on the particle size distribution curve (ASTM D., 2014; 6913–04, 2009) presented in Figure 4, CT has been classified under zone-IV (IS 383, 2016). Its various physical properties, such as bulk density, specific gravity, and water absorption, are enlisted in Table 2.

IS 1542 (1992). Copper Tailing (CT) was partially used to replace the fine sand by 10% volumetric quantity, and thus, the replacement percentage is from 0% to 50%. The various components required to prepare one cubic meter of mortar are given in Table 3. Indeed, it has been demonstrated that above 50% replacement level, mixing in trial mixtures becomes more difficult and requires extra water to attain appropriate workability. Thus, mixtures with more than 50% replacement level were beyond the scope of this part of the study. The mixed variation is divided into five series, namely 3CT0 to 3CT5. Mortar compositions were prepared using a volumetric ratio of 1:3 with CT in Series 3CT. Tests were performed based on workability, fresh density, mechanical strength, durability and microstructural

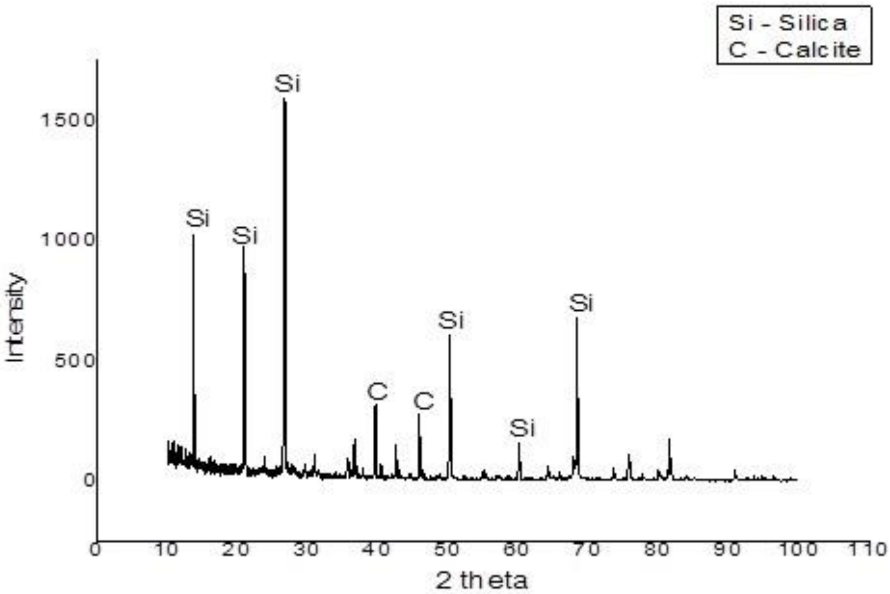

Figure 3. X-ray Diffraction Spectra (XRD) of River Sand (FS).

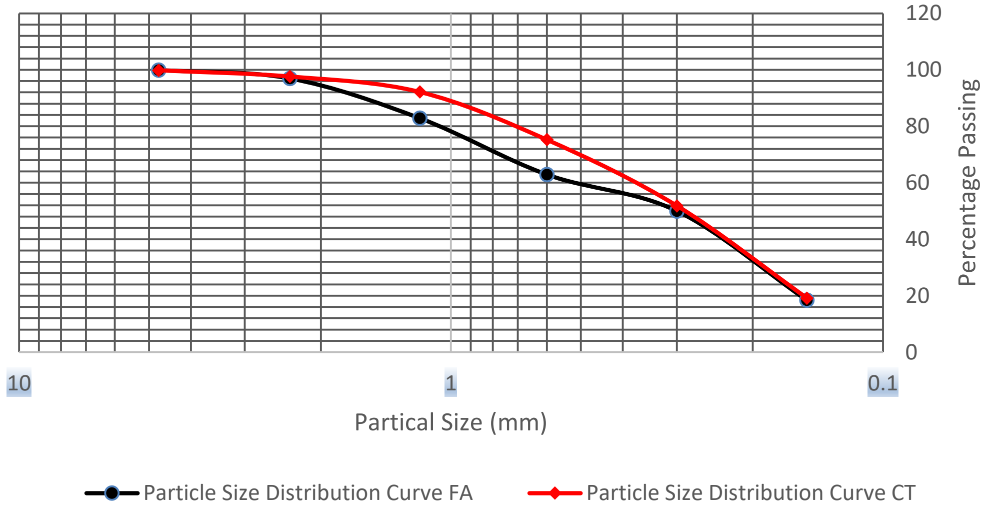

Figure 4. Particle Size Distribution of River Sand and Copper Tailings (CT).

Mixing compositions for cement mortar

At this stage, the control mortar was prepared using Fine Sand (Zone IV), conforming to IS 2116 (1980) and

properties. The properties were interpreted with respect to the control mortar.

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Table 3. Sand, CT Cement and Water Mixing as per Replacement of Copper Tailings (CT).

Mix Id	% of Sand	% of CT	Cement (kg)	W/C Ratio	Water (kg)	Sand (kg)	CT (kg)
3CT0	100	0	350	0.87	305	1494	0
3CT1	90	10	350	0.92	322	1345	129
3CT2	80	20	350	0.98	343	1195	259
3CT3	70	30	350	1	350	1046	389
3CT4	60	40	350	1.1	385	896	519
3CT5	50	50	350	1.15	403	747	648

Figure 5. Sample Casting for Different Tests.

The experimental study for various properties was conducted at Poornima University, Jaipur.

Result and Discussion

In this regard, trial mixes of rich cement mortars using air-dried copper tailing were studied, and their characteristics were compared to those of the reference mortar. Further, the necessary fine aggregate properties in mortar were achieved by incorporating copper tailing in a gradation as per BIS 1542 and BIS 2116. The mix ratio of CT suggested was 50:50. Finally, the mechanical and durability aspects of mortar mixes with 1:3 mixes were studied. Detailed information about the findings and discussion is presented in the subsequent sections.

Workability of Fresh Mortar

It was found out from the test results that the addition of CT up to 20% with river sand increased the w/c ratio by approximately 12%. Indeed, the test results indicate that with lower amounts of increment in water, up to 20% CT is able to achieve workability similar to the control mix. This, in turn, made available water more effective due to improved utilization of cement hydration of the mortar paste. While at 30% replacement level, the water-to-cement ratio in the replacement mortar remained approximately the same. Beyond that, however, the paste's larger surface area took more water to provide the needed flow of 205-215 mm as shown in figure 6.

For the reference mortar specimen, the desired flow in the 1:3 mix was achieved with a w/c ratio of 0.87, while this ratio increased to 1.15 for the mix containing 50% CT. The w/c ratio increased by a significant value of 32% in the case of the addition of a maximum amount of CT in mortar.

Fresh Bulk density

Figure 7 presents the trend of fresh bulk density of cement mortars with increasing CT consumption. It has been noticed that as CT replaced RS in cement mortars, mortar density increased for all mix quantities. The reference mix at a 1:3 proportion had a maximum density of 1.68 gm/cc and the mortar prepared with 50% CT in

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the same proportion had a minimum density of 1.91 gm/cc. In 1:3 mixtures, the fresh bulk density of mortars with 50% CT was increased by 12%. The main reason for this increment in density was due to the specific gravity of CT, which was 3.20, compared to FS, which was 2.60. The other reason for the lower bulk density was that mixtures containing CT had higher water content. The similar trend was reported when on replacing fine aggregate in concrete with copper tailing of higher specific gravity. There was an increment in the bulk

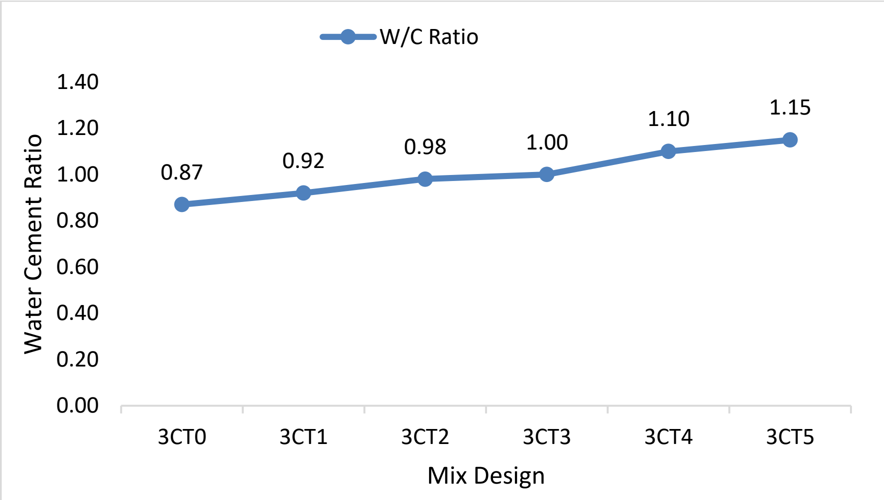

Figure 6. Water Cement Requirement of Mortar with Varying % of Copper Tailing.

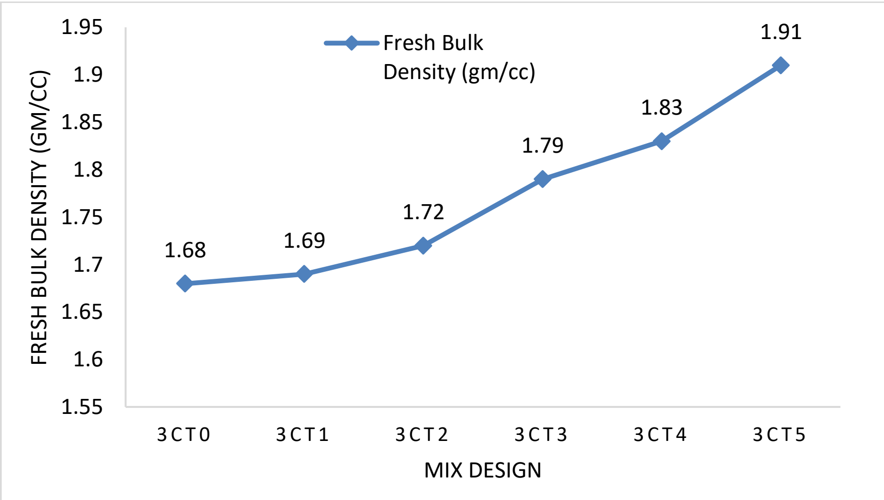

Figure 7. Fresh Bulk Density of Mortar with Varying % of Copper Tailing.

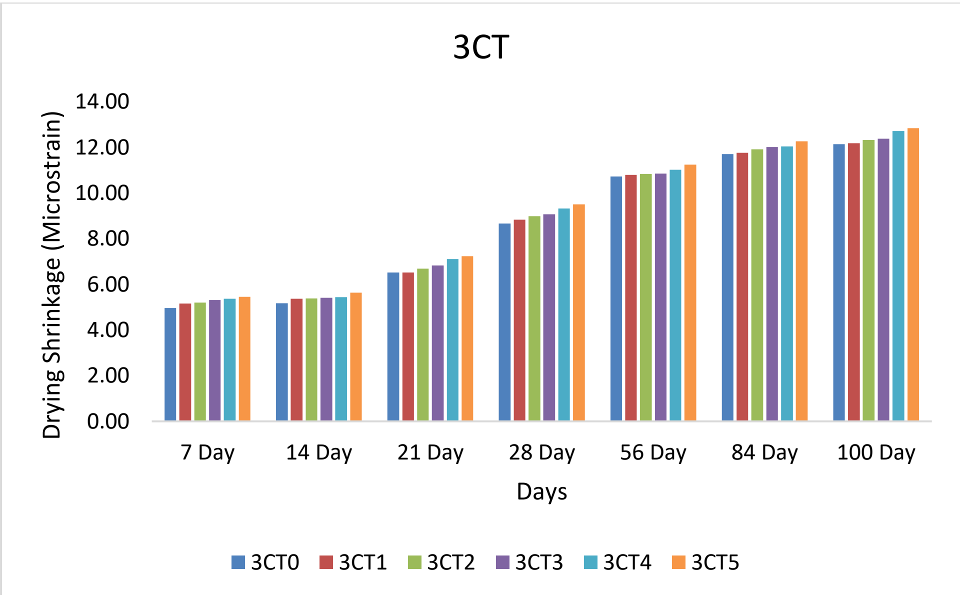

Figure 8. Drying Shrinkage of Mortar Mix (1:3) after 7 to 100 Days Curing with Varying % of Copper Tailing.

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density of the composite paste made from cement (Thomas et al., 2013).

Drying Shrinkage of Mortar Mix 1:3 with CT

The matrix of the mortar mixture loses its water content and consequently undergoes drying shrinkage. Figure 8 shows the variations in drying shrinkage of the test specimens of mortar. It can be understood from this figure that the 1:3 proportion mixes showed larger shrinkage. This mainly can again be attributed to the presence of a higher amount of cement in 1:3 mixes.

A similar trend was found that the finer dimensions of the copper tailings particles, which are increased surface area of copper tailings, will most likely increase drying shrinkage in a mortar (Siddique et al., 2010).

Sorptivity Test of Mortar Mix 1:3 and 1:6 with CT

The amount of water absorbed by capillary suction for 1:3 mix ratios, tested for 15 minutes, 1 hour, 4 hours, and 24 hours, is shown in Figures 8. The mortars containing CT showed better water absorption due to capillary suction in the mix ratio of 1:3. For the 3CT5 mortar, the maximum absorption obtained was 86.07 gm/100 cm² after a 24-hour duration. The voids of mortars containing CT at 1:3 ratio have a marginal gain compared to the control mortar. This increase in voids could be attained from the fact that the CT particles seemed in good quantity, uniform grading with particle sizes over 600 microns as mentioned in the particle size distribution curve. From these results, a trend appeared according to the phenomenon of absorption during immersion.

Salt Crystallization Testing on Mortar Mix

Among all mixtures, CT20 mixtures recorded the highest improvement in compressive strength after salt crystallization, with increases of 26% for 3CT2. Its higher density can explain this exceptional strengthening effect in the CT20 mixtures. The strength increased from 17% to 27% for the remaining mix. This was found to be attributable to crystallization of sodium sulphate that occupied the voids within mortar.

Fire Test on Mortar Mix

The samples were subjected to real fire conditions inside a furnace to study the effect of fire on mortar. The temperatures generated inside the furnace were governed

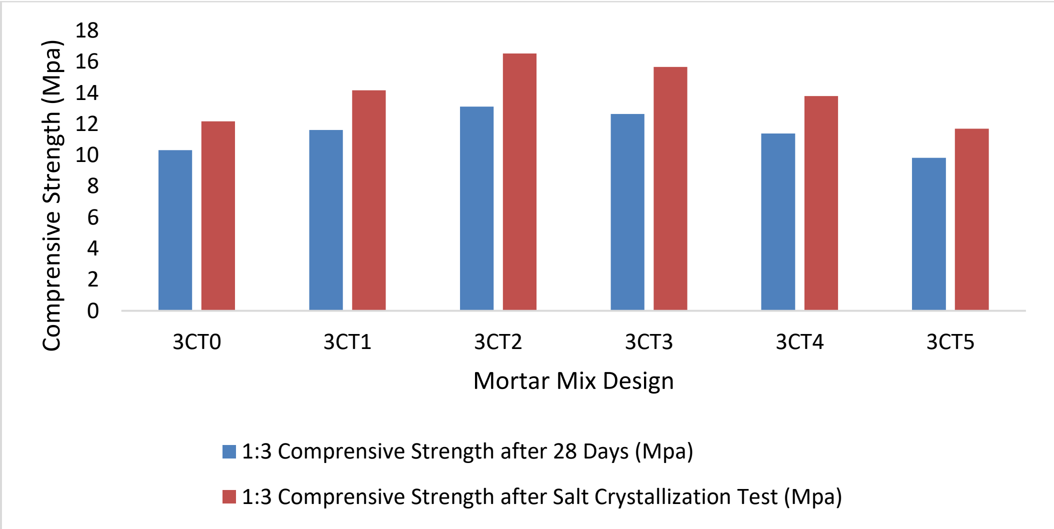

Figure 9. Compressive Strength of Mortar Cubes (1:3) after test for Salt Crystallization.

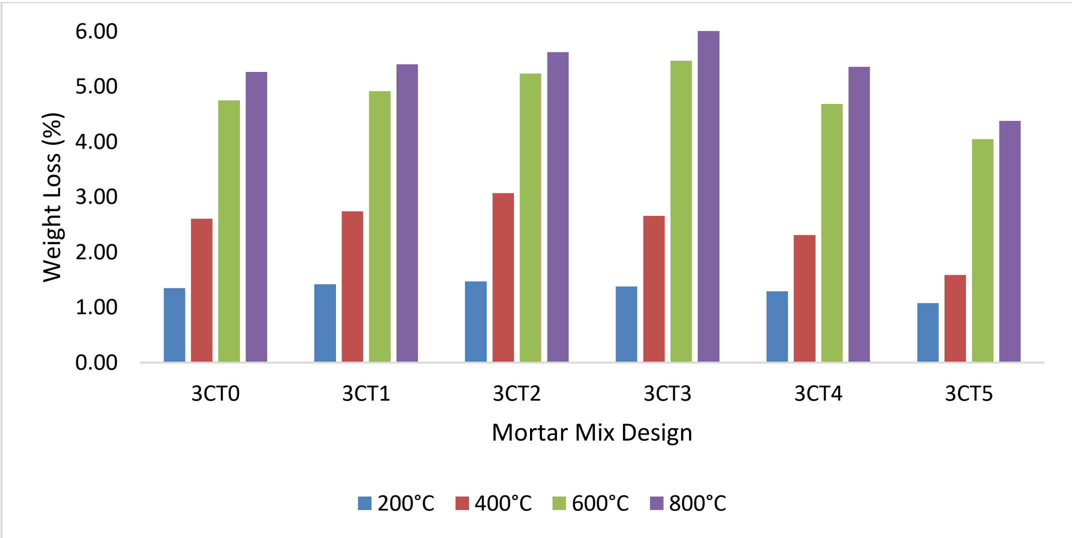

Figure 10. Weight Loss (%) of Mortar Cubes (1:3) when tested for Direct Fire.

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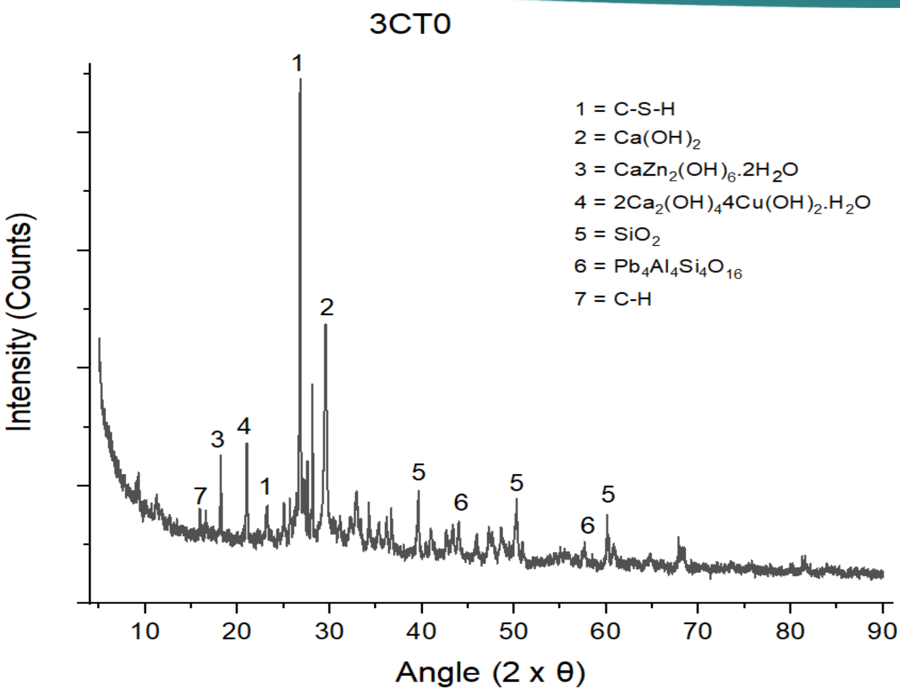

Figure 11. X-ray Diffraction (XRD) Analysis of 3CT0.

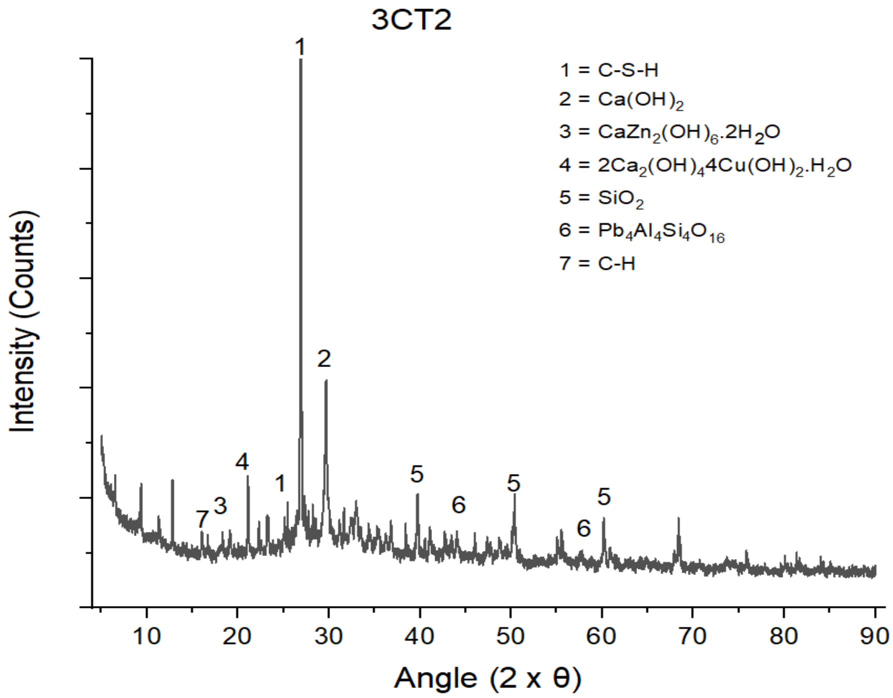

Figure 12. X-ray Diffraction (XRD) Analysis of 3CT2.

using the standard fire curve as per ISO 834. In this process, the test specimens were exposed to a maximum temperature of 800°C throughout the exposure to fire. The weight loss of the 1:3 mortar series is presented for a temperature range of 200°C to 800°C in Figures 10. Mortars made with CT had a smaller weight loss. This can be explained by the lower amount of portlandite present in CT mortars, which was transformed into C-A-S-H. Portlandite dehydrates during heating, producing water and lime (CaO). As a result, a reduction in water loss leads to a smaller overall amount of weight loss.

$$Ca(OH)_2 = Heat = CaO + H_2O \ldots\ldots\ldots\ldots\ldots\ldots.(i)$$

X-ray Diffraction (XRD) Testing and Analysis of Mortar

The mortar mix sample was ground into powder for X-ray diffraction studies, taking a small section from inside the mortar specimen after removing a 10 mm layer from all sides. The X-ray diffractograms for 3CT0, 3CT2, and 3CT5 mixtures are shown in Figures 11 through 13. During hydration of C-S-H, C-H, SiO_2 and $Ca(OH)_2$ are formed. As long as C-S-H is amorphous, it cannot be easily analyzed by using X-ray diffractograms. Hence, the consumption of C_3S to form C-S-H is estimated with reference to a diffraction angle of 26°.

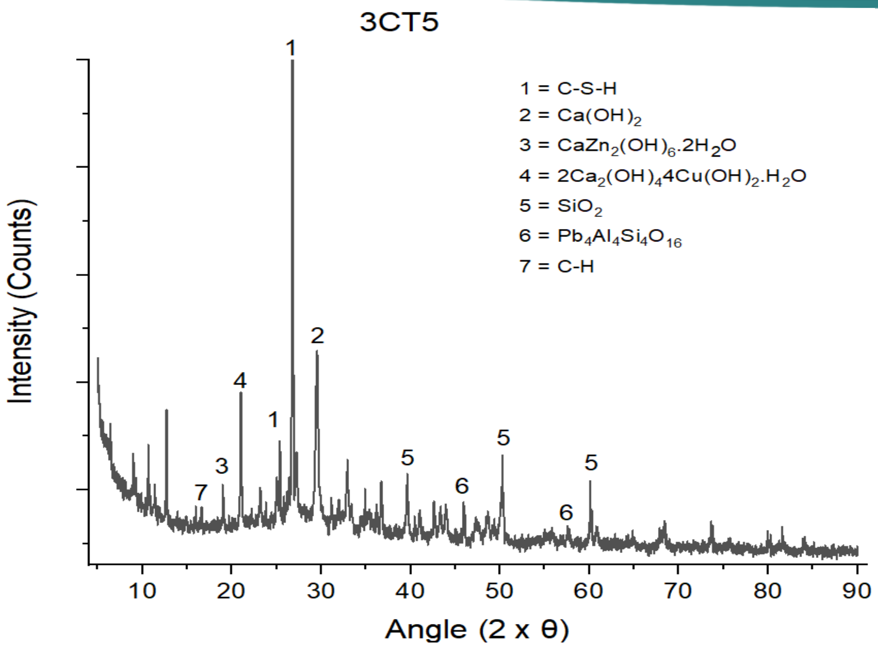

Figure 13. X-ray Diffraction (XRD) Analysis of 3CT5.

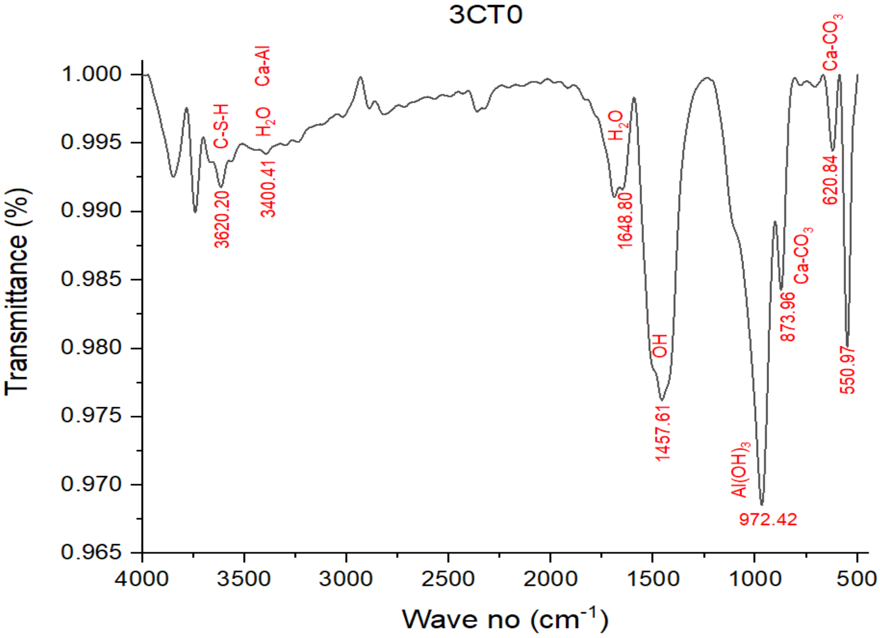

Figure 14. Fourier Transform Infrared Spectra Analysis of 3CT0.

The cement peaks for 3CT0, 3CT2, and 3CT5 mixes were at 350.52 at 26.8°, 219.40 at 26.52°, and 213.1 at 26.7°, respectively. The degree of hydration is greater for copper tailing-added mixes, as can be appreciated from the peak value of cement in copper tailing-added mortars, which is lower than that of control mortars.

The mullite of fly ash and the C-H formed by the hydration of C_3S combine to form the C-A-S-H, characterized by its 16.6° diffraction angle. The 15.92° gave the intensity of 84.86 counts for 3CT0, 182 for 3CT2, and 120.15 for 3CT5, indicating that more C-A-S-H was produced in mixes containing CT.

Analysis by Using Fourier-Transform Infrared Spectroscopy (FTIR) of Mortar

Fourier-transform infrared spectra obtained from mortar mixes with copper tailing were analysed. The equipment used for this test was 'Spectrum Two,' and it ran eight scans at 4 cm^{-3} resolution using a K-Br beam splitter. A powdered mortar sample was used for the test (Chouhan et al., 2019). The FTIR spectra of 3CT0, 3CT2, and 3CT5 mixes are given in Figures 14 to 16. A band at approximately 1000 cm^{-1} reflects hydration of the cement; this band appears at 972.42 cm^{-1}, 968.32 cm^{-1}, and 970.52 cm^{-1}.

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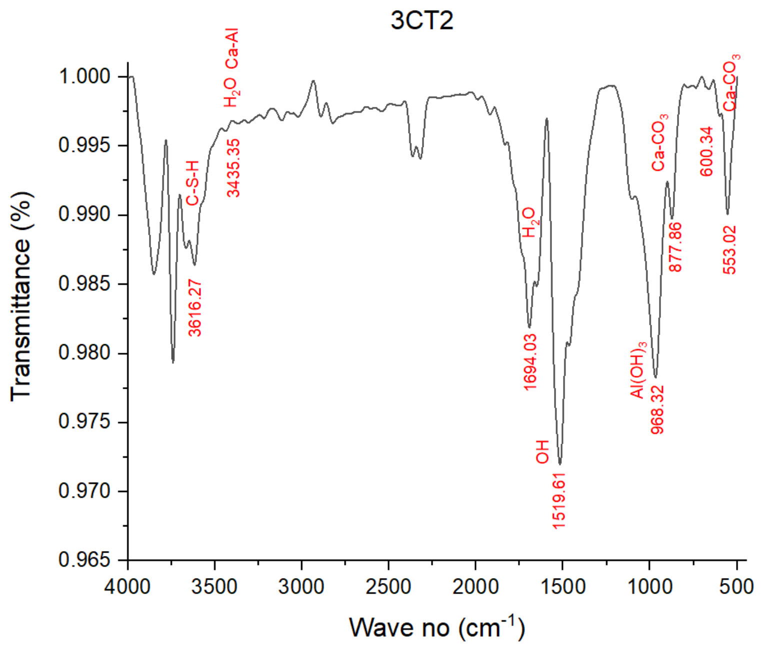

Figure 15. Fourier Transform Infrared Spectra Analysis of 3CT2.

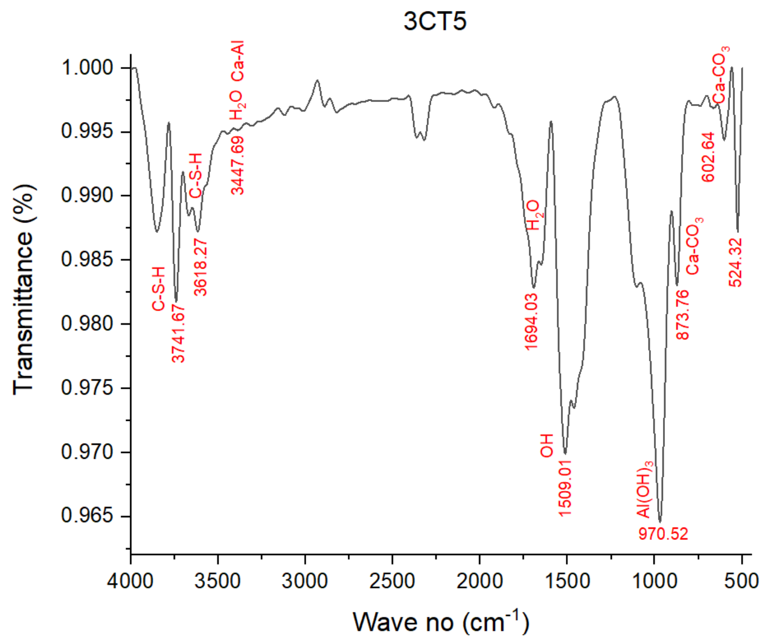

Figure 16. Fourier Transform Infrared Spectra Analysis of 3CT5.

The band assigned to portlandite is situated around 3600-3700 cm⁻¹. The spectra of the mixtures 3CT0, 3CT2, and 3CT5 have recorded values at 3620.20, 3616.27, and 3741.67 cm⁻¹, respectively. It can be observed that the FTIR spectrum presents a slight influence of the addition of CT replacing natural fine aggregate on the hydration of the clinker and portlandite cement (Ghazi et al. 2022).

Also, CT replacement to natural fine aggregate intensifies the band at approximately 1000 cm⁻¹. Therefore, this test suggests that adding copper tailing modifies the hydration reaction, which had also been supported by the XRD test and improves the strength of the studied mortars.

Conclusion

In this study, waste copper tailings were used as a replacement for river sand in the synthesis of cement mortar, and investigations into the effects of copper tailings on cement mortar strength characteristics were performed. The comprehensive conclusion of this study is given below:

- The addition of CT in cement mortars increases the fresh bulk density by 12% at 50% replacement of CT due to the fact that CT has higher specific gravity than river sand.
- Workability is improved with little water increment up to 20% addition of CT, but beyond 30%, the increased surface area of CT increases the

water-to-cement ratio substantially to as high as 32% increase at 50% replacement of CT.

- The drying shrinkage was higher for the 1:3 mixtures of mortar mix due to their higher surface area and also high cement contents. The finer copper tailing also contributed to increasing the shrinkage.
- The maximum water absorption for the mortars containing copper tailings was 86.07 gm/100 cm^2 for mix ratio 1:3, obtained after 24 hours.
- The compressive strength gain after salt crystallization was most significant in the CT20 mixtures, reaching up to a 26% gain for the 3CT2 mixture due to increased density and filling of the voids within the mortar owing to sodium sulfate crystallization.
- Mortars made from copper tailings showed a decrease in the amount of portlandite, which reduced the weight loss after heating up to 800°C.
- Compared with control mixtures, mortars containing copper tailings showed higher C-A-S-H intensity with lower intensity of cement peaks, indicating improved hydration. This was confirmed using X-ray diffraction analysis.
- The FTIR study, supporting the XRD data, proved that copper tailing improves hydration reaction in mortar; increasing especially the intensity of a band around 1000 cm^{-1} increased the strength in mortar.

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Conflict of Interest

The authors state that they have no known competing financial interests or personal relationships that could have influenced the work presented in this study.

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