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Use of the rice and corn husk ashes as an innovative pozzolanic material in ceramic tile adhesive production

Richard Ahumada[a], Holman Ospina-Mateus[b], Katherinne Salas-Navarro[c]*

[a]*Corporación Universitaria del Caribe (CECAR), Sincelejo, Colombia*
[b]*Department of Industrial Engineering, Universidad Tecnológica de Bolívar, Cartagena, Colombia*
[c]*Department of Productivity and Innovation, Universidad de la Costa, Barranquilla, Colombia*

Abstract

In this study, the use of agro-industrial waste from rice and corn husk ashes as a pozzolanic addition for cement substitution in the manufacture of ceramic tile adhesive is presented. The cement replacement, at 15%, 20%, 25%, and 30%, shows good results, especially for the case of rice husk ash with an index of pozzolanic activity equal to 0,94. The physical-chemical treatment of the ashes is essential. In this case, it was carried out through a calcination process at 800°C and a subsequent grinding process. Test of consistency, open time, and adhesive resistance to traction, were carried out to evaluate the quality of the ceramic mortar.

Keywords: Pozzolanic; index of pozzolanic activity; adhesive resistance to traction; ceramic tile adhesive.

1. Introduction

The contribution to the economic development of modern societies by Portland cement has been significant due to the remarkable population growth. Cement production involves high consumption of natural and energy resources and CO2 emissions, and other pollutants [1]. Investigations of materials that can partially meet the need to use cement are currently required. Pozzolans are an alternative, where promising studies have focused in recent years. New research is required to use new materials that promote innovation, technological and scientific development, and above all, sustainable development [2-4]. Pozzolans are siliceous or alumino-siliceous materials that, in the presence of water, chemically react with calcium hydroxide to form compounds with cementing properties [5]. The latest research has focused on pozzolans obtained by calcining Silico-aluminous clays and residues from the steel

* Corresponding author: Katherinne Salas-Navarro
E-mail address: ksalas2@cuc.edu.co

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industry. Research on pozzolans from agricultural and mining waste (non-metallic) is limited. Most studies are applied to the manufacture of structural concretes and conventional mortars [6, 7].

Pozzolans can be classified as natural (volcanic ash) and artificial (industrial processes). In the most common natural: blast furnace slag, fly ash fuel pulverized, calcined clays, among others [8]. The artificial pozzolans are divided into two groups, the natural silicates of clayey and schist condition, which acquire the pozzolanic character by thermal processes, and another result of agricultural and industrial operations [9, 10]. Pozzolans of artificial origin constitute one of the most encouraging experiences searching for more ecologically and economically sustainable substitutes for cement [11]. In recent years, agricultural residues (bagasse from sugar cane and rice husks) have been studied to obtain pozzolans, finding silica contents higher than 80% [12-14]. The test to evaluate these materials' pozzolanic activity is proposed by the ASTM [5, 15-18], and consists of manufacturing mortars under standardized conditions evaluating their tensile strength at ages 7, 28, and 56 days.

The pozzolanic material is evaluated by carrying out partial replacements in cement and analyzing the mortars' resistance. The analyzes are reported with the Strength Activity Index (SAI) ASTM C109/C109M – 12 [16-19]. When a material has values greater than 75%, the material exhibits pozzolanic properties. The products of the pozzolan/lime reaction are generally the same type as the hydration products of Portland cement: Hydrated Calcium Silicates (HCS), Hydrated Calcium Aluminates (HCA), and Hydrated Calcium Aluminates (HCA)[20]. Currently, studies aimed at the effects of the use of pozzolanic additions from agro-industrial waste (AW) to produce structural mortars and concrete have shown excellent results [21]. De Sensale [13] studied the effects of rice husk ash on durability in cementitious materials. Also, ash additions of various residues such as oil palm, sugarcane bagasse, rice husk, rice and wheat straw, and corn husk would have been studied to improve the physical and chemical characteristics in concrete, demonstrating once again that an ecological and sustainable concrete can be obtained. [12, 2-2-24]. Many studies highlight the importance of the novel use of materials such as pozzolans in recent years [25, 26]. The use of cement in construction is very diverse and is not limited solely to concrete and structural mortars. On the contrary, it is widely used as a plaster material, as a bonding material in masonry, and as an adhesive material for ceramics. In this research, the emphasis is placed on two types of common Agro-industrial Waste from the department of Sucre, Colombia, such as rice husks and corn husks. Its potential to be used as a pozzolanic addition of adhesive mortars for type C ceramics will be analyzed. Adhesive mortars are classified according to their functional characteristics, finding cementitious adhesives (C), dispersion (D), and reactive resins (R). Additionally, type C can also be subdivided into several classes, according to their characteristics following NTC 6050-2 [27] or its equivalent, ISO 13007-2 [28].

2. Method

The present study is experimental - descriptive. In the first stage of the study, the areas of Sucre (Colombia) with the highest availability of agro-industrial residues in rice husks and corn husks were identified. Subsequently, the collection of samples was carried out, and the methods of preparing the samples for use as pozzolanic additions were established. The agro-industrial waste was subjected to a calcination process under controlled conditions and then to comminution or reduction in size as recommended by previous studies. In the next stage, it was sought to characterize the ashes' physicochemical properties to evaluate their potential. The ash obtained was subjected to humidity tests, organic matter content, density, loss on ignition, and screening. In the next research stage, a mixture of ceramic adhesive mortar was made, making partial substitutions of cement for the ash obtained. In the experiment, the cement replacement percentages were varied to establish the recommended percentage to obtain the best performances. In the study, quality tests were performed on the adhesives according to the Colombian technical standard NTC 6050, thus identifying the type of waste with the highest pozzolanic potential.

3. Materials

- Cement: A general-purpose type I Portland cement was used. Within the considerations, it was evidenced that this material complied with the Standard Test Method for Density of Hydraulic Cement (ASTM C188-14)[17].
- Bermocoll ME 1000X: One of the fundamental additives for adhesive mortar development for ceramics is a cellulosic thickener. It allows in the mixture the viscosity and water retention necessary for the formulation of an

optimal paste. Thus, methyl ethyl hydroxyethyl cellulose was used. The technical information of the product is observed in table 1.

Table 1. Technical information of the Bermocoll ME 1000X

Characteristics	Data
Particle Size	98% < 450μm
Residual moisture	Max. 4%
Salt content	Max. 6%
pH (1% solution)	Neutral
Viscosity (1% solution)	11.0– 14.000 mPas

Table 2. Technical information of Elotex 60W

Characteristics	Data
Bulk density	500 – 650 g/l
Residual moisture	Max. 1%
pH (Dispersion 10% in water)	6.3 – 8.3

Elotex 60W: Another of the additives necessary for the mixture corresponds to a re-dispersible polymer. This additive improves the adhesion between the surface of the mixture and the ceramic material. A copolymer of vinyl acetate and ethylene was used. Table 2 lists the technical data of the product.

- Aggregates: Sand from the Sucre region (Colombia) was used, which meets the Colombian technical standards' specifications (NTC) to produce ceramic mortars. It required an additional sieving process using the # 100 sieve.
- Rice husk ash: The rice husk used in this research was obtained in San Marcos' municipality (Sucre). To activate the residue's silica, a controlled calcination process at 800 ° C was necessary. Subsequently, the calcined material was comminuted to fine granulometry (# 200 sieve). See table 3. Additionally, essential physicochemical characteristics of the ash were determined, see table 4.
- Corn husk ash: The corn husk used in the trials was obtained from the Department of Sucre's rural areas. A calcination process of the pozzolanic material was carried out at 800 ° C to reduce the particle size. The physicochemical data and the granulometric profile are presented in tables 4 and 5, respectively.

Table 3. Rice husk ash granulometry.

Sieve	Diameter	Retained			% Accumulated	% Filtered out
(Inches)	(Millimetres)	Weight (Grams)	Corrected weight (Grams)	%		
No 4	4.76	0.00	0.00	0.00	0.00	100.00
No 10	2.00	0.00	0.00	0.00	0.00	100.00
No 20	0.84	1.10	1.21	0.61	0.61	99.40
No 40	0.43	28.60	28.71	14.36	14.96	85.04
No 60	0.25	53.10	53.21	26.61	41.57	58.44
No 80	0.18	51.10	51.21	25.61	67.17	32.83
No 100	0.15	19.10	19.21	9.61	76.78	23.23
No 200	0.07	26.00	26.11	16.06	89.83	10.17
Fund	---	20.30	20.30			

Table 4. Physicochemical characteristics of rice husk ash and corn husk ash.

Determination	Method	Rice husk ash	Corn husk ash	Average
Density (g/cm3) ASTM C188	Volumetric	0.30	0.70	-
Humidity (%)	Gravimetric	0.86	0.22	-
Organic Material (%)	NTC 5403	1.01	0.55	2.00 – 4.00
Lost By ignition (%)	Gravimetric	87.50	92.50	-

Table 5. Corn husk ash granulometry.

Sieve	Diameter	Retained			% Accumulated	% Filtered out
(Inches)	(Millimetres)	Weight (Grams)	Corrected weight (Grams)	%		
No 4	4.76	0.00	0.00	0.00	0.00	100.00
No 10	2.00	0.10	0.28	0.14	0.14	99.86
No 20	0.84	9.30	9.48	4.74	4.88	95.12
No 40	0.43	40.00	4.018	20.09	24.96	75.04
No 60	0.25	36.40	36.58	18.29	43.25	56.75
No 80	0.18	39.90	40.08	20.04	63.29	36.71
No 100	0.15	15.40	15.58	7.79	71.08	28.93
No 200	0.07	32.10	32.28	16.14	87.22	12.78
Fund	---	25.50	25.50			

According to the results, it is observed that the contents of organic material and humidity are low, which favors experimentation. These results avoid additional reactions that impair the cement's hydration reactions, modifying the setting or reducing the resistance. A low humidity value guarantees an optimal environment for the cement and the cellulosic thickener, avoiding an early reaction of the material in a dry state (Powder). According to the granulometry, the initial amount of ash corresponds to 20% of the material that could be filtered or sieved (# 100). In this sense, 3 kg of rice husk and 1.8 kg of corn husk were obtained for the adhesive mortar composition. Both ashes' granulometric profiles were developed considering the similarity with all aggregate materials' granulometry to guarantee the replacement by partial percentages in the mixture. From this, four mixtures were designed with partial replacements of cement by the pozzolanic material obtained in percentages of 15%, 20%, 25%, and 30% to determine the optimal ash addition. See table 6.

Table 6. Content in grams of the ceramic mortar mixtures.

Materials	Grams			
	15%	20%	25%	30%
Gray Cement	836	784	735	686
Ash	147	196	245	294
Silica mesh # 100	3000	3000	3000	3000
Elotex 60W	8	8	8	8
Bermocoll ME 1000x	8.80	8.80	8.80	8.80
TOTAL	4996	4996	4996	4996

The conventional mixing process was carried out as follows: 1. Dry mixing of aggregates (Bermocoll 1000X and Elotex 60W). 2. Dry mixing of the cementitious material with the pozzolanic material. 3. Pouring cementitious material into the dry aggregate mix. 4. Hydration with water until obtaining the correct consistency. 5. Finally, free time, tensile adhesive strength, and pozzolanic activity index tests were performed.

4. Results and analysis

4.1. Consistency and open time

The amount of water required for each mortar was determined to obtain a paste with a suitable consistency. It was established that in 5 parts by volume of the ceramic mortar, two equal parts by volume of water should be implemented. A thin layer of the prepared adhesive was applied to a concrete surface with a straight side. Later a thicker layer was added again, forming grooves with a notched trowel. After 5, 10, 15, 20, 25, 30 minutes, test ceramic tiles were placed on the adhesive mortar for at least 30 seconds apart, applying a 20 ± 0.05 Newton (2 kg). Finally, the sample units are detached until not less than 30% of the material adhered to the unit is observed, which indicates that the material begins its drying process. It is emphasized that this was an experimental observation control method. The results are tabulated in tables 7 and 8. The results were classified between workable, non-workable, and harden.

Table 7. Open time test in mortar with rice husk ash.

Time (min)	15%		20%		25%		30%		STD
	Sample 1	Sample 2	Sample 1	Sample 2	Sample 1	Sample 2	Sample 1	Sample 2	Sample 1
5	W	W	W	W	W	W	W	W	W
10	W	W	W	W	W	W	W	W	W
15	W	W	W	W	W	W	NW	NW	W
20	W	W	W	W	NW	NW	NW	NW	W
25	W	W	W	W	NW	H	H	H	NW
30	NW	NW	NW	NW	H	H	H	H	NW

* W: workable; NW: Non-workable; H: Harden.

Table 8. Open time test in mortar with corn husk ash.

Time (min)	15%		20%		25%		30%		STD
	Sample 1	Sample 2	Sample 1	Sample 2	Sample 1	Sample 2	Sample 1	Sample 2	Sample 1
5	W	W	W	W	W	W	W	W	W
10	W	W	W	W	W	W	W	W	W
15	W	W	W	W	W	W	NW	NW	W
20	W	W	W	W	NW	NW	NW	NW	W
25	NW	W	W	NW	NW	H	NW	H	NW
30	NW	NW	NW	NW	H	H	H	H	NW

*W: workable; NW: Non-workable; H: Harden

The longer the time available in which the adhesive can be worked, the greater the margin of correction in the event of an error when placing a ceramic material. Therefore, the percentages between 15% and 20% yield the best results. The test with the standard sample indicates that the free time is approximately 20 minutes, which allows us to infer that the addition of ash in the mortar structure favours its free time. The drying capacity is related to the mortar's water retention, which is linked to the effect of the cellulosic thickener, whose function is focused on giving the material an adequate viscosity, which implies an optimal amount of retained water. Percentages more significant than 20% are affected by the function of this substance in the adhesive mortar.

4.2. Adhesive Tensile Strength

A thin layer of the prepared adhesive mortar was applied to a concrete surface with a straight trowel. Later a thicker layer was added again, forming grooves with a notched trowel. After 5 minutes, test ceramic tiles with dimensions of 5cm x 5cm were placed on the adhesive for at least 30 seconds, spaced apart, and applying a force of 20 ± 0.05 Newton (2 kg). After 7 and 14 days of storage at standard conditions, pull heads were bonded to the tiles with a high strength epoxy adhesive. Twenty-four hours later, the tensile adhesive strength was calculated using a force of 250 ± 50 N using a weight-pulley system (NTC 6050 - 2). See Figures 1 and 2.

This property's measurement allowed to establish that the rice husk ash in a partial replacement of cement with 20% obtained excellent results. A smaller difference is observed in the results concerning the standard sample established at 0.188 N and 0.392 N. In the results with corn husk ash, a decrease is identified for the rice's resistance values husk ash. The decrease is due to the different levels of silica in the rice husk. Both materials present a perfect opportunity to carry out partial cement replacements in type (C1) adhesive mixtures. The optimal replacement values range between 20% for rice husk ash and 15% for the case of corn husk. It is essential to highlight that the two materials exceed the reference standard after the test days (14 days) for the tensile adhesive strength.

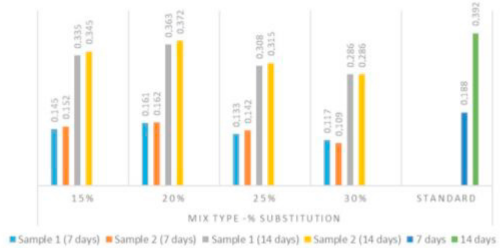

Fig. 1. Adhesive tensile strength (N) in mortars with rice husk ash.

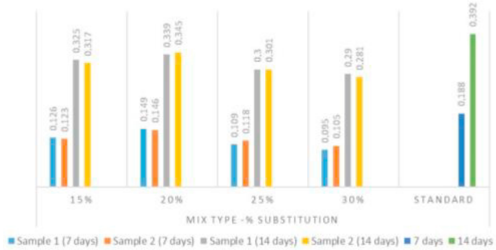

Fig. 2. Adhesive tensile strength (N) in mortars with corn husk ash.

4.3. Pozzolanic Activity Index

The pozzolanic activity index is defined as the ratio of tensile strengths of a mortar with pozzolanic material and that of a cement-based mortar only (standard). These indices were calculated for mortars with partial replacement by rice husk and corn husk ash. See Figures 3 and 4.

Fig. 3. Pozzolanic Activity Index in mortars with rice husk ash.

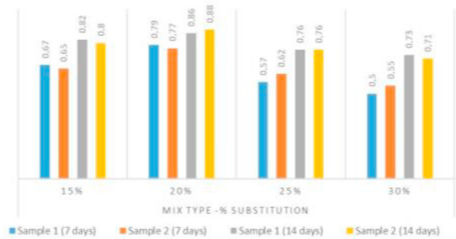

Fig. 4. Pozzolanic Activity Index in mortars with corn husk ashes.

The best results are recorded for a replacement percentage of 20% in both cases. For values more significant than this, a decrease in the adhesive strength is observed, suggesting that the cement setting reactions are affected by interferences in the surface area by particles of the pozzolanic material. Rice husk ash has more significant potential as a pozzolanic material. Rice ash has a higher pozzolanic activity index (0,94), the values recorded from its tests being the closest according to the standard sample.

5. Conclusions

It was found that the use of ashes obtained from the agro-industrial residues of rice husk and corn leaf represents the great potential in its use as a pozzolanic addition in the preparation of adhesive mortars for ceramics. The use of ash has a positive ecological impact since it implies a partial reduction in cement use, which creates an opportunity to reduce the environmental impact and generates an innovation process. Due to their high silica content, Rice husk ash-es become the material with the highest pozzolanic activity index (0.94), which implies improvements in properties such as available time and adhesive resistance. Their values are very similar to the standard sample that uses 100% cement. It is highlighted that the properties are optimized when partial replacements are made in the mixture between the percentages of 15% and 20%. Concentrations higher than the previous ones deteriorate the cement setting reactions. It is essential to highlight that the tests carried out on the mortar were at 7 and 14 days. It implies a medium resistance of the cement (0.392N). Finally, the calcination process's importance (800 ° C) and sieving (# 100 mesh) is highlighted as incidental factors in developing a quality adhesive mortar for ceramics. The water absorption tests established that the mortars' quality is high due to their proximity to the standard sample.

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