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NaOH alkali-activated class F fly ash: NaOH molarity, Curing conditions and mass ratio effect

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Abstract
Alkali-activated fly ash has been prepared by mixing Moroccan fly ash (ASTM class F) and sodium hydroxide (purity, 97%). The samples were cured at two different curing conditions (80 and 100°C) for 4 days in different experimental conditions. XRD analysis and laser granulometry were used to characterize fly ash. Microstructure of paste and and their compressive strength were investigated. The results reveled that compressive strength of geopolymers depends on NaOH concentration, curing conditions, and also fly ash to sodium hydroxide mass ratio. Compressive strength of 30 MPa was obtained when the mixture was prepared with 14M NaOH, fly ash to sodium hydroxide ratio of 1.4, and cured at 80°C.

Key words: geopolymers, alkaline activations, class F fly ash, compressive strength

1. Introduction
Producing cement required a lot of energy (from 750 to 1450°C to produce clinker), and rejecting large quantities of CO_2 in the atmosphere. The production of cement in 2005 accounted for around 7% of CO_2 world emission[1]. Reduction in the use of Portland cement must reduce its negative environmental impact. Geoplymers are an effective solution to replace Portland cements.
Fly ash is a by-product form thermal power stations are using pulverized coal to produce electricity.
Accorded to its pozzolanic properties, fly ash can be used as material source to produce geopolymers. However, to make geopoolymers a source of alumino-silicate material(fly ash, slag...) must be activated with high alkali solutions[2]. Combinations of sodium hydroxide solution (NaOH) and sodium silicate solution (Na_2SiO_3) with different Na_2SiO_3/NaOH mass ratios are the most used[3,4]. Geopolymer can be made at different curing conditions. At ambient temperature of around 25 °C, the fly ash geopolymer gains strength slowly[5].To obtain reasonable strength fly ash geopolymers, temperature curing at 40–75°C is normally required[6].

2. Materials details
2.1. Materials
Fly ash was obtained from Jorf Lasfar power station plant in Morocco. The physical properties and the chemical composition of fly ash are given in Table1 and Table2, respectively. Sodium hydroxide pellet was dissolved in deionised water to obtain NaOH solution. To investigate the effects of NaOH concentration on geopolymer paste samples, concentrations of 10, 12, and 14M were used. To avoid silica contamination polyethylene molds were used in the experimentation. Mass ratios of mixtures, fly ash to sodium hydroxide are given in table 3.

2.2. Synthesis
To prepare the geopolymer paste, Fly ash was activated with a sodium hydroxide solution for 15 min. Sodium hydroxide/fly ash mass ratios of 1, 1.2, 1.4, 1.6, and 1.8 were experimented at the present study. Additional water was added to mass ratio of 1.8 to provide good workability of the paste. To cast the geopolymer samples,

polyethylene molds 20 x 40mm diameter and height, respectively, were used. The samples were cured in oven at temperatures of 80°C and 100°C for four days, and kept at room temperature until testing time.
Compressive strength tests were determined at 30 days, following the procedure described in ASTM D1633[7].

Table1: Physical properties of fly ash

Materials	Median particle size(µm)
Fly ash (FA)	24.5

Table2: Chemical composition of fly ash

Chemical composition (%)									
SiO_2 t	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	K_2O	Na_2O	PF	total
52,44	26,54	5,22	3,90	1,63	0,22	2,37	0,37	5,47	98,15

2.3. Mictostructure analysis

To provide information on used fly ash structure, an X-ray analysis was performed. After determining the strength, different specimens were investigated to perform the development of geopolymer reaction. Thus, to record molecular absorption and transmission to create a molecular imprint of the samples, FT-IR analysis was used.

3. Results and discussion

3.1. Physical and chemical properties of fly ash

The results shown in Fig.1 performed the particle size of the fly ashes. Therefore, the fly ash median particle size is (24.5µm). The given chemical composition of the fly ash SiO_2 + Al_2O_3 + Fe_2O_3 of 84.2 %, SO_3 of 0.22 %, and CaO of 3.90 %, shown in Table 2, indicate that our fly ash is a class F fly ash as described in ASTM C618-08a[8].

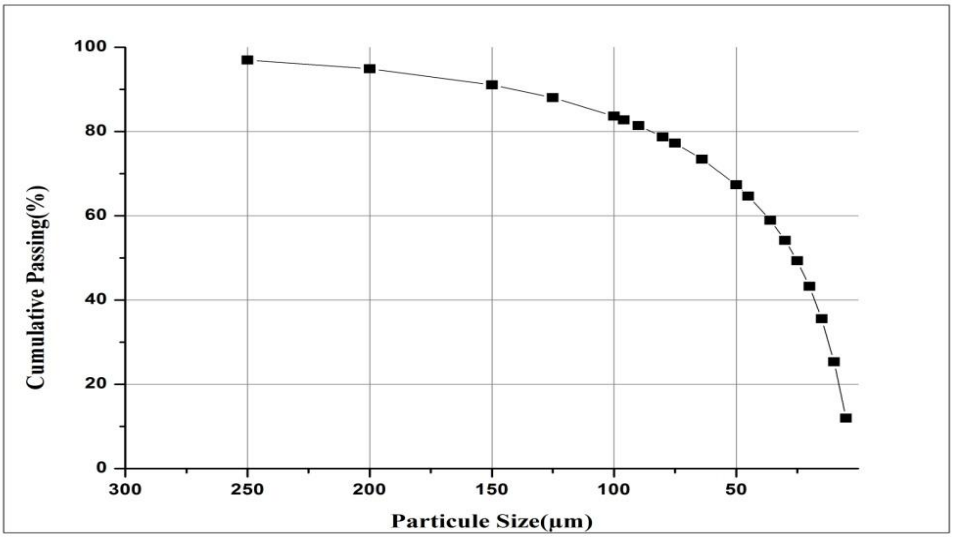

Figure1: Particle size distribution of fly ash

3.2. X-ray diffraction of fly ash

Result of XRD analysis of fly ash material is shown in the fig1. The X-ray diffraction pattern, shows that fly ash consist mainly of amorphous alumino-silicate products, and slightly of crystalline products, predominantly quartz and mulite.

Table3: Initial mass ratios of fly ash to sodium hydroxide in the mixtures

Mix	NaOH concentration (M)	Fly ash/sodium hydroxide (mass ratio)	Temperature
10NaOH	10	1	80-100°C
	10	1.2	80-100°C
	10	1.4	80-100°C
	10	1.6	80-100°C
	10	1.8	80-100°C
12NaOH	12	1	80-100°C
	12	1.2	80-100°C
	12	1.4	80-100°C
	12	1.6	80-100°C
	12	1.8	80-100°C
14NaOH	14	1	80-100°C
	14	1.2	80-100°C
	14	1.4	80-100°C
	14	1.6	80-100°C
	14	1.8	80-100°C

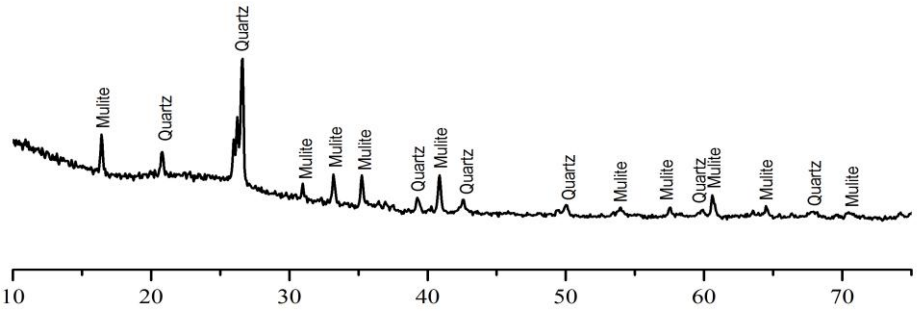

Figure 2: XRD profile of fly ash

3.3. Compressive strength test

Compressive strengths of geoplymer specimens are given in the fig.3 and fig.4. Compressive strengths increased with an increase of NaOH concentration. The use of 12M and 14M give relatively high strength geopolymer. However, the use of 10M gave low compressive strength as a result of low leaching of Si and Al ions in NaOH solution[9][10]. Further away, a weak chemical reaction resulted by the use of a low alkali solution[11]. Mass ratios (fly ash/NaOH) affected directly the strength of geopolymer. Thus, mass ratio of 1.0 gave low compressive strength 14MPa of 14M NaOH;100°C and 8MPa of 10M, 12M, 14M NaOH for 80°C fig.2, compared to ratios of 1.2; 1.4; and 1.6 for 80°C and 100°C fig.3 and fig.4.

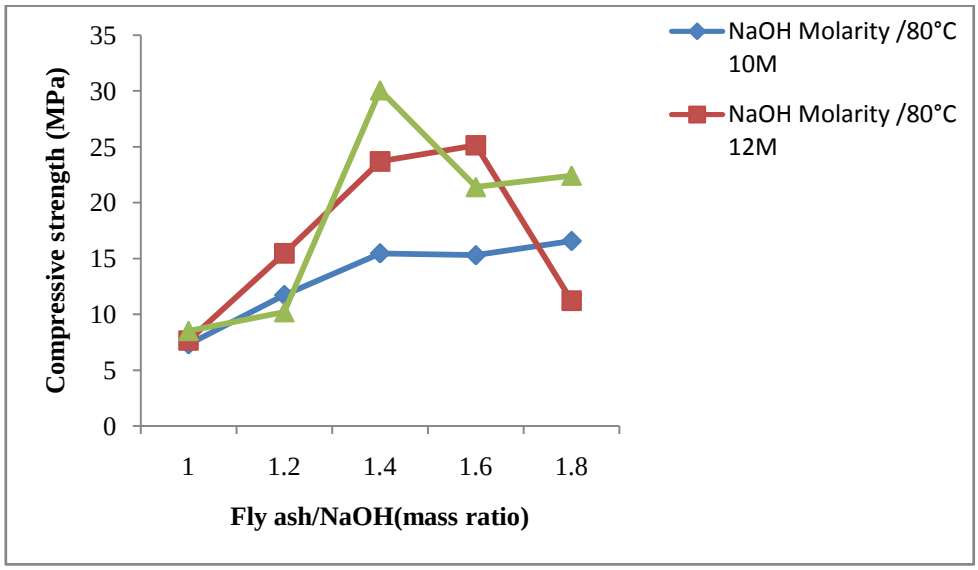

Figure 3: Compressive strength of alkali-activated fly ash at 80°C

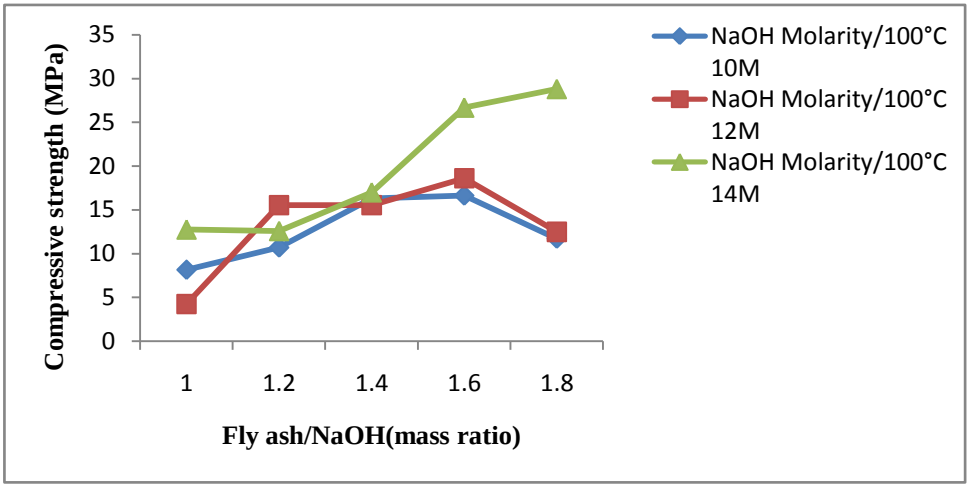

Figure 4: Compressive strength of alkali-activated fly ash at 100°C

The results also give a comparison between two curing time conditions fig.2 and fig.4. Specimens cured at 80°C and specimens at 100°C were tested. Compared to specimens cured at 80°C fig.3, Compressive strength of specimens cured at 100°C still are less than 20MPa for all ratios used, expected ratios of 1.6 and 1.8 of 14M NaOH fig.4. This results lead to say that curing conditions have an effect on compressive strength. Increasing temperature, increases water evaporation of geopolymers specimens, consequently decreases the compressive strength of geopolymers .

3.4. FTIR analysis results

The results of infrared spectroscopic for fly ash and alkali-activation products are shown in fig.5. An articulate peak appears on IR spectrum of fly ash at 451 cm^{-1} associated with the Si-O-Si bending vibration and another intense band around 1050 -994 cm^{-1} associated with Si-O-Si and Si-O-Al asymmetric stretching vibration[12][13]. With the increase of NaOH concentrations of 10-14M, Peaks of bands around 451 cm^{1} are unchangeable. Compared to the band 993 cm^{-1} of fly ash, the peak of gopolymer pastes is 996-993cm^{-1}. The formation of this new amorphous aluminosilicate gel phase suggests depolymerisation and structural reorganization of the amorphous phases in the alkali-activated fly ash[14].The board bands around 1681cm^{-1} are attributed to the stretching vibration of –OH, and Board bands around 3487cm^{-1} are associated to bending vibration of O-H-O[15].

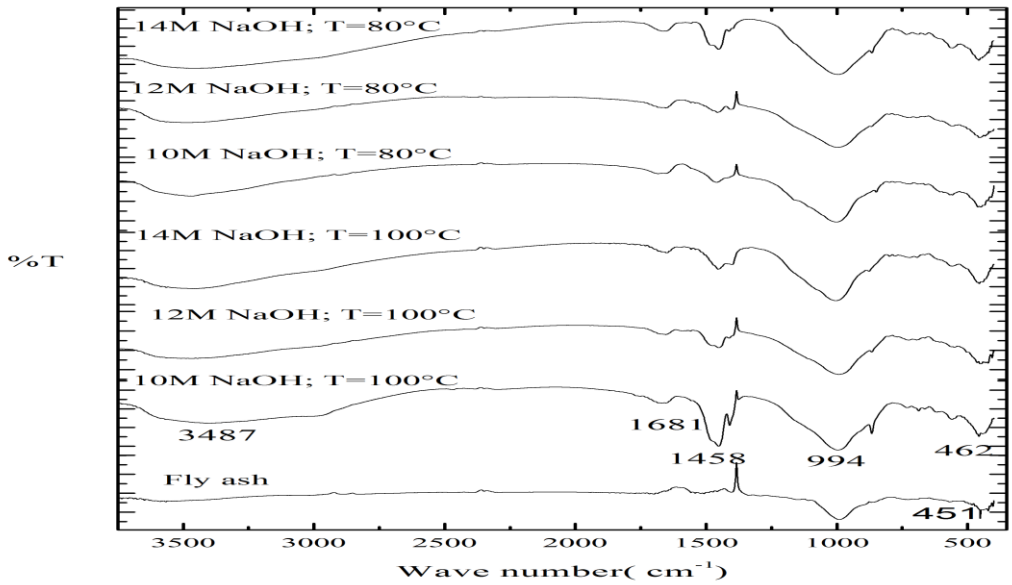

Figure5: infrared analysis of fly ash and alkali activated fly ash at different NaOH concentrations under two curing conditions (80 and 100°C)

Conclusion

Reasonable compressive strength geopolymers can be produced at different NaOH concentrations and different curing conditions. NaOH concentration affected directly compressive strength of alkali activated fly ash. Compressive strengths at 80°C were much better than that obtained at 100°C. Compressive strength of 30MPa was obtained with 14M NaOH at 80°C. FTIR study showed that geopolymerization reaction occurred with high alkaline conditions.

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