

ENGINEERING PROPERTIES OF GEOPOLYMER CONCRETE BASED ON ALKALI ACTIVATED NATURAL POZZOLAN

D. Bondar¹, C.J. Lynsdale², N. Milestone³, N. Hassani⁴, A.A. Ramezaniapour⁵

¹Ph.D. candidate of Sheffield University and P.W.U.T. Research Associate

²Dept. of Civil and Structural Engineering, (c) Dept. of Material Engineering University of Sheffield,
Mappin Street, Sheffield, S1 3JD, UK

³Research Center of Natural Disasters in Industry, P.W.U.T., Tehran, Iran

⁴Dept. of Civil Engineering, Amir Kabir University, Tehran, Iran

ABSTRACT

The development of alkali activated binders with superior engineering properties and longer durability have emerged as an alternative to OPC. It is possible to use alkali-activated natural pozzolans to synthesize environmental friendly sound geopolymeric cementitious construction materials. The main benefit of geopolymeric cement is the reduction in environmental impact in harmony with the concept of sustainable development. This paper presents a comprehensive summary of the extensive studies conducted on the results of experimental investigations on the engineering properties of geo-polymer concrete using activated Iranian natural pozzolan namely, Taftan. Experimental work was conducted to determine mechanical strength; modulus of elasticity; ultrasonic pulse velocity and shrinkage of different concrete mixtures. Test data are used to identify the effects of salient factors such as water to binder ratios and curing conditions that influence the properties of the geopolymer concrete. The results show that mortar and concrete made with alkali activated natural pozzolan develop moderate to high mechanical strength and modulus of elasticity and shrink much less than ordinary Portland cement (OPC) concrete.

Keywords: alkali-activated binder; geopolymeric cement; natural pozzolan

1. INTRODUCTION

Portland cement concrete is a major construction material worldwide use is said to be second only to water. Unfortunately, the production of Portland cement releases large amount of CO₂ in to the atmosphere. This gas is a major contribution to the greenhouse effect and the global warming of the planet. To reduce greenhouse gas emissions, efforts are needed to develop environmentally friendly construction materials. Unlike with regular concrete the chemical reactions that form geopolymer concrete alternative do not give off carbon dioxide or require high temperatures, which also lead to CO₂ emissions.

In geopolymer concrete, the geopolymer paste serves to bind the coarse and fine aggregates, and any un-reacted material. Geopolymer concrete can be utilized to manufacture pre-cast concrete, structural and non-structural elements, to make



concrete pavements, immobilize toxic waste, and produce concrete products that are resistant to heat and aggressive environments [5, 6].

This paper presents the technology of making geopolymer concrete using natural pozzolan as its source material and presents the results of laboratory tests conducted on this material. The research data presented in this paper are useful to understand the engineering properties of geopolymer concrete.

2. PREVIOUS RESEARCH ON GEOPOLYMER MATERIAL

The term “geopolymer” describing a family of mineral binders those have a polymeric silicon-oxygen-aluminum framework structure. The mechanism of geopolymerisation may consist of dissolution, transportation or orientation, and polycondensation [15], and takes place through an exothermic process [3, 8].

Different pathways for preparation of a synthetic geopolymer, including the order of addition of the raw materials, show different evolutions of compressive strength of the materials. The best method is to prepare an alkaline solution (mixing MOH and water and stirring for 2 minutes), adding pozzolan to alkaline solution for 15 minutes in a mixer, followed by sodium silicate, and mixing for 15 minutes [10].

The geopolymer paste serves to bind the coarse and fine aggregates and any unreacted material to prepare geopolymer concrete [5, 6].

The nature of the fresh geopolymer concrete is stiff paste with high viscosity hence it tends to have low workability [5, 6].

A geopolymer mix can be timed to set either fast or slow, by adjusting the mixture components. Depending on the synthesis conditions, structural integrity and reasonable strength were attained in a short time, sometimes in as little as sixty minutes [14]. With the use of granulated blast furnace slag as the source material with the addition of metakaolinite, Cheng and Chiu (2003) found that the setting time of geopolymer paste was affected by curing temperature, type of alkaline activator and the composition of source material. They stated that the setting time of above geopolymer paste was between 15 to 45 minutes at 60°C. The laboratory experience by Hardjito, Wallah, Sumajouw and Rangan (2004) showed that the fresh geopolymer concrete could be handled for at least 120 minutes after mixing, without any sign of setting and degradation in compressive strength. These have mostly depended on the compounds in the source material, for instance the higher the content of CaO, the faster the setting. The presence of compounds other than Al_2O_3 and SiO_2 in the source material may also delay the setting. The pfa based geopolymers show faster initial setting time at higher temperatures and the final setting of these mortars occur from 15 to 25 minutes after the initial setting [7].

There are many different views as to which main parameters affect the compressive strength and other mechanical properties of geopolymer concrete. Palomo et al. (1999) stated that the significant factors affecting the compressive strength are the type of alkaline activator, the curing temperature and the curing time [5, 6]. Other researchers have reported that the important parameters for satisfactory polymerization are the relative amounts of Si, Al, K, Na, molar ratio of Si to Al in solution, the ratio of alumina silicate mineral to kaolinite, type of alkaline activator, water content, and curing temperature [1, 12, and 15]. The presence of silicate ions



in the aqueous substantially improves the mechanical strength and modulus of elasticity values but has a slightly adverse effect on the otherwise very strong matrix/aggregate and matrix/steel bond [4]. Experimental results show that the H_2O/M_2O molar ratio in the mixture composition is a significant parameter affecting the compressive strength of fly ash based geopolymer concrete, whereas the influence of the Na_2O/SiO_2 molar ratio is less significant. An increase of the H_2O/M_2O molar ratio and water to geopolymer solids ratio decreases the compressive strength of geopolymer. In addition, Van Jaarsveld et al. (2002) found that curing at elevated temperatures for long periods of time may weaken the structure of hardened material. The research on fly ash-based geopolymer binder, Palomo, Grutzeck, and Blanco (1999) have confirmed that curing temperature and curing time significantly influenced the compressive strength but seems not to be same for different alumina silicate. Longer curing time and higher curing temperature increased the compressive strength in fly ash based geopolymer concrete, although the increase in strength may not be significant for curing at more than $60^\circ C$ and for periods longer than 48 hours [5, 6]. In most cases 70% of the final compressive strength is developed in the first 4 hours of setting. Because the chemical reaction of the geopolymer paste is a fast polymerization process, the compressive strength does not vary with the age of concrete, after it has been cured for 24h. This observation is in contrast to the well-known behaviour of OPC concrete, where the hydration process extends over a long period and hence strength increases over time [5, 6]. Another Kinetic difference between the Portland and alkaline systems is the existence of a relatively low threshold temperature in the former, above which thermal curing can have an adverse effect on the mechanical development and even on material durability. In an activated ash, on the contrary, a suitable choice of reaction time and curing temperature can yield different reaction product without detracting from material durability, because according to Fernandez, Palomo, and Hombradoz (2006) increases in the curing temperature go hand-in-hand with decreases in the amount of Al incorporated into the final product and a concomitant improvement in mechanical properties. Such improvements parallel the formation of a homogeneous alumina-silicate matrix [3].

When alkali-activated slag cement concrete is cured in water, compressive strength of the concrete keeps increasing until 365 days. However, if the concrete is cured in sealed condition the strength stopped increasing at about 90 days. This may be attributed to the lack of moisture available for the hydration of slag inside the concrete. The concrete exposed to air exhibit the lowest strength all the time and strength retrogression at ages greater than 28 days. The strength reaches a maximum after 14 to 28 days of hydration, and then starts to decrease [13].

Puertas et al. (2003) reported that the elastic modulus of OPC mortars was 5679 MPa, also higher than the values for activated PFA mortars (4441 MPa).

Fernandez-Jimenez et al. (2006) found that the addition of soluble silicates in the alkali solution improves the modulus of elasticity. However, this improvement was not sufficient and the alkali activated PFA concrete showed a much lower static modulus of elasticity than expected. The values presented for OPC concrete ranged



from 30.3 to 32.3 GPa while for geopolymeric concrete ranged from 10.7 (without silicate) to 18.4 GPa (with silicate). Hardjito et al. (2004) observed better elastic modulus results for a concrete samples made in similar conditions: 22.95 to 30.84 GPa.

Geopolymers also attain shrink much less on setting (for 7days 0.2% & for 28days 0.5% of OPC) [14]. The explanation for this behaviour is to be found in the micro structural characteristic of the new binder and the main reaction product of the alkali activation of fly ash which causes a zeolite-type phase. Zeolite properties and microstructure are widely known to be unaffected by the loss of the water incorporated during their synthesis because not only water loss is reversible in most zeolites but also they are able to absorb water from the humidity in atmosphere [4].

3. EXPERIMENTAL WORK

3.1. Material and Mixing Procedure

In this research, Taftan andesite was selected as the most reactive natural pozzolan in Iran, used to produce Portland pozzolan cement by Khash Cement Factory. The chemical composition was analysed by XRF and presented on Table 1.

Potassium hydroxide was used as pellets to produce the alkaline solution for geopolymeric concrete production. It was a 98% pure KOH supplied by MERK International Ltd.

Sodium silicate was also provided by Iran Silicate Industrial Company in the form of granules, powder and solution (water glass). The chemical composition of the solution provided by the manufacture was:

8.5% of sodium oxide (Na_2O), 26.5% of silicon oxide (SiO_2) and 65% of water. Aggregate used in this study was obtained from deposits of Karaj River in northwest of Iran comprising 14mm and 4.75mm coarse aggregates and fine sand. The fineness modulus of combined aggregates was 2.08.

The proportioning of the concrete mixture was based on the BRE method targeting a 40 MPa (28 days) compressive strength and a slump of 70 mm. Then, the amount of cement was substituted with the same quantity of natural pozzolan and the amount of water was ignored since there was already water in the alkali solution. The BRE method was used only to decide what is a common ratio of binder, sand and coarse aggregates, it was not expected that the actual 28 days compressive strength or the slump would be in accordance with the values designed. The details of the different mixes are presented in Table 2 and the notation for the mixes is as follows:

CM1: PC control mix with $w/c=0.45$

CM2: PC control mix with $w/c=0.55$

ATAF1: Activated Taftan pozzolan with $w/c=0.45$

ATAF2: Activated Taftan pozzolan with $w/c=0.55$

The mixing of the geopolymeric concrete was carried out in two different mixers sequent. The paste was prepared in a Hobart mixer (2 litre capacity) and added to a horizontal pan mixer (20 litre capacity) which contains the aggregates.

Taftan specimens were de-moulded 24 hours after casting. Then they were cured in two curing regimes and at three different temperatures:



1. Sealed curing: Three series of specimens were sealed wrapped in a special plastic covering which was tested to be impermeable and stored in a controlled room kept at three different temperature equal to 20 ± 2 , 40 ± 2 and $60\pm 2^\circ\text{C}$.

Table 1: Chemical composition (oxide percent) of the materials used in this investigation reported by Kansaran Binaloud X-ray laboratory in Tehran, Iran (2005-2006)

Material	LOI	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	TiO ₂	K ₂ O	Na ₂ O
Taftan andesite	1.85	61.67	15.90	4.32	7.99	2.04	0.438	2.12	3.21

Table 2: Concrete Mix Proportion

Mix No.	Natural Pozzolan (Kg/M ³)	OPC (Kg/M ³)	Alkaline Hydroxide (Kg/M ³)	Water Glass (CC/M ³)	Water (Kg/M ³)	Total Water (Kg/M ³)	Total Binder (Kg/M ³)	Fine Agg. (Kg/M ³)	Coarse Agg. (Kg/M ³)	W/B%
CM1	—	403	—	—	180	181.3	403	577.82	1227.88	0.45
CM2	—	357	—	—	195	196.35	357	702	1119.65	0.55
ATAF1	391	—	66.27	34.18	157.78	180	403	578.17	1228.8	0.45
ATAF2	344	—	71.82	36.9	171	195	357	702	1121	0.55

2. Steam curing: Three series of the specimens were put in the steam curing chamber set at three different temperatures equal to 20 ± 2 , 40 ± 2 and $60\pm 2^\circ\text{C}$ for measuring compressive and splitting tensile strength and one series was put at $40\pm 2^\circ\text{C}$ for other measurements.

3.2. Testing Procedures

In order to determine the compressive strength of geopolymeric concrete each of the subsequent mixtures were prepared in 100x100x100 mm cubes and the compressive strength for these samples were tested according to BS EN 12390-3:2000. Details of casting and curing are described in section 3.1. Three samples of each condition were tested for 1, 7, 14, 28, 90, 180 and 365 days, and the average compressive strength values reported as the results.

PUNDIT was used to measure the ultrasonic pulse velocity in accordance with BS 1881: part 203: 1986. The measurement was conducted on the 100mmx100mm end face of prism with a length of 500mm. Duplicate sets of samples were tested at 28, 90, 180 days. For measuring ultrasonic pulse velocity, a pulse of longitudinal vibration is produced by means of 54 kHz an electro acoustical transducer of 50mm diameter and picked out by another transducer after travelling a known path length.

The splitting tensile strength of all mixes was measured using 100mm Φ x 200mm length cylinders. The samples were prepared and splitting tensile tests performed as described in BS EN 12390-6:2000. The specimens were tested in duplicate sets at 7, 14, 28, 90, 180 and the average results are reported.

The static modulus of elasticity is determined according to BS1881-121:1983



standard by subjecting a 100mm Φ x200mm cylinder specimen to uni-axial compression and measuring the deformation by means of dial gauges fixed between certain gauge lengths. Dial gauge reading divided by gauge length will give the strain while load applied divided by area of cross section will give the stress. A series of readings are taken and the stress-strain relationship is established. The modulus of elasticity so found out from actual loading is called static modulus of elasticity.

In the present work the changes in length of 75x75x280mm concrete prisms were measured by commonly used mechanical equipment. Predrilled metal studs were fixed to either end of the concrete specimen with the adhesive at a preset spacing with the aid of a standard calibration bar. The distance between each two pins located into the stud holes was measured by an accuracy of about 0.0025 mm at certain times. One end of the reference rod was designed as the top and it was kept uppermost during all measurements. The prisms were placed in the apparatus with the marked end uppermost and were rotated slowly around the contact surfaces to measure the changes in length. For each mix two specimens were cast and cured for 3 and 7 days. The prisms were then left in a room controlled at 20°C and 70% humidity room and chemical shrinkage was measured using the length comparator in accordance with BS 812: Part 120: 1989.

4. RESULTS AND DISCUSSION

4.1. Compressive Strength

The results of the compressive strength tests on geopolymeric concrete using activated natural pozzolan and control Portland cement concrete mixes are presented in Figures 1.

In all cases, the strength of the concretes increased with age. The rate of strength gain is high at early ages and gradually decreases at longer ages. Geopolymeric concrete mixes mostly showed lower strengths than OPC control mixes at early ages, but they reached the same and even higher strengths than OPC mixes after long-term aging. ATAF1 have the highest compressive strength equal to 43.5 MPa. While ATAF2 mix has resulted compressive strength equal to 39.1 MPa after 365 days which is close to the amount resulted for OPC control mix.

It is well known that the lack of curing greatly affect the strength development. Figure 1 clearly shows the effect of different curing temperatures in two conditions of curing: sealed and steam curing. It generally sealed condition gives the best results in long term the same as OPC control concrete although the difference between the two conditions is not significant.

The results suggest that the optimum temperature for curing alkali-activated Taftan pozzolan is 60°C at early ages but curing at 40°C under sealed conditions gave the highest strength results in the long-term.

An increase of the water to binder ratio decreases the compressive strength of geopolymer concrete significantly (Figure 2).

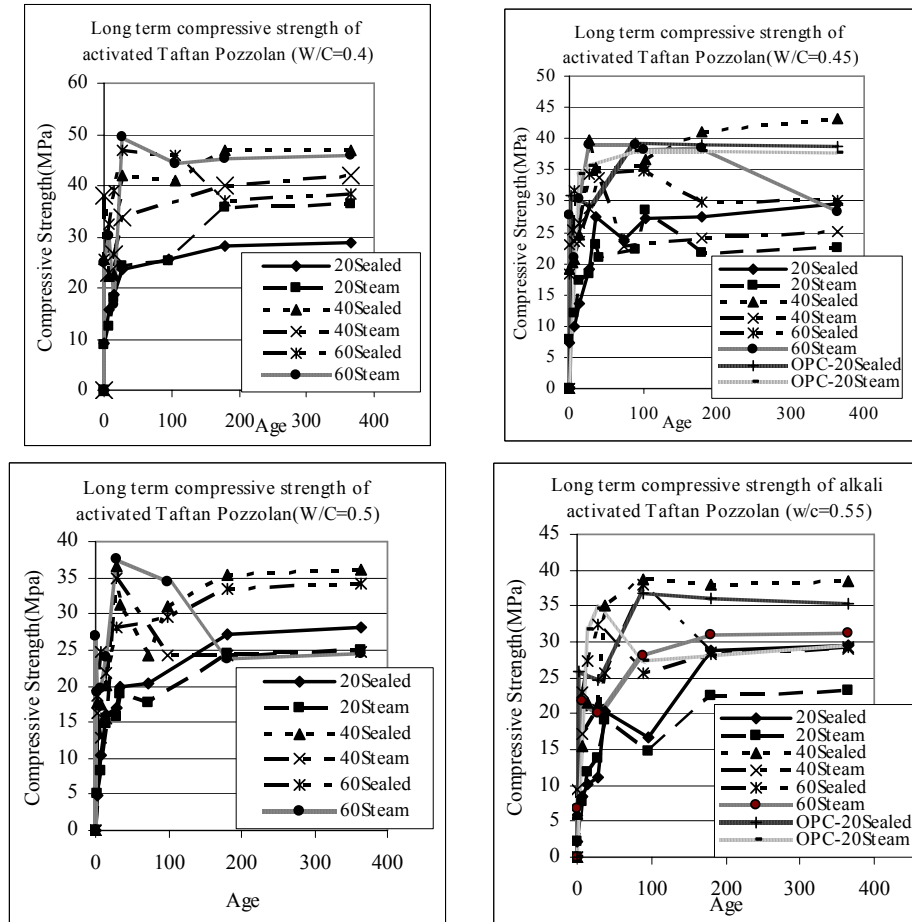


Figure 1. Effect of different curing condition and curing temperature on compressive strength development for activated Taftan pozzolan with different water to binder ratio

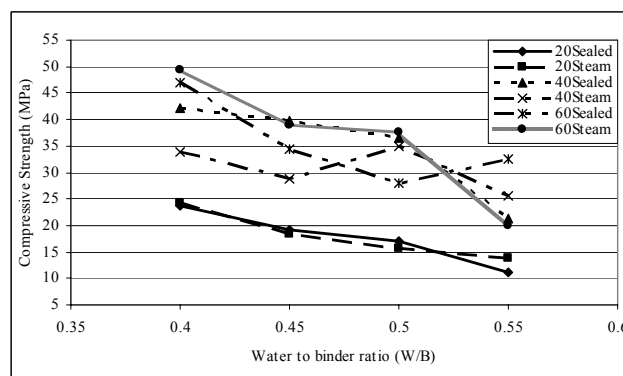


Figure 2. Compressive strength at 28 days versus water to binder ratio (W/B) for alkali activated Taftan pozzolan at different curing temperature



4.2. Ultrasonic Pulse Velocity

Figure 3 shows the results for the ultrasonic pulse velocity test for all mixes. The Figure shows that ATAF1 achieved the highest values followed by ATAF2. Tabular form of the results is shown in Table 3. Comparing the results with Table 4, which gives the pulse velocity rating as suggested by Central Water and Power research Station, Khadakwasla (India), presents lower velocity corresponding to the same compressive strength. It seems that in geo-polymeric concrete due to its lower density the velocity of pulses are lower than OPC concrete.

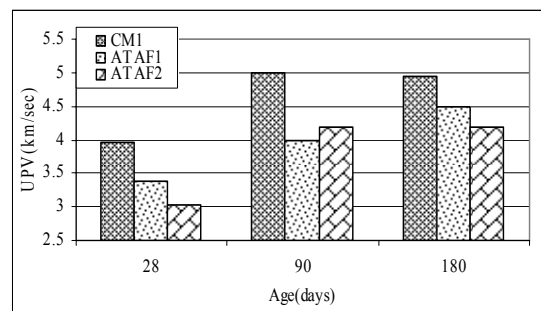


Figure 3. Ultrasonic Pulse Velocity for different mixes

Table 3: The pulse velocity and the corresponding compressive strength of different mixes

Mixes	Age(days)	Velocity(km/sec)	Compressive strength(MPa)
CM1	28	3.95	29.8
	90	5.0	39.23
	180	4.95	39
CM2	28	3.5	24.78
	90	4.7	36.78
	180	4.6	36
ATAF1	28	3.39	39.7
	90	4.0	35.6
	180	4.5	40.97
ATAF2	28	3.03	21.36
	90	4.2	38.72
	180	4.2	38.06

Table 4: Quality Criteria Suggested by Central Water and Power Research Station Khadakwasla (India)

Velocity (km/sec)	Classification (Quality)	Compressive strength(Kg/cm ²)
4.0 and above	Very good	300 to 350
3.5 to 4.0	Good	250 to 300
3.0 to 3.5	Medium	200 to 250
3.0 and below	Poor	150 to 200



4.3. Indirect Tensile Strength

The results of the indirect tensile strength tests up to 180 days are shown in Figure 4. The trend in tensile strength is similar to that obtained for compressive strength. Tensile strength increases as time proceeds. Figure 6 illustrates a difference development in tensile strength of different mixes. As far as the geopolymeric concrete mixes based on activated natural pozzolan are concerned, higher strength was observed at longer ages in comparison with control mixes. At early age, ATAF2 shows lower tensile strength results than OPC control mix. Although, the tensile strength of ATAF1 geopolymeric concrete mix is 3.57 MPa after 28 days and higher than the OPC control mix. The results of long term tensile strength show that Taftan geopolymeric concrete mixes have higher tensile strength than OPC control mix equal to 3.69 and 3.0 MPa after 365 days.

The tensile strength of this type of concrete as compared to its compressive strength, is more sensitive to improper curing, the same as OPC concrete. Figure 4 illustrates the effect of curing conditions and temperatures on tensile strength of concrete based on activated natural pozzolans. The optimum temperature of curing is 40°C, the same as that found for compressive strength.

Figure 4 shows that the higher water to binder ratio results in lower tensile strength, same as OPC mixes.

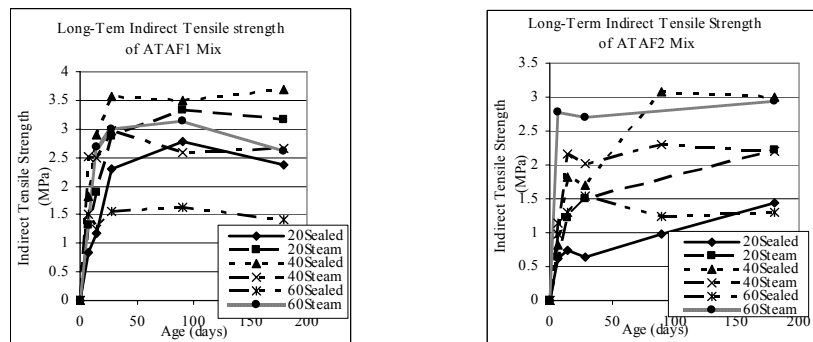


Figure 4. Effect of different curing condition and curing temperature on Indirect Tensile strength development for ATAF1 and ATAF2 mixes

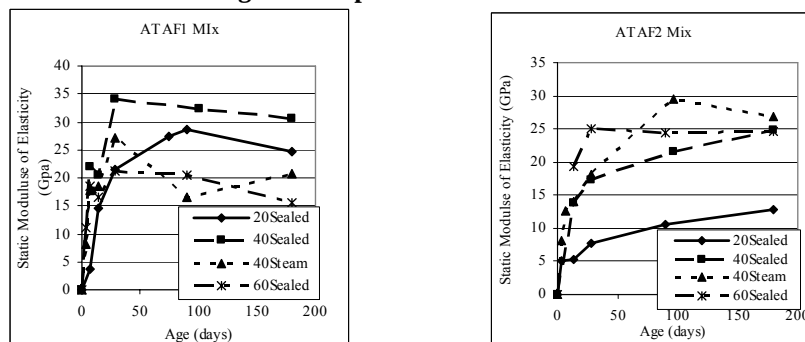


Figure 5. Effect of different curing condition and curing temperature on Static Modulus of Elasticity development for ATAF1 and ATAF2 mixes



4.4. Static Modulus of Elasticity

Results of the static modulus of elasticity are shown in Figure 5. Similar to the compressive strength results of the mixes, static modulus of elasticity increases with age. This improvement is fast in the first 28 days as the most of the modulus value is generally achieved in this period. During the first 14 days the mixes made with activated natural pozzolans have mostly shown lower values of static modulus of elasticity than OPC concrete mixes, except ATAF1 mix. The static modulus of elasticity for ATAF1, ATAF2 mixes after 14 days is 33.96, 14.033GPa, respectively with that for the CM1 mix is 26.55GPa. Long term results show that the static elastic modulus of some of alkali activated natural pozzolans such as ATAF1 are around 5% to 20% more than OPC mixes. The long term static modulus of elasticity of ATAF1, ATAF2 mixes were resulted at 32.664, 26.805GPa in compare with OPC concrete mixes which resulted 29GPa.

The elastic modulus was affected by the curing temperatures and conditions. Early age static modulus of elasticity increases with increasing the curing temperature to a limit which seems to be related to the water to binder ratio. For ATAF1 with water to binder ratio equal to 0.45, the elastic modulus increases with increasing curing temperature up to 40°C and then decreases when the curing temperature rises up to 60°C. This optimum temperature to achieve higher static modulus of elasticity raises to 60°C for ATAF2 mixes with water to binder ratio equal to 0.55. However, the long term results drop to the same amount resulted for the mix cured at 40°C.

4.5. Drying Shrinkage

The shrinkage time curves are shown in Figures 6. From this investigation the following observations are made:

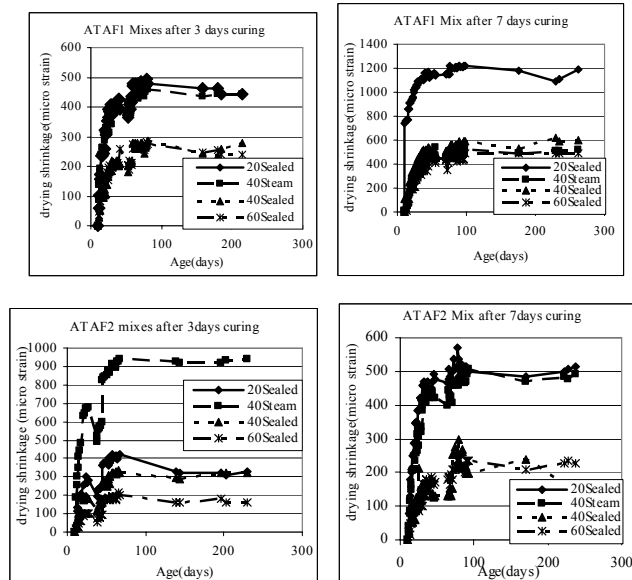


Figure 6. Effect of length, temperature and condition of curing on drying shrinkage development with age for ATAF1 and ATAF2 mixes



- 1) The graphs show that the magnitude of shrinkage increases with time and the rate of shrinkage decreases rapidly with time. The rate of shrinkage in Taftan pozzolan mixture was similar but not as rapid as the rate of development of strength and, seems to be constant after 60 days.
- 2) In OPC concrete one of the important factor which influences the magnitude of shrinkage is water to cement ratio of concrete and the values of shrinkage increases with the increasing of this ratio. The results indicate that the total water to binder ratio has a significant effect on the shrinkage properties of geopolymer concretes as well and seems to be contrary to the behaviour of OPC concrete, where the higher the water to binder ratio lowers the amount of drying shrinkage resulted. The maximum amount of final drying shrinkage for ATAF2 mix observed is 43% of that for ATAF1. The shrinkage of ATAF1 and ATAF2 mixes at 180 days at same curing conditions were 1185×10^{-6} and 514×10^{-6} , respectively.
- 3) The results show that at a given water to binder ratio, the drying shrinkage at all ages varied with different curing regimes and temperatures. In concrete based on alkali activated natural pozzolan, the higher the curing temperature the lower the amount of drying shrinkage resulted. The lowest amount of drying shrinkage for different curing temperatures correspond to ATAF1 and ATAF2 mixes was 239×10^{-6} and 161×10^{-6} , respectively and achieved for mixes cured at 60°C. It can be observed that steam curing shows higher amount of drying shrinkage. This phenomenon may be related to the pozzolan nature minerals and because of swelling properties of its minerals during absorbing moisture. When the samples are subjected to wetting condition, they start swelling. Swelling is due to the adsorption of water by the natural minerals in pozzolan gel. The water molecules act against the cohesive force and tend to force the gel particles further apart as a result of which swelling takes place. In addition, the ingress of water decreases the surface tension of the gel. The property of swelling when placed in wet condition, and shrinking when placed in drying condition. While in OPC concrete the magnitude of shrinkage is less sensitive to moisture movement in concrete.
- 4) The length of curing affects the amount of drying shrinkage as well. The specimens cured for a period of three days seem to absorb environmental humidity similar to zeolites but after 7days the property of water absorption reduces. Thus, the former show lower amount of shrinkage than the latter. In calcined Shahindej, this phenomenon is not observed.

5. CONCLUSION

The main conclusions drawn from the investigation of engineering properties of geopolymeric concrete based on activating natural pozzolans (i.e. alkali activated natural pozzolan or AANP) are summarized as follows:

- 1) Geopolymeric concrete mixes based on activated natural pozzolans have mostly shown lower strength than OPC mixes at early ages, but they have reached the same and even higher strength than OPC mixes after long-term.
- 2) It seems that the ultrasonic pulse velocity in the geopolymeric concrete due to



its inherent property caused lower density is lower than in OPC concrete having the same compressive strength.

- 3) During the first 14 days the mixes made with activated natural pozzolans have mostly shown lower values of static modulus of elasticity than OPC concrete mixes. However, long term results show that the static elastic modulus of alkali activated natural pozzolans concrete is generally around 5 to 20% more than OPC mixes.
- 4) The elastic modulus of AANP concrete was affected by the curing temperatures. Early age static modulus of elasticity increases with increasing the curing temperature to a limit which seems to be related to the water to binder ratio. That means if there be the lack of water due to its evaporation at higher temperature before the full strength is gained the static modulus of elasticity decreases at higher temperatures.
- 5) The AANP concrete mixes may exhibit lower drying shrinkage in comparison with the OPC mixes at the same water to binder and cement to aggregate ratio.
- 6) The results indicate that the total water to binder ratio have a significant effect on the shrinkage properties of geopolymer concretes and seems to behave differently to OPC concrete in this respect. Where, higher the water to binder ratio for geopolymer concrete the lower is the amount of drying shrinkage.
- 7) In concrete made with alkali activated natural pozzolan, the higher the curing temperature, the lower the amount of drying shrinkage resulted.
- 8) It can be observed that steam curing shows higher amount of drying shrinkage. This phenomenon may be related to the pozzolan natural minerals and because of swelling properties of its minerals during absorbing moisture.

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