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### **Original Article**

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# Structural characterization of post-consumption sludge as aggregates in construction materials

Caracterización estructural de lodos pos-consumo como agregados en materiales de construcción

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### ABSTRACT

### **Keywords:**

DRX Minerals Mechanical resistance **SEM** 

The present work covers the physical, mechanical and chemical characterization of pozzolanic materials. The pozzolans were obtained by mixing red clay, commercial bentonite and residual aluminous sludge, from a drinking water treatment plant. Ceramic cubes were manufactured, which were sintered at 1100 ° C and subsequently, were structurally characterized by DRX, SEM and EDS. Its compressive strength was also evaluated before and after being subjected to different environments. The results showed that the relations with which the standards for nonstructural bricks according to NTC 4017 (100 Kgf / cm2) are maintained are those that contain 10%, 20% and 30% of mud with respect to the clay maintaining an aggregate of bentonite 10% with resistances of 123.62 Kgf / cm2, 112.90 Kgf / cm2 and 107.32 Kgf / cm2, respectively. Likewise, for units exposed to corrosive environments, the 10% replacement mixture is the only one that meets the criteria established by the standard with a resistance of 112,80 Kgf/cm2 and in abrasive environments mixtures of 10% and 20% with resistance of 116,75 Kgf/cm2 and 101,96 Kgf/cm2 respectively.

### **RESUMEN**

### Palabras clave:

DRX Minerales Resistencia mecánica **SEM** 

El presente trabajo abarca la caracterización física, mecánica y química de materiales puzolánicos. Las puzolanas fueron obtenidas por la mezcla de arcilla roja, bentonita comercial y lodos aluminosos residuales, provenientes de una planta de tratamiento de agua potable. Se fabricaron cubos cerámicos, los cuales fueron sinterizados a 1100 °C y posteriormente, fueron caracterizados estructuralmente por DRX, SEM y EDS. También se evaluó su resistencia a la compresión antes y después de ser sometidos a diferentes ambientes. Los resultados demostraron que las relaciones con las cuales se mantienen los estándares para ladrillos no estructurales según la NTC 4017 (100 Kgf/cm2) son los que contienen 10%, 20% y 30% de lodo respecto a la arcilla manteniendo un agregado de bentonita del 10 % con resistencias de 123,62 Kgf/cm2, 112,90 Kgf/cm2 y 107,32 Kgf/cm2, respectivamente. Así mismo para las unidades expuestas a ambientes corrosivos la mezcla con sustitución del 10% es la única que cumple el criterio establecido por la norma con una resistencia de 112,80 Kgf/cm2 y en ambientes abrasivos las mezclas de 10% y 20% con resistencias de 116,75 Kgf/cm2 y 101,96 Kgf/cm2 respectivamente.

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### 1. Introduction

**Pozzolanics** are commonly siliceous aluminosilicate materials which are capable of improving the properties of different products, this makes them useful in the formation of clay pieces which are subjected to a sintering process [1]; In Colombia, red, black clays and as a complement sand are commonly used as raw material for the formation of masonry products, these materials are found in rivers due to displacements due to climatic factors (such as rain and air) and are considered as secondary character for not being in its place of origin, due to this physical processes are produced which provide certain properties such as a lower grain size, acquisition of organic material, feldspar among others. During the last years the study of materials has focused on the incorporation of byproducts of the industry or pozzolanics, considered waste [2], for the production of materials that are more environmentally friendly, one of these aluminosilicates is the sludge from the drinking water treatment plants (PTAP).

In the PTAP during the water purification process, an amount of coagulant is added, commonly aluminum sulfate which contributes to the destabilization of the suspended particles, followed by the formation of flocks in the flocculation stage and subsequent sedimentation, thus generating sludge formation considered waste; these sludges are mostly made up of inorganic substances, feldspars, silts, fine sands and are characterized by having a high concentration of silicon oxide and aluminum oxide; Approximately 0.3% to 1.0% of these occur in a PTAP with respect to the total volume of treated water [3]. In Colombia there is currently no regulation for the final disposal of sludge, which is why the PTAP discharges them downstream of the water streams from which they initially acquire their waters [4], although most of the composition of sludges are inorganic substances, they also contain iron, metal oxides and hydroxides, which in the long term cause a decrease in photosynthetic activity in aquatic plants, in turn the high concentrations of aluminum present form sediments that isolate the benthic layer making it difficult to life of marine species that develop at this level.

In Colombia, the reuse of Pozzolan materials mostly covers the construction sector, but in the case of sludge resulting from water treatment, the information reported is scarce, being thus used as additives for the production of Clinker, cement Portland and as a raw material in the recovery of aluminum [1,3–7], in turn due to the characteristics they possess; Many authors have studied the feasibility of reusing them in the ceramic industry by categorizing them as secondary clays; To do this, a sludge dehydration process is carried out, which consists in eliminating the greatest amount of surface water and that found within the crystalline network of the different minerals present in the mud. For the manufacture of masonry units in Colombia, the Colombian Technical Standard (NTC) [8] must be taken into account, as this specimens created in structural and non-structural construction units can be classified.

In this study, the compressive strength of nonstructural type pozzolanic materials was evaluated before and after chemical attacks with hydrochloric acid and sodium hydroxide, followed by an X-ray diffraction (DRX) characterization of the crystalline phases present in the units, scanning electron microscopy (SEM) and X-ray dispersive energy (EDS).

### 2. Materials and methods

For the respective characterizations, mixtures of a standard clay with mud doped with commercial bentonite were made in the following proportions, 100: 0: 0, 80:10:10, 70:20:10 and 60:30:10 respectively; for the trials involving evaluation of mechanical strength, solid cubic units of 5 cm3 sintering them to 1100° C.

# Mechanical characterization, compressive strength

The mechanical resistance of all units was carried out according to Colombian Technical Standard 4017 "Methods for sampling and testing of masonry units and other clay products", This indicates that the compressive strength of solid clay units must be at least 100 Kgf/cm<sup>2</sup>[8]; This is calculated by dividing the breaking or failure load over the area of the unit analyzed, in turn the mechanical strength of the specimens was evaluated after being subjected to corrosive and abrasive environments [9].

# Morphological and chemical characterization

The chemical and morphological characterization of the units were carried out through x-ray diffraction (DRX), these data were taken in a powder diffractometer brand Bruker model D8 advance with DaVinci geometry, using a polymethylmethacrylate (PMMA) sample holder, a copper radiation (Cu  $K\alpha \lambda = 1.5406 \text{ Å}$ ) and a LynxEye linear detector; the data was recorded with a step of  $0.02035^{\circ}$  (20) between  $3.5^{\circ}$  -  $70.0^{\circ}$  (20) with a speed of 0.8 seg/paso, operating with a voltage of 40 kV and a stream of 40 mA and scanning electron microscopy (SEM) for which each sample was coated with graphite, in a Quorum 150ES coating equipment, the micrographs were obtained in a Quanta Feg 650 microscope with an FEG type electron source (Field Emission) Gun), the equipment operated at low vacuum with an acceleration voltage of 25 kV and a scattered electron image detector type SSD. For the EDS type chemical analysis, the EDAX APOLO X detector was used whose resolution is 126.1 eV, using Mn radiation, and obtaining the semi-quantitative information using the EDX Genesis software.

# 3. Results and Discussions

# Mechanical characterization, compressive strength

The results from the compressive strength for the units of different clay mixtures: sludge: bentonite

according to NTC 4017 are recorded in Table I. In this it can be seen that changing the concentration of clay: mud with constant bentonite; as the concentration of sludge in the unit increases, the compressive strength decreases approximately 10 units. For mixtures with a higher percentage of sludge, the quality standards described by the standard for non-structural materials [8] would not be met. At the same time, it is observed that the replacement of 10% of bentonite in the samples generates a considerable increase in the mechanical resistance compared to the clayonly sample; Therefore, it is considered that these types of minerals provide structural properties of vital importance. Without this doping, it would be expected that units with some percentage of mud in their structure would not comply with the provisions of the standard.

Table I. Mechanical resistance of mortars

Mixture	Mechanical strength [Kgf/cm <sup>2</sup> ]
100:0:0	112,24
80:10:10	123,62
70:20:10	112,90
60:30:10	107,32

Due to the resistance presented by the materials, the resistance to corrosive and abrasive media [9] was carried out at 4 units, the results obtained are recorded in Table II.

Table I. Mechanical resistance of mortars after chemical attack

Mixture	HCl attack Mechanical strengtha [Kgf/cm²]	NaOH attack Mechanical strength [Kgf/cm²]
100:0:0	106,34	109,02
80:10:10	112,02	116,75
70:20:10	99,65	101,96
60:30:10	89 35	98.65

During the attacks with hydrochloric acid and sodium hydroxide it could be seen that the immersed material initially absorbs a large amount of solution which decreased as the concentration of substituent material increased, this due to the porosity presented by the material after the sintering process. The materials have a much lower resistance when subjected to acidic environments than the base environments, which may indicate that the material is highly corrosive, not suitable for acidic environments. In turn, it is observed that as the percentage of sludge

in the sample increases, the resistance decreases by approximately 10 units.

Finally, it is taken as regards abrasive media that the units with a 30% replacement of mud do not meet the minimum strength stipulated in the standard, while in corrosive media only the blank and the replacement of 10% meet the stipulated value.

# Morphological and chemical characterization

Through DRX, the mineralogical composition present in the mixtures was observed, the presence of feldspar-type minerals being found in all three units. The diffractogram for the 60:30:10 mixture is shown in Figure 1. It can be seen as a majority phase quartz (Q) from both mud and clay and a high percentage of albite (A) from bentonite, in turn the phases illite (I), muscovite 2M (M), microcline (Mi), hematite (H) and magnesiohornblende (Mg).

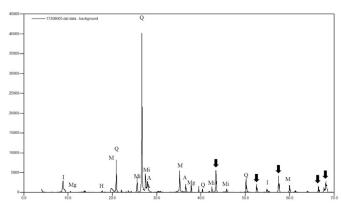


Figure 1. Mix diffractogram 60:30:10

The surface morphology of sintered specimens was performed using SEM. In Figure 2, the clay micrographs are shown. It is observed that the present morphology is varied due to the presence of crystals with different sizes and shapes, thus giving an irregular surface with high porosity as expected, since the organic matter present in the clay has undergone an oxidation process [10].

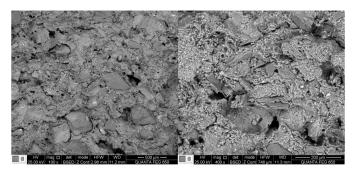


Figure 2. SEM micrograph of clay

In Figure 3, the micrograph of the 60:30:10 mixture is shown. It can be seen the presence of crystals of different sizes from both clay and mud, it is also appreciated that there is a decrease in porosity attributed to the presence of bentonite, since it is amorphous and has a smaller particle size, which makes it possible to be located around the crystals forming a protective layer that when subjected to high temperatures causes these particles to become porous and spongy [11].

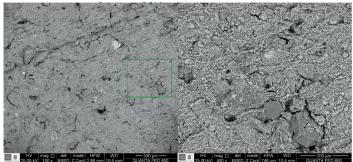


Figure 3. SEM micrograph of the mixture 60:30:10

Finally, a chemical analysis was performed. Figures 4 and 5 show the spectra of dispersed energies of the clay and the mixture 60:30:10 respectively. They show that they consist mainly of Si and Al and some traces of Ca, Na, Mg, K, and Ti; these traces come from the different crystalline phases that are found in each of the raw materials being dispersed and randomly in the microstructure There is also the presence of iron oxides, which is responsible for the brown tones of the final pieces and titanium which is usually added to ceramic materials [12].

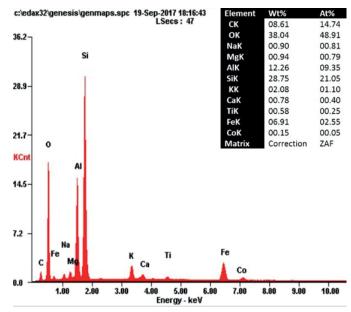


Figure 4. EDS of clay

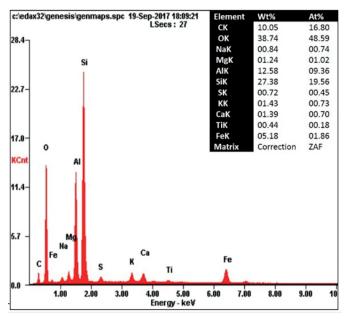


Figure 5. EDS of the mixture 60:20:10

## 4. Conclusions

Waste sludge from drinking water treatment, serve as aggregates in pozzolanic materials in substitutions of 10%, 20%, and 30% since they meet the minimum values required by the Colombian technical standard.

The additions of bentonite in the manufactured units confer mechanical properties to them, generating an increase in compressive strength.

Units created with percentages of 10% and 20% of mud can be exposed to abrasive environments; while only 10% can be subjected to corrosive environments.

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