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Effect of Slag on the Physical and Mechanical Properties of Microwave-Sintered Floor Tile

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ABSTRACT

Copper slag is a solid waste generated during the industrial production of copper, and its accumulation in the environment poses significant challenges. To date, copper slag has been utilized in various industries, including construction, where it serves as clinker in cement, aggregate in asphalt, a component in glass compositions, and as a raw material in tile and brick manufacturing. Its application in these areas is primarily due to its chemical composition, which contains oxides of calcium, iron, silica, and alumina—components commonly found in cement clinker, asphalt aggregate, glass, tiles, and bricks. Therefore, the use of copper slag not only mitigates environmental waste but also reduces the consumption of non-renewable natural resources. One of the objectives of the present study is to explore the incorporation of copper slag in the tile industry to decrease slag reserves. The results indicate that adding slag to the tile composition increases the density and reduces the porosity of the floor tile after sintering. Specifically, the addition of 5 wt% slag allowed the sintering temperature to decrease from 1200°C to 1170°C, while maintaining comparable density and porosity values. The addition of slag had a negligible effect on the hardness of the samples; however, it significantly influenced flexural strength. The incorporation of 15 wt% slag resulted in an approximately 30% increase in flexural strength compared to tiles without slag.

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1. INTRODUCTION

In industrial tile production, firing is typically conducted in tunnel furnaces using gas over prolonged heating cycles, which contributes significantly to air and environmental pollution. The use of non-fossil energy sources, such as microwave energy, can mitigate environmental impact and reduce production costs by lowering the firing temperature and duration. The global increase in industrial activities has generated substantial quantities of waste and by-products, raising both environmental and economic concerns. Copper slag, a by-product of copper smelting, is produced in large volumes—typically between 2.2 and 3 tons per ton of copper extracted. Its chemical composition varies depending on the ore source and production technique, but generally consists of oxides of iron, silicon, aluminum, and calcium. Typical compositional ranges

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are FeO (30–40%), SiO_2 (35–40%), Al_2O_3 (0–10%), CaO (0–10%), and copper oxide (0.5–2.1%). Given the environmental burden of its accumulation and the inefficient utilization of natural resources, copper slag has attracted increasing interest for potential reuse (Gorai et al., 2003; Klaffenbach et al., 2023; Kundu et al., 2023).

Extensive research has investigated the use of copper slag in construction materials. It has been demonstrated that copper slag can replace up to 40% of conventional fine aggregates in concrete, achieving optimal mechanical performance. Coarser slag particles have been incorporated into asphalt mixtures, contributing to the development of durable paving materials (Raposeiras et al., 2018; Sahu et al., 2024). Copper slag has also been used as a complete sand substitute in concrete, achieving compressive strengths up to 150 MPa. Unrefined copper slag can be classified as a pozzolanic material, with SiO_2 + Al_2O_3 + Fe_2O_3 exceeding 70%, making it suitable as a substitute in cement clinker production (Aly et al., 2018; Feng et al., 2019; Prem et al., 2018).

Despite these applications, only a small fraction of copper slag is currently reused, with the majority being landfilled or stockpiled, resulting in environmental and social challenges. This underscores the need for further studies and innovative strategies to expand its utilization. Recently, partial substitution of clay with copper slag in brick and ceramic tile production has emerged as a promising reuse strategy (Hou et al., 2023). Copper slag has also shown potential in glass and ceramic industries; however, the release of sulfur gases at high temperatures limits the slag content to a maximum of 40 wt%. Among tested compositions, the most effective mixture containing 40 wt% copper slag achieved excellent performance after sintering at 1025 °C for one hour, with a flexural strength of 57 MN/m², water absorption of 2%, Vickers hardness of 750, and strong acid resistance (Kim et al., 2016; Lassinantti Gualtieri et al., 2018; Luz & Ribeiro, 2007; Mohaddes Khorassani et al., 2020). Research on the application of copper slag in structural ceramics remains limited. Beyond reducing waste and dependence on virgin raw materials, incorporating industrial by-products like copper slag into ceramics provides environmental benefits, notably by reducing surface excavation typically required for natural resource extraction (Shi et al., 2008; Wang et al., 2021). Consequently, the use of slag in building ceramics is expected to be more sustainable than in structural ceramics, with additional advantages such as reduced transportation costs and lower sintering temperatures compared to conventional raw materials. Microwave energy has been demonstrated to be highly effective for the uniform and rapid synthesis of ceramic materials (Ebadzadeh, 2010). Unlike infrared energy, which is initially absorbed at the material surface and gradually penetrates conductive objects, microwave energy is absorbed volumetrically from the outset of radiation. This is due to the increasing dielectric loss coefficient of ceramics with temperature, which facilitates rapid internal heating.

In the present work, floor tile composition powder prepared under factory conditions was used to prepare the samples. The effects of adding copper slag on the sintering behavior, microstructure, and phase development of the floor tile composition were examined. In addition, the influence of slag on the physical and mechanical properties of the pressed samples was investigated.

2. Experimental Procedures

The chemical composition of the floor tile and slag is presented in Table 1. Different levels of copper slag (5, 10, and 15 wt%) were incorporated into the floor tile mixture. The resulting mixture was milled in an alumina mill with alumina balls in an aqueous medium for 2 hours. After drying, the powder mixture was pressed into bar-shaped samples with dimensions of 4.25 × 2.7 × 5 mm under a pressure of 5.5 MPa in a steel die. Sintering of the samples was performed in a microwave furnace (900 W, 2.45 GHz) at temperatures of 1150, 1170, and 1200°C without soaking time.

Phase analysis of the sintered samples was carried out using a Siemens D500 X-ray diffractometer with Cu Kα radiation. Bending strength measurements were performed on samples measuring 30 mm in length, 10 mm in width, and 8 mm in thickness. At least three samples were tested in each case, and the reported values represent the average. Scanning electron microscopy (SEM) studies were conducted on polished specimens using a Leica 360 Stereoscan, Cambridge LTD.

3. Results and discussion

Figure 1 shows the powder mixture of floor tiles containing 15 wt% of slag. As can be seen, the powder

TABLE 1. The chemical analysis of the floor tile and slag

Wt%	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	K_2O	Na_2O	SO_3	L.O.I
Floor tile	65	18	4.8	2.5	1	1.5	1.8	-	5.4
Slag	43.7	9.3	24	5.4	4.2	1.5	9.5	2.4	-

mixture contains irregular shapes of agglomerates (Fig. 1a) in the range of 30 to 40µm, which are placed among smaller agglomerates. A closer look at the agglomerates at high magnification, shown in Figure 1b, demonstrates that the large agglomerates are covered with fine 'cauliflower-like' agglomerates.

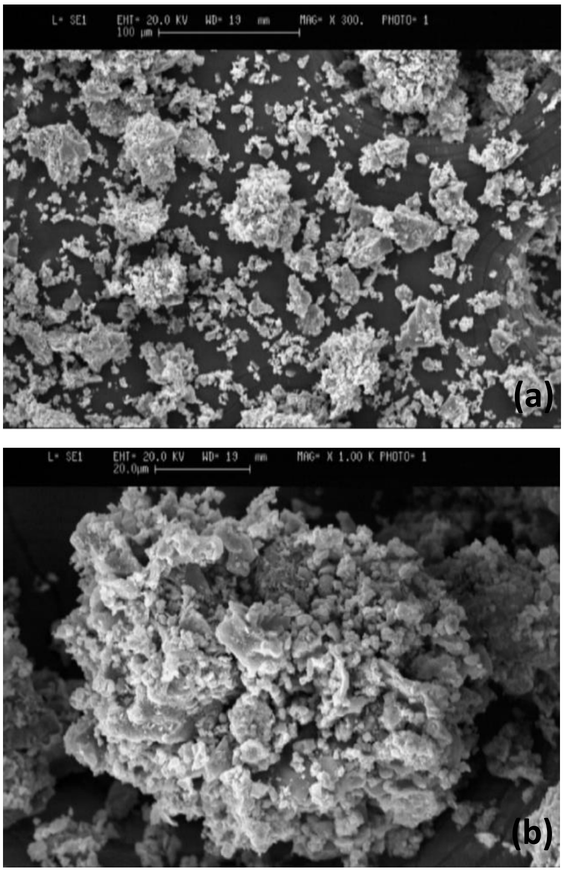

Figure 1. Electron microscope images of the powder mixture of tile and slag (15 wt%) after grinding, (a) low and (b) high magnification

Figure 2 shows the appearance of sintered specimens at 1170 and 1200°C. As observed, samples sintered at 1200°C, particularly those with higher slag content, exhibited swelling and increased surface roughness due to the formation of a liquid phase at elevated temperatures. For this reason, mechanical properties were measured only for the samples sintered at 1170°C.

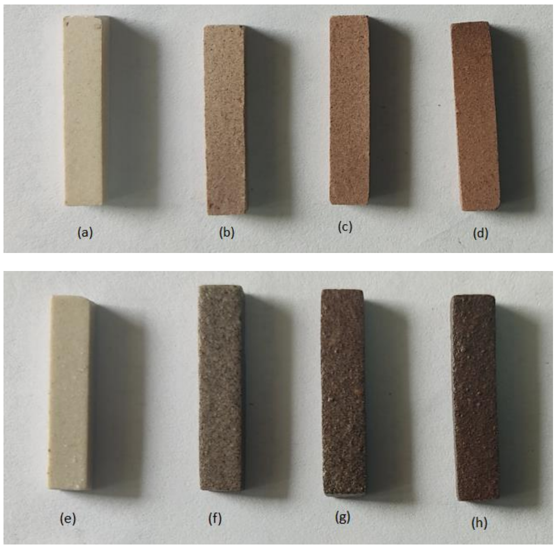

Figure 2. Slagless sample (a, e), sample containing 5 wt% (b, f), 10 wt% (c, g) and 15 wt% (d, h) of slag sintered at 1170°C (a, b, c and d) and 1200°C (e, f, g and h)

The X-ray diffraction patterns of samples containing different amounts of slag and sintered at 1170°C are shown in Fig. 3. As observed, increasing the slag content from 5 to 15 wt% did not alter the phases present. All samples containing slag exhibited the same phases as the slag-free sample. A closer examination of the peak intensities in Fig. 3 indicates that the intensities of the quartz and anorthite peaks decrease with increasing slag content. This reduction may result from the formation of a greater amount of liquid phase at the sintering temperature as the slag content increases, which ultimately leads to the dissolution of more quartz and anorthite particles into the liquid phase. The X-ray diffraction patterns of the samples sintered at 1200°C were not included due to surface melting.

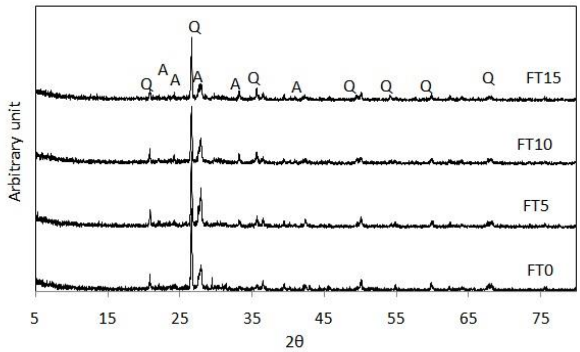

Figure 3. X-ray diffraction patterns of samples sintered at 1170°C without holding, FT0 sample without slag, FT5, FT10 and FT15, samples containing 5, 10 and 15 wt% slag

Changes in density and porosity with varying slag content and sintering temperature are presented in Figures 4(a) and 4(b), respectively. The results indicate that, in all samples, increasing the sintering temperature leads to an increase in density and a decrease in porosity. An exception occurs for the sample sintered at 1200°C, where porosity increases as the slag content rises from 10 to 15 wt% due to the increased formation of liquid phase and sample swelling; however, the porosity remains lower than that of the samples sintered at 1150 and 1170°C. From the trends in density (Fig. 4(a)) and porosity (Fig. 4(b)), it is evident that adding 5 wt% slag allows the sintering temperature to be reduced from 1200 to 1170°C while achieving nearly the same density (1.96 and 1.98 g/cm³, respectively) and porosity (25.4% and 25.1%, respectively).

Furthermore, at a fixed sintering temperature of 1170°C, increasing the slag content from 0 to 15 wt% raises the density from 2.02 to 2.25 g/cm³ and reduces the porosity from 18.4 to 15.2%. When both the sintering temperature is varied from 1150 to 1200°C and the slag content from 0 to 15 wt%, the sample density increases from 1.96 to 2.33 g/cm³ (a 15.9% increase), while the porosity decreases from 25.4 to 6.7% (a 73.6% reduction).

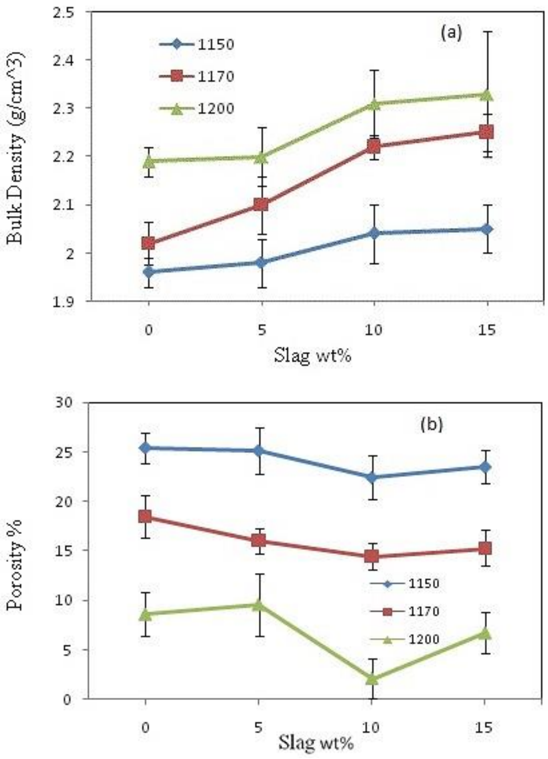

Figure 4. Changes in density (a) and porosity (b) of samples sintered at different temperatures with changes in the amount of slag

The microstructural images presented in Figure 5 show that porosity decreases with increasing slag content, which confirms the density and porosity measurements reported in Figures 4(a) and 4(b).

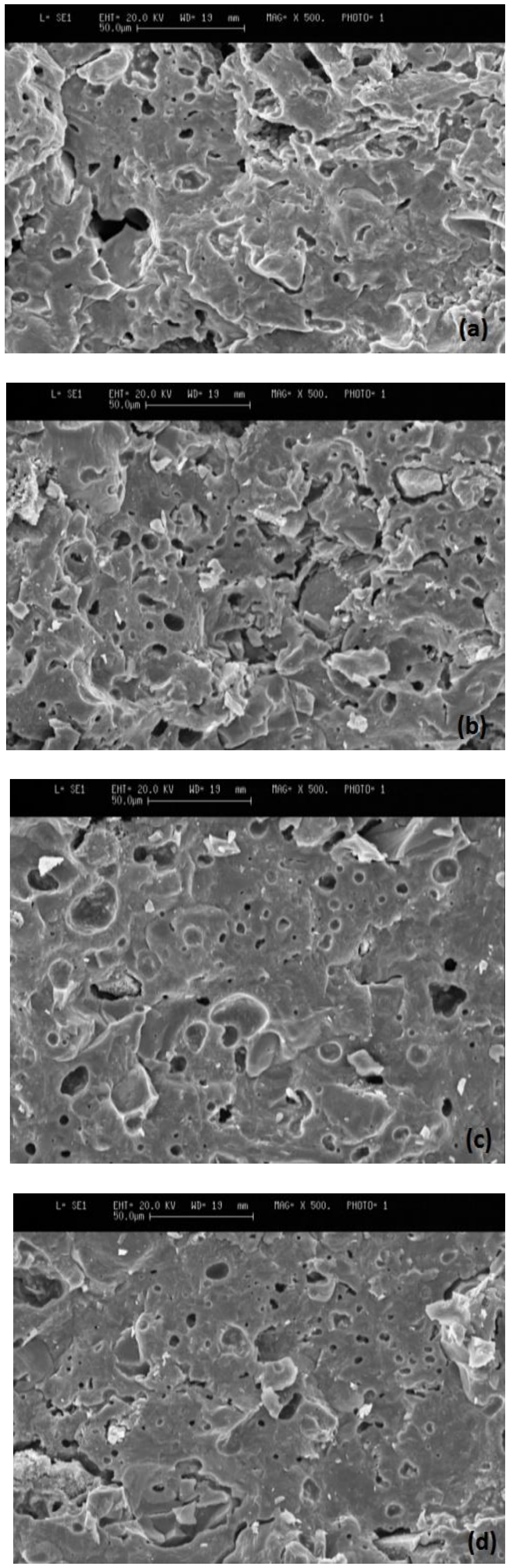

Figure 5. Electron microscope images of the sample sintered at 1170°C containing (a) 0, (b) 5, (c) 10 and (d) 15 wt% slag

Table 2 presents the hardness and flexural strength of samples without slag and with varying amounts of slag. As observed, changes in hardness with varying slag content are negligible. The slightly lower hardness values in the slag-containing samples compared to the slag-free sample can be attributed to the partial dissolution of anorthite and quartz phases into the liquid phase formed by the addition of slag during the sintering process. This is supported by the X-ray diffraction patterns shown in Figure 3, where the intensities of the anorthite and quartz peaks decrease with increasing slag content.

Microwave sintering of a floor tile sample with the same composition used in previous work (Ebadzadeh, 2009) showed that after sintering at 1056°C without holding time, a density of 2.22 g/cm³ and a porosity of 11.93% were obtained. In the present study, the sample containing 10 wt% slag and sintered at 1170°C exhibited the same density and slightly higher porosity (2.22 g/cm³ and 14.5%, respectively). Although a higher sintering temperature was employed in the present work, increased porosity was observed compared to the previous study (Ebadzadeh, 2009). This may be attributed to the presence of compounds such as $FeSO4$ and $CuSO4$, whose sulfur oxide removal occurs at high temperatures of 680°C and 560°C, respectively. Consequently, the removal of sulfur oxides at these temperatures is likely to induce additional porosity despite the increase in sintering temperature.

Table 2. Changes in hardness and flexural strength of samples sintered at 1170°C containing different amounts of slag

Slag wt%	0	5	10	15
Hardness (kN/mm^2)	5.8±1.1	5.4±1.0	5.5±1.0	4.7±1.7
Flexural strength MPa	42.2±4.9	42.4±11.3	45.8±3.2	60±6.5

4. Conclusions

The results of this study demonstrate that the addition of slag increases the density and reduces the porosity of the floor tile composition. Specifically, by adding 5 wt% slag, the sintering temperature can be reduced from 1200 to 1170°C while achieving nearly the same density and porosity as the sample sintered at 1200°C. Furthermore, at a constant sintering temperature of 1170°C, increasing the slag content to 15 wt% raises the density from 2.02 to 2.25 g/cm³ and decreases the porosity from 18.43 to 12.28%. When both the sintering temperature is increased from 1150 to 1200°C and the slag content from 0 to 15 wt%, the density increases from 1.96 to 2.33 g/cm³ (a 15.9% increase), and the porosity decreases from 25.4 to 6.7% (a 73.6% reduction).

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