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Recycling of Firewood Ash Waste in Ceramic Floor Tiles with Low Water Absorption

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This work aims to evaluate the recycling of firewood ash waste as a procedure to provide sustainable raw material for ceramic floor tiles with low water absorption. For this purpose, firewood ash waste coming from a red ceramic company in Campos dos Goytacazes-RJ (Brazil) was selected as an alternative raw material into a ceramic floor tile body, replacing natural quartz material by up to 10 wt.%. Floor tile pieces containing up to 10 wt.% of firewood ash waste were prepared by the dry process, pressed, and fired between 1190 ºC and 1250 ºC using a fast-firing cycle. The floor tile pieces were tested to determine their properties (linear shrinkage, water absorption, apparent porosity, apparent density, and flexural strength). The results showed that the partial replacement of quartz with firewood ash waste, in the range up to 10 wt.%, allows the production of ceramic floor tiles with low water absorption (WA) (BIa group - WA < 0.5% and BIb group - 0.5 < WA ≤ 3%; ABNT NBR ISO 13006:2020 Standard) in different amounts of firewood ash waste at lower firing temperatures.

Keywords: *Firewood ash, Solid waste, Valorization, Floor tiles, Properties.*

1. Introduction

Brazil has an expressive red ceramic industry responsible for the manufacture of materials for civil construction such as bricks, ceramic blocks, and roofing tiles, among others. The red ceramic companies are spread across all geographic regions of the national territory, which contribute to great job creation and social development. For example, the municipality of Campos dos Goytacazes-RJ (southeast region of Brazil) is home to an important red ceramic industrial hub that brings together more than 100 companies with great economic impact. In this red ceramic hub, the fuel most used in the kilns during the firing process is eucalyptus firewood[1] due to its calorific value, availability, and cost. Despite its economic repercussions, the consequence of using firewood as fuel is the large-scale generation of a solid waste, henceforth referred to as firewood ash waste. Considering that, Brazil has severe environmental legislation[2], thus the firewood ash waste produced in the red ceramic industry cannot simply be discarded into the environment without any treatment. Up to now, the Brazilian red ceramic industry does not have a definitive technological solution for the friendly recycling of this abundant polluting solid waste. In this context, it is of paramount importance to develop new researches that can contribute to friendly recycling, but at the same time, also add value to the firewood ash waste.

The firewood ash waste is the product resulting from the direct combustion of firewood, which is characterized as a fine particulate material. It exhibits wide chemical and mineral variability, depending on many factors such as the origin of the firewood (i.e., type of tree from which the firewood was produced), combustion temperature, and type of kiln[3,4]. Composition of firewood ash wastes contains variable amounts of silica (SiO_2 = 1.86 – 68.18%), alkaline earth oxides (CaO = 5.30 – 72.39% and MgO = 1.10 – 13.11%), and alkaline oxide (K_2O = 4.51 – 24.00%), as well as several others oxides in smaller amounts[4-7]. So, to a certain extent, there are compositional similarities between firewood ash wastes and the natural raw materials used in the manufacture of ceramic products. Based on the literature[5], it is estimated that approximately 476 million tonnes per year of biomass ash wastes are produced worldwide, including firewood ash wastes. Because of these circumstances, several researches have been directed towards the recycling of firewood ash wastes as a cheap alternative raw material in the production of ceramic materials. Indeed, the firewood ash wastes have been applied to clay bricks, ceramic blocks, soil-cement bricks, porcelain, mortars, concretes, etc[1,3,6,8-14].

In 2020, world production of ceramic tiles was 16,093 million square meters[15]. In this global scenario, Brazil is one of the main players in the world market for ceramic tiles, currently occupying the third position in production in the world. In 2020, Brazil produced 840 million square meters[15]. The Brazilian ceramic tile industry manufactures several types of ceramic floor and wall tiles, including low absorption ceramic tiles. According to the ABNT NBR ISO 13006:2020 standard[16], dry-pressed ceramic floor tiles with low water absorption are classified as BIa and BIb groups. Such low water absorption ceramic floor tiles are increasingly used in civil construction due to their superior quality and aesthetic beauty. They are manufactured using typically triaxial formulations composed of kaolin, clays, feldspars,

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and quartz[17]. In addition, after firing, these ceramic floor tile classes are characterized due to their highly vitrified nature, dense microstructure, and superior properties (mechanical strength, water absorption, and durability). It is well known that there have been several research efforts on the use of solid wastes in triaxial ceramic formulations for low water absorption ceramic floor tiles[18]. Despite this, it appears that there is little information about the recycling of firewood ash wastes generated in the red ceramic industry in the production of high quality ceramic floor tiles. In fact, Olokode et al.[19] showed that it is feasible to partially replace clay with firewood ash waste in a triaxial formulation for BIa floor tile, while Santos[20] showed the possibility of partially replacing feldspar with mesquite firewood ash waste for BIb floor tile. In this context, new researches aimed at the effects of firewood ash wastes on the characteristics of the ceramic formulations and technical properties of low absorption ceramic floor tiles are relevant and desirable.

The aim of this work was to investigate the recycling of firewood ash waste generated in the red ceramic industry as partial substitute of quartz in low water absorption ceramic floor tile formulations.

2. Experimental Procedure

Four triaxial floor tile formulations composed of mixtures of kaolin, albite, and quartz + firewood ash waste were prepared, as shown in Table 1. To carry out this work the following starting raw materials were used: i) commercial kaolin, albite, and quartz; and ii) firewood ash waste generated during the firing process collected from a red ceramic company (Campos dos Goytacazes-RJ, Brazil). For the purpose of comparison, the reference floor tile formulation (M1 formulation) used is free of firewood ash waste[21]. In the other formulations, however, quartz was partially replaced by up to 10 wt.% of firewood ash waste.

All raw materials were separately beneficiated in terms of drying at 110°C, dry milled, and then sieved using an ASTM no. 325 sieve, with aperture of 45 µm. Then, the floor tile formulations described in Table 1 were mixed, homogenized, and granulated by the dry process[17].

The chemical analyzes of the raw materials were determined by using an energy-dispersive X-ray spectrometer (Shimadzu, EDX 700). The X-ray diffraction experiments were performed in a conventional powder diffractometer (Shimadzu, XRD 7000) by using Cu-Kα radiation at a scanning speed of 1.5° (2θ)/min. The plasticity index (PI) of the tile formulations was determined by the Atterberg method according to PI = LL - PL, where LL is the liquid limit and PL is the plasticity limit. The liquid limit and plasticity limit were determined following the Brazilian standards NBR 6459[22] and NBR 7180[23], respectively. The bulk density was determined according to the NBR 6508[24] standard. The Hausner ratio of the granulated tile powders was determined as the ratio of the tap density to apparent density. The screen residue corresponding to the fraction > 63 µm has been also obtained.

The floor tile formulations (Table 1) were moistened with 7 wt.% water, pressed into rectangular bars of approximate dimensions of 115.0 x 25.4 x 7.0 mm^3 at 50 MPa, and dried at 110 ºC for 24 h. Finally, the green floor tile specimens were fast-fired in air at 1190 ºC, 1210 ºC, 1230 ºC, and 1250 ºC with a soaking time of 6 min to simulate fast firing cycle. The fast-firing cycle used in this research was less than 60 min, including cooling.

The following physical and mechanical properties of the fired floor tile specimens (five test specimens for each formulation and temperature) were determined: linear shrinkage, apparent density, apparent porosity, water absorption, and flexural strength. Linear shrinkages upon drying and firing were measured from the variation in the length of rectangular tile specimens according to ASTM C 326-09[25] and calculated using equation (A) given by:

$$LS = Ld - Lf / Ld \times 100 \quad (A)$$

where LS is the linear shrinkage after firing (%), Ld is the dried length of test specimen (mm), and Lf is the fired length of test specimen (mm).

Apparent density, water absorption, and apparent porosity were measured based on the Archimedes method described in ABNT NBR ISO 10545-3[26] and calculated using the equations (B), (C), and (D) given by:

$$AD = Md / Msa - Mi \quad (B)$$

$$WA = Msa - Md / Md \times 100 \quad (C)$$

$$AP = Msa - Md / Msa - Mi \times 100 \quad (D)$$

where AD is the apparent density (g/cm^3), WA is the water absorption (%), AP is the apparent porosity (%), Md is the mass of test specimen dried at 110 ºC (g), Msa is the mass of test specimen saturated with water (g), and Mi is the mass of test specimen immersed in water (g).

The flexural strength was determined by a three-point bending test using a universal mechanical testing machine (Instron, model 5582) according to ABNT NBR ISO 10545-4[27] and calculated using the equation (E) described as follows:

$$FS = 3PL / 2bh^2 \quad (E)$$

where FS is the flexural strength after firing (MPa), P is the breaking load (N), L is the distance between supports

Table 1. Compositions of the floor tile formulations (wt.%).

Formulation	Kaolin	Albite	Quartz	Firewood ash
M1	40.0	47.5	12.5	0.0
M2	40.0	47.5	10.0	2.5
M3	40.0	47.5	7.5	5.0
M4	40.0	47.5	2.5	10.0

(mm), b is the test specimen width (mm), and h is the test specimen thickness (mm).

3. Results and Discussion

The crystalline mineral phases identified by XRD analysis of the starting raw materials used in the floor tile formulations are provided in Table 2. As expected, commercial kaolin, albite, and quartz exhibited typical mineral phase compositions. The firewood ash waste sample from eucalyptus firewood showed a typical mineral composition, which is characterized by the mixture of several minerals such as quartz, calcite, potassium carbonate, Hydrate magnesium sulfate, hematite, portlandite, and gypsum. Calcite is the main mineral phase. This result is in agreement with literature data[5,6].

The chemical analysis and loss on ignition of the raw materials are given in Table 3. The obtained results are consistent with the mineral phases described in Table 2. Kaolin is essentially composed of SiO_2 and Al_2O_3. Albite contains mainly SiO_2, Al_2O_3, and Na_2O. The used quartz is essentially composed of SiO_2. The firewood ash waste is mainly composed of SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, and K_2O, with a predominance of CaO. Thus, the obtained results in this work are in line with those reported in the literature[5,6,10,28,29]. In terms of chemical composition, quartz and firewood ash waste are quite different. Note that, in addition to quartz (SiO_2), firewood ash waste contains appreciable amounts of fluxing oxides (Fe_2O_3, CaO, MgO, and K_2O). This means that the partial replacement of quartz with firewood ash waste tends to enrich ceramic floor tile formulations with a higher amount of fluxing components. Therefore, important repercussions on the densification behavior and technical properties of ceramic floor tiles are expected.

Table 4 displays important physical properties of the granulated floor tile powders prepared by the dry process. As can be seen, the partial replacement of quartz with firewood ash waste only caused small differences in the plasticity index (PI = 12.1 to 13.1%) of the floor tile formulations. Such results indicate that all tile formulations have suitable plasticity characteristics for the manufacture of ceramic floor tiles with low water absorption. The bulk density (BD) of the tested tile formulations decreased with the increase in the incorporation of the firewood ash waste. This effect occurred because the quartz used in this work has a bulk density of 2.61 g/cm^3, while that of the firewood ash waste is 2.49 g/cm^3. It was also observed that all floor tile powders exhibited low value of screening residue (SR), indicating good level of comminution of the starting raw materials. The floor tile powders presented Hausner ratio (Hr) values between 1.05 and 1.26. It is noted that the incorporation of firewood ash waste decreased the Hausner ratio, indicating better flowability characteristics of the floor tile powders[30,31].

The drying properties of the floor tile specimens are given in Table 5. It was found that the effect of incorporating the firewood ash waste was to decrease the drying shrinkage of the studied formulations. However, all tile specimens showed low linear shrinkage values (0.21 - 0.42%), indicating good dimensional control in the dry state. It was also observed

Table 2. Crystalline phases identified in the raw materials used.

Raw material	Crystalline mineral phases
Kaolin	Kaolinite ($Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$) and quartz ($SiO_2$)
Albite	Albite ($NaAlSi_3O_8$) and quartz (SiO_2)
Quartz	Quartz (SiO_2)
Firewood ash	Calcite ($CaCO_3$), quartz (SiO_2), potassium carbonate (K_2CO_3), magnesium sulfate heptahydrate ($MgSO_4 \cdot 7H_2O$), hematite (Fe_2O_3), portlandite ($Ca(OH)_2$), and gypsum ($CaSO_4.2H_2O$)

Table 3. Chemical compositions of the raw materials used (wt.%).

Sample	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	K_2O	Na_2O	SO_3	MnO	$^+$LoI
Kaolin	49.07	33.74	0.22	0.01	0.30	0.06	1.97	0.52	-	-	14.01
Albite	69.55	18.82	0.14	0.02	0.17	0.09	1.47	9.63	-	-	0.32
Quartz	98.97	0.41	0.01	0.02	0.01	0.01	0.18	0.13	-	-	0.26
Ash	9.55	6.63	4.38	0.83	40.77	10.79	7.35	-	1.68	0.72	17.30

$^+$LoI – loss on ignition

Table 4. Physical properties of the floor tile formulations.

Formulations	PI (%)	BD (g/cm^3)	SR (%)	Hr
M1	12.1	2.55	4.0	1.26
M2	13.0	2.47	4.2	1.05
M3	12.9	2.46	1.7	1.09
M4	13.1	2.45	0.7	1.17

PI = plasticity index; BD = bulk density; SR = screening residue; Hr = Hausner ratio

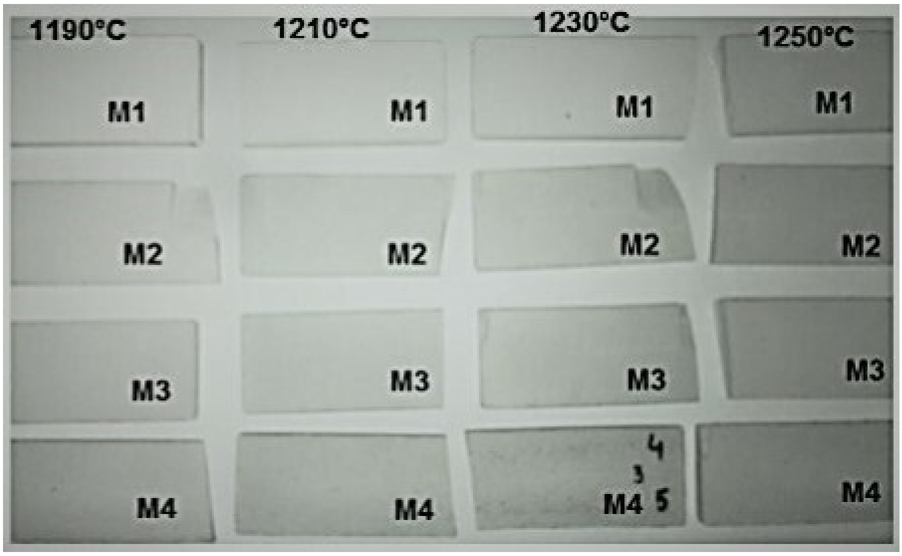

Figure 1. Typical appearance of the produced ceramic floor tiles.

Table 5. Drying properties at 110 ºC of the floor tile specimens.

Formulation	Drying Shrinkage (%)	Drying Density (g/cm^3)
M1	0.42	1.80
M2	0.36	1.78
M3	0.32	1.77
M4	0.21	1.72

that the drying density of the floor tile specimens decreased with increasing amount of firewood ash waste added. This effect can be explained due to the lower bulk density value of the firewood ash waste.

Figure 1 shows the visual appearance of the fired floor tiles. It can be observed that all specimens of floor tiles fired between 1190 ºC and 1250 ºC showed a light firing color, regardless of the amount of firewood ash waste added. However, a tendency towards darker hue at higher firing temperatures was observed[17,32].

The effects of firewood ash waste as a partial replacement for quartz in low water absorption floor tile formulations were evaluated in terms of physical and mechanical properties of technological interest (Figures 2-6).

Figure 2 shows the linear shrinkage of the fired floor tile specimens. The linear shrinkage values obtained within the 4.71 - 9.66% range are favorable to produce floor tiles with low water absorption[33]. However, the linear shrinkage values were influenced by both firing temperature and added firewood ash waste amount. In general, increasing the firing temperature tends to increase the linear shrinkage. This finding is due to the higher degree of vitrification of the floor tile specimens at higher temperatures. It should also be noted that, at any firing temperature, there was a gradual increase in linear shrinkage with an increase of up to 5 wt.% of firewood ash waste. This effect was mainly due to the presence of fluxing oxides in the firewood ash waste (Table 3), which tend to reduce the viscosity of the liquid

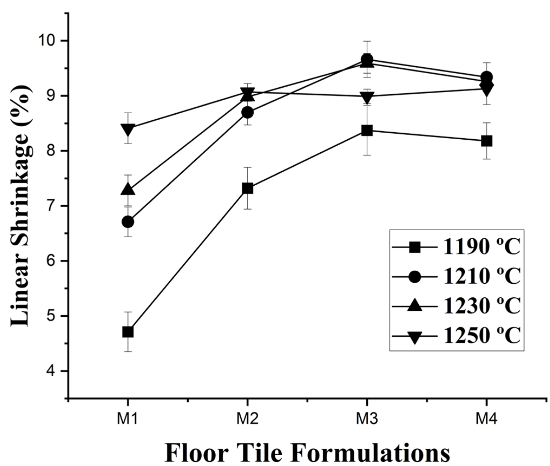

Figure 2. Linear shrinkage of the fired floor tile specimens.

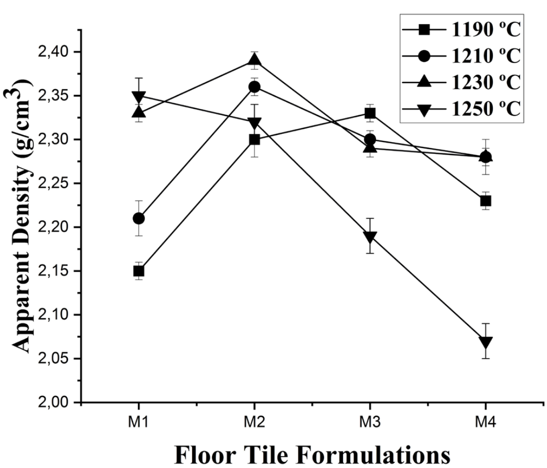

Figure 3. Apparent density of the fired floor tile specimens.

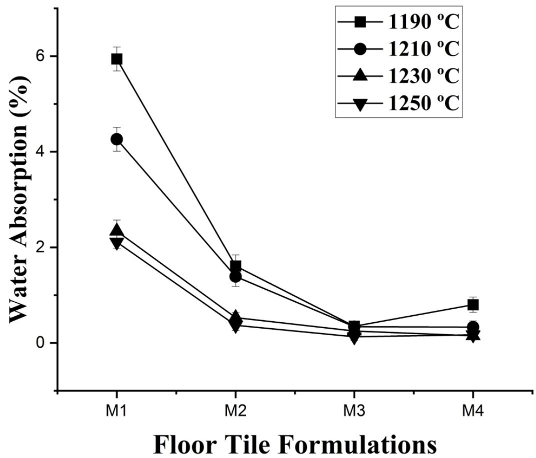

Figure 4. Water absorption of the fired floor tile specimens.

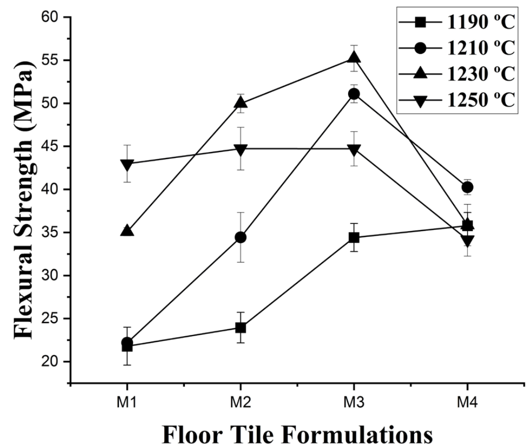

Figure 6. Flexural strength of the fired floor tile specimens.

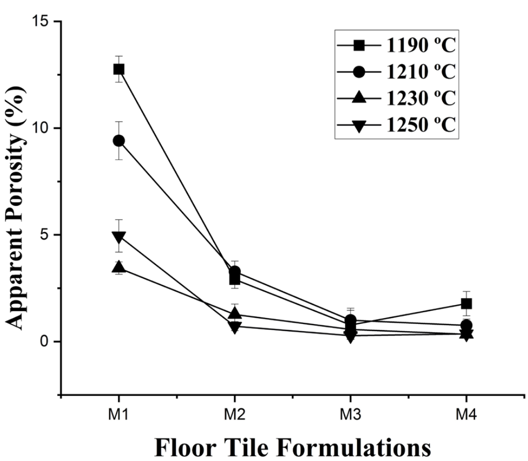

Figure 5. Apparent porosity of the fired floor tile specimens.

phase and accelerate the vitrification process. A further increase of firewood ash waste (M4 Formulation), however, a tendency towards decreasing linear shrinkage can be seen. The reason for this may be related to the expansion effect of the tile specimens with the formation of gas bubbles during firing step[34,35].

The apparent density of the floor tile specimens is shown in Figure 3. The floor tile specimens produced had a wide variation in apparent density (2.07 – 2.39 g/cm^3). In general, the floor tile specimens had a trend towards lower apparent density at higher firing temperature and higher amount of firewood ash waste added. This result is important as it indicates the creation of closed porosity due to the formation of gas bubbles[1,9,36]. In addition, it also indicates that the partial replacement of quartz with high amounts of firewood ash waste in floor tile formulations should be avoided.

The water absorption that determines the open porosity level of the floor tile specimens is shown in Figure 4. The water absorption values found range from 0.13 to 5.94%. As can be seen, all formulations showed a similar trend of decreasing in water absorption with increasing firing temperature. Such a trend is associated with the formation of higher amount of liquid phase that infiltrates the open pores of the structure, resulting in floor tile specimens with low water absorption. It is worth mentioning that the firewood ash waste also contributes to the reduction of water absorption, since it tends to reduce the viscosity of the formed liquid phase. In fact, when compared to the water absorption evolution of the reference formulation (5.94% to 2.11%), the waste-containing formulations (M2, M3, and M4 formulations) showed lower water absorption (1.61% to 0.13%). A similar behavior was also observed in the apparent porosity values[14], as shown in Figure 5.

The flexural strength of the floor tile specimens is shown in Figure 6. The floor tile specimens produced showed a wide range of flexural strength between 21.79 MPa and 55.21 MPa. As expected, both the firing temperature and the amount of firewood ash waste influenced the flexural strength behavior. The flexural strength of the tile specimens prepared with the M1 formulation (waste-free reference formulation) increased as the firing temperature was increased. This behavior reflects the higher degree of sintering with concomitant reduction of open porosity (lower water absorption) in the structure of the tile specimens. The flexural strength of the M2 and M3 formulations (containing 2.5 and 5% firewood ash waste, respectively) tends to increase up to 1230 °C, and then decreases when the temperature is increased to 1250 °C[20]. Now, the flexural strength of the M4 formulation (containing 10% firewood ash waste) decreased for temperatures above 1210 °C[20]. Thus, in general, there was a tendency towards a decrease in mechanical strength with the combined use of a higher amount of firewood ash waste and a higher firing temperature. These results were well correlated with the physical properties (apparent density (Figure 3), water absorption (Figure 4), and apparent porosity (Figure 5)).

In this work, the feasibility of the recycling potential of the firewood ash waste in the production of ceramic floor tiles was determined in terms of water absorption (WA) and flexural strength (FS). The dry-pressed ceramic floor tiles are classified into the following groups according to ABNT NBR ISO 13006[16]: BIa (WA < 0.5% and FS > 35 MPa), BIb (0.5% < WA ≤ 3.0% and FS ≥ 30 MPa), BIIa (3.0% < WA

Table 6. Classification of the floor tiles incorporated with firewood ash waste.

Formulation	1190 ºC	1210 ºC	1230 ºC	1250 ºC
M1	BIIa	BIIa	BIb	BIb
M2	BIIa	BIb	BIa	BIa
M3	BIa	BIa	BIa	BIa
M4	BIb	BIa	BIa	BIa

≤ 6.0% and FS ≥ 22 MPa), and BIIb (6.0% < WA ≤ 10.0% and FS ≥ 18 MPa). However, only BIa and Bib groups are considered ceramic floor tiles with low water absorption. Table 6 summarizes the different types of ceramic floor tiles produced. Relevant changes were found in the quality of the floor tiles due to the incorporation of firewood ash waste. The M1 formulation (waste-free) made it possible to obtain a BIb-type ceramic floor tile only at 1230 ºC and 1250 ºC. The M2 formulation allows to obtain BIb at 1210 ºC and BIa at 1230 ºC and 1250 ºC. The M3 formulation allowed to obtain BIa at all firing temperatures. The M4 formulation allowed obtaining BIb at 1190 ºC and BIa between 1210 ºC and 1250 ºC. Based on these results, it is evident that the proposed solution for the recycling of firewood ash waste from the red ceramic industry can be highly attractive, as it allows obtaining dry-pressed ceramic floor tiles with low water absorption (i.e., superior quality ceramic floor tiles) at lower firing temperatures[37].

4. Conclusions

In this work, the firewood ash waste produced in a red ceramic company was successfully recycled as partial replacement for quartz in a ceramic floor tile formulation. The firewood ash waste is rich in CaO, but also contains appreciable amounts of SiO_2, Al_2O_3, Fe_2O_3, MgO, and K_2O. Thus, its incorporation tends to enrich the ceramic floor tile formulations with a higher amount of fluxing oxides. Due to this, the partial replacement of quartz by firewood ash waste positively influenced the technical properties and final quality of the ceramic floor tiles. It was found that the partial replacement of quartz by firewood ash waste, in the range up to 10 wt.%, allows the production of ceramic floor tiles with low water absorption (BIa group and BIb group; ABNT NBR ISO 13006 Standard) at lower firing temperatures. Therefore, the recycling of firewood ash waste in ceramic floor tiles with low absorption could be highly attractive in environmental and economic terms.

5. Acknowledgements

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