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DOI
10.1016/j.jksus.2023.102855

Publication date
2023

Document Version
Final published version

Published in
Journal of King Saud University - Science

Citation (APA)
Ahmed, M. M., Kassim, A., Ragab, A., EL-Razaz, Z., Al-Enizi, A. M., Yousef, A., Abdelrady, A., & El-Naggar, K. A. M. (2023). Enhanced strength, durability, and thermal shock resistance of clay roof tiles substituted with ferrosilicon slag. *Journal of King Saud University - Science*, *35*(8), Article 102855. https://doi.org/10.1016/j.jksus.2023.102855

Journal of King Saud University – Science 35 (2023) 102855

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Original article

Enhanced strength, durability, and thermal shock resistance of clay roof tiles substituted with ferrosilicon slag

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A R T I C L E I N F O

Article history:
Received 24 September 2022
Revised 13 August 2023
Accepted 14 August 2023
Available online 20 August 2023

Keywords:
Ferrosilicon slag
Kaolin
Mechanical strength
Cristobalite
Corundum
Roof tile

A B S T R A C T

The present investigation involved the production of roof tile samples through the replacement of kaolin clay with varying proportions of Ferrosilicon slag (FS) (0%, 10%, 15%, 20%, and 25% by weight) at different firing temperatures (900 °C, 1000 °C, and 1100 °C). The present study investigated the impact of incorporating FS slag waste on durability, mechanical strength, thermal shock resistance, and thermal properties. Furthermore, an examination of the microstructure of the fired roof tiles was conducted through SEM analysis. The properties of the roof tiles exhibited enhancement as the percentage of FS slag increased, reaching a maximum of 15%, and the firing temperature increased up to 1000 °C. This can be attributed to the formation of significant amounts of corundum phase. Increased temperature and a higher percentage of FS slag are associated with the generation of a significant quantity of cristobalite phase, resulting in a reduction in the mechanical properties of roof tiles. The roof tile samples fabricated with up to 15% FS slag at 1000 °C exhibited low water absorption and porosity. Increases in temperature and FS slag, on the other hand, resulted in an increase in water absorption and porosity. There were no observable impacts on water absorption and apparent porosity at 900 °C. The firing temperature of 1000 °C and a slag percentage of 15% resulted in a minimum water absorption of 9.8%. This value meets the standard requirements for moderate weather resistance. Notwithstanding the increase in density of roof tiles containing elevated proportions of FS slag, they continue to fall within the limits of lightweight roof tiles as stipulated by determined standards. The experimental results indicate that the incorporation of 15% FS slag and firing at a temperature of 1000 °C resulted in a significant increase of 34.9% in the transverse breaking strength (TBS) of the clay roof tiles when compared to the conventional sample. This suggests that the structural properties of the clay roof tiles were improved through the addition of FS slag.

1. 1.Introduction

Expanding on the production of clay roof tiles as a substitute to the asbestos sheet is a vital requirement due to the adverse health effects of asbestos (Sultana, 2015; Dondi, 1997). Roofing materials directly face the sunlight, which could be affected by the indoor thermal environment compared to other building components (Weerasuriya, 2014). Thus, the selection of suitable roof tiles could improve indoor thermal comfort. Clay roof tiles have several advantages as compared to asbestos roofing sheets, such as good appearance, thermal absorption, and human safety (De Silva and Surangi, 2017). The substituting of clay with waste materials (e.g., waste sugarcane bagasse, waste rice husk ash, sewage sludge) could have an effect on the clay roof tiles' characteristics (e.g., strength, durability, and thermal performance) (De Silva and Mallwattha, 2018). Several authors have examined the possibility of recycling various materials into clay roofing tiles. Souza et al.

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Peer review under responsibility of King Saud University.

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https://doi.org/10.1016/j.jksus.2023.102855

(Souza, 2011) investigated the possibility of using bagasse as an additive up to 60% as replacement for the clay in clay roof tiles and found that, after being fired at 1200C, the physical and mechanical characteristics increased. The possibility of using rice husk ash as a partial substitute for clay in 900C-fired roof tiles was explored by Da Silva et al. (Souza, 2011). They discovered that a 46% boost in breaking strength occurred at a 10% replacement level due to the presence of silica in the ash. The tiles' thermal resistance was also shown to enhance somewhat as a result of this. The enhanced strength may be attributed to the production of mullite, which was prompted by the presence of fine silica in the ash. A comparable result was attained by Sungkono (Sungkono, 2018) through the replacement of clay with rice husk ash at weight proportions of up to 7.5%. The statement mentioned that the addition of 7.5% ash resulted in a 15% enhancement in strength, which was attributed to the fine silica present in the ash that filled the clay pores. Costa etal. (Costa, 2009), demonstrated the utilized of different percentages of alpha (sieve size ~0.088 to 0.125 mm) and beta (sieve size ~0.037 to 0.088 mm) glass powder waste (0, 5, and 10 %) as the replacement for clay. The samples have been fired at different five temperatures (800, 900, 1000, 1100, and 1200 °C). The samples have been showed the reduced in the water absorption and apparent porosity for the samples composed of 10% go alpha and beta glass powder, which demonstrated the water absorption of < 7.5% and apparent porosity < 15%. At a temperature of 1200 °C, the Flexural strength of specimens containing glass powder alpha was observed to be higher than that of specimens containing glass powder beta. Furthermore, the incorporation of 10% glass powder resulted in a significant increase in the Flexural strength value, which was estimated to be approximately 17 MPa. Additionally, Delaqua etal. (Delaqua et al., 2020), investigated the possibility of using biomass like Salvinia auriculata Aublet as an additive to clay mixtures to create red ceramic bodies. The biomass's reasonable calorific value was used to estimate the energy savings that would occur from its use in the fire process. Contrarily, a variety of mineral byproducts were substituted for clay in roof tiles. Torres et al. (Torres, 2009), replaced different proportions of rock waste with clay. They found that by substituting 10% granite waste replaced clay, they could get the best results in terms of water absorption and flexural strength. A similar methodology was assumed by Shanjida Sultana et al, (Sultana, 2015), as fabricated roof tiles used different proportions of rock dust (10–50%) and different sintered temperatures (850–1100 °C). They obtained a strength that is more than the standards with low water absorption. They recommended a fire temperature of 900 °C and a rock dust percentage ranging from 20 to 30% in order to get a achieved the results agreement with the standard. Ferrosilicon slag (FS) is produced when quartz of high purity is reduced with coal in an electric arc furnace in the silicon and ferrosilicon industries. Due to its cheap cost, it is widely used in industries such as steelmaking and Portland cement manufacturing (Acchar, 2005). Huge amount of ferrosilicon slag is produced. It's not only very effective in building, but it's also quite cheap (Ahmed, 2021; Li, 2022; Amin, 2022). The presence of a significant amount of silicate in ferrosilicon slag leads to the formation of vitreous silicates during the maturation of clay bodies. These silicates function as fluxes, thereby reducing the maturation temperatures of clay bodies. This observation provides convincing proof that the incorporation of ferrosilicon slag into raw clay materials could enhance the firing performance of clay bodies. Consequently, the utilization of ferrosilicon slag in this manner represents a value-added implementation (Costa, 2009). For the first time, FS slag is recycled to partially substitute kaolin clay in the preparation of clay roof tiles in this study. The aim of this research is to evaluate the potential use of FS slag as a substitute for kaolin clay in roof tiles. Five FS slag proportions were used (0, 10, 15, 20, and 25% wt). Durability (water absorption, porosity of the fired tiles, bulk density, saturation coefficient, and linear shrinkage) and strength (modulus of rupture, transverse breaking strength) have been examined as a function of FS slag fractions and firing temperatures (900 °C, 1000 °C, and 1100 °C).

2. Materials and methods

The materials and methods details have been explained in the supplementary file.

3. Results and discussions

3.1. Characterization of raw materials

Sieve analysis results, shown in Fig. 1, show the particle size distributions of both raw material powders. The median particle size (D50) of clay is 0.2 mm (Fig. 1**A**) and 1.2 mm (Fig. 1**B**) less than that of FS. Fig. 2 shows an XRD pattern of FS slag and clay particles. Slag's mineral comprises a low-alpha quartz phase (Fig. 2**A**) (Ahmed, 2021). FS slag exhibits diffraction peaks mostly including silicon within the chemical formula (Si), quartz (SiO_2). Furthermore, ferdisilicite ($FeSi_2$) may be found in FS. Kaolinite ($AlSi_2O_5(OH)_4$) and illite (Fe_2O_3) may be seen in the XRD pattern of clay, as shown in (Fig. 2**B**) (SiO_2).XRF analysis has been performed to achieve the chemical compositions of the FS slag and clay. Table 1 shows the results obtained. The clay is mainly composed of aluminosilicate, whereas the FS slag is mostly composed of silica. The high silica content of FS slag contributes to an increase in the SiO_2/Al_2O_3 ratio in the clay tile casting mixtures (De Silva and Perera, 2018).

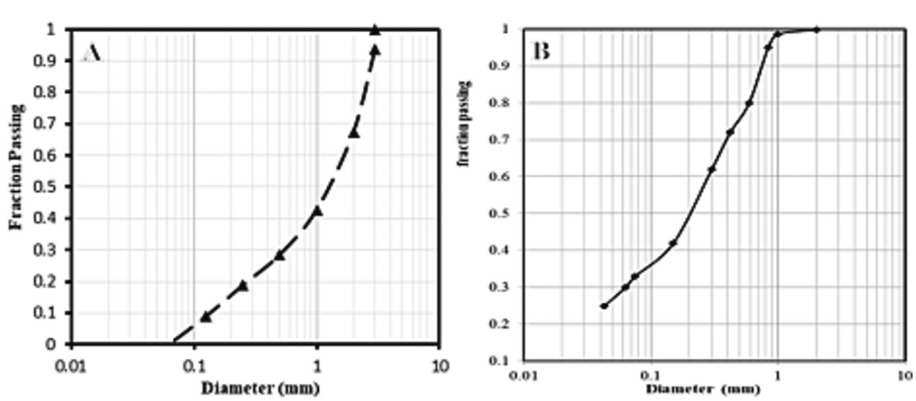

Fig. 1. Particle size distribution of raw materials (A) clay and (B) FS slag.

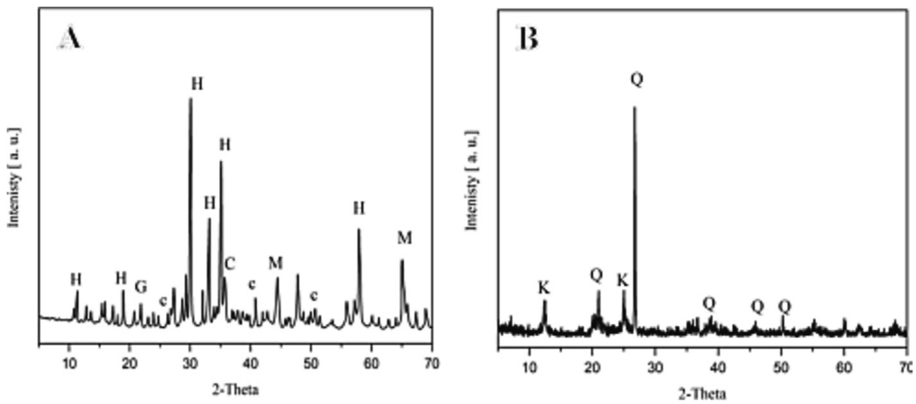

Fig. 2. XRD Analysis of FS slag (A); H. Hexagonal alumina, C. Cryolite, c. rhombohedral corundum, G. goethite and XRD Analysis of kaolin clay (B); K. kaolinite, Q. quartz.

3.2. Green body properties

The plasticity index (PI) is reduced from 32% to 20% as the percentage of FS slag increases in the mixture from 0% to 30% using 18% water content (on a dry basis) (Fig. 3**A**). This implies that up to 25% of FS slag may indeed be exploited for tile production without losing its plasticity. The decrease in the PI is expected due to the non-plastic nature of the FS slag which substitutes the plastic clay. A similar effect is observed when the clay is replaced by sugarcane bagasse in the fired clay bricks (Sungkono, 2018). Furthermore, PI increases as particle size refinement increases (Amin, 2022). However, the particle size of clay is coarser than that of FS slag which results in lower plasticity. The shrinkage depends on the clay's properties such as its mineralogical nature and particle size. As the FS slag replacement in the tile mixtures incresased dry shrinkage percent has been decreased (Fig. 3**B**), which is beneficial for walling materials (De Silva and Perera, 2018). This might be attributed to the non–plastic nature of the FS slag as well as its large particle size compared to that clay. As a result, increasing the FS slag replacement will result in lower particle mobility, and hence decreased drying shrinkage (Amin, 2022). Bigot's calculations predict a 4–8% overall shrinkage throughout the drying period (Mancuhan, 2016). If the material shrinks by more than 8% during the drying process, it is possible for the bricks to develop cracks and fractures on the inside (Mancuhan, 2016). As a direct consequence of this, the investigation into the green samples drying process has to take into account the shrinkage issue. Accordingly, the dry shrinkage of the fabricated green samples is in the range of 1.7–2.7%. The bending strength (Fig. 4**A**) and modulus of rupture (Fig. 4**B**) of green tile samples have behaved similar to dry shrinkage and plasticity, as the FS slag increased both of them decreased. The obtained strengths are 692.05, 634.4, 505.9, 491.3 and 484.7 N for 0%, 10%,15%, 20%, and 25% respectively. Similar characteristics were observed when fly ash was used as a replacement for clay (Amin, 2022; Mancuhan, 2016; Sharma and Baredar, 2018).

Table 1
Comparison with ASTM C 1167 Standards for Grade 2 clay roof tiles.

property	15% FS fired at 1000	ASTM1167 /grade 2
Apparent porosity	10%	13%
Dry breaking strength	5016.05 N	890 N
Saturation coefficient	0.726	0.86
efflorescence	No efflorescence	pass

3.3. Sintering properties of fired bodies

The density, XRD, microstructure, water absorption and total porosity, and saturation coefficient have been explained in the supplementary file.

3.3.1. Linear firing shrinkage

Fig. 5 represents the variations in linear firing shrinkage percentage with increasing FS slag percentage and firing temperature. Increasing in the firing temperature from 900 °C to 1000 °C promotes linear firing shrinkage up to 15% slag addition. It is linearly increasing with FS slag percentage and firing temperature up to 15 % and 1000 °C. An increase in firing shrinkage percentage may be due to the formation of fluxing oxides [1, 29]. This is could be

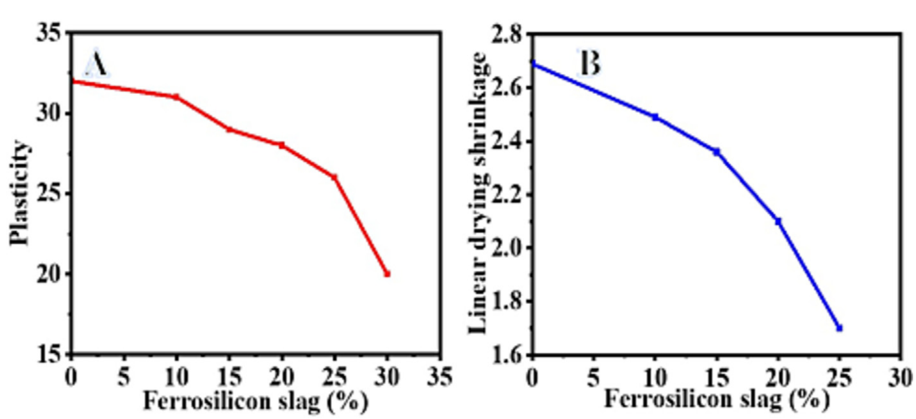

Fig. 3. The influence of FS addition on the Plasticity (A) and dry shrinkage (B).

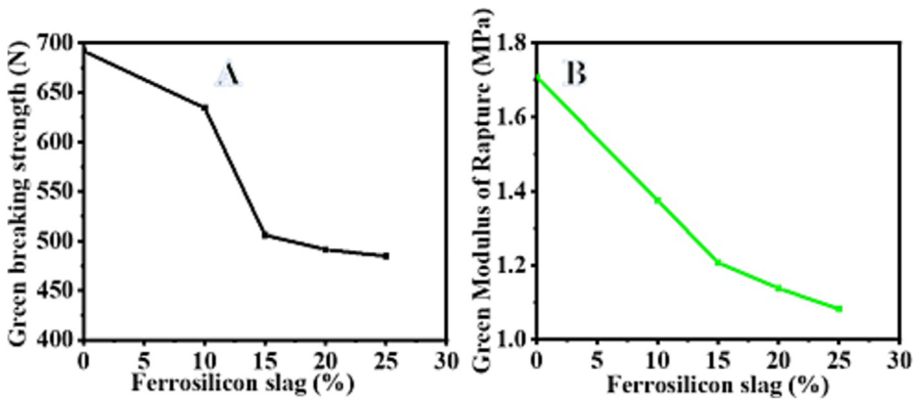

Fig. 4. The influence of FS addition on the green breaking strength (A) and green Modulus Of Rapture (B).

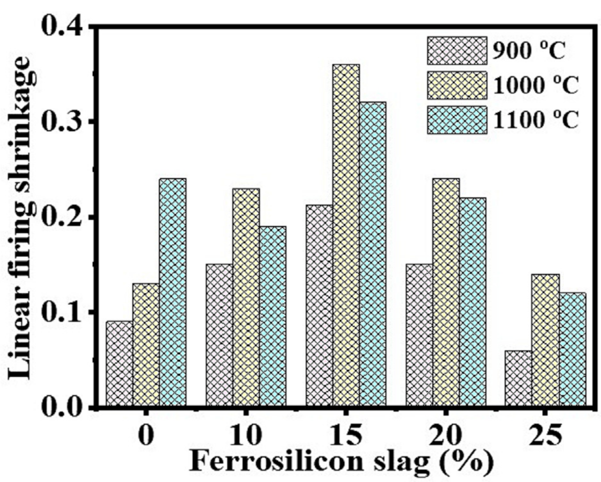

Fig. 5. The influence of FS slag addition and firing temperatures on linear firing shrinkage.

due to the formation of a high amount of corundum phase compared to that formed at 1100 °C. Higher amounts of slag addition and higher temperature of 1100 °C decrease linear firing shrinkage may be due to the formation of high pores, which leads to decreases linear firing shrinkage (Ahmed, 2021). Sultana etal. (648, A.C., Standard Test Method for breaking strength of ceramic tile., 2020), replaced red clay with hard rock dust in roof tiles production. They found the linear firing shrinkage at 1000 °C is higher than fired at 1100 °C. They proved their point with a larger quantity of corundum produced at 1000 °C. Since expansion coefficients are negatively affected by the high corundum content. Reduced tensions from this point on provide for better behavior all the way through the cooking procedure.

3.3.2. Transverse breaking strength (TBS) and modulus of rupture (MOR)

The fired tiles' compressive durability is assessed in two ways: first, the averageTBS is obtained for each sample, which is the standard measure of their strength. After that, the MOR is determined. The strength of roofing materials depends heavily on their porosity and mineral make-up, two characteristics that are often associated with their durability. TBS is raised when FS slag is used in lieu of clay and then fired. The higest TBS clay roof tiles may be produced with 15% FS slag at a kiln temperature of 1000 °C (Fig. 6**A**). This could be due to the formation of a low amount of cristobalite and higher amounts of corundum at 15 wt% FS slag and 1000 °C that enhancing the mechanical properties (Cely Illera et al., 2018). According to ASTM C1167 minimum required TBS is 890 N for type III roof tile [27]. Interestingly, all samples have TBS values higher than the standard value. For the 15% FS slag incooperated tiles at 1000 °C, the TBS was found to be 5016.05 N, indicating a 34.9% enhancement in the TBS compared to that of the conventional fired clay tile, which has the TBS of 1297.92 N. This might be due to the formation of great corundum phase. Compared to 1000 °C, the samples fired at 1100 °C and 900 °C showed the low strength. This could be due to the formation of great cristobalite phase at 1100 °C as well as SiO_2 react with free oxides and formed glassy phase at fired temperature above 950 °C (Costa, 2009). The FS slag used in this study was the predominately amorphous phase with high silica content. The presence of a considerable quantity of silica in RHA leads to an increase in strength during

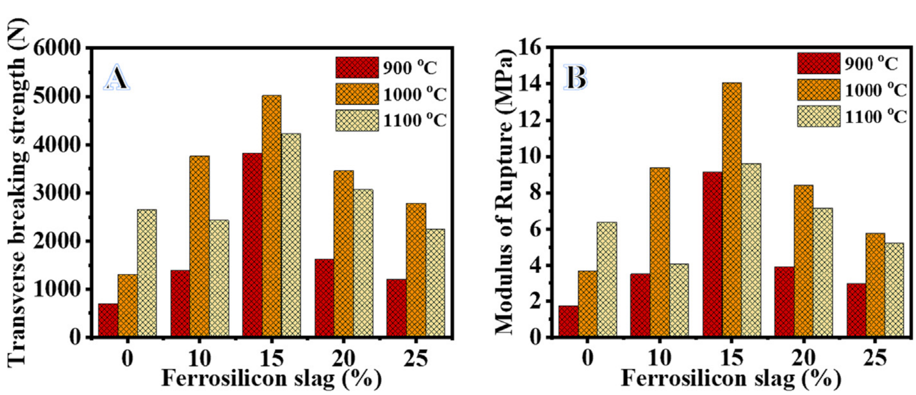

Fig. 6. The influence of FS slag addition and firing temperatures on TBS (A) and MOR (B).

the geopolymerization process. However, a significant amount of silica mixing with clay is undesirable since, as De Silva and Crenstil (Ahmed, 2021) found, raising the SiO_2/Al_2O_3 ratio up to 3.4–3.8 is predominantly responsible for the considerable strength gain at later stages of the geopolymerization process. Increased silica concentration was associated with higher SiO_2/Al_2O_3 ratios, indicating a reduced degree of geopolymerization (Ahmed, 2021). Because of the greater amount of unreacted components, higher percentages of clay replacement with RHA are associated with a looser brick structure. As a result, the resultant microstructure is weak and porous [35]. Increasing the proportion of FS slag causes a decline in compressive strength over 25%. However, with up to 25% RHA in the tiles, the TBS is greater than 890 N for type III roof tile. Strength becomes optimum when the ratio of $SiO_2/Al_2O_3 = 1.9$ with (5016.05 N) is much higher than that obtained by replacing clay with rice husk (1136 N) which was obtained at a ratio of $SiO_2/Al_2O_3 = 3.7$ (De Silva and Mallwattha, 2018) and also higher than (1427 N) obtained by replacing clay by ceramic sludge (Souza, 2011). MOR is calculated and plotted against the FS slag percentage at different firing temperatures (Fig. 6**B**). The MOR was increased with an increase in FS slag percentage up to 15% then decreased. The maximum MOR (14 MPa) is obtained at 15% FS slag and 1000 °C. No standard requirements for MOR are available. TBS and MOR show the same trend. De Silva et al. (Souza, 2011) replaced clay with different percentages of ceramic sludge (0%, 10%, 15%, 20%, and 25%). The maximum TBS (1427 N) and MOR (4.274 MPa) are obtained at 25% ceramic sludge.

3.3.3. Efflorescence

No efflorescence was observed on the sample composed of 15% FS slag and fired at 1000 °C. Calcium oxide (CaO) is considered the main component that causes efflorescence. However, it cannot be showing any efflorescence effect below 2% [39]. Interestingly, the mineralogical composition of the used raw materials demonstrates low CaO content (2.47%).

3.3.4. Thermal shock resistance

15% FS slag percentage and 1000 °C firing temperature samples were chosen for themal shock resistance test. Thermal shock resistance was evaluated using the water quenching technique. Thermal shock experiments were carried out by quenching brick samples in a water bath from 600 °C to 25 °C. The three-point flexural strength after thermal shock was tested. The appearance of tiny fractures in the tile surface is an indication that its flexural strength has decreased after being subjected to a temperature shock of 600 °C. Good resistance against thermal shock is shown by a reduction in flexural strength from 5016.05 N to 2372.28 N. In the end, it was determined that up to 15% of the FS in the initial mixture could be substituted with clay and yet provide the same final result after being fired at 1000 °C. Roofing tiles burned at this temperature met the requirements of ASTM C 1167 for grade 2. Table 1 shows a comparison of the attributes of tile samples containing 15% FS fired at 1000 °C and the standards of ASTM C 1167.

4. Conclusion

The study concluded that FS slag could potentially be used as a substitute for clay. The findings are summarized as follows:

- o The produced roof tiles utilize varying percentages of FS slag (0%, 10%, 15%, 20%, and 25%) at varying burned temperatures (900, 1000, 1100 °C).
- o Fabricated clay roof tiles perform optimally when subjected to a firing temperature of 1000 °C and a sample composed of 15% FS slag.
- o It was also shown that the cristobalite phase developed at a substantially smaller percentage (5.6%) in the 15% FS and 1000 °C than it did in the 1100 °C (17.8%). As compared to the sample fired at 1100 °C, the amount of corundum phase obtained (19.1%) is much larger. The mechanical characteristics are improved by an increase in corundum phase development during sintering. This may be because corundum assists in the closing of pores, resulting in a reduction in porosity.
- o The optimal density (1.812 g/cm^3) was achieved at 15% FS slag and 1000 °C. This is lighter than the lightweight roof tiles made by substituting rice husk ash for clay (density = 1.903 g/cm^3). In this setting, all parameters pertaining to strength and durability characteristics are satisfied.
- o Using 15% FS slag at 1000 °C reduces the water absorption of a conventional roof tile (0% FS slag) from 16.8% to 9.8%, meeting the standards set by ASTM C-1167.
- o It is also noted that the apparent porosity decreases with increasing sintering temperature, which may be a result of the sample being denser. At 15% FS slag and 1000 °C, the apparent porosity is at its lowest (17.45%).
- o The TBS (5.016 KN) of the sample made with 15% FS slag and fired at 1000 °C is much greater than that of standard roof tiles (2.2 KN). Additionally, the 15% FS slag and 1000 °C firing temperature increase durability and strength in comparison to the conventional tiles and are in line with the requirements of ASTM C-1167.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgments

The authors extend their sincere appreciation to the Researchers Supporting Project number (RSP2023R55), King Saud University, Riyadh, Saudi Arabia for the financial support.

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