

# Temperature for geopolymerization of fly ash. Mechanical behaviour

Sergio Apodaca-García, Susana P. Arredondo-Rea, José M. Gómez-Soberón, Jorge L. Almaral-Sánchez, Ramón Corral-Higuera

**Abstract**— Alkali activation of fly ash (FA) is a chemical process which allows the transformation of the amorphous structure which makes up most of its particles, in compact cementitious skeletons geopolymers are called solid materials usually consist of a alkali hydroxide and solid precursor high content in silicon (Si) and aluminium. This paper presents the effect of temperature and the curing time in the alkali activation in FA pastes with 8M NaOH, geopolymers curing was carried out at room temperature, 60 °C and 80 °C at aged 7 14 and 21 days. Compression tests were conducted to obtain the strength of the geopolymer and analysis by X-ray diffraction to determine the new phases present resulting in higher compression resistance with higher temperature and curing time, a change was detected in the diffractogram because new phases present in the alkaline activation product.

## I. Introduction

The concrete is a material widely used for the development of civil works and building; between its components, cement production has increased in the last decade of the order of 1.5 to 2.2 billion tons, producing for each ton of this material a ton of CO<sub>2</sub> [1-2]. Therefore, the search for new alternative materials and friendly to the environment is a pressing need, these materials have been grouped into so-called supplementary cementitious materials (SCM), among which the fly ash (FA) seem to be able to be used to produce any comparable applications.

Advances in research aimed to achieve production with CV established MCS called geopolymer [3], which consist of main form of aluminosilicates, they tend to form alkali hydroxides from its activation with the precursor base material (CV, silica fume and blast furnace slag) [4-5]

It has been established that the activation process requires temperature [6], and it can be decisive to ensure optimum mechanical product with satisfactory results; however, this has not been established or determined to be regarded as specific and accurate process; therefore, their study is crucial to validate and establish their affectation and involvement in the SCM.

Sergio Apodaca-García, Susana P. Arredondo-Rea, Jorge L. Almaral-Sánchez, Ramón Corral-Higuera.  
Universidad Autónoma de Sinaloa  
México

José M. Gómez-Soberón,  
Universidad Politécnica de Cataluña.  
Spain.  
josemanuel.gomez@upc.edu

## II. Methodology

It was used as materials a FA from coal-fired stem power plant of "José López Portillo" located in Coahuila, Mexico, classified as type F as specified in ASTM C618. FA as activator, was used 8M NaOH. The mixtures were designed with an ash/solution ratio 0.5.

The mixing process for the realization of specimens consisted in put the FA in a plastic container and then add the activator solution, applying a constant manual mixing until obtaining a homogeneous mixture. The result of the mixture was used to prepare cubic samples of 5 cm (Fig. 1a) which were then used for compression tests in accordance with the specifications and ages in the ASTM-C109 mortars standard, but in this case used to pastes; mechanical compression tests were conducted using a press INSTRON universal Units (Fig. 1b) and applying the above standard.

The cured specimens were performed ages 7, 14 and 21 days, three different curing temperatures: ambient, 60 °C and 80 °C (for the last two temperatures, the relative humidity (RH) was 99%). To ensure constant conditions curing, the specimens were coated with plastic wrap and then introduced into bags sealed airtight plastic, to finally be put in sealed aluminium containers filled with distilled water, a condition that lasted until moments before your test.



Figure 1. Experiment performed. a) Processing specimens, b) Specimen ready for stripping, c) Unmold specimens, d) Curing chamber, e) Compression test specimens.

Compression tests indicate the direct state of the geopolymer, while XRD is used for the identification of new phases formed, to observe the development of the initial materials used when they reacted, and to determine if the temperature influences effect favoring the formation of hydrate crystals. For these tests, a PANalyticalX'Pert PRO diffractometer equipped with an X-ray source which uses CuK $\alpha$  radiation was used, the range of 2 $\theta$  used for this study was between 5° and 90°.

### A. COMPRESSIVE STRENGTH

The test procedure presented in Figure 2. In Figure 2a), the computer can be seen prior to the test load, the gantry to load compressive strength applied by a piston on a lower deck which reacts with the upper fixed. The Figure 2b) the provision of pre-loaded specimen is observed. Finally, in Figure 2c), the mode of failure resulting from the specimens studied, which was characterized by narrowing in the central zone of the specimen, forming two cones with bases at the ends of the faces and is presented of inclination 45 ° (plane of maximum shear)

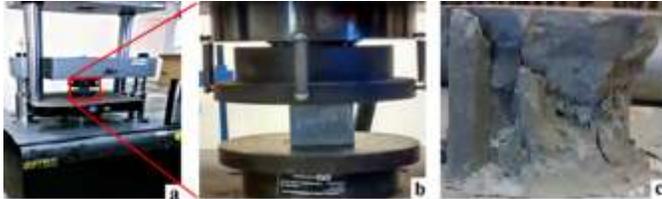


Figure 2. Experiment performed, a) Placement of the specimen in the press, b) Carrying out the experiment, c) Specimen tested.

### B. XRD

In order to identify new crystalline phases present in the diffractogram, product activation and changes the amorphous halo, DRX is used in the base material and the geopolymeric FA pastas. The technique was carried out on a diffractometer equipped with a PANalyticalX'Pert PRO X-ray source CuKa radiation testing was performed in the range 5 ° to 70 ° of 2θ in steps of scanning 0.05s (Figure 3).

For the preparation of the sample a piece of geopolymers tested in compression, which was ground in an agate mortar until it reached the particle size of talc was used; 10 g of this material was placed in the sample holder and then was introduced in the X-ray diffractometer.



Figure 1 Diffractometer PANalyticalX'Pert PRO

## III. Results

The development of the geopolymers depends largely on the type of FA used, its concentration, the type of alkali activator, curing duration, the ratio of materials (silica-aluminium), and possibly curing temperature between other [7].

The mechanical behaviour of geopolymers (particularly compressive strength) reports when something goes wrong to ages at which the gel matrix is created (curing process with different temperatures); therefore the behaviour correlated with this matrix microstructure can be an intuitive hypothesis, and for which the use of XRD can help understand this process, since it allows to discern: the existence in crystalline formation, increasing its peaks, bands and gaps in changes in their crystalline phases delimitation.

### A. COMPRESSIVE STRENGTH

In Figure 3 the compression results obtained are presented for the specimens cured at different temperatures and for ages study. It can be seen that the variables GFA60°C and GFA80°C reach more resistance than the control variable; Moreover, one can also say that the overall behaviour of the variables is evolving over (more marked in the GFA80°C case) time and the variables are more consistent over long ages, that at early ages (anomaly to 7 days GFA60°CRT).

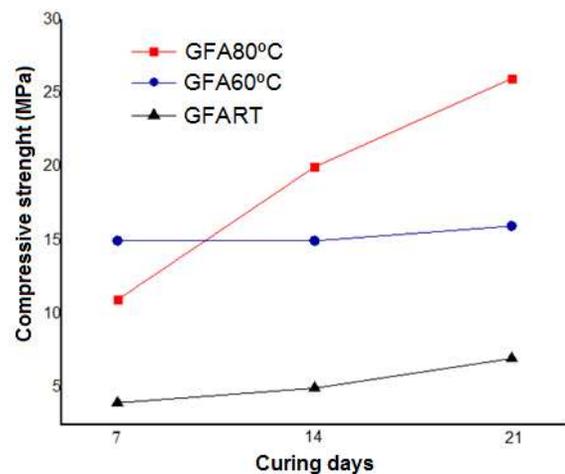


Figura 3. Compressive strength

With respect to the evolution of resistance gains due to the temperature, in Table 1 the percentages of each variable gain representing on the basis of study are presented to GFART; in this, can be see that by the age of 7 days GFA80°C and GFA60°C C have a strength gain of 275% and 375% after 14 days 400% and 300% and at 21 days 371% and 228% respectively.

Table 1. Comparison of resistance with reference to the control sample.

Geopolymers	Results MPa compressive		
	7 days	14 days	21 days
GFA80°C	275%	400%	371%
GFA60°C	375%	300%	228%
GFART	100%	100%	100%

Therefore, it can be inferred that the temperature is critical for the compression behaviour of the geopolymer, and that this effect can be attributed to the reaction kinetic of compounds

which develop cementitious phases in the case of alkali activation—for the increase in temperature— [8]. Thus, it can be said that there is a significant difference between the geopolymers curing with high temperature and those carried out at room temperature, since for this chemical reaction is necessary a temperature of 35 ° C [9].

## B. XRD

The crystalline phases were identified using XRD technique for pasta to ages of 7 and 21 days (see Figure 4); also include diffractogram without activating the CV as a reference or change identification thereof. As shown, all variables studied presented in the diffractograms, that with respect to the reference, reported noticeable changes. As differentiating parameters, shown:

1. Halo present (zone A) of the FA is 18 ° to 30 ° 2 θ and the geopolymer about 23 ° is traversed at 40 ° 2 θ as shown in Area C (imputed these structural changes suffering the material forming the aluminosilicate network).
2. For Geopolymers it present an intensity between 5 ° and 14 ° 2 θ (zone B) is when the reaction takes, is attributed to the formation of the crystal lattice of the crystalline phase attributed to gel.

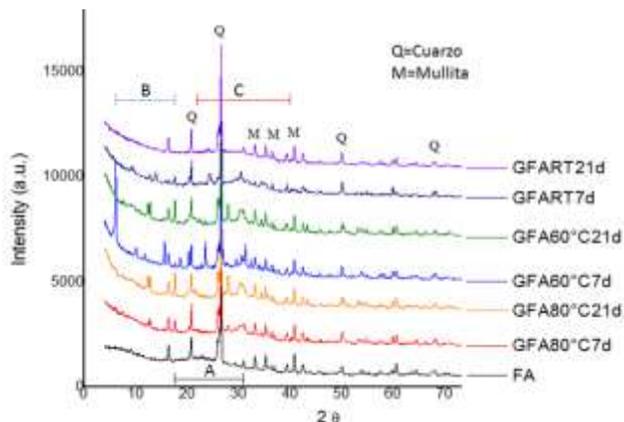


Figure 4. Geopolymers diffractogram.

## IV. Conclusions

The Results of mechanical pastes of CV demonstrate that polymers with higher temperature and curing time increase the mechanical strength due to alkali activation that develops with more effective.

Besides time and temperature, relative humidity conditions in the initial curing processes also have an important role in the manufacture of pasta de FA activated at the beginning. If moisture conditions are not adequate affects the development of the alkali activation and not is performed properly.

The research conducted allowed to validate the feasibility of alkali activation at room temperature of the FA, being indispensable to the creation of geopolymer matrix is present the decisive factor of the increase in temperature.

## 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, CIMAV-Chihuahua and to the scholarships program for Master Degree studies by CONACYT.

## References

- [1] Pacheco, J., Fajardo, G., Valdez, P., & Badillo, A. (2007). El rol de las puzolanas naturales en el mejoramiento de la durabilidad del concreto reforzado. (The role of natural pozzolans in improving the durability of reinforced concrete) ciencia fic.
- [2] Hanehara, S., Tomosawa, F., Kobayakawa, M., & Hwang, K. (2001). Effects of water/powder ratio, mixing ratio of fly ash, and curing temperature on pozzolanic reaction of fly ash in cement paste. *Cement and Concrete Research*, 31(1), 31-39.
- [3] Provis, J. L., & Van Deventer, J. S. J. (Eds.). (2009). *Geopolymers: structures, processing, properties and industrial applications*. Elsevier.
- [4] Pacheco-Torgal, F., Castro-Gomes, J., & Jalali, S. (2008). Alkali-activated binders: a review. Part 2. About materials and binders manufacture. *Construction and Building Materials*, 22(7), 1315-1322.
- [5] Van Jaarsveld, J. G. S., Van Deventer, J. S. J., & Lukey, G. C. (2002). The effect of composition and temperature on the properties of fly ash-and kaolinite-based geopolymers. *Chemical Engineering Journal*, 89(1), 63-73.
- [6] Krivenko, P. V., & Kovalchuk, G. Y. (2002, August). Fly ash based zeolite cements. In *Innovations and Developments in Concrete Materials and Construction: Proceed. Intern. Conf. "Challenges of Concrete Construction"*, Dundee (pp. 123-132).
- [7] Joseph, B., & Mathew, G. (2012). Influence of aggregate content on the behaviour of fly ash based geopolymer concrete. *Scientia Iranica*, 19(5), 1188-1194.
- [8] Palomo, A., Grutzeck, M. W., & Blanco, M. T. (1999). Alkali-activated fly ashes: a cement for the future. *Cement and concrete research*, 29(8), 1323-1329.
- [9] Chithiraputhiran, S., & Neithalath, N. (2013). Isothermal reaction kinetics and temperature dependence of alkali activation of slag, fly ash and their blends. *Construction and Building Materials*, 45, 233-242.



**S. Apodaca-García**

**Title:** Civil engineer and candidate to Master in Materials at Autonomy University of Sinaloa, Mexico.

**Research areas:** Concrete Technology, sustainable concretes characterization techniques applied to pastes, mortars and concretes, and sustainability in construction



**S. P. Arredondo-Rea.**

**Title:** PhD in Materials Science Research Center for Advanced Materials, Mexico.

**Research areas:** Concrete Technology, sustainable concretes characterization techniques applied to pastes, mortars and concretes, and sustainability in construction



**J. M. Gómez-Soberón.**

**Title:** PhD from the Technical University of Catalonia.

**Research areas:** Concrete recycled, porous structure of concrete, sustainable construction, minimization, reuse and recycling in construction, ICT in teaching, virtual campus teachers.



**J. L. Almaral-Sánchez.**

**Title:** PhD in Science (Materials), CINVESTAV-Querétaro, Mexico.

**Research areas:** Properties and modification of materials, inorganic-organic hybrid materials, zeolites, nanotechnology, sustainable materials and steel corrosion.



**R. Corral-Higuera.**

**Title:** PhD in Science (Materials), CIMAV-Chihuahua, Mexico.

**Research areas:** Concrete Technology, sustainable concretes characterization techniques applied to pastes, mortars and concretes, and sustainability in construction.