International Journal of Material Science & Engineering– IJMSE
Volume 2 : Issue 1 [ISSN : 2374-149X]
Publication Date : 30 April, 2015

Analysis of the maturation process of geopolymer mortars

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***Abstract*— Mechanical and maturation of geopolymers mortars based on fly ash –FA– (FAM), properties are studied. The values of FAM in compressive strength were lower than conventional Portland cement mortars (PCM). For geopolymers were achieved in some cases compressive strengths up to 24.42 MPa, but in another study variables an abnormal behaviour was identified with loss of compressive strengths and elastic modulus (reaching only 14.51 MPa and 1.24 GPa). This behaviour can be attributed to a possible reaction between the aggregate and the alkaline medium, which appears to increase with +age of curing. Involvement implies that the reaction impedes or disrupts the geopolymerization process of FA, causing less compressive strength**.

***Keywords*—Geopolimerization, compressive strength, elastic modulus, Fly ash**

I. Introduction

Important research in the field of sustainable materials that can be used in the construction is underway in recent years; among the most commonly used, the cement is considered one of the main ones that affect the environment, because of its volume of production (produces on the order of one ton of CO_2 for every ton of cement) [1]. So as an alternative to its use, different materials with cementing capacity are being investigating [2,3] (Cementitious Supplementary Material, CSM); among these, the fly ashes (FA) transformed through their activation (called inorganic polymers or geopolymers), may be able to replace the cement even in its totality [4].

The term geopolymers was introduced for the first time by Davidovits [5]. These materials developed from industrial wastes are considered as inorganic binders similar to zeolitic materials with amorphous structures. The process of geopolymerization involves a rapid reaction of silica-aluminous materials in alkaline conditions, resulting in a chain polymerized in three dimensions with Si-O-Al-O bonds [6]. For the activation of these materials, are often used alkaline solutions with concentrations higher than 8M, the most commonly used is the sodium hydroxide (NaOH). The activa tor has the function to start the geopolymerization process by dissolving the constituents of silica and aluminum that contain the starting material.

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Currently there are investigations of geopolymer pastes [7, 8]; however, for more complex matrices as a mortar, just some basic physical properties have been reported [9, 10] therefore, this research proposes the study of this type of complex matrices of mortars. So, it is necessary to determine their physical properties, thus allowing correlate them with previous studies, and mechanical as its resistance to compression and dynamic modulus of elasticity that are still unsolved in its totality. In this way, will be achieved to establish a friendly material with the environment and able to replace cement.

II. Methodology

A. *Materials*

The FA used was from the power plant Rio Escondido, Chihuahua, Mexico; this was classified as type F in accordance with ASTM C618 standard. The cementing material used like reference was an ordinary Portland cement Type III according to ASTM C150 or compound Portland cement (CPC 30R - regulation NMX-C-414). Standardized sand defined in ASTM C33 standard was used as fine aggregate (AG). Finally, an alkaline solution with a concentration 8M based at sodium hydroxide (NaOH) with a purity of 97% was used as activator of FA; which was prepared with NaOH flakes which were dissolved in distilled water under continuous stirring for 5 minutes until their disintegration (at a ratio of 320 g of NaOH per liter of distilled water). They were used after 24 hours of dissolution, ensuring the dissipation of the chemical heat generated.

B. *Preparation of mixture*

The mortars mixtures design was performed following the proportions of the standard ASTM C642, they are manufactured with a ratio solution/cementitius material of 0.5. Prior to use, the FA was crushed in a steel ball mill and sifted through mesh # 325, and then was mixed with the activator solution 8M for 5 min. manually in a nonreactive bowl and using a spatula, after which the FA was incorporated and mixed for 5 min. The variables of the study were grouped in two groups: those who used FA (FAM7, FAM14 and FAM28) and those who serve as reference using Portland cement PC: (PCM7, PCM14 and PCM28). In both cases the number indicates the age at which samples were tested.

After performed mixtures, test specimens were fabricated, these were then covered in plastic wrap to prevent loss of activator solution and introduced to the oven for a period of 24 hours at a temperature of 80 °C; after this period, the specimens were unmolded. The specimens were placed in sterile sealable bags to prevent the penetration of curing water.

For curing, the samples were introduced into sealed containers with distilled water and these were placed in an oven at 80 °C until the test age (see Figure 1). PC based mortars were produced as controls.

Figure 1. Preparation of mortars process: a) preparation of mixture; b) filling of molds; c) acceleration of curing process at 80 °C; d) unmolding; e) specimens in watertight containers with water; f) curing at 80 °C.

C. *Mechanical behavior*

For the evaluation of the compressive strength, three specimens were manufactured with dimensions of 5cm x 5cm x 5cm, for each variable of study; which were tested as indicated by the ASTM C109, and using a universal press INSTRON 600DX model. In the case of the modulus property of elasticity the specimens used were performed on specimens of 4cm x 4cm x 16cm. The applied test used the nondestructive dynamic method by using the frequency of resonance produced by an impact according to ASTM C215; the assay consists of excite the specimen with a hammer blow and capture the frequency response produced by wave using an accelerometer (see Figure 2).

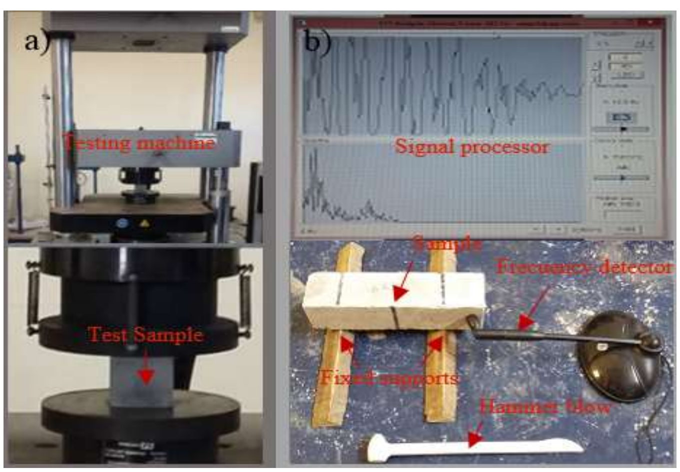

Figure 2. Test of the mechanical behavior of mortars: (a) compression test; (b) elastic modulus test.

D. *Scanning electron microscope (SEM)*

The SEM characterization was performed on a JEOL JSM-6510. The samples analyzed were FAM7, FAM28, FAM7 and FAM28. The sample preparation consisted of: extraction of the interest region by cutting with sierra, encapsulated of samples with epoxies resin [Bisphenol-A-(epichlorohydrin)], obtain a study area with an ultrafine polished, applied a thin film of carbon on the polished surface, and placing silver conductors to achieve conductivity (see Figure 3).

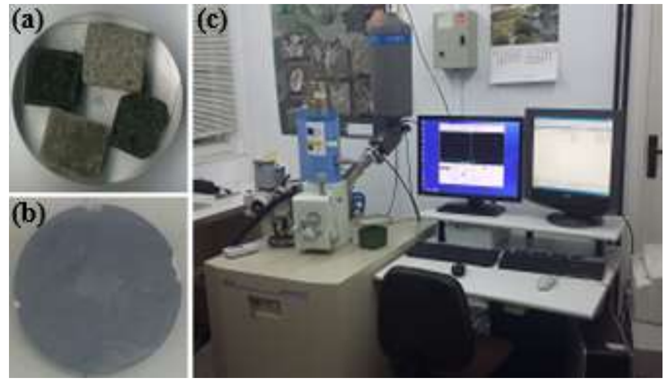

Figure 3. SEM characterization: a) encapsulated and polished; b) surface with carbon; c) test equipment (CCiT of the UB).

Observations made include backscattered electron images (BEC) and secondary (SEI) at 100x, 200x and 500x to obtain the characterization of the transition areas between aggregates and pastes (ITZ). The test constants were: voltage 20kV, 75µA current load, vertical distance from the filament to the polished face 13 nm and a spotsize SS40 size 60.

III. Results

The chemical composition of the FA was determined by x-ray fluorescence (XRF) in order to avoid possible variables that could affect the mechanical behavior of the studied mortars. The results indicated the presence of the majority compounds: SiO_2, Al_2O_3, Fe_2O_3 and CaO (see Table 1), all of them are usual and used for the proposed research.

Table 1. Chemical composition of the FA

Component	Al_2O_3	CaO	Fe_2O_3	K_2O	MgO
%	26.02	4.098	5.551	1.50	0.728
Component	MnO	NaO	SiO_2	SO_3	PPI
%	0.026	0.052	56.79	0.72	2.99

The Brunauer Emmett Teller (BET) technique was used at the FA in order to determine their specific surface, resulting in 1.6 m^2/g, and with an average particle size of 16.52 µm (lower than those of cement –with more reaction capacity–).

A. *Compressive strength*

As representative property of the mortars behavior, the compressive strength of these was evaluated in specimens tested at a constant speed of 50 kg/s to rupture, prior cessation of its curing process (24 hours before the test).

The results obtained (see Table 2) report that at the age of 7 days, the reference mortar outperforms the geopolymer in (3.7 MPa). At the age of 14 days, the trend is more sensitive

(5.64 MPa), cement seems to have more reactive capacity. In addition, is in this age where maximum limit that was capable to reach the geopolymer variable (depletion of their hydraulic capacity). This allows guarantee of a good behavior of the geopolymers:

1. Resistance is a direct function that gives and provides the own gel (sodium aluminosilicate hydrate N-A-S-H, reaction product).

2. The connection between the geopolymeric matrix and adding depend on the ability of dissolution of the FA (ITZ).

In the case of the geopolymer at long ages, the loss of resistance should be explained by the content of alkali present in the solution, which can be reactive with the addition used [11].

Table 2. Mechanical results of samples

Compressive strength			
Sample	Result (MPa)	Sample	Result (MPa)
FAM7	19.35	PCM7	15.65
FAM14	30.06	PCM14	24.42
FAM28	31.33	PCM28	14.51

Finally, at the age of 28 days the gap between the variables of study comes to be such, that in the case of the geopolymers are reported values even below the same variable but at lower ages (abnormal behavior) (see Figure 4). In this last case, the hypothesis that allows its explanation should be discussed from the ability that may have the FA for reacting.

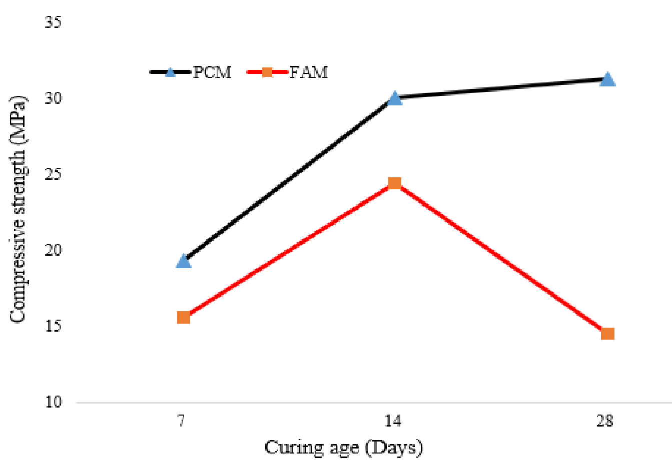

Figure 4. Mortar compressive strength

B. *Elastic Modulus*

The elastic behavior is a property that indicates the ability of a material to be deformed when a mechanical load acts on it.

The dynamic method nondestructive results indicated that PCM can obtain values of the order of the 26.75 Gpa, which places them above the FAM (5.99 Gpa) (see Figure); and for both variables, their respective values do not report significant changes with the passage of curing time studied. Therefore, for this property, as in the case of simple compression, the age of 28 days maintains their abnormal behavior. Finally, the set of variables, it is evident that the FAM will form components less rigid than the PCM, consideration that must be taken in their mechanical applications and services.

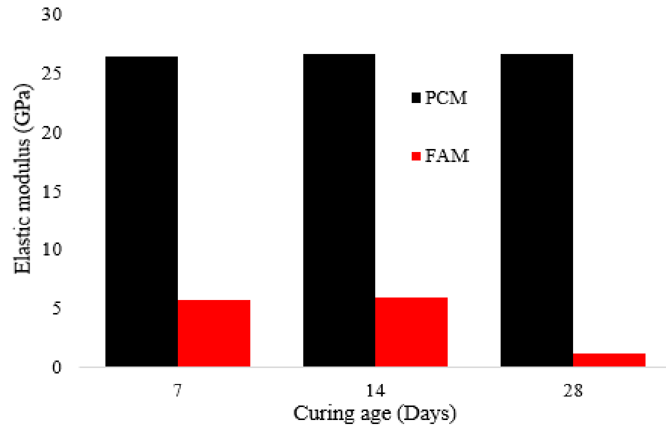

Figure 5. Elastic modulus of mortars

According to the results of the assays to compression and elastic properties, the FAM studied showed abnormal behavior at an age of 28 days. The following SEM images show the morphology of the matrix developed in the process of geopolymerization of the mixtures: Figure 6 presents the composition of a matrix geopolymeric extracted from a previous parallel study with similar specifications of materials and tests; in it, can be distinguished sharply different phases that compose it (aggregate, paste and transition zone), and in what respects to its morphology and reported behavior, this was established as okay and normal in mixtures of this type [12].

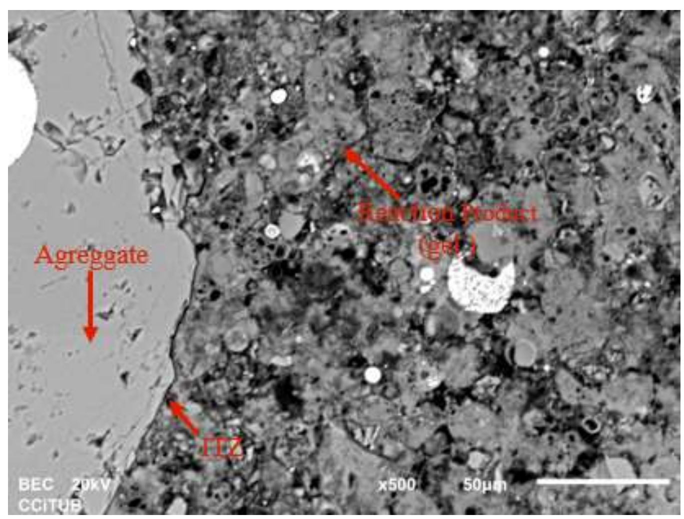

Figure 6. Micrograph of a normal matrix for a mortar base FA.

On the other hand, in Figure 7 study FAM7 variable matrix, shown on it that there is divergence in the morphology of the added component; which comes accompanied by an apparent general cleft of the aggregate, and suggest as a cause a reaction with the alkaline medium which causes decrease of their mechanical properties. However, for short ages it seems that the reaction is dominated by the geopolymerization of the ashes and not for the abnormal reaction of the aggregate with the environment; this is, that the mechanical properties are affected at the beginning, but the process of the geopolymerization is that establishes the mechanical behavior.

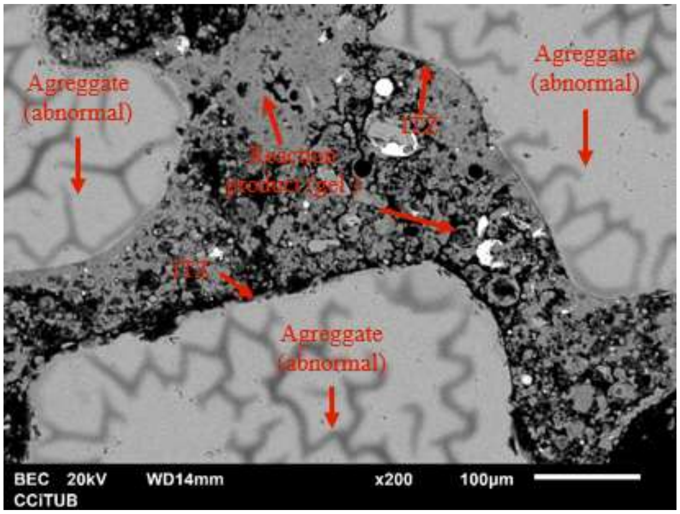

Figure 7. Micrograph of FAM7

Figure 8 shows the micrograph of the FAM28 variable (signals of abnormal behavior); in this you can observe the general composition of the matrix structure, identifying features that govern the scheme and allow understand the mechanical behavior of this:

1 Formation of radial anomalous elements in the outline of the aggregates ("tree branches-shaped" elements).

2 Orientation of the "tree branches" in direction from the outside to the inside.

3 Thickness of similar "tree branches" or constant value in the middle of the "tree branch"; while in the same end zone is reduced or is thinner.

4 Occurrences of the "branches of the tree" with the presence of fissures of the own aggregate.

5 In general terms, the orientation of the "branches of the tree" are perpendicular to the contour of the aggregate.

6 "Clean" areas of impacting areas located in the interior of the aggregates.

This allows mark a conjecture of the FA geopolymerized studied, with increasing curing age, cause that the abnormal reaction becomes in the principal cause of the behavior, to be the cause of the effect abnormal of the mechanical behavior reported.

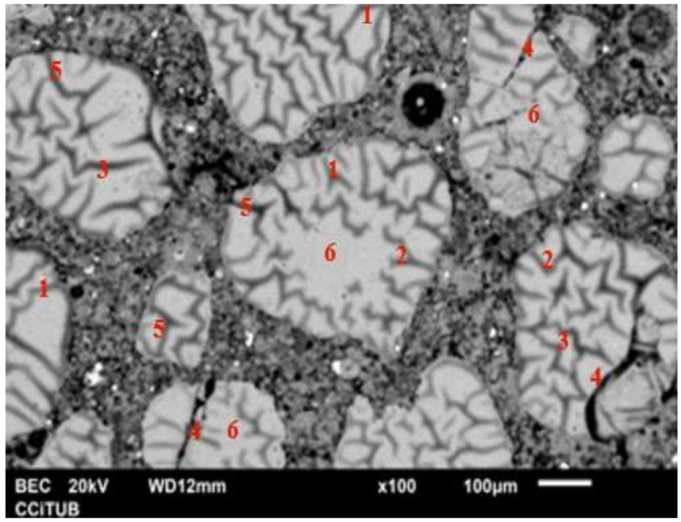

Figure 8. FAM28 micrograph (signs of the behavior of the areas)

IV. Conclusions

The use of FA to develop binding materials through solutions alkaline represents a possible alternative as a replacement of the PC, because these manage to develop resistance to compression similar to the PC.

The geopolymers showed similar mechanical behavior to the PCM to short curing ages, however values were below. In addition, long ages can present an abnormal behavior, due to a possible reaction of the aggregate with the alkaline medium; Therefore is necessary to investigate more on the subject to clarify that behavior.

In general the geopolymers demonstrated to develop mechanic and elastic properties that placed them as one of the main alternative materials to the PC.

Acknowledgment

The authors express thanks to: research project S-01117 from CTT-UPC, to EPSEB-UPC, to the Department CAII-EPSEB-UPC, to FIM-UAS and to the scholarships program for Master Degree studies by CONACYT.

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