

Influence of masonry residue (RM) as supplementary cementitious material in the production of Mortars¹

Influencia del residuo de mampostería (RM) como material cementicio suplementario en la elaboración de morteros

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Abstract

Portland cement production is responsible for about 5 % of global emissions of carbon dioxide (CO₂) emissions worldwide. An important contribution to the sustainability of this industry could be the use of supplementary cementitious materials (SCMs) that reduce the content of Portland cement when making concrete, especially if the material used comes from construction and demolition residues. This paper shows the effect of masonry residue as an alternative material to the commonly used SCMs. This study is presented the experimental results of the characterization of a masonry residue (RM). First, the RM was subjected to a grinding to be analyzed by X-ray fluorescence (XRF), X-ray diffraction (XRD), thermogravimetric analysis (TG) and scanning electron microscopy (SEM). Also, it was evaluated the effect of the incorporation of different levels of RM (from 0 % to 50 %) on the compressive strength in Portland cement mortars at different ages of curing (7, 28, 65 and 90 days), and the density and porosity were evaluated only at 28 days of curing. The results show that this residue (RM) has a pozzolanic behavior and its optimum percentage of replacement in Portland cement mortars is 20 %.

Keywords: residue of masonry; portland cement; pozzolanic activity; mortars; compressive strength.

Introduction

The construction and demolition waste (CDW) are becoming more common in modern society, these are usually composed of different materials among which are residues of concrete, ceramics, wood, plastics, metals, and others (Vegas et al., 2015; Gomes et al., 2015; Özalp et al., 2016). The volume of this waste (CDW) is generated in large

quantities, mainly in urban areas, causing environmental damage due to the lack of adequate sites for disposal, and also due to the neglect of society (Poon, 2007). Among the materials that make up the RCD, concrete and ceramics are those that occur in greater quantity, and they are the masonry residue (RM).

In the city of Cali, Colombia about 2480 m³ of CDW are produced daily, of which 23.4 % is contributed by home remodeling (Ortiz & Silva, 2013). The most common practice to eliminate them is the disposal in authorized landfills that can not supply them and therefore much of this waste is left in green areas, parks or lots. It is estimated that there are 108 chronic garbage dumps and debris in the city of Cali as well as critical points (El País, 2013), which generate problems due to environmental and landscape deterioration in urban areas.

The recycling and reuse of the CDW have become an issue of global interest and there is a high need to have alternative applications for the different materials that make up this waste. Numerous studies have been carried out with acceptable results on the use of CDW as recycled aggregate for applications in road bases and sub-bases (Leite, Motta, Vasconcelos & Bernucci, 2011; Rahman *et al.*, 2015; Xuan, Molenaar & Houben, 2015), as coarse aggregate for the production of a new concrete (Shi-cong, Baojian & Chi-sun, 2012; Xuan, Zhan & Poon, 2016) and as the replacement of natural fine aggregates in concrete (Fan, Huang, Hwang & Chao 2016; Bogas, De Brito & Ramos, 2016).

Another way to use the CDW and more specifically the masonry residue (clay brick with mortar adhered) as partial replacement of Portland cement, since its main component, the clay brick has been subjected to a calcination (burning temperature between 500 °C and 900 °C), where the dehydroxylation of the clay minerals takes place and the formation of metakaolin is produced, which is an amorphous material with high reactivity. Therefore, the residue of ground clay brick (GCB) could be used as supplementary cementitious material, generating environmental benefits since by taking advantage of these wastes, it is possible to reduce the land occupation for these materials, in addition to reducing the emissions of CO₂ generated from cement industry (Ioannou, Iliá & Philokyprou, 2009). In general, clays contain kaolinite (Al₂O₃·2SiO₂ · 2H₂O) that is transformed into metakaolin by calcination (Siddique & Klaus, 2009; Ilić *et al.*, 2016). The reaction between the amorphous silica and the alumina of the metakaolin with the calcium hydroxide produced in the hydration of the Portland cement leads to the formation of

calcium-silicate-hydrate gel (CSH) and calcium aluminate hydrate (C₄AH₁₃), which promote in the mixtures (mortar or concrete) high strength and low permeability (Singh & Garg, 2006).

The objective of this research was to study the feasibility of producing Portland cement mortars using masonry residue (RM). Consequently, this will help reduce the cost of producing the mortar and reduce the environmental impact produced by the CDW and the cement industry. The pozzolanic reactivity of the RM from construction and demolition waste was evaluated and characterized. The effect of the addition of RM up to 50 % on the compressive strength, Absorption and porosity were studied in the Portland cement mortars. Additionally, the optimum content of incorporation of this material according to the compressive strength was established.

Methodology

The methodology used in this study is presented in Figure 1. In the first instance, a sampling of the masonry residue was carried out in a house in demolition of the Cali area, which was built in clay bricks. with Portland cement mortar as binder (Figure 2). Subsequently, the adequacy of waste (RM) was made by reducing the size initially using a jawbreaker and then ground by the ball mill. In the ball mill, a grinding study was carried out where after the third hour samples were taken every 60 minutes until reaching 5 hours of grinding as shown in Figure 3. In this, a bimodal behavior can be observed. obtain different average particle sizes (D [4.3]) for each grinding time, as shown in Table 1. After grinding, characterization of this material was carried out (RM).

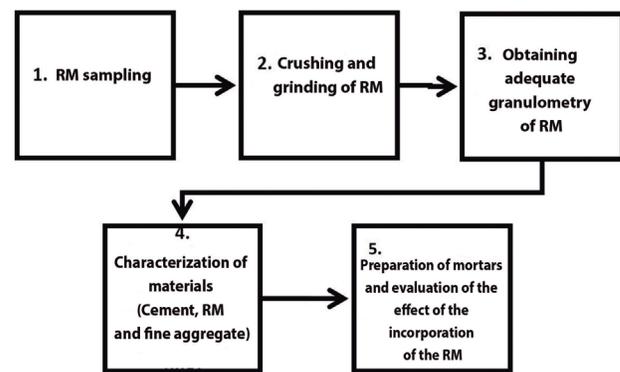


Figure 1. The methodology developed for the characterization and evaluation of masonry waste (RM)

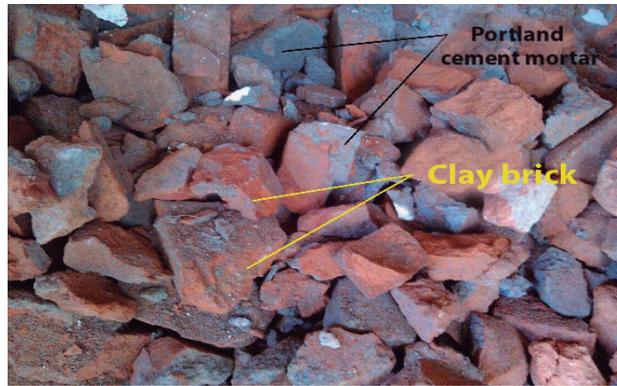


Figure 2. Masonry residue without grinding

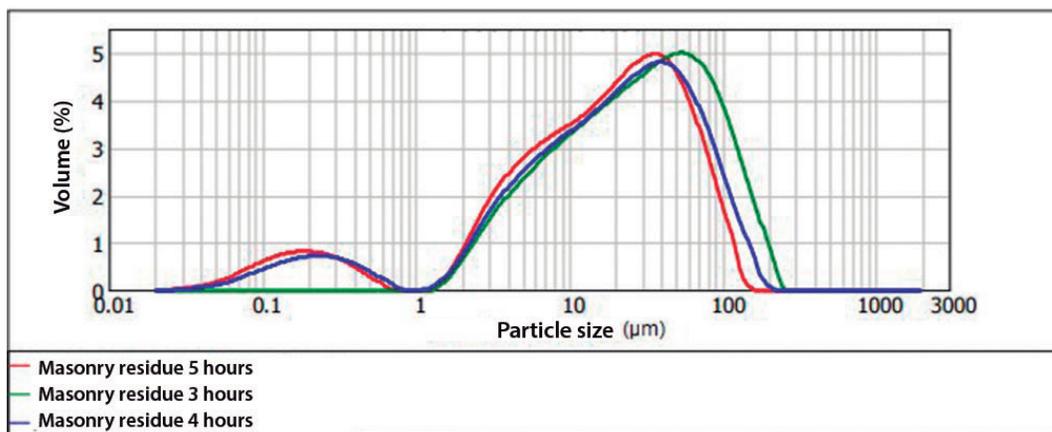


Figure 3. RM granulometry curve at different grinding times

Table 1. Granulometric distribution values of the RM at different grinding times

RM	Grinding time		
	3 hours	4 hours	5 hours
average diameter (µm)	44.16	31.65	24.08
Diameter d(0.1) (µm)	4.99	2.19	1.80
Diameter d(0.5) (µm)	29.01	20.01	15.89
Diameter d(0.9) (µm)	106.95	78.75	65.04

Materials

Cement

Two types of Portland cement were used, one without addition, known as “concrete” cement for the pozzolanic characterization of the RM, and another Portland cement of general use for the preparation of the mortars. The chemical composition of the Portland cement was obtained using X-ray fluorescence analysis (XRF).

Masonry residue (RM)

The masonry residue obtained from the milling process was characterized by different techniques. The RM was investigated in terms of their chemical and mineralogical composition using X-ray fluorescence analysis (XRF) and X-ray diffraction (XRD). On the other hand, scanning electron microscopy (SEM) was used

to determine the morphology of the particles. Finally, a thermogravimetric analysis (TGA) was used to determine the mass loss or gain of the sample, and the Derivative thermo-gravimetric (DTG) to observe the variation of weight versus temperature. (-dm/dt).

Evaluation methods of pozzolanic activity of RM

The evaluation of the pozzolanic activity of masonry the residue (RM) was determined by two methods:

1. Strength activity index according and chemical requirements to ASTM C311 / ASTM C 618

The strength characteristics were determined to observe for possible pozzolanic activity and the determination of the Strength Activity Index (SAI) (ASTM C311). This standard prescribes the use of 20 % by weight of a pozzolan to replace Portland cement. In addition, the ASTM C618 standard presents the chemical and physical requirements for natural fly ash and natural pozzolan. The most important criteria for pozzolanic activity are: (1) the sum of the chemical components, ie $\text{SiO}_2 + \text{Al}_2\text{O}_3 + \text{Fe}_2\text{O}_3$ and (2) the Strength activity index (SAI), which is defined as the relationship of the compressive strength of mortar with the replacement of the pozzolanic material (in this case RM) by Portland cement, with a control mortar (100% Portland cement). For the elaboration of these mortars a ratio of binder to fine aggregate (standardized Ottawa sand) was set constant at 1:2.75 by weight in accordance with ASTM C311. Control mortar was cast using 100% by weight of OPC as a binder, while the RM mortars were cast using a binder in which OPC was replaced by RM at each fineness at a rate of 20 % by weight of binder and a water / binder ratio of 0.55.

2. Chemical method

The chemical method used to determine the pozzolanic activity of the RM was the Frattini test at 7 and 28 days of curing, described in standard NTC 1512. This test evaluates accurately the calcium hydroxide (CH) consumption by the pozzolanic reaction. The Frattini test is a commonly used direct method that involves chemical titration to determine the dissolved Ca^{2+} and OH concentrations in a solution containing OPC and the test pozzolan (Donatello, *et al.*, 2010).

Effect of masonry residue on the hardened state of mortar

The effect of the incorporation of RM was determined through mortars produced with different RM replacement ratios (0 %, 10 %, 20 %, 30 %, 40 %, and 50 %) by the weight of Portland cement. After mixing, fresh mortar mixtures were cast into their cubic molds (50.8 x 50.8 mm x 50.8 mm) and kept there until the end of 24 ± 2 h under humid conditions. After removed from their molds, they were moved into a water tank and cured there until the completion of pre-defined total curing periods of 7, 28, 65 and 90 days, in order to monitor the pozzolanic reactions. In addition, the Absorption and porosity were determined using the procedure described in the ASTM C642 standard, for this the specimens were dried in an oven at a temperature of 105 ± 5 °C for 24 hours. After removing the specimen from the oven, it was allowed to cool in dry air and weighed. The specimen was then immersed in water for 48 h and the saturated surface dry (SSD) weight of the specimen was measured. Then the specimen was immersed in water and boiled continuously for 5 hours. It was taken out, cooled, surface moisture was removed by a towel and then the saturated surface dry (SSD) weight was measured. As a final step, the hydrostatic weight of each of the samples was taken. The water to binder (PC or PC + RM) ratio of mortars was 0.65 and the ratio of binder to fine aggregate was set constant at 1:2.75 by weight.

Experiment design

Table 2 shows the design conditions, where one factor was considered: type of mixture, with 6 levels, which generated 6 treatments. Each of the treatments was performed at random under identical conditions. The response variable in the statistical analysis was the compressive strength at 28 days of curing.

Table 2. Experimental design conditions for optimization of MR replacement in mortars

Factors	Levels (%OPC-%RM)	Treatments	Experimental unit	Response variable
Type of mixture	M1: (1-0)	6	Mortar specimens	Compressive strength (28 days curing)
	M2:(0.9-0.1)			
	M3:(0.8-0.2)			
	M4:(0.7-0.3)			
	M5:(0.6-0.4)			
	M6:(0.5-0.5)			

For the analysis of the results, a completely random design (DCA) is proposed, where the response variable (y_{ij}) is the compressive strength, μ is the general average, α_i is the effect due to the mixture and ϵ_{ij} is the experimental error. The model is given by:

$$y_{ij} = \mu + \alpha_i + \epsilon_{ij} \quad \text{Equation 1}$$

$$i = 1, 2, 3, 4, 5, 6. \quad j = 1, 2, 3.$$

The comparison of the mixtures was carried out by means of an analysis of variance (ANOVA), bosanova tests, and a graph of main effects. The hypotheses associated with D.C.A are $H_0: \mu_1 = \mu_2 \dots = \mu_6$, vs. $H_a: \mu_i \neq \mu_i'$, which are tested by means of ANOVA. The information processing was performed using Minitab 16 statistical package.

Results and discussions

Portland cement

The chemical composition of the two types of Portland cement was determined by FRX and are presented in Table 3. A greater loss by ignition (PI) is observed in the cement of general use (CG), attributed to the calcareous addition (limestone) that presents this cement in its manufacture.

The particle size was determined by laser granulometry and the granulometric distribution values are recorded in Table 4. In this, it is observed that 50% of the cement particles CG and CA are below 15.821 and 17.079 respectively, presenting a percentage greater of fine particles the CG.

Table 3. Chemical composition of general purpose cement (CG) and cement without addition (CA)

Component	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	SO ₃	PI
CG % in weigh	19.39	4.13	4.70	55.68	1.70	0.31	0.28	3.9	9.21
CA % in weigh	17.99	3.88	4.76	62.28	1.71	0.23	0.32	4.03	4.14

Table 4. Granulometric distribution values of the two Portland cement (CG and CA)

Portland Cement	CG	CA
average diameter (µm)	20.670	21.650
Diameter d(0.1) (µm)	1.964	3.594
Diameter d(0.5) (µm)	15.821	17.079
Diameter d(0.9) (µm)	46.818	46.555

Masonry Residue (RM)

The chemical composition of the masonry residue is presented in Table 5, where it can be seen that it is composed mainly of SiO₂, Al₂O₃, Fe₂O₃ and CaO. Red clay is characterized by low calcium content and a higher content of alumina compared to yellow or black clay (Eliche-Quesada & Leite-Costa, 2016). In this case, the

masonry residue ground when having mortar of Portland cement in its composition can present a variation in the components when compared with a clay that is used for the manufacture of bricks. Figure 4 shows the scanning electron microscopy images of the RM taken at 500X and 1000X. The morphology of the RM shows different particle sizes no greater than 30 µm. Some particles finer than others, with irregular morphology.

Table 5. Chemical composition of masonry waste

Component	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	Na ₂ O	K ₂ O	SO ₃	PI
% in weigh	56.859	15.528	7.636	7.881	2.954	2.492	1.362	0.557	3.39

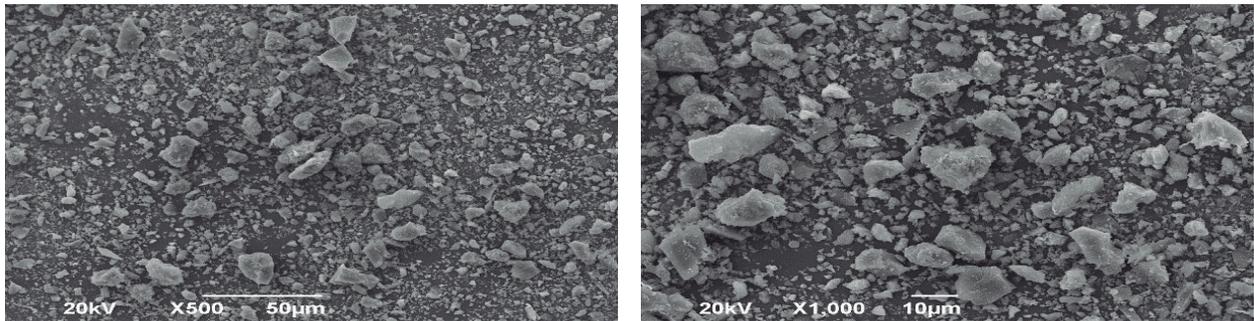


Figure 4. SEM micrograph of masonry waste a) 500X and b) 1000X

Fig. 5 presents the XRD pattern of RM. There are prominent peaks corresponding to quartz (characteristic peaks in 2θ - 24.26; 31.03; 46.16; 49.72; 58.94; 64.69) and peaks of other minerals with a lower intensity such as those of albite, cordierite, anorthite, and hematite. The peak of greater intensity indicates that the sample has a large quantity of quartz (SiO₂), corresponding to the sand present in the mortar paste and to the quartz found in the brick powder.

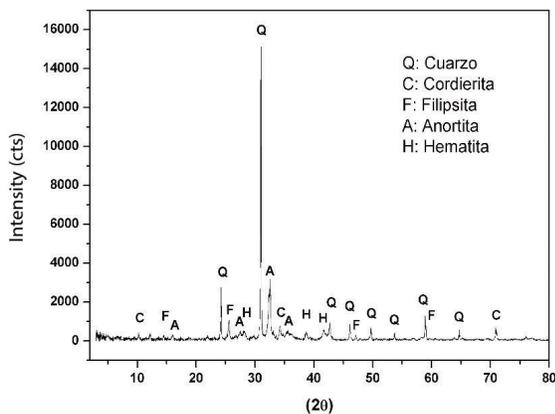


Figure 5. XRD pattern of masonry residue

Figure 6 presents the results of the RM subjected to a simultaneous analysis of thermogravimetry (TGA / DTG) detecting the changes associated with loss of mass by temperature. With the TGA / DTG, it was possible to determine the weight loss at different heating stages up to 1100 °C. A weight loss was detected in the range of 25 °C to 150 °C associated with the humidity present in the sample. Weight loss between 550 °C and 660 °C is associated with the endothermic reaction due to the transformation of the most common materials present in clay: kaolinite and illite (Matias, Faria & Torres, 2014).

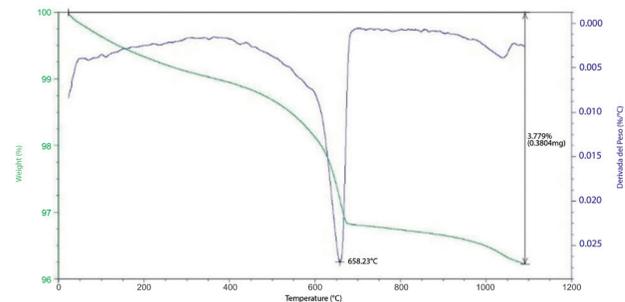


Figure 6. Results of differential thermal analysis of masonry waste

Evaluation of the pozzolanic activity of RM

Pozzolanic activity according to ASTM C618 / ASTM C311

ASTM C618 specifies that the strength activity index (SAI) of pozzolan mortar should not be less than 75 % of the reference mortar. SAI is one of the most important criteria for the determination of pozzolanic performance. The SAI was calculated according to the relationship between the compressive strength of the mortar added with RM (20 %) and the mortar without addition based on that described in the ASTM C311 standard. According to Table 6, the RM complies with the requirement of ASTM C618, the sum of SiO₂, Al₂O₃ and Fe₂O₃ is more than 80 % and the SAI after 28 days of curing exceeds the limit value established in the standard, so it can be affirmed from the point of view of chemical composition and mechanical performance that the RM has potential to be used as a pozzolanic addition. However, the Pozzolanic behavior of material must be corroborated by other techniques that allow establishing the reactive behavior of the RM.

Table 6. Chemical and physical properties of pozzolans according to ASTM C618

Requirements	Puzolana	
	class N, ASTM C618	RM
Chemical Requirement		
SiO ₂ +Al ₂ O ₃ +Fe ₂ O ₃	min. 70.0	80.023
Sulfur trioxide (SO ₃)	Max. 4.0	0.557
Moisture content (%)	Max. 3.0	0.21
Loss on ignition (%)	Max. 10.0	3.39
Physical Requirement		
Strength activity index (SAI), at 28 days, percent of control	min. 75	84.36

Chemical Method - Frattini

In order to complement the results obtained from the pozzolanic activity in accordance with the provisions of the ASTM C618 standard, the chemical method of Frattini was used. This method evaluates the pozzolanic activity and consists of an evaluation of the concentration of hydroxide ions (OH⁻) and calcium ions (Ca²⁺) expressed as calcium oxide (CaO) in a mixture containing 80 % Portland cement and 20 % of RM for a given period (7 and 28 days). The bottled mixture was kept in a thermostatically controlled oven at 40 °C. Then, the concentration of these ions was compared with the solubility isotherm pattern of Ca(OH)₂ at same temperature. If the [Ca²⁺] and [OH⁻] concentrations are below the lime solubility curve, the mineral addition is considered chemically active (Amar *et al.*, 2017).

The results of the frattini test are presented in Figure 7. At 7 days of contact between the materials, pozzolanic activity is not observed, because the consumption of calcium ions and total alkalinity were not enough, so the RM is located above the isotherm of solubility of Ca(OH)₂. However, at 28 days, the concentration of CaO in the solution decreases as well as the alkalinity, so the RM was able to locate in the pozzolanic region or low saturation, this behavior indicates that a large proportion of calcium hydroxide generated during the hydration of Portland cement it has been consumed by the pozzolanic reaction (Tironi, Trezza, Scian & Irassar, 2012), in this way the results obtained by the mechanical method (SAI) are corroborated.

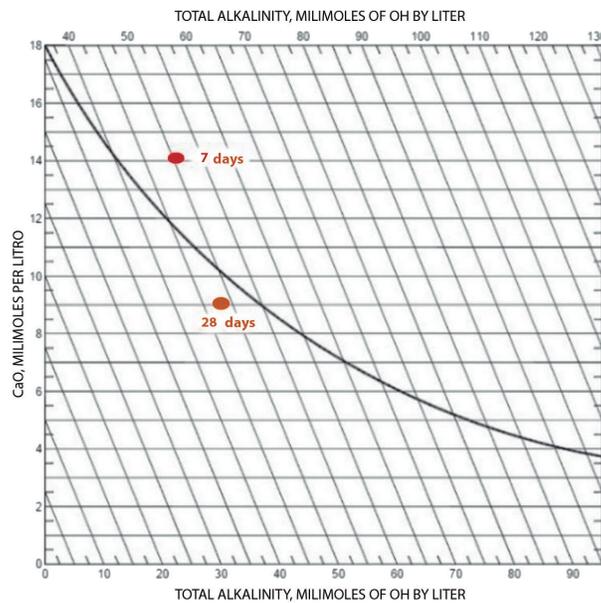


Figure 7. Diagram of the Frattini test for the masonry residue

Effect of the incorporation of the masonry residue in different levels in the properties of the mortar in the hardened state

After evaluating the pozzolanic behavior of the RM by means of physical and chemical characteristics, six (6) different mortar samples were prepared. The mixtures were designated as follows: the control mixture - (M1), the mixture with 10 % RM - M2: (0.9-0.1); with 20 % RM-M3: (0.8-0.2); with 30 % of RM-M4: (0.7-0.3); with 40 % RM-M5: (0.6-0.4) and 50 % of RM-M6: (0.5-0.5). The compressive strength of the mixtures was evaluated at different ages of curing (7, 28, 65 and 90 days) and the results are presented in Figure 8. It is observed that the compressive strength is influenced by the percentage of substitution of the RM. The control mixture gained the highest strength and the strength decreased with increasing RM replacement rates, however in the case of mortars with replacements up to 20% of RM the difference is not so high. The reduction in resistance becomes greater as the replacement level increases. The influence of the decrease is more important at shortages (7 and 28 days of curing), after this time, this difference decreases because the resistance of the reference mortar became almost stable, But the average strength development of mortar with RM increased continuously until 90 days of curing. In mortars with RM, when the substitution level changed from 0 to 10 %, 20 %, 30 %, 40 % and 50 % the compressive strength decreased by 9.4%, 19.3%, 32.3%, 48.5% and 59.9 % respectively at 28 days and 7.1 %, 13.1 %, 24.4 %, 38.43 % and 48.42 % at 90 days. The observed loss of strength could be attributed to the low pozzolanic activity mainly at early ages of RM with consequential dilution of active phases contained in cement (dilution effect) so that the pozzolanic effect of the mineral admixtures was evident after 90 days of curing, decreasing the difference between the strength of the control mixture and the mixtures that contain RM.

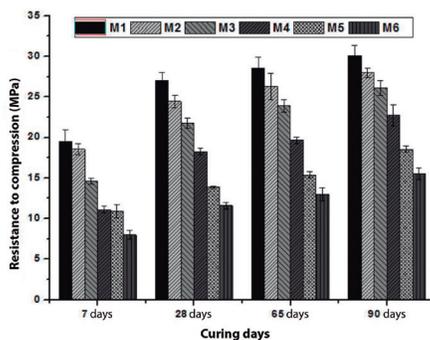


Figure 8. Compressive strength of mortars with RM

On the other hand, in the analysis of the statistical design (Table 7), the following results were found:

- The model was proposed to satisfy the assumptions of error (normality and homogeneity) at significance levels greater than 10 %.
- With an R^2 of 98.75 %, which indicates that good control of the experiment was exercised since the variation is being explained by the mixture and not by the errors, a result that is also evident in the sum of squares
- With respect to the comparison of the mixtures, a level of significance 0.0000 there is a difference between the means of the mixtures (M1, M2, M3, M4, M5, and M6), i.e. that at least there is a pair of stockings that have a different effect on the compressive strength of mortars.

Table 7. Analysis of variance of the resistance to compression (MPa)

Source of variation	Sum of squares	p-value
Mezcla	544.3	0.0000
Error	4.83	
Total	249.16	

In order to establish that between mixtures there are differences, and which is the optimum mixture of portland cement with RM in mortars after 28 days of curing, a ANOVA test was performed using the Tukey test at a level of significance of 5 % (Table 8). It was found that the M2 and M3 mixtures are above the average (located in the same group of the control mix-M1), as shown in Figure 9, indicating that up to 20 % of incorporation RM of this material present appropriate results exceeding 21 MPa at 28 days of curing. In addition, the mortar with 20 % of RM has an environmental and economic advantage. Since it is giving an appropriate use to this waste, generating a decrease in the consumption of cement portland, which leads to lower CO₂ production derived from the manufacture of this binder, in addition, it make use of the large volumes of construction and demolition waste, which avoids the need for more landfills for final disposal.

Table 8. Post-Anova Test using the Tukey method

Type Mix	N	Subset					
		1	2	3	4	5	6
M6	3	11.52					
M5	3		13.89				
M4	3			18.26			
M3	3				21.51		
M2	3					24.46	
M1	3						26.99

Source: the authors

Other important properties that were studied: absorption, apparent density and the volume of permeable pore space (%), which were calculated according to the ASTM C642 standard after 28 days of curing. The results are shown in Table 9. The addition of the RM generates an increase in the percentage of absorption and volume

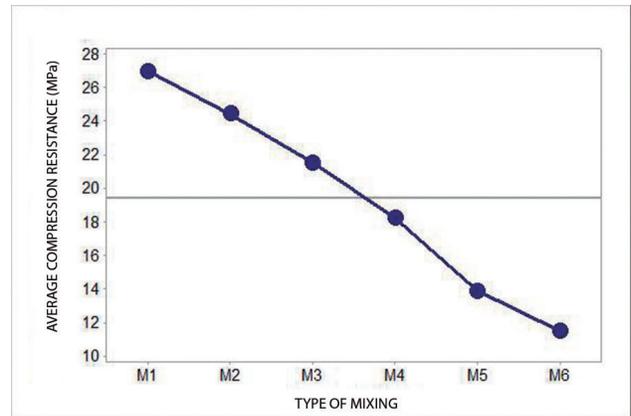


Figure 9. Graph of main effects of the resistance to compression at 28 days of curing

of permeable pores; and a decrease in apparent density. This can be attributed to the fact that the water does not completely react in the mixture, as a consequence of the presence of the RM, and some part water remains free in the structure and when evaporating leaves pores in the mixture.

Table 9. Absorption, density and porosity results according to the ASTM C642 standard

Mix	% absorption after immersion	Apparent density (kg/m ³)	% of Volume of permeable pores
M1	11.62	2599	22.59
M2	11.91	2554	22.77
M3	12.28	2537	23.01
M4	12.70	2536	24.16
M5	13.4	2525	25.21
M6	13.54	2524	25.52

Conclusions

This study aimed at investigating and characterize the masonry residue (clay brick and Portland cement mortar) in the city of Cali, Colombia. This material from construction and demolition waste were incorporated into Portland cement mortars as partial replacement of cement and its influence on the mechanical behavior of the mortars with different percentages was analyzed, obtaining the following conclusions:

The characterization of the RM with respect to its physical and chemical behavior reveals that this material can be considered as pozzolanic material, since the SAI was higher than 75 % as it is specified in the standard ASTM C618 and the Frattini test shows the positive fixation of calcium, placing it below the saturation curve at 28 days.

The compressive strength of the mortars with RM in their composition, presented lower values than the reference mortar, regardless of the percentage of replacement. This difference became smaller as the age of cure increased, which allows us to conclude that the RM is a slow reaction admixture mineral since at early ages it does not contribute to the evolution of resistance, on the contrary, it affects these developments due to the effect dilution.

the use of RM (reddish color) generates a coloration in the mortar mixtures, which can be interesting for conservation purposes, rehabilitation and in some cases do without the need for paintings, for example in architectural elements. Therefore, the replacement of Portland cement by RM seems to be a promising solution.

Acknowledgments

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