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Characteristics of fly ash geopolymer based on alkaline ratio of activators for building materials: *A review*

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ABSTRACT

The geopolymer introduced by Davidovits is a new binder for making concrete as an alternative to ordinary Portland cement. One of the materials used to make geopolymers is fly ash. The annual worldwide production of fly ash is estimated to be around 500 Mt. In making geopolymers with fly ash, scientists usually use activators such as silicate and aluminate solutions, as well as with kaolinite. The geopolymerization process is related to the metal oxide ratio of the activator solution. Geopolymers can withstand temperatures up to about 600°C, so they are fire/heat resistant. This paper describes the role of the comparison of dominant elements in a mixture of geopolymer paste with fly ash and alkali which produces the best quality based on compressive strength, where there is an effect of the ratio of alkali on the compressive strength of geopolymer paste. Almost all of the highest compressive strength obtained did not follow the Davidovits rule. This is caused by other factors that affect the compressive strength such as the content of elements such as Si and Al in fly ash and the curing temperature. However, the volumetric and mechanical properties of the artificial aggregate (geopolymer fly ash application) meet the requirements as a road material. It's just that the absorption value is above the requirements (>3%). Seeing the properties of these geopolymers, there is hope to be developed into materials for building materials.

ABSTRAK

Geopolimer yang diperkenalkan oleh Davidovits adalah pengikat baru untuk membuat beton sebagai alternatif pengganti semen *Portland* biasa. Salah satu bahan yang digunakan untuk membuat geopolimer adalah *fly ash*. Produksi tahunan *fly ash* seluruh dunia diperkirakan sekitar 500 Mt. Dalam membuat Geopolimer dari *fly ash* para ilmuwan biasanya menggunakan aktivator seperti larutan silikat dan aluminat, serta dari kaolinit. Proses geopolimerisasi berkaitan dengan rasio oksida logam dari larutan aktivator. Geopolimer dapat menahan suhu hingga sekitar 600°C, sehingga tahan api/panas. Makalah ini menjelaskan tentang peranan perbandingan unsur-unsur dominan pada campuran pasta geopolimer dengan *fly ash* dan alkali yang menghasilkan kualitas terbaik berdasarkan kuat tekan, dimana terdapat pengaruh perbandingan alkali terhadap kuat tekan pasta geopolimer. Hampir seluruh kuat tekan tertinggi yang didapatkan tidak mengikuti aturan Davidovits. Hal ini disebabkan oleh faktor lain yang mempengaruhi kuat tekan seperti kandungan unsur-unsur seperti Si dan Al dalam *fly ash* dan temperatur curing. Namun, *properties volumetric* dan mekanik agregat buatan (aplikasi geopolymer *fly ash*) memenuhi persayaratan sebagai bahan jalan. Hanya saja nilai penyerapannya di atas persyaratan (>3%). Melihat sifat-sifat geopolimer tersebut, membuka harapan untuk dikembangkan menjadi material untuk bahan bangunan.

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1. Introduction

Nowadays, waste is in demand for research as a building material. Research by utilizing mine dust waste in an amount not exceeding 5% can increase the ability of lateritic soils as subgrade [1]. Another waste, namely fly ash, is currently being studied as a building material in the form of geopolymers to be made into concrete. Fly ash is used as a cement substitute. Although the compressive strength and tensile strength are lower, the workability of concrete increases [2]. Research on geopolymer paste based on the comparison of Na_2SiO_3 with NaOH on the compressive strength of the paste, in realizing the idea of making artificial aggregates, shows that the compressive strength of fly ash based geopolymer paste can be increased by modifying the alkali activator and NaOH molarity [3]. Research on the volumetric characteristics of fly ash based geopolymer, as an artificial aggregate with a focus on the ratio of alkali activator, has shown an effect on the specific gravity and absorption of the aggregate [4]. Aggregate made of geopolymer fly ash, meets the requirements for hardness, strength and durability, as a pavement material with an alkaline activator ratio of less than 3.0 [5].

The Geopolymer was originally introduced by Davidovits. Geopolymer is a new binder for making concrete as an alternative to ordinary Portland cement. Describe an inorganic aluminosilicate polymer produced from the synthesis of natural materials such as kaolinite clay or industrially producing fly ash and slag with highly alkaline activators [6]. Geopolymer from iron ore tailings (IOT), is a microevolution of geopolymer by combining fly ash as starting material with IOT and activated with sodium silicate and sodium hydroxide solution [7]. In an effort to reduce CO_2 emissions in the cement manufacturing process, the use of fly ash as a geopolymer material is very interesting. Every year a large amount of coal ash is produced [8]. Annual worldwide coal ash production is estimated to be around 600 Mt, of which about 500 Mt is fly ash [6]. One approach to using fly ash is to use it as an artificial aggregate to replace natural aggregate. The reasons that support this approach are: (1) fly ash accounts for 90% of the total ash produced, (2) the presence of natural aggregates is dwindling, and (3) the large amount of aggregate demand for civil engineering infrastructure is continuously increasing. For example, to make concrete about 70-80% is the mass of aggregate [7].

The use of aggregate is very wide not only for concrete. Therefore, the manufacture of fly ash aggregates not only reduces the impact of fly ash disposal on the environment but also provides great benefits to the economy [8]. Fly ash geopolymer is getting more attention and interest from researchers because it has high mechanical strength, high-temperature resistance, and higher durability when compared to ordinary cement. An investigation of the durability of fly ash based geopolymer with metakaolin at high temperature and acid concluded that the geopolymer has better durability than ordinary Portland cement under the same conditions [7]. This type of alkaline activator plays an important role in the geopolymerization process and has a significant effect on the mechanical strength of the geopolymer. Currently, the most commonly used activating base is a combination of sodium hydroxide solution (NaOH) and sodium silicate solution (Na_2SiO_3) with different mass ratios of Na_2SiO_3/NaOH. The addition of Super Plasticizer (SP) without alkaline elements did not cause a significant change in the strength of the activated fly ash paste and did not increase the workability [6].

Geopolymerization using various fly ash compositions with oxide molar ratios, revealed that fly ash geopolymer depends more on the internal properties of the fly ash itself than on the molar ratio of the oxide. The workability and geopolymer strength of a mixture of sand and high calcium fly ash (Class C fly ash) depends on the amount of sodium silicate and sodium hydroxide in the geopolymer gel. In a mixture, these two alkaline activator components affect the workability of the gel and the strength of the geopolymer. Geopolymerization is highly dependent on the physicochemical properties of fly ash, the availability of silicate and aluminate solutions, and the concentration of added sodium hydroxide [8].

In the production of geopolymers from fly ash, scientists usually refer to the manufacture of geopolymers from activators such as silicate and aluminate solutions, as well as from kaolinite. Geopolymerization of the activator solution, related to the metal oxide ratio of the activator solution such as SiO_2/M_2O, SiO_2/Al_2O_3, M_2O/H_2O and M_2O/Al_2O_3, where M is sodium or potassium [8]. In addition to the alkali ratio and atomic content in fly ash, temperature also affects the compressive strength of geopolymers [9]. The compressive strength of geopolymers increases with increasing curing temperature. Geopolymer can withstand exposure to temperatures up to about 600°C, so this material is a fire-resistant material [10]. This gave rise to the idea of making building materials with geopolymer for civil engineering construction. Based on the results of several studies, this paper aims to explain the role of comparison of the dominant alkaline activator element in a mixture of geopolymer paste with fly ash which produces the best quality.

2. Fly ash content

Fly ash is industrial waste that can be found all over the world. Fly ash is a material that has distinctive properties and is a source of aluminosilicates. Fly ash is an industrial product that is proven to be suitable for producing geopolymer materials. The geopolymer properties of fly ash have been studied extensively in the last decade. A finding indicates that fly ash is suitable for making geopolymers, and the resulting geopolymer presents high mechanical properties and durability [11]. Fly ash has pozzolanic properties and is the result of power plant fuel. Fly ash mainly consists of unburned carbon and aluminosilicate particles as well as mineral constituents of fly ash including the glass phase and the crystalline phase [7]. Table 1 shows that the atomic content in fly ash varies.

Table 1. Variation of mineral content in fly ash.

Source	Fansuri et al. [8] FA-A	Fansuri et al. [8] FA-B	Fansuri et al. [8] FA-C	Fansuri et al. [8] FA-D	Fansuri et al. [8] FA-E	Nematollahi & Sanjayan [6]	Duan et al. [7]	Pu et al. [11]	Khan et al. [12]	R. N. Thakur and S. Ghosh [13]
SiO_2	69.60	67.20	66.10	55.70	52.30	48.80	29.47	29.47	51.11	56.01
Al_2O_3	24.40	26.40	30.70	26.60	32.40	27.00	51.72	51.72	25.56	29.80
Fe_2O_3	1.80	1.40	0.54	10.80	11.00	10.20	2.25	2.25	12.48	3.58
CaO	0.29	0.35	0.06	1.10	1.00	6.20	5.21	5.21	4.30	2.36
MgO	0.30	0.30	0.13	0.65	0.80	1.40	0.15	0.15	1.45	0.30
Na_2O	0.22	0.29	<0.05	0.23	0.10	0.37	0.05	0.05	0.77	0.61
K_2O	2.50	2.90	0.28	0.47	0.22	0.85	0.35	0.35	0.70	0.73
TiO_2	1.00	1.10	2.10	1.60	2.10	1.30	1.83	1.83	1.32	1.75
Mn_2O_3	0.03	0.02	<0.02	0.03	0.20	0.15	0.03	0.03	0.15	-
SO_3	<0.02	0.03	<0.02	0.03	<0.02	0.22	-	-	0.24	-
P_2O_5	0.09	0.08	0.07	1.40	0.07	1.20	-	-	0.885	0.44
BaO	0.05	0.06	0.04	0.32	0.04	0.19	-	-	-	-

Source	Fansuri et al. [8] FA-A	Fansuri et al. [8] FA-B	Fansuri et al. [8] FA-C	Fansuri et al. [8] FA-D	Fansuri et al. [8] FA-E	Nematollahi & Sanjayan [6]	Duan et al. [7]	Pu et al. [11]	Khan et al. [12]	R. N. Thakur and S. Ghosh [13]
SrO	0.03	0.03	<0.02	0.33	<0.02	0.16	-	-	-	-
ZnO	<0.02	<0.02	0.08	0.05	<0.02	-	-	-	-	-
V_2O_5	0.02	0.03	0.05	0.03	0.02	-	-	-	-	-
LOI*	-	-	-	-	-	-	8.58	8.58	0.57	0.40

3. The role of alkalis in geopolymer mixture

Geopolymer is a type of cement material with alumino-silicate content. The exact geopolymerization mechanism is not well understood because the process involves rapid chemical reactions [14]. Base activation is a chemical process that gives rapid changes to certain structures [15]. However, the predicted mechanism includes three stages: (1) separation of Si and Al atoms from the material through the action of hydroxide ions, (2) condensation of precursor ions in monomers, (3) arrangement/polymerization of monomers into polymer structures. The atomic ratio of Si:Al in the polysalatal structure determines the field of application. Low Si:Al ratios 1, 2, and 3 form a very rigid 3D network. The high Si:Al ratio, higher than 15, gives the geopolymer material a polymeric character r [14].

Alkaline activation of fly ash is different from the hydration of Portland cement. The most widely used alkaline activator is a mixture of sodium or potassium hydroxide (NaOH, KOH) and sodium silicate or potassium silicate. Alkaline liquids can be used to activate silicon (Si) and aluminum (Al) in natural or manufactured materials to produce binders [15]. Sodium hydroxide increases the amount of silicate and aluminate solutions in the geopolymer mixture. Solubility tests on various fly ash samples showed that the solubility increased when the sodium hydroxide concentration in fly ash increased, which also increased the strength of the resulting geopolymer aggregate. The compressive strength of geopolymer aggregates also increases to a maximum before decreasing again when the amount of sodium silicate increases [8]. The geopolymer, introduced by Davidovits in 1970, is synthesized by mixing aluminosilicate reactive materials such as fly ash or iron ore tailings and a strong alkaline solution (such as NaOH or KOH) at room temperature. Alkali activation of waste materials, was first introduced in the 1940s is the origin of geopolymer research [7].

4. Compressive strength of geopolymer paste

Geopolymerization is highly dependent on the physicochemical properties of fly ash, the availability of silicate and aluminate solutions, and the concentration of added sodium hydroxide [8]. This is evidenced by using 6 types of fly ash and 6 variants of alkaline ratios, the paste that produces the highest compressive strength are obtained for Fly ash A = 44.5 MPa, Fly ash B = 13 MPa, Fly ash C = 16.5 MPa, Fly ash D = 13 MPa and Fly ash E = 3.36 MPa. Different fly ash can give different reactivity because the chemical composition varies. The effect of NaOH molarity, fly ash and alkali activator ratio, NaSiO/NaOH ratio, and curing temperature are very important to achieve optimal geopolymer strength [15]. The addition of SP did not cause a significant change in the strength of the alkali activated fly ash paste, and did not increase the workability [6].

The study on geopolymers prepared from iron ore tailings was tested after exposure to 3 and 7 thermal cycles at different heating target temperatures of 200^0C, 400^0C, and 800^0C. Fly ash was replaced in stages from 0% to 30% at intervals of 10% by weight. Under the influence of thermal cycling, the compressive strength decreases significantly, especially after 7 thermal cycles. The presence of IOT increases the thermal resistance of the geopolymer so that there is an increase in compressive strength. The gradual replacement of fly ash up to 30% leads to a reduction in porosity and the microcrackings become much denser, resulting in increased compressive strength [7]. The compressive strength for geopolymer specimens with different alkali content shows that the compressive strength increases almost linearly with increasing alkalinity of the mixture. Increasing the alkali content of the geopolymer mixture, results in a decrease in the number and size of unreacted fly ash particles, indicating the formation of more aluminosilicate gels resulting in an increase in compressive strength [13]. Table 2 shows the compressive strength of the paste from several previous studies.

Table 2. The ratio of Na_2O/ SiO_2, SiO_2/ Al_2O_3 and H_2O/ Na_2O and the highest compressive strength

Source	Fansuri et al. [8]	Nematollahi & Sanjayan [6]	Duan et al. [7]	Pu et al. [11]	Khan et al. [12]	R. N. Thakur and S. Ghosh [13]
Na_2O/SiO_2	1.016	0.915	2.898	0.363	0.180	0.155
SiO_2/Al_2O_3	0.845	1.654	2.062	0.299	2.120	4.000
H_2O/Na_2O	0.385	12.676	2.969	2.674	10.320	8.170
Highest Compressive Strength	44.5 Mpa	96.2 MPa	50 MPa	70 MPa	103 MPa	37.27 MPa

5. Discussion

The ratio of Na_2O/SiO_2, SiO_2/Al_2O_3, and H_2O/Na_2O in the geopolymer is known by calculating the total weight of the content of each compound. After knowing the content of each in weight, it can be calculated the ratio in moles by dividing the weight of each by the atomic mass. For example, for a mixture of A-1 to find the ratio of Na_2O/SiO_2, the total weight of Na_2O and SiO_2 must first be found both from fly ash and from reactants (alkali). The total weight of Na_2O = 9.703 grams and SiO_2 = 21.563 grams. Then converted into moles by dividing the total weight of each by the mass of each atom. The atomic mass of Na_2O = 62 gram/mol and SiO_2 = 60.09 gram/mol, then Na_2O = 9.073 gram/62 gram/mol = 0.157 mol and SiO_2 = 21.563 gram/60.09 gram/mol =

0.359 mol. Furthermore, the ratio of Na_2O/SiO_2 = 0.157 mol/0.359 mol = 0.436 is obtained. The ratio of the whole mixture is based on the results of previous studies [8][6][7][11][13].

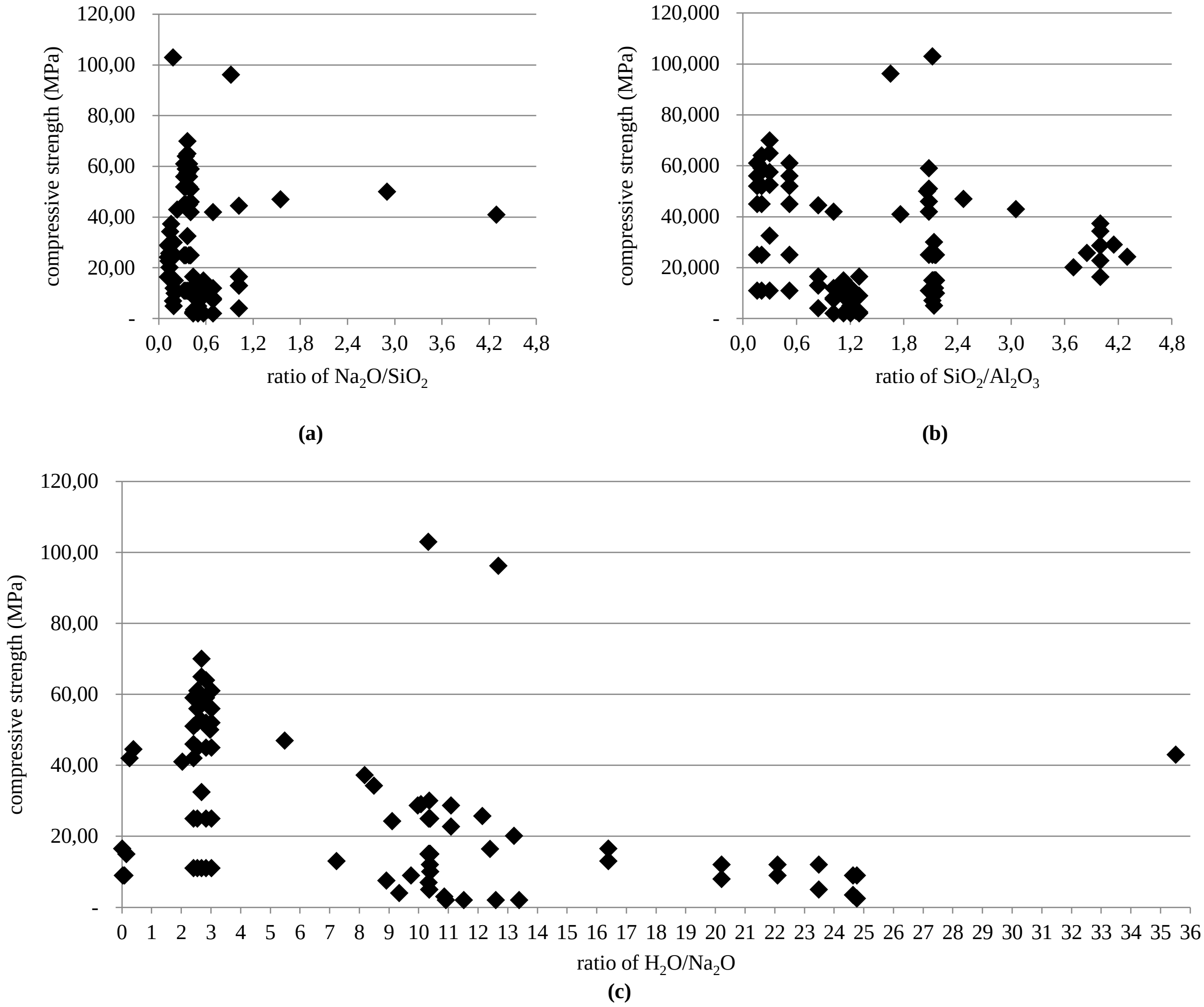

Figure 1. (a) ratio of Na_2O/SiO_2 to compressive strength; (b) ratio of SiO_2/Al_2O_3 to compressive strength (c) ratio of H_2O/Na_2O to compressive strength

Figure 1 shows, that with the ratios of Na_2O/SiO_2, SiO_2/Al_2O_3, and H_2O/Na_2O, there are some discrepancies with the intervals given by Davidovit. High compressive strength polarization occurs at more than one interval. At the Na_2O/SiO_2 ratio, it can be seen that the polarization of compressive strength above 50 MPa is concentrated at the interval of 0.321-0.405, partly scattered at the ratio of 0.18 and 0.915. In the SiO_2/Al_2O_3 ratio, it can be seen that the polarization of compressive strength above 50 MPa is concentrated at the interval of 0.162-0.522, partly scattered at the ratio of 1.654, 2.083, and 2.120. At the H_2O/Na_2O ratio, it can be seen that the polarization of compressive strength above 50 MPa is concentrated at intervals of 2.408-3.006, partly scattered at the ratio of 10.320 and 12.676. The results above show that with the same ratio of Na_2O/SiO_2, SiO_2/Al_2O_3, and H_2O/Na_2O, the compressive strength obtained is different. Davidovits limits the geopolymer alkali ratio at the following intervals [8]:

a) $0.2<Na_2O/SiO_2<0.28$;
b) $3.5<SiO_2/Al_2O_3<4.5$;
c) $15<H_2O/Na_2O<17.5$.

Based on the interval above for the Na_2O/SiO_2 ratio, the average compressive strength in that interval is 21.60 MPa, at the SiO_2/Al_2O_3 ratio, the average compressive strength in the interval is 26.715 MPa, and 14.75 MPa is the strength press the average for the interval H_2O/Na_2O ratio. Compressive strength greater than 50 MPa was found at the average ratio of Na_2O/SiO_2 = 0.382, SiO_2/Al_2O_3 = 0.695 and the ratio of H_2O/Na_2O = 3.743. This shows that the alkali ratio is not the only determinant of geopolymer performance. The highest compressive strength of the ratio of Na_2O/SiO_2, SiO_2/Al_2O_3 and H_2O/Na_2O (Table 2). However, it is not necessarily the best ratio that can be used. This is because the compressive strength value is obtained by different methods. The difference in curing temperature and fly ash used can also affect the compressive strength.

In the study of geopolymer pastes with class C fly ash, low water to fly ash (W/F) ratio could satisfy fluidity. Increasing the sodium hydroxide concentration decreases the fluidity of the mixture. In addition to the W/F ratio, the difference in curing temperature also has an effect. With a curing temperature of 70°C for 24 hours, the geopolymer paste experienced a much higher compressive strength. This is because the structure becomes denser, where the density of the paste structure contributes to the increase in compressive strength [16]. Even the composition of lime (Ca) in the geopolymer also has an effect. The addition of lime sand increases the compressive strength of fly ash based geopolymer because it can change the composition of the gel phase in the geopolymer structure and with physical strengthening due to good sand grain bonding. The compressive strength of the geopolymer paste is lower than that of the geopolymer mortar because in the gel phase the geopolymer mortar has a Ca content which is much higher than the Ca content of the geopolymer paste. The value of this parameter increases with an increasing dose of alkali and silicate. At one point, the maximum value is reached and then it starts to decrease. The compressive strength increases with increasing NaOH concentration and the mass ratio w (Na_2SiO_3)/w(NaOH), reaches its maximum value at one point and then decreases. Fly ash based geopolymer can be considered as a brittle material based on the maximum compressive strength and minimum flexural strength values. On the other hand, from the increase in compressive strength, there was a slight decrease in the flexural strength of the fly ash geopolymer. In addition, the presence of good physical bonding of the sand grains also contributes to the strength of the fly ash based geopolymer [17].

Artificial aggregate is one application of geopolymer fly ash as a building material (road pavement material). The ratio of alkaline activator between Sodium Silicate and Sodium Hydroxide used is 1.5, 2, 2.5, and 3. Aggregate is made using a granulator. The volumetric and mechanical properties of the aggregate indicate that the aggregate meets the requirements for use as a road pavement material. The weakness found is that the aggregate absorption is greater than 3% (i.e. as low as 6.1% at 2.5% alkali ratio) [5][4].

6. Conclusion

The conclusion of the discussion of the results of several studies on the use of geopolymers as building materials is as follows:

1) There is an influence of the alkali ratio on the compressive strength of geopolymer paste, but the highest compressive strength almost entirely does not follow the Davidovits rule. This is due to other factors that affect the compressive strength such as the content of elements such as Si and Al in fly ash and the curing temperature.
2) The application of geopolymer as an artificial aggregate for road materials, shows weakness in its absorption (>3%), while other characteristics meet the requirements.

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