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Coffee Husk as Feldspar Substitute in the Manufacture of Enameled Ceramic Tile*

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Abstract: This study researches the effect of adding coffee husk (CH) and coffee husk ash (CHA) as partial feldspar substitutes to the manufacture of enameled ceramic tiles. Clays and other paste additives were characterized using XRD and XRF. The designed prototypes were pressed, dried, and fired at 1,120 °C. The physical (drying and firing shrinkage, apparent density, water absorption) and mechanical (flexion resistance) properties were evaluated and compared with standard fired specimens. Although the physical and mechanical properties of the specimens with 5 and 10 % CH and CHA make them suitable for the manufacture of BIIb ceramic tiles, it was concluded that CH and CHA under the working conditions do not replace feldspar in the preparation of enameled ceramic tiles due to surface quality defects.

Keywords: Coffee husk; feldspar; ceramic tiles; red clay; sustainable construction

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Cisco de café como sustituto del feldespato en la fabricación de baldosas cerámicas esmaltadas

Resumen: en este estudio, se investigó el efecto de la adición de cisco de café (cc) y de la ceniza de cisco de café (ccc) como sustitutos parciales del feldespato en la fabricación de baldosas cerámicas esmaltadas. Las materias primas utilizadas en la pasta se caracterizaron mediante DRX y FRX. Los prototipos moldeados por prensado se cocieron a 1 120 °C para evaluar el comportamiento de las propiedades físico-cerámicas (contracción en seco y cocido, densidad aparente, absorción de agua) y mecánicas (resistencia a la flexión) con el aumento de adiciones de cc y ccc. A pesar de que las propiedades físicas y mecánicas de los especímenes con 5 % y 10 % de cc y ccc los hacen aptos para la fabricación de baldosas cerámicas BIIb, se concluyó que el cc y la ccc bajo las condiciones de trabajo no reemplazan al feldespato en la elaboración de baldosas cerámicas esmaltadas debido a los defectos en la calidad de la superficie.

Palabras clave: cisco de café; feldespato; baldosas cerámicas; arcilla roja; construcción sostenible

Introduction

Coffee is one of the leading products sold worldwide; approximately 10.25 million tons were produced in 2018 [1]. This production sector generates large amounts of waste after processing, with values that can range from 30 to 50 % of the total weight of the product; the husk, also known as coffee husk (CH), constitutes about 12 % of the weight of the grain on a dry base [2]. Traditionally, this waste has been used as fuel in brick kilns, stoves, peasant homes, and silos for coffee drying [3]. It has also been used in the production of biopesticides for the control of mosquitoes [4], composting [5], bio-absorbents [6], among others [7], [8].

The chemical composition of CH depends on the process of harvesting and production of the coffee bean [9]; however, it is mainly composed of cellulose (35.98 %), hemicellulose (17.8 %), lignin (25.4 %), and ash (2.4 %). Approximately 82.7 % by weight of this waste is volatile and has a porosity close to 79 % [10]. These characteristics suggest the possible potential of the waste to produce lightweight or absorbent materials.

The use of CH and other waste products in the production of construction materials has become a new trend that, besides adding value to new products, is conceived as an environmental alternative for waste disposal [11]-[13]. In the field of ceramics, CH has been used to produce bricks, tiles, and other non-enameled ceramic products, obtaining lightened materials with thermal insulation properties [14]-[16].

Sodium-potassium feldspar is the flux material traditionally used in the manufacture of ceramic tiles [17]. This mineral contributes to decreasing the plasticity, permeability, and solidification of the paste. Additionally, it favors the formation of high volumes of liquid phase during firing [18], intervening in the definition of the firing temperature and the pyro-plastic deformation [19] that the product may suffer.

The reason for selecting CH as a partial replacement for feldspar is supported by the fact that this material, within its chemical composition, contains alkaline potassium and alkaline earth (Ca and Mg), elements that could favor the liquid phase formation [20]. Based on this, this research proposes the manufacture of enameled ceramic tiles replacing different proportions of feldspar with CH and coffee husk ashes (CHA) in the ceramic mixture (red clay). Some of their main physical and mechanical properties (density, shrinkage, water absorption, and flexural strength) are analyzed to establish the feasibility of their use.

Materials and Methods

Materials

The CH was provided by a local company (Cúcuta, Colombia) belonging to the National Federation of Colombian Coffee Growers. The other raw materials used to make the ceramic paste were four clay types (CL01, CL02, CL03, CL04), feldspar (Feld), limestone, raw, and fired break. A local ceramic company (Cúcuta, Colombia) provided these raw materials to conduct the study with the standard mixture formula, which has been designated as "P." The feldspar was replaced with increasing amounts of CH and CHA.

Obtaining CHA

CH underwent two different treatments before being used in pulp. First, to obtain a uniform particle size, CH was milled in a rotary hammer mill and passed through a 74 µm mesh. Second, uncut kerosene was burned by imperfect combustion in a temperature-controlled oven (650 °C) for two and a half hours to remove the highest content of organic matter present in the material. The carbonized husks were brought back to 700 °C for two and a half hours to guarantee the total decomposition of organic matter [21]. After combustion, the CHA was crushed and passed through a 74 µm sieve.

The chemical composition of raw materials was determined using the X-Ray Fluorescence (XRF) technique and a sequential X-ray fluorescence spectrometer of 4 KW dispersive wavelength (Bruker S8 Tiger model). The main crystalline phases of the feldspar and CH were defined by X-ray diffraction (XRD) using a Bruker D8 Advance powder diffractometer with DaVinci geometry. Phase quantification was performed by Rietveld refinement using corundum as the reference phase for the calculation of the amorphous phase [22].

Evaluation of CH as a Feldspar Substitute in the Manufacture of Ceramic Tiles

Blends were prepared with 78.5 % clays (CL01, CL02, CL03, CL04), 16.5 % feldspar, 2 % limestone, 1 % raw break, and 2 % fired break (P0). Six more mixtures were prepared, in which the proportion of feldspar was replaced by 5, 10, and 100 % CH (CH5, CH10, CH100) and CHA (CHA5, CHA10, CHA100); the percentages for the other components remained constant. Each 400 g mixture was prepared by wet milling (36 % by weight of water), using a Gabrielli fastball mill, with 400 g of alumina grinding bodies for 2 minutes and 45 seconds.

After grinding, the mixtures were dried at 110 °C for 24 hours and subsequently ground to diameters less than 1 mm in a Servitech rotary hammer mill. Ten specimens of each mixture were prepared using as a base 80 g of semi-dry powder (humidified at 5.6– 5.8 % by weight) for each specimen; uniaxial pressing at 26.7 bar was performed on a GABTEC press using a steel mold (110 mm x 55 mm x 8 mm). The specimens were dried to constant weight at 110 °C. Subsequently, the drying shrinkage (DS) percentage was determined for its newly formed lengths. The specimens were then enameled using a bell-type application system to be subsequently baked in an industrial roller kiln at a maximum temperature of 1,120 °C with a full cycle of 34 min. For the ten fired specimens, losses by calcination, firing shrinkage, water absorption, bulk density, and mechanical resistance to bending were determined.

Losses of ignition (LOI) (physically adsorbed water still present, dehydroxylation, decomposition of carbonates, and organic matter) were determined by evaluating the mass difference of dry and fired test pieces. The firing shrinkage (FS) of the obtained specimens was calculated by determining the linear shrinkage of the specimens after firing them compared to the dry dimension. The water absorption percentage (E), the boiling in water method) and bulk density q (ρ) were established as outlined in the NTC 4321-3 standard [23]. Afterward, all the specimens were dried at 110 °C at a constant weight to determine the mechanical flexural strength (S) and modulus of rupture (R) according to NTC 4321-4 [24].

Results and Discussion

Characterization of Raw Materials

Table 1 shows the chemical composition of the raw materials determined by XRF. The results of microstructure analysis by XRD are presented in Table 2. From the chemical point of view, the clay materials that make up the paste (CL01, CL02, CL03, and CL04) are characterized by being rich in silicon and aluminum. The mineralogical composition allows establishing quartz as the predominant crystalline phase, followed by the presence of phyllosilicate minerals such as kaolinite and muscovite. These two phases are responsible for explaining the higher content of Al and Si. The potassium content is associated with the presence of muscovite and the microcline-type feldspar that is a minority phase. The iron content is associated with the presence of hematite in clay materials and possibly in muscovite. The presence of titanium correlates with the existence of anatase in the mineralogical composition of Table 2.

Due to its contents of Fe_2O_3, Na_2O, K_2O, MgO, and CaO, clay materials can be considered suitable for the manufacture of stoneware red, with some variations in their tones by the content of titanium oxide; these colors are characteristic of the region, especially the metropolitan area of Cúcuta [25], [26]. High silica content in the form of quartz (SiO_2) is observed. It acts as a degreaser in the paste, favoring the degassing of impurities and the elimination of water during the drying process and reducing linear shrinkage and deformation during firing [27].

Table 1. Chemical composition of raw materials

Raw Materials	Weight Percentage (%)						
	CL01	CL02	CL03	CL04	Feld	CH	CHA
SiO_2	59.67	58.58	60.28	60.75	71.61	0.62	
Al_2O_3	21.72	21.81	21.55	20.68	15.2	0.17	
Fe_2O_3	5.29	6.22	5.26	6.09	1.02	0.05	
K_2O	1.98	1.71	2.07	1.84	5.58	0.24	
TiO_2	0.95	0.92	0.88	0.98	0.18	-	
MgO	0.67	0.86	0.67	0.77	0.26	0.24	
P_2O_5	0.57	0.6	0.62	0.61	0.54	0.07	
Na_2O	0.35	0.28	0.3	0.33	3.08	-	
CaO	0.38	0.27	0.47	0.32	0.45	0.24	
*LOI	8.12	8.48	7.59	7.4	1.78	98.24	20.04

Source: Own elaboration.

Table 2. Mineralogical composition of raw materials

Crystalline Phase	Weight Percentage (%)					
	Feld	CH	CL01	CL02	CL03	CL04
Quartz	44.50	0.40	34.80	34.00	39.50	40.60
Muscovite	5.10	-	11.70	10.50	13.20	13.30
Anatase	-	-	0.80	0.50	0.70	0.80
Kaolinite	-	-	23.20	16.90	18.60	16.60
Hematite	-	-	0.90	1.90	1.20	1.40
Microcline	19.60	-	1.80	0.40	1.30	1.80
Albite	25.30	-	1.50	0.40	-	1.10
Calcite	-	-	-	-	-	0.20
Total crystal	94.50	0.40	74.70	64.60	74.50	75.80
Amorphous and others	5.50	99.60	25.30	35.40	25.50	24.20

Source: Own elaboration.

The content of alumina of the analyzed raw materials is approximately 21 %, enough quantity to provide the clay with high melting temperature, low thermal conductivity, high resistance to corrosion, low expansion coefficient, and hardness [28]. The results obtained from the feldspar show that it is an alkaline potassium-sodium type, with a low sodium proportion and traces of barium compared to potassium.

For CH, small traces of quartz (SiO_2) are identified as crystalline phases with XRD (Table 2 and Fig. 1), revealing that CH is a practically amorphous material. The CHA shows higher contents of iron and potassium oxides, which will be responsible for the reddish color of the fired bodies and the sintering at low temperature, respectively. The high content of K_2O allows inferring that this material will have a strong flux effect compared to feldspar, whose K_2O content is 5.58 % by weight.

The presence of microcline and albite (sodium and potassium feldspar) in the feldspar used is compelling from the ceramic point of view since, if only potassium feldspar prevails, the vitreous mass will remain very viscous, even at high temperature. If sodium predominates, the decrease in viscosity with the increase in temperature will be faster.

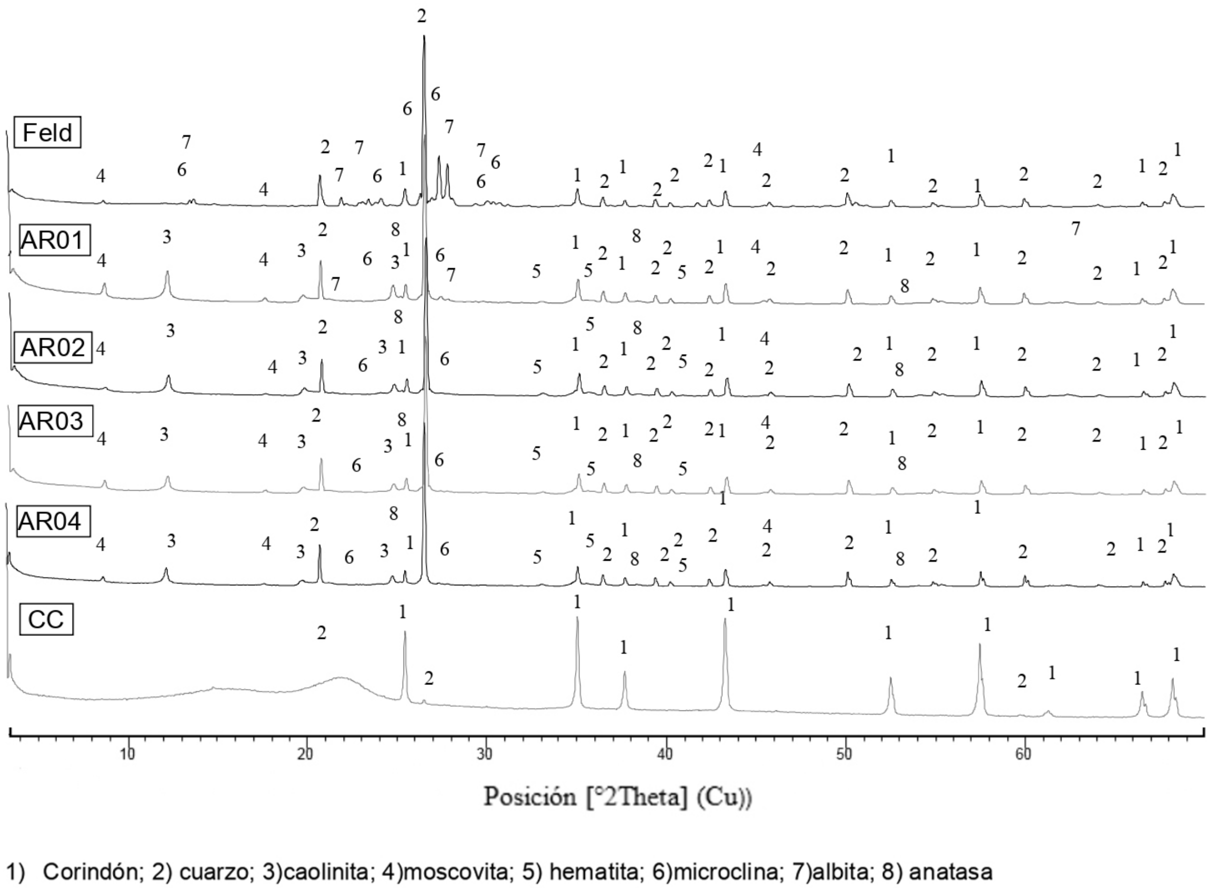

Fig. 1. XRD pattern of raw materials.

Source: Own elaboration.

Evaluation of the physical-mechanical behavior of mixtures

The use of CH and CHA in the manufacture of ceramic tiles affected the final properties of these products. Table 3 presents the results of the properties evaluated. The viscosity of the paste increased with the incorporation of larger quantities of CH. Therefore, the CH100 mixture, whose viscosity after wet milling was so high that it could not flow, was not tested further since it did not guarantee the adequate discharge of mills, the transport to storage tanks, and atomization at the industrial level [29].

Table 3. Physical-ceramic properties of the specimens tested

Mixture	DS (%)	FS (%)	LOI (%)	E (%)	ρ (gr/cm3)
P	0.23	3.42	5.50	9.87	2.06
CH5	0.21	2.53	7.11	8.21	1.86
CH10	0.23	1.26	7.77	8.05	1.63
CHA5	0.16	3.19	6.60	9.80	1.95
CHA10	0.15	3.40	7.14	9.76	1.94
CHA100	0.06	4.19	13.32	22.62	1.64

Source: Own elaboration.

Table 3 indicates that in the ceramics obtained at 1,120 °C, as CHA replaced feldspar, there was an increase in the percentage of firing linear shrinkage, being very similar to the P mixture with the 10 % CHA addition. On the contrary, for mixtures with CH, the linear firing shrinkage decreased at higher amounts of CH as feldspar replacement. The apparent density of the fired pieces decreased with the percentage of the addition of CH and CHA in replacement of feldspar, the CH10 and CHA100 mixtures having the lower apparent density, even inferior to the standard mixture (20.9 % and 20.4 % respectively).

For CH fired pieces, at higher additions of CH, the percentages of water absorption decreased significantly, even getting reductions of 16.8 % and 18.4 % for the CH5 and CH10 mixtures, respectively, in comparison to the P mixture. The mixtures with CHA presented a similar behavior with additions of up to 10 %, whose values are very similar to that of the standard mixture. On the chemical composition of CHA, the literature reveals the presence of potassium oxides (23–46 %), phosphorus (3–25 %), magnesium (4–22 %), calcium (10–18 %), sodium (0.1–8 %), and quartz (1–17 %) [30]–[32]. This composition explains why the more significant the proportion of CHA, the higher the content of fluxes (K_2O, CaO), which increased the amount of liquid phase during sintering, decreased porosity, and helped to increase the mechanical strength. However, for the mixture with 100 % feldspar replacement by CHA, water absorption increased up to 1.3 times compared to P.

Based on the results of e and the criteria in [33], the fired specimens of mixtures CH5, CH10, CHA5, and CHA10 are classified within group BIIb (6 % < E < 10 %, S > 500 N), ceramic tiles of colored and porous support without application of glazes or decorations. Different surface defects in the enameled prototypes, probably due to the degassing process of the inorganic matter that mainly compose CH (99.6 % by weight), are the principal causes of porosities that contribute to decreasing bending resistance and increasing water absorption, as suggested [30]. Fig. 2 shows that mixes CH5, CHA10, and CHA5 presented the best bending resistance, exceeding the flexural strength of the pattern mix by 17.2 %, 7.5 %, 2.3 %, respectively.

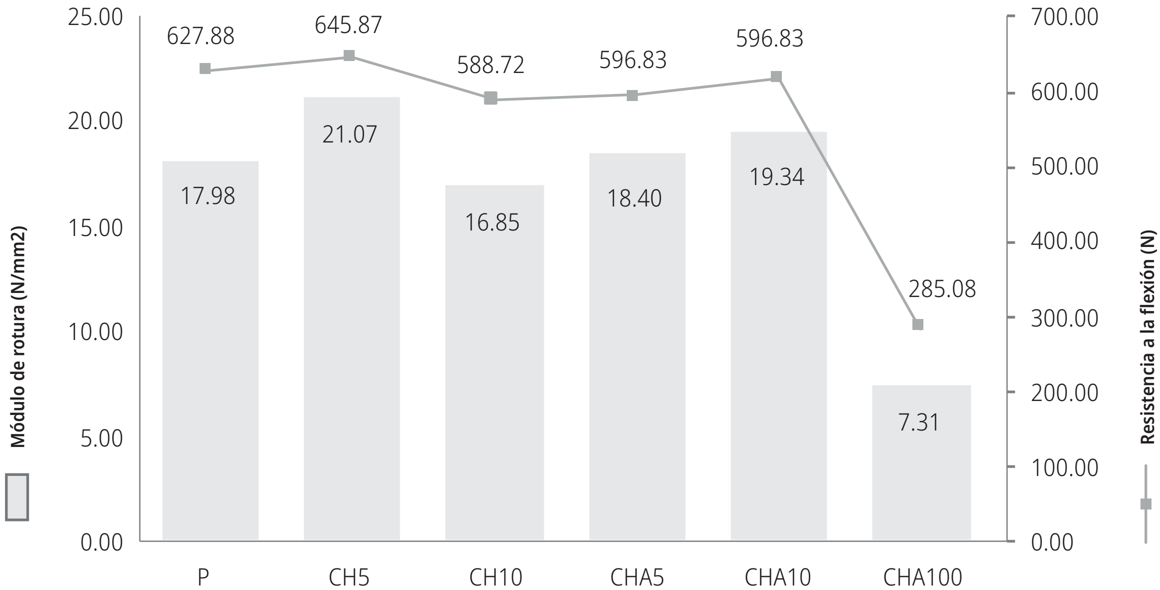

Fig. 2. Flexural strength and modulus of rupture of fired specimens.

Source: Own elaboration.

Also, blackheart gradually increased as the CH and CHA content increased; its presence additionally caused swelling of the prototypes, deterioration of the technical and aesthetic characteristics of the enamel, and change of tone in the support and enamel materials, especially CHA100 prototypes. This behavior can be attributed to the rapid firing cycle handled by the roller kiln and the impossibility of gases (CO_2 and CO), which have developed inside the ceramic piece, to go outside because of the surface waterproofing condition.

Conclusions

Based on the results obtained, it is concluded that CH and CHA under the working conditions are effective in reducing the density of ceramic products. The higher the addition, the lower the density, which may be due mainly to the massive amounts of inorganic matter present, which, after burning, causes higher losses, greater porosity, and lower weight. Lower densities are obtained with additions of CH in comparison with additions of CHA.

CH and CHA showed to have flux aptitude; therefore, they can replace the feldspar of a standard composition for ceramic tiles without requiring significant changes in the production process and obtaining products with similar physical-mechanical behavior to a standard formulation. However, the additions of these materials do not allow obtaining glazed tiles with high aesthetic value.

Although the surface quality of the specimens with 5 % addition of CH (CH5) in replacement of feldspar is not ideal, the results of drying shrinkage (0.21 %), firing shrinkage (2.53 %), water absorption (8.21 %), bulk density (1.86 g/cm^3), and flexural strength (645, 87 N) are better than those determined in the standard mixture. These characteristics classify them within group BIIB (S > 500 N, E 6–10 %), according to the requirements established in NTC 919.

The mixture with 5 % CHA allowed to obtain specimens with better physical and mechanical properties compared to the standard mixture, and with a lower number of surface defects compared to the CH5 mixture, being classified within the BIIB group as well.

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