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EFFECT OF CURING TEMPERATURE ON THE PROPERTIES OF 100% CLAY-BASED GEOPOLYMER CONCRETE

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Substantial researches have already been carried out on alkali-activated binders for the production of geopolymer concrete but studies on clay-based geopolymer concrete is still insufficient. The aim of this paper is to identify the effect of curing temperature on the properties of 100% clay-based geopolymer concrete. Clay pre-treated at 80°C and 120°C for 24 hours were selected as the source binder material. Four Activator Modulus (AM); 1.0, 1.25, 1.5 and 1.75 for each of two Sodium Oxide (Na_2O) dosages of 10% and 15% were selected as the activator material for this investigation. X-ray diffraction (XRF) was applied to characterize the clay material. Specimens were cured at 80°C and 120°C for 24 hours. Specimens were tested under compression at seven, 14, 28 and 40 days. Specimens cured at 80°C took a longer duration (>28 days) to achieve structural integrity while the specimens cured at 120°C achieved structural intigrity within seven days. Compressive strength of specimens prepared with AM of 1.0 for both of the Na_2O dosage of 10% and 15% exhibited superior performance to other AMs investigated.

Keywords: Clay, Compressive strength, Activator modulus.

1 INTRODUCTION

According to the report of Intergovernmental Panel on Climate Change (IPCC) 2014, the growth of world cement production had increased sevenfold by 2010 compared to that in 1970, and from 2000, this growth has been increasing sharply (Fischedick *et al.* 2014). It has been also reported that the cement industry was responsible for more than 13% of global emission (emissions from cement-forming reactions only) of CO_2 in 2010. The IPCC has also suggested substituting geopolymer concrete in place of OPC concrete to reduce CO_2 emissions (Fischedick *et al.* 2014).

Until now, fly ash is the main source of binder material to produce geopolymer concrete, although other source materials are being examined. An alarm is that the manufacturing of fly ash will decline in the future as power generation will shift from coal to other renewable forms of electricity generation. It has also been reported that not all fly ash is suitable for geopolymer concrete production. A naturally occurring material like clay which is abundant throughout the world, could be an opposing alternate source material of OPC to fulfil the future requirement. It is possible to reduce the global warming up to 40% if clay-based geopolymer is used as a substitute material of OPC (Heath *et al.* 2014). Researchers are revising a range of different raw materials for geopolymerization such as rice husk ash (He *et al.* 2013), natural zeolites (Villa *et al.* 2010), lignite bottom ash (Sathonsaowaphak *et al.* 2009) and calcinated paper sludge (Santa *et* *al.* 2013). Recently, blended geopolymer which is derived from the mixture of two or more industrial by-products has drawn attention from a number of researchers (Bhutta *et al.* 2014, Xu *et al.* 2014, He *et al.* 2013).

Although clay-based binder possesses significant potential to be an alternative material to OPC, research on the utilization of clay as an alternate binder material for geopolymerization system is still in its infancy. The effect of varying curing temperature on the performance of 100% clay-based geopolymer has been reported in this paper through an experimental investigation. Four Activator Modulus (AM), namely 1.75, 1.50, 1.25 and 1.00 for each of two Na_2O dosages of 15% and 10% were selected for this analysis. AM and Na_2O Dosage is defined as follows:

AM = (SiO_2 in Alkaline Activator Solution) / (Na_2O in Alkaline Activator Solution)

Na_2O Dosage (%) = (Na_2O in Alkaline Activator Solution) / (Mass of Binder).

2 EXPERIMENTAL PROCEDURE

2.1 Materials Used

In this study, the clay material used as binder material was collected from the Enfield area about 30 km from Ballarat, Victoria, Australia. The chemical composition along with phase composition supplied by Selkirk Company is presented in Table 1. A mixture of D-grade liquid Na_2SiO_3 and 15 Molar NaOH solution was used as activator solution. River sand of specific gravity of 2.5 and fineness modulus of 3.0 was used as fine aggregate.

Table 1. Chemical and phase composition of clay material.

Chemical Composition		**Phase Composition**	
Oxide	**%**	**Phase**	**% (Weight)**
Al_2O_3	14.83	Illite	24.98
SiO_2	69.88	Kaolinite	27.17
CaO	0.24	Quartz	43.05
Fe_2O_3	3.88	Rutile	1.67
K_2O	2.75	Chlorite	1.42
MgO	1.09	Montmorillite	0.17
MnO	0.015	Albite	0.87
Na_2O	0.23	Goethite	0.6
TiO_2	0.82		
Cl	475 ppm		

2.2 Proportioning of the Materials

The mass proportioning of ingredients of different AM for both Na_2O dosages of 10% and 15% has been presented in the Tables 2 and 3. The proportioning of ingredients was calculated following an established mix design of Adam (2009). The mass ratio of sand to binder was fixed at 2.75 (ASTM C109/ C109M-07). The w/s (water/solid) ratio of 0.37 was used to ensure consistent workability of the geopolymer mortars. The quantity of water in the mix is the sum of the water contained in the sodium silicate, sodium hydroxide and the added water, while the quantity of solid is the sum of the mass of clay, and the solid contained in the alkaline activator solution.

Table 2. Mix design details based on AM for Na_2O dosage = 15%.

AM	Clay (kg)	Sand (kg)	Na_2SiO_3 (kg)	NaOH (kg)	Added Water (kg)	Na_2SiO_3/NaOH
1	1	2.75	0.51	0.24	0.07	2.12
1.25	1	2.75	0.64	0.17	0.044	3.61
1.5	1	2.75	0.76	0.11	0.018	6.65
1.75	1	2.75	0.89	0.06	0	14.24

Table 3. Mix design details based on AM for Na_2O dosage = 10%.

AM	Clay (kg)	Sand (kg)	Na_2SiO_3 (kg)	NaOH (kg)	Added Water (kg)	Na_2SiO_3/NaOH
1	1	2.75	0.34	0.15	0.17	2.17
1.25	1	2.75	0.42	0.11	0.153	3.69
1.5	1	2.75	0.51	0.07	0.135	6.95
1.75	1	2.75	0.6	0.04	0.118	14.63

2.3 Specimen Preparation and Testing

Clay was processed for two different pretreatment conditions: at 80°C and 120°C for 24 hours. After pretreatment, the clay material was powdered using Ball Mill Grinder Machine. The mill comprised of a total of 15 balls and 5000 cycles were applied to make the clay powder. The powdered clay was sieved through a 106 µm size sieve. Four Activator Modulus (AM); 1.0, 1.25, 1.5 and 1.75 for each of two Sodium Oxide (Na_2O) dosages of 10% and 15% were selected as activator material. The clay powder was mixed with sand for 4 minutes using 5-liter Hobert mixer. A 15 Molar NaOH solution and D-grade liquid Na_2SiO_3 of 1.52 g/cc density with composition of 14.7% Na_2O, 29.4% SiO_2 and 55.90% water were premixed 15 minutes before the additional water was added to the activator solution. This activator solution was then added to the mixture of clay/clay powder and sand. After manual mixing for 1 minute, the whole mix was blended by Hobert mixer machine with two rotating speeds: 150 and 300 rev/min for four and two minutes respectively. The mix was then placed in 50x50x50 mm Teflon moulds followed by 30 second vibration on vibrating table. After allowing 24 hours at room temperature, the moulds were kept in oven at 80°C and 120°C for another 24 hours. The moulds were then demoulded and cured at room temperature until testing. Compressive strength measurements of mortars were performed on a TCM Testing Machine in accordance with ASTM C109/C109M-13 with a loading rate of 0.34 $N/mm^2/S$. Three cubes were tested for each data point.

3 RESULTS AND DISCUSSION

The specimens were tested for compression at seven, 14, 28 and 40 days. The 40-day test was carried out to assess the rate of strength reduction as significant falloff strength between 14 and 28 days was observed. The compressive strength of geopolymer mortars are presented in Tables 4-9. It can be seen from the Tables 4-7 that high temperature (> 80°C) is needed for achieving the structural integrity of clay-based geopolymer mortars. Curing at 80°C for 24 hours was not sufficient to set the specimens at even 28 days. Similar types of behavior were obtained for the specimens prepared from the clay pre-treated at 120°C also (Tables 6-7). The specimens achieved structural integrity within seven days when the curing temperature was increased at 120^0C for both the cases of pre-treatment of clay: at 80°C and at 120°C. Testing at three days was not possible as the specimens had not achieved structural integrity. This indicates that clay-based geopolymer mortar takes longer duration to achieve better strength than that of normal cement mortar or fly ash based geopolymer mortar. A normal Portland cement would be expected to set within 24 hours (Neville 2011) at room temperature. He *et al.* (2013) also found that red mud (RM) and rice husk ash (RHA) based geopolymer paste took at least 35 days to achieve complete geopolymerization. They mentioned three possibilities: dominant crystalline solid phase acts as unreactive filler, larger particle size slows down the dissolution rate and the presence of impurities may have a detrimental effect on the rate of geopolymerization process. Hanjitsuwan *et al.* (2014) pointed out that at higher NaOH concentration (up to 18 M) the geopolymerization process occurs at a slower rate resulting in a longer setting time. Ming *et al.* (2016) also mentioned that to initiate the geopolymerization reaction by exceeding the thermal activation of reaction, heat is obligatory for clay based geopolymers.

Table 4. Compressive strength of specimens of clay pre-treated at 80^0C, curing temperature of 80^0C and Na_2O Dosage of 15%.

AM	**7 days strength (MPa)**	**14 days strength (MPa)**	**28 days strength (MPa)**	**40 days strength (MPa)**
1	-	-	-	<1
1.25	-	-	-	<1
1.5	-	-	-	<1
1.75	-	-	-	<1

Table 5. Compressive strength of specimens of clay pre-treated at 80^0C, curing temperature of 80^0C and Na_2O Dosage of 10%

AM	**7 days strength (MPa)**	**14 days strength (MPa)**	**28 days strength (MPa)**	**40 days strength (MPa)**
1	-	-	-	<1
1.25	-	-	-	<1
1.5	-	-	-	<1
1.75	-	-	-	<1

Table 6. Compressive strength of specimens of clay pre-treated at 120^0C, curing temperature of 80^0C and Na_2O Dosage of 15%.

AM	**7 days strength (MPa)**	**14 days strength (MPa)**	**28 days strength (MPa)**	**40 days strength (MPa)**
1	-	-	-	<1

Table 7. Compressive strength of specimens of clay pre-treated at 120^0C, curing temperature of 80^0C and Na_2O Dosage of 10%.

AM	7 days strength (MPa)	14 days strength (MPa)	28 days strength (MPa)	40 days strength (MPa)
1	-	-	-	<1

Table 8. Compressive strength of specimens of clay pre-treated at 80^0C, curing temperature of 120^0C and Na_2O Dosage of 15%.

AM	7 days strength (MPa)	14 days strength (MPa)	28 days strength (MPa)	40 days strength (MPa)
1	9.58	10.80	9.40	11.72
1.25	10	11.34	9.74	7.83
1.5	12.86	13.45	9.45	7.64
1.75	20.02	11.96	12.04	11.05

Table 9. Compressive strength of specimens of clay pre-treated at 80^0C, curing temperature of 120^0C and Na_2O Dosage of 10%.

AM	7 days strength (MPa)	14 days strength (MPa)	28 days strength (MPa)	40 days strength (MPa)
1	9.68	14.5	10.42	13.88
1.25	10.87	14.21	10.06	13.25
1.5	11.36	10.97	9.75	12.5
1.75	14.92	10.36	9.96	9.69

The results also show that (Tables 8-9) the strength generally increases from seven to 14 days for both the 10% and 15% Na_2O dosages. The only exception being the 15%, 1.75 AM which achieves the maximum strength (20.02 MPa) at seven days which reduces significantly at 14 days (11.96 MPa).

The highest strength (20.02 MPa) was achieved for AM of 1.75 at seven days whereas AM of 1.5 provided the highest strength (13.45MPa) at 14 days for Na_2O dosage of 15%. For this dosage, compressive strengths of specimens at 28 days were almost similar for all AMs except for AM of 1.75. For the case of Na_2O dosage of 10%, the optimum strengths at 14, 28 and 40 days were displayed by specimens prepared with using AM of 1.0. It can also be seen from Tables 8-9 that strengths of specimens prepared with Na_2O dosage of 10% are more consistent than those associated with Na_2O dosage of 15%. Malolepszy (1993) mentioned that the solubility of Na+ ion is very low in alkali activated binder material because of the formation of Na_2O-CaO-SiO_2-H_2O. Results in this study also indicate that the more is the Na_2O content the lower the strength with time.

4 CONCLUSIONS

The following conclusions were drawn from the above discussion:

- Elevated curing temperature (> 80°C) is needed for achieving the structural integrity of 100% clay-based geopolymer mortar and its setting time is longer than that of FA based geopolymer mortar or normal OPC mortar.

- The clay-based geopolymers generally showed an increase in strength up to 14 days. After this a slight increase in strength was observed for the specimens with a 10% Na_2O dosage, while a decrease was observed for those with a 15% Na_2O dosage, other than with an AM of 1.0.

- Compressive strength of specimens prepared with AM 1.0 for both of the Na_2O dosage of 10% and 15% exhibited superiority over other AM.

- Excess Na_2O content has a detrimental effect on the strength gain of clay-based geopolymer mortar i.e. the higher the Na_2O content, the lower will be the strength with time.

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