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Evaluation, comparison and differentiation of geopolymers by studying microstructural

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Abstract— Geopolymers are materials derived from an activation process materials with high content in silicon and aluminum, that in contact with an alkaline solution can acquire cementitious properties, and whose added value is to be environmentally friendly.

This research have like objective to determine the efficiency of geopolymers using two different types of fly ash (FA) as a possible replacement of an alternative material to cement Portland (CP). The manufacturing process consisted of the FA activation using NaOH, the tests conducted included the chemical characterization of the FA through X-ray fluorescence (XRF) and X-ray Diffraction (XRD); as well as the determination of the compressive strength at various ages curing according to ASTM C10 and development of crystalline phases by XRD. The results showed that with increased age curing is achieved the development of resistance and the generation of crystalline phases, also acquiring compression strength above of 20 MPa at the age of 14 days cure.

Key words—Fly ash, geopolymer, alkaline activation, compressive strength

Introduction

The high growth in the population also creates an increased demand in construction materials, the above in turn has led to need to be investigated new materials that can meet this need, and likewise, that they be cheap and friendly to the environment. Among them, one of the most used, and most polluting in this sector is the CP (one ton of CP requires on the order of 1.5 tonnes of non-renewable resources, and emit around of 0.8 to 1.1 tonnes CO_2) (1-3).

As an alternative to this problem, is usual the proposal of supplementary cementitious materials produced of wastes in industrial applications such as silica fume, blast furnace slag and FA; the latter mostly with origin of thermal power plants, which, by alkaline activation (geopolymer) can get to get cementitious value equivalent to CP.

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José M. Gómez-Soberón. Universidad Politécnica de Cataluña. Spain. Researches on geopolymers have increased from the 90's (4) and mostly, they have provided information of their mechanical properties, the activator solutions, the optimum temperatures of activation and the study of the formation of its hydration products; without be studied to considered possible existing variations depending of the source original FA used. Therefore, be propose in this paper a comparative through parallel study using two of them, including in addition to activation of both, their characterization by XRD, XRF techniques and its relation to behaviour compression mechanical at different ages.

п. Methodology

Two CV were selected for this study: black fly ash (BFA) and white fly ash (WFA); both from coal plants located in Coahuila, Mexico.

Their characterization was performed by measuring the content of chemicals compounds using the spectrometric X-ray fluorescence (XRF) technique (Table 1) and ASTM C618 standard (when the sum of $SiO_2 + Al_2O_3 + Fe_2O_3 > 70\%$ and CaO <10% is classified type F, and as a class C when the minimum amount of $SiO_2 + Al_2O_3 + Fe_2O_3$ is 50% and CaO> 10%). The XRF technique is based on the irradiation of the sample with a beam of X-rays to excite the elements present and that these emit their own characteristic fluorescent X-rays. Based on the results obtained both FA are classified as class F.

Table 1 Resulted of FRX.

| Percentage de oxides | BFA | WFA |
|----------------------|-------|-------|
| SiO_2 | 56.89 | 59.11 |
| Al_2O_3 | 26.24 | 20.31 |
| Fe_2O_3 | 5.566 | 4.63 |
| CaO | 4.253 | 9.918 |

The stages performed for geopolymers manufacturing were: a) grinding the FA through a steel ball mill until a size reduction of 45µm, b) activation by alkaline solution of NaOH 8 molar with a ratio solution/FA equal to 0.5, c) performing cubic samples 5cmx5cmx5cm of paste, d) curing specimens at a temperature of 80 $^{\circ}$ C, with relative humidity (RH) 99%; for which the specimens were placed in sealed bags to avoid contact with the water of outside and then immersed in water at temperature studio for ages 3, 7, 14 and 21 days (Figure 1), completed the curing time, mechanical compression testing and characterization by XRD technique were performed.



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Figure 1 Manufacturing process of geopolymers: a) crushing, b) activation, c) specimens c) curing

A. XRD

XRD is a technique that was used to detect the amorphous nature and the phases present of the origin FA; as well as to identify the creation of new phases produced by alkaline activation solution. The tests were performed on a diffractometer PRO PANalyticalX'Pert (see Figure 2) equipped with a source CuKa radiation X-rays; the assay was performed in the range 5 $^{\circ}$ to 70 $^{\circ}$ of 20 in steps of scanning 0.05s. Samples were prepared by grinding material from the specimens tested in mechanical testing in an agate mortar until obtaining a similar fineness of the talcum, the resultant powders were prepared in the sample holder and introduced into the diffractometer for analysis.



Figure 2 Diffractometer PANalyticalX'Pert PRO

B. Simple compression resistance

Mechanical tests (Figure 3) were performed in accordance with ASTM C109 standard, making an adaptation of this (because there is now a specific for this type of material). These variations were to not use natural fine aggregate and use application speed of the load applied to 50kg/s. The tests were conducted in a universal press mark INSTRON 600DXR2081



Figure 3 Realization of the compression test: a) before loading, b) specimen tested

III. Results

A. XRD

Figure 3 shows the differences between starting FA and geopolymer activated produced, demonstrated that the generation of new phases is being produced by alkali activation, whereas on the other hand, the existing phases unchanged preserved. This also is validated by the displacement in the halo, attributed to the amorphous material of the FA that appeared between $^{\circ}18^{\circ}-30^{\circ}$ 20, and that after the activation process is travelled at higher values of 2θ (of 22° to 40°), the above is explained by the alkaline cations of the activator solution which promote dissolution of the silicoaluminate material on FA to generate to forming product reaction gel (5-6).

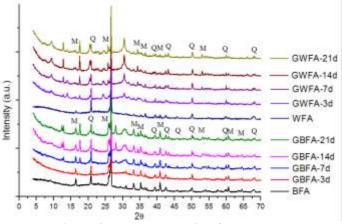


Figure 4 XRD. M = mullite, Q = quartz

The differences that can be observed between GBFA and GWFA is the presence of peaks appearing in GWFA at 8° of 2θ , which are attributed to zeolitic phases, as well as the peak at 28° 2θ related with the presence of sodium carbonates in GBFA. Besides the above, in both geopolymer is observed the presence of the other peak at 13° that increases with increasing days of curing, attributed to the generation of the formation of crystal lattice of the gel. The above shows that more cure days can be obtained greater reaction products (7-8).

B. Simple compression resistance

In the Table 2 are presented the results of compressive strength for the study variables. The sample of GWFA it was obtained higher strength (36% compared to GBFA at age of 21 days curing), this increase was related to the presence of elements or simple compounds present in FA source, since the SiO_2 / $\mathrm{Al}_2\mathrm{O}_3$ ratio for WFA became 2.91 while for BFA only reached a value of 2.16. The explanatory logic of behaviour indicates that most relationship between these compounds can reach an increase in the development of its reaction products. Similarly, the production of the Si-O may become greater than



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Al-O bond, so that it results a more resistant to mechanical stress gel (9-10).

Table 2 Compression test results.

| Compressive strength (MPa) | | | | |
|----------------------------|--------|--------|---------|---------|
| Geopolymer | 3 days | 7 days | 14 days | 21 days |
| GBFA | 8.30 | 10.70 | 23.50 | 26.00 |
| GWFA | 8.80 | 15.20 | 30.00 | 40.90 |

On the other hand (see Figure 5), it is observed that the resistance of both gepolymers is always incremental throughout the curing period studied, can be said that the extension of thermal curing is a positive factor for the development and improved mechanical properties**Error! Reference source not found.** (11-12).

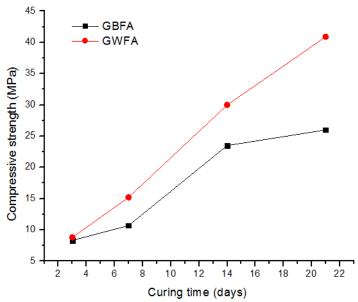


Figure 5 simple compression of the geopolymer.

IV. Conclusion

From the results obtained in this study, it can be said that the production of the geopolymer based in FA may be a choice of suitable material for the production of prefabricated structural elements in the construction, since they reported strength values similar compression to those obtained with CP, and since in this case the prefabricated building techniques used in the accelerated curing at high temperatures (80 ° C), the manufacture of these using active FA may be feasible. Moreover, the FA require an activation process using alkaline solutions, their management must be done with safety and security measures that only an industrialized process can be facilitated Finally, the use of FA is only feasible if there is a previous study of the behaviour of these, as these are considered heterogeneous because its composition is always variable and content of oxides is essential in the process of

activation, guarantor of you can get good mechanical properties.

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