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IJIE

Journal homepage: http://penerbit.uthm.edu.my/ojs/index.php/ijie
ISSN: 2229-838X e-ISSN: 2600-7916

The International Journal of Integrated Engineering

The Effect of Dry Mix Sodium Hydroxide onto Workability and Compressive Strength of Geopolymer Paste

A. Z. Mohd Ali^{1*}, N. A. Jalaluddin¹, N. Zulkiflee¹

¹Faculty of Civil Engineering and Built Environment, Universiti Tun Hussein Onn Malaysia, Parit Raja, 86400, Johor, MALAYSIA

*Corresponding Author

DOI: https://doi.org/10.30880/ijie.2020.12.09.014

Received 28 May 2020; Accepted 18 October 2020; Available online 26 December 2020

Abstract: The production of ordinary Portland cement (OPC) consumes considerable amount of natural resources, energy and at the same time contribute in high emission of CO₂ to the atmosphere. A new material replacing cement as binder called geopolymer is alkali-activated concrete which are made from fly ash, sodium silicate and sodium hydroxide (NaOH). The alkaline solution mixed with fly ash producing alternative binder to OPC binder in concrete named geopolymer paste. In the process, NaOH was fully dissolved in water and cooled to room temperature. This study aims to eliminate this process by using NaOH in solid form together with fly ash before sodium silicate liquid and water poured into the mixture. The amount of NaOH solids were based on 10M concentration. The workability test is in accordance to ASTM C230. Fifty cubic mm of the geopolymer paste were prepared which consists of fly ash to alkaline solution ratio of 1: 0.5 and the curing regime of 80°C for 24 hours with 100% humidity were implemented. From laboratory test, the workability of dry method geopolymer paste were decreased. The compressive strength of the dry mix of NaOH showed 55% and the workability has dropped to 58.4%, it showed strength reduction compared to the wet mix method.

Keywords: Geopolymer paste, workability, compressive strength, fly ash, sodium hydroxide, sodium silicate, alkaline solution

1. Introduction

Geopolymer paste is the binder that bind all the materials in geopolymer concrete matrix. By producing geopolymer it can overcome the usage of Ordinary Portland Cement (OPC) in construction industries. Nowadays, the cement industry is certainly is one of the major contributors to the emission of greenhouse gasses like carbon dioxide. In addition, one of the advantages of geopolymer is that it has excellent reaction resistance to both conditions which is in acid and salt environment hence the strength escalated as the curing time and temperature increased. In geopolymer, the main materials on enhancing the activation while the polymerization process are held including parameters such as the molarity of alkaline, the ratio of alkaline activator composers and the addition of other materials [1]. Geopolymer is indeed a new material which is in need of developing it to the standard design procedure of its constituent material mixing up until now but with a correct mix design and formulation development, concrete that are made from geopolymer definitely have been proven to be found more durable than the usual concrete which is the Portland cement concrete [2]. Many practicing and professional engineers are interested on trying to create a measurement of the strength and other physical properties of the binder paste. This paste will bind the matrix containing coarse aggregate and fine aggregates namely called geopolymer concrete. In this matter, fly ash and alkaline solution is used as a binder [3].

Researches have been searching and experimenting every different source material in developing geopolymer binders. Theoretically, any source of amorphous silica could be mixed to manufacture geopolymer binders. Geopolymers are a type of inorganic polymer that can be created by using industrial waste or by products which that can enhance and has similar functions to Ordinary Portland Cement (OPC) [4]. Moreover, the geopolymer can be used in applications to fully replace OPC with environmental and technical advantages. Geopolymer binder is made from fly ash, sodium silicate (Na₂SiO₃) and sodium hydroxide (NaOH) in this experiment to test on the workability and strength of the geopolymer concrete itself. Hence, geopolymer paste will be formed to test on the achievement of the concrete strength. Generally, concrete is regarded as very-low viscosity inorganic resins and it hardened like a thermosetting resin, but it portrays a very high strength and are ceramic-like in their properties.

Geopolymer is an inorganic polymer based on alumino-silicates and can be produced by synthesizing pozzolanic compounds source materials with exceedingly high alkaline solutions [5]. One of the primary advantages of these geopolymer concretes are they can possess impressive physiochemical and mechanical properties because it can exhibit a various variety of valuable characteristics than the OPC [6]. Furthermore, it also known to show excellent performance with respect to rapid hardening, high and early compressive strength including low creep and drying shrinkage in such severe environment [7]. Unique properties of the geopolymer concrete does attracts the industries to choose this as a substitute for OPC and moreover, it also reduces the emissions of the CO₂ by cement industries and utilizes the industrial wastes more efficiently [8].

2. Materials and Methods

The cement industry is a major contribution to the environment health and safety issues associated with cement productions. It requires a large amount of non-renewable resources like raw materials and fossil fuels which donates to the cumulation of dust and gases. The consumption of raw materials. There are two principle constituents of geopolymer which is namely the source materials and the alkaline liquids.

2.1 Fly Ash

Fly ash is chosen as one of the usable raw material in geopolymer due to its attributes as one of the cheapest wastes that can be obtained easily by cement manufacturers. Fly ash are recognized as valuable substances which can be develop desirable characteristics and stabilization process. Table 1 showed the quantities of fly ash produced among other wastages.

The geopolymer reaction is based on aluminosilicate reaction to form a binder as it is one of the main sources on where the aluminosilicate reaction is produced. Class F type of fly ash is used in this study and it was obtained from Manjung Quarry Power Plant in Perak. It was selected due to its low calcium and it does not interfere with the polymerization process and change the microstructure of the geopolymer [9]. Table 2 showed the chemical composition between Class F of fly ash that were used in this study.

Chemical Compound	Low calcium fly ash Class F
Silicon dioxide (SiO ₂)	54.90
Aluminium oxide (Al ₂ O ₃)	25.80
Iron oxide (Fe ₂ O ₃)	6.90
Calcium oxide (CaO)	8.70
Magnesium oxide (MgO)	1.80
Sulfure trioxide (SO ₃)	0.60
Sodium oxide (Na_2O) and Potassium oxide (K_2O)	0.60

Table 1 - Chemical composition between Class F type of fly ash

Class F is an ideal cementitious material in mass concrete and can achieve high strength mixes. Fly ash contains a high content of silica hence for many applications in the civil engineering field, a low Silica to Alumina (Si: Al) ratio is suitable.

2.2 Sodium Hydroxide (NaOH)

Sodium hydroxide (NaOH) is a material that is highly soluble in water and absorbs carbon dioxide and moisture present in the air. Sodium hydroxide can be obtained in pellets and liquids. Basically, the type of constituents is important and sodium hydroxide are one of the widely used for synthesis of geopolymer rather than potassium hydroxide (KOH). It was proven that sodium hydroxide (NaOH) is having smaller ionic size of its Na+ which is 116

pm compares to K+ ionic size which is 152 pm. Hence, the Na+ is more active and will intensify the dissolution process of alumino-silicate minerals. Concisely, the main influence on a geopolymer is its activator. In Fig. 1 and Table 3 below shows the figure and table of chemical composition of NaOH.



Fig. 1 - NaOH pellet

•
Percentage (%)
98
0.9
0.003
0.001
0.004
0.015
0.04
0.004

Table 2 - Composition of NaOH

2.3 Sodium silicate (Na₂SiO₃)

Sodium silicate readily available in liquid form. The sodium silicate is colorless or plain white and have a high viscosity due its glassy properties. In geopolymer, geopolymerization is known as the process of hardening of fly ash based that is achieved by the presence of alkaline medium. The alkaline liquid is used as it reacts easily to both Al-Si oxides in the low calcium fly ash to form a geopolymer paste that bonded the loose coarse aggregates, fine aggregates and other un-reacted materials combined to create a geopolymer. The molarity is determined by the number of dissolved material molecules in one liter or ml of the solution [10]. Higher molarity and viscosity of the end solution will make the compression strength increased. Fig. 2 and Table 4 shows the chemical composition of sodium silicate.



Fig. 2 - Sodium silicate

Table 3 - Chemical composition of sodium silicate

Chemical composition	Percentage (%)
Sodium Oxide (Na ₂ O)	17
Silicate Oxide (SiO ₃)	35
Total Solid Content	52

2.4 Methods Preparation

The geopolymer paste mix was determined by casting the geopolymer paste in 50mm 3 mold in order to obtain the workability and compressive strength of paste. It is based on the ratios of alkaline solution to fly ash (AL/FA) and sodium hydroxide (solid or liquid). The molarity used was 10M. The ratio of Al/FA is 1: 0.5, SiO/NaOH is 1: 2.5. There are two types of mix which are wet mix and dry mix. The wet mix are the conventional method used in geopolymer. For the dry mix, the NaOH solid are mixed with the fly ash without water. In this dry method, the sodium hydroxide solid were mixed with fly ash. Next, both materials of sodium silicate and water were mixed to make an alkaline medium. The dry materials of NaOH solid and fly ash are mixed into the alkaline medium and mixed in the bowl mixer for 3 minutes. The workability of geopolymer paste was measured using flow test accordance to the ASTM C230, immediately after mixing. The paste was molded in six samples of cubes and placed in the oven for curing of 24 hours at 80°C with 100% humidity.

3.2 Workability Test

Due to the high viscosity of geopolymer paste, the workability of the geopolymer mix was tested using the flow test. Geopolymer paste were mixed, poured and compacted into 3 layers into a brass cone mold with the inner diameter base of (d_0) of 80mm. Each of the layers was tamped for 25 times using a brass tamper. The mold was lifted, and the mix could flow [11]. The diameter is taken based on d_1 and d_2 of the flowed mix was measured after one minute. The slump flow (Sf) of the final slump are measured on the average of two perpendicular diameters $(d_1$ and $d_2)$ after the cone lifting. Relative slump, rp was calculated by using the equations below.

$$Sf = (d_1 + d_2) / 2$$

 $do = 80mm$ (1)
 $rp = (sf/d_0) - 1$

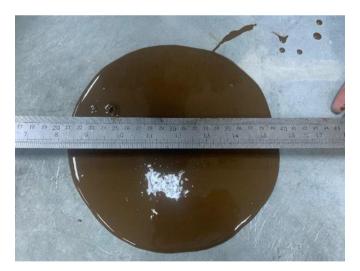


Fig. 3 - Measurement of d_1 and d_2 for geopolymer paste

3.3 Compressive Strength Test

All specimens were casted in 50mm cubes, it is molded and tested after 24 hours by using a compactor machine. The hardened geopolymer paste cubes were cooled for 24 hours before tested. The machine will control the displacement of rate 1 mm/min until the sample fails. The compressive axial force was recorded throughout the process and analyzed. The mean value obtained from the six cubes was taken as the cube compressive strength for each geopolymer paste mix. The compressive strength of the geopolymer paste is calculated by dividing the maximum load obtained from the test.

3. Results and Discussions

4.1 Effect of The Sodium Hydroxide Solid on Workability in between Wet and Dry Mix Method

The effect of the sodium hydroxide solid on workability was investigated by six different samples. The results of the workability test were showed that the relative slump differences between the mixes were 0.97. The dry mix workability has dropped 58.4% compared to wet mix. It has been observed that the flow diameter decreases due the solid NaOH does not mixed with sodium silicate that it made the dry mix viscosity increased and resulted in reduction of the of flow diameter. The reduction of workability for dry method effected in the strength of geopolymer paste cube due to the presence of void [12].

	Table 4	- Relative	slump	summary
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Type of Mix	Diameter 1 (d ₁), mm	Diameter 2 (d ₂), mm	Relative Slump (rp)
Wet Mix	210	215	1.66
Dry Mix	140	130	0.69

4.2 Comparison of Physical Analysis Between Dry and Wet Method

There is significance reduction of strength between geopolymer paste cube when casted with dry method. One of the main differences geopolymer paste made by using dry method is the presence of undissolved sodium hydroxide [13]. The wet NaOH geopolymer paste showed absence of sodium silicate and sodium hydroxide well mixed with the fly ash. The results proved that the compressive strength of wet method geopolymer paste cubes were excellent while the geopolymer paste cubes made by using dry method showed the presence of white spots in the geopolymer paste. The white spots are the undissolved NaOH solids. The undissolved NaOH solids were not reacted with the sodium silicate for aluminosilicate reaction that provide the strength of geopolymer paste [14]. It is also can be observed that sodium silicate presence around the NaOH solids made the paste looked damp.





Fig. 4 - Physical observation of wet sodium hydroxide





Fig. 5 - Physical observation of dry sodium hydroxide

4.3 Effect of Sodium Hydroxide Solid on Compressive Strength in Dry Mix

The graph in Fig. 6 showed the results taken; the reduction of the strength was 55% as a result between the dry and wet mix. The strength deterioration was affected by the solid NaOH which did not dissolve together during the process. However, there are aluminosilicates reaction happen which involved alumino and silicate from fly ash and NaOH that

dissolved in free added to the geopolymer paste made by dry method. These reactions that provide the strength observed in dry method geopolymer paste cube compressive strength. In general, geopolymer paste effect mixed with NaOH prove that the wet mix gained more strength than the dry mix of NaOH due to complete reaction between alumino and silicate elements from fly ash and both NaOH and sodium silicate [15].

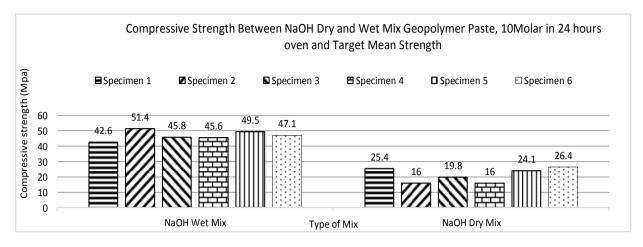


Fig. 6 - Compressive strength between NaOH in dry and wet mixes (Specimens 1-6)

Based on the results showed, the specimen 1 to 6 were mixtures which are mixed in the same mould as showed in the figure below. It is categorized for NaOH Wet Mix and also NaOH Dry Mix but the results showed differently even they were in the same mould due to the methods of pouring and tamping during the process including the way they were placed in the oven. This is because the heat in the oven were not equivalently spread onto the specimens during the heating process. Hence, the results showed significant different between the 6 specimens in the mould.





Fig. 7 - The geopolymer are labelled as Specimen 1-6 in the mould

4. Conclusions

This study has proved that it is impossible for the sodium hydroxide solid to obtain the optimum of workability and compressive strength for the geopolymer mixture. The geopolymer paste made by using the sodium hydroxide solid was the difference and the undissolved sodium hydroxide resulted in non-complete reaction between alumino and silicate reaction in due to silicate did not happened in the geopolymerisation process [16]. The dry method geopolymer paste strength came from the reaction of alumino-silicate reaction process from fly ash and NaOH since no sodium silicate reacted in the mix. The time can't be reduced for the cooling down of the reaction of NaOH with water hence the solid hydroxide is not recommended.

Acknowledgment

Authors are thankful to Universiti Tun Hussein Onn Malaysia (UTHM) and Ministry of Education Malaysia for providing the resources and facilities for the research. The authors also want to thank to Research Management Centre

(RMC UTHM) for providing research fund under the Postgraduate Research Grant (GPPS) project no. U946 for this research.

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