

Fresh Properties and Early Strength of Diatomaceous Earth Based Geopolymer Pastes

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Abstract. The fresh properties of concrete are crucial for ensuring it is easy to produce and process while achieving optimal performance during and after hardening. This research investigates the fresh properties of geopolymer paste made from diatomaceous earth with molarities of 10M and 12M. The liquid-to-solid ratio of the paste is 0.6, using NaOH and Na₂SiO₃ alkali activators in a 1:1 ratio. The fresh properties tested include normal consistency, flow, density, setting time, and 3-day compressive strength. The normal consistency and flow tests show that the 10 M diatomaceous paste, with 25% water from the binder, achieves a flow diameter of 12 cm. Meanwhile, the 12 M paste, with 24% water, has a smaller flow diameter of 11.8 cm. Setting time data indicate that the 12 M paste hardens faster than the 10 M paste, reaching a higher level of hardness at 45 minutes. The 3-day compressive strength test reveals that the 10 M paste has a strength of 19.91 MPa, while the 12 M paste is slightly stronger at 20.02 MPa. The density of the 10 M paste is 1.235 g/cm³, while the 12 M paste has a higher density of 1.292 g/cm³. Overall, this research demonstrates that the molarity of the alkali solution significantly influences both the fresh and mechanical properties of the geopolymer paste, with the 12 M paste showing faster setting time, higher density, and greater compressive strength than the 10 M paste.

1. Introduction

Diatomaceous earth is known by various terms such as diatomite, tripolite, or fossil flour, and has a chemical formula of (SiO₂, nH₂O) with a high silica content. Geopolymer is an environmentally friendly cement material with a molecular network structure similar to macromolecular polymers [1-2]. In the geopolymer process, a chemical reaction occurs between aluminosilicate oxides (Si₂O₅, Al₂O₃) and alkaline polysilicate, producing Si-O-Al polymer bonds [3]. Factors such as temperature, time, and the ratio of alkali used in the mixing process will affect the strength of the geopolymer [4]. Sodium silicate (Na₂SiO₃) and sodium hydroxide (NaOH) are used as alkaline solutions to achieve good compressive strength for the geopolymer [5-6].

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Diatomaceous earth (DE) significantly affects the properties of paste, increasing viscosity, reducing flow diameter, lowering bleeding rate, shortening setting time, while also reducing heat of hydration and accelerating peak heat [7]. Diatomite as a mixing material in cement paste can increase water demand as the percentage of diatomite content rises. Initial setting time, flowability, and flexural strength decrease with higher diatomite content. The use of 10% and 20% diatomite does not result in significant changes in compressive strength. Several previous studies applied it in composite cement production [8-12], and high-performance concrete [13].

The research off fresh properties and early strength of diatomaceous earth-based geopolymer pastes aims to evaluate properties that affect the production process and performance, such as normal consistency, flow test, setting time, unit weight, and 3-day compressive strength. This testing is important to ensure ease of mixing, the ability of the paste to flow into molds, appropriate setting time, density that affects strength, and the early-stage strength development of the material, thereby optimizing composition and mixing techniques to produce high-performance geopolymer pastes. The selection of alkali molarities at 10 M and 12 M was based on technical considerations reported in previous geopolymer studies. Alkali molarities below 8 M often lead to insufficient dissolution of silica and alumina, resulting in incomplete geopolymerization and low early-age strength, while molarities higher than 14 M tend to increase paste viscosity, accelerate setting excessively, and reduce workability. Therefore, the molarity range of 10–12 M was selected as a practical and effective range to achieve a balance between workability, geopolymerization kinetics, and early-age strength development in diatomaceous earthbased geopolymer paste.

The significance of this research lies in the importance of understanding the fresh properties and early strength of diatomite-based geopolymer pastes in the context of production and performance of construction materials. This research will provide valuable data to ensure ease of mixing, optimal flow ability of the paste into molds, and appropriate setting time, as well as improve the density and strength of the material at the early stage. Additionally, this research will help optimize the composition and mixing techniques, thereby producing diatomite-based geopolymer pastes with better performance and broader application potential in the construction industry.

2. Research Methodology

2.1 Materials

Diatomaceous earth was obtained directly from Desa Beureunut, Kec. Seulimeum, Kabupaten Aceh Besar, Provinsi Aceh. At first, the diatomaceous earth was in large blocks, which were crushed with a hammer into small pieces. The diatomaceous earth pieces were dried using an oven at 150°C for 24 hours. After 24 hours of drying, the diatomaceous earth was ground using a Los Angeles Machine into fine powder, which was then sieved using a No. 200 (0.075 mm) sieve. Finally, the fine powder was calcined at 650°C for 5 hours.

2.1.1 Particle Size Analysis

Particle Size Analysis (PSA) for diatomaceous earth is important in the production of geopolymer paste because particle size affects reactivity and the rate of reaction in

geopolymerization. Finer particles with a larger surface area accelerate the reaction with alkali activators, improving the performance of the geopolymer paste. Conversely, coarser particles can affect the strength and stability of the paste. PSA also allows for the adjustment of material proportions and mixing processes to achieve optimal performance, as well as ensuring the quality and consistency of diatomaceous earth raw materials. Figure 1 shows the particle size distribution of diatomaceous earth material. Figure 1 shows the distribution of diatomaceous earth material particle size. Figure 1 shows the particle size of diatomaceous earth material. From Figure 1, it can be seen that the largest particle size that passes through is 143 μm . This indicates that most of the diatomaceous earth particles are finer, which can affect their reactivity in the geopolymerization process. This knowledge helps in adjusting the mix for optimal geopolymer paste performance.

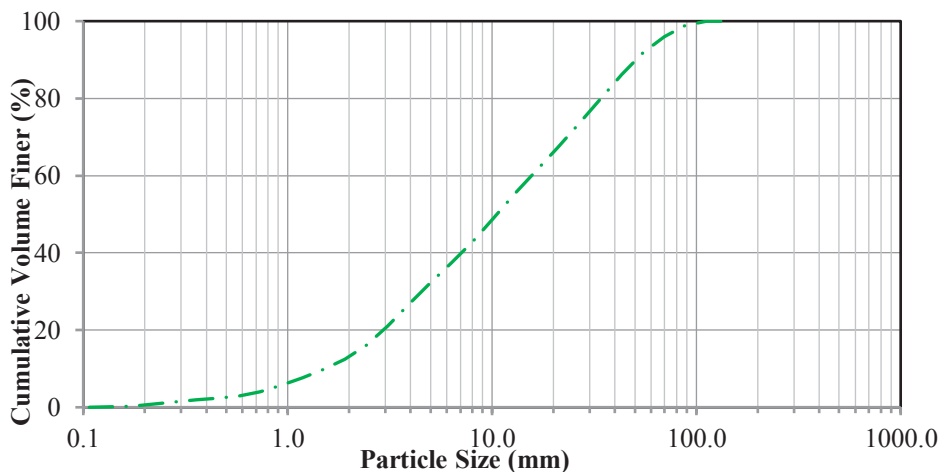


Fig.1. Particle size distribution of diatomaceous earth

2.1.2. Scanning electron microscopy (SEM) test

Morphology and texture of the processed diatomaceous earth binder were captured using scanning electron microscope (SEM) images. This study utilized a JSM-6510 LA device manufactured by JEOL. The SEM image on Figure 2 shows the microstructure of calcined diatomaceous earth at a magnification of 10,000 \times . The characteristic porous frustule structure of diatomaceous earth is clearly observed, consisting of a regular arrangement of circular and sub-circular pores distributed across the silica skeleton. This highly porous morphology results in a large specific surface area, which enhances the dissolution of silica and alumina during alkali activation and promotes geopolymerization reactions. Similar porous frustule structures of diatomaceous earth and their contribution to increased reactivity have been reported by [8]. The angular and irregular particles surrounding the frustules indicate fragmented diatomite and partially reacted phases, which are commonly observed in alkali-activated aluminosilicate systems and contribute to early geopolymer gel formation and strength development, as reported [5],[14].

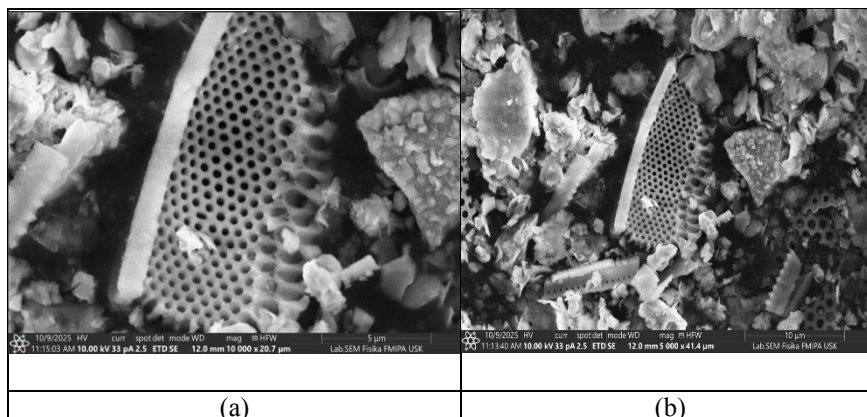


Figure 2. SEM results of diatomaceous earth at 5000x magnification (a) and 10000x magnification (b)

2.1.3 X-ray fluorescence (XRF) test

Composition of the calcined diatomaceous earth binder chemical was analyzed using X-ray fluorescence (XRF) testing, which enables precise identification of chemical elements. In this study, the XRF Analyzer TORONTOTECH TT-EDXPRT.XRF was used to perform the testing and obtain data on the elemental content of the diatomaceous earth sample.

Based on Table 1, diatomaceous earth has a lower silica content (71.4%), but a relatively high aluminum oxide (Al_2O_3) content of 8.46% and calcium oxide (CaO) content of 12.9%. Al_2O_3 acts as a reactive component in the geopolymerization process, while the significant amount of CaO can support the bond of calcium silicate hydrate (CSH), which enhances the early strength of the geopolymer paste. This combination makes diatomaceous earth more advantageous for applications requiring high mechanical properties during the early stages of hardening.

Table 1. Chemical composition of diatomaceous earth based on XRF test results.

<i>Chemical Composition</i>	
<i>MgO</i>	<i>0.685</i>
<i>Al₂O₃</i>	<i>8.46</i>
<i>SiO₂</i>	<i>71.4</i>
<i>P₂O₅</i>	<i>0.0759</i>
<i>SO₃</i>	<i>0.106</i>
<i>Cl</i>	<i>0.527</i>
<i>K₂O</i>	<i>1.29</i>
<i>CaO</i>	<i>12.9</i>
<i>TiO₂</i>	<i>0.539</i>
<i>MnO</i>	<i>0.0292</i>
<i>Fe₂O₃</i>	<i>3.89</i>
<i>NiO</i>	<i>0.0113</i>

<i>Chemical Composition</i>	
<i>ZnO</i>	<i>0.0121</i>
<i>Rb2O</i>	<i>0.0063</i>
<i>SrO</i>	<i>0.0434</i>
<i>ZrO2</i>	<i>0.017</i>

2.2 Methods

The mix design was planned by considering the paste’s liquid-to-solid ratio of 0.6, using alkali activators NaOH and Na₂SiO₃ in a 1:1 ratio. The test specimens produced have dimensions of 50 mm x 50 mm x 50 mm. The water requirement for the mixture was determined through a normal consistency test, resulting in a water content of 25% for 10 M diatom paste and 24% for 12 M diatom paste. To improve workability, 2% of the solid volume was added in the form of viscocrete or superplasticizer. This mix design aims to optimize the performance of the geopolymer paste at 10 M and 12 M molarities while ensuring good strength and stability for each variation tested.

The initial step in geopolymer paste production involves preparing all materials required for the research. The main material used in this study is diatomaceous earth, which serves as the primary binder. Before mixing the materials, the diatomaceous earth must first be calcined to remove unnecessary compounds, thereby improving the silica content in the material. Calcination can be carried out in the materials laboratory using a furnace. In addition to the binder, alkali activators are used to trigger the reaction of alkali present in the binder. This is crucial, as previous studies have shown that increasing the concentration of alkali compounds can enhance the bonding strength and compressive strength of geopolymer paste.

The alkali activators are mixed according to the NaOH and Na₂SiO₃ ratio specified in the mix design, with molarities of 10 M and 12 M. After the diatomaceous earth undergoes calcination, the material is mixed using a mixer until homogeneity is achieved. The alkali activator is gradually added to ensure all materials are thoroughly mixed. Once the mixture is ready, it is poured into molds with the planned dimensions. To enhance workability, 2% of the total solid volume is added as superplasticizer during the mixing process. The mixture is then placed into molds. After the test specimens harden, they are removed from the molds and subjected to a curing process before being tested at the age of 3 days.

3 Test result

3.1 Normal consistency and flow

The normal consistency and flow test is used to measure the workability of diatom paste, indicating how easily the paste can flow. In the 10 M diatom paste, with of 25% water by the weight of the binder, a flow diameter of 12 cm was obtained. This indicates that the 10 M paste has a more liquid consistency, making it easier to flow and apply in the geopolymer production process.

Meanwhile, the 12 M diatom paste, which contains 24% water, resulted in a smaller flow diameter of 11.8 cm. This indicates that the 12 M paste is thicker compared to the 10 M paste, with a slightly more limited flow, but still sufficient to meet the requirements of the

geopolymerization process. The normal consistency and flow test results can be seen in Figure 3.

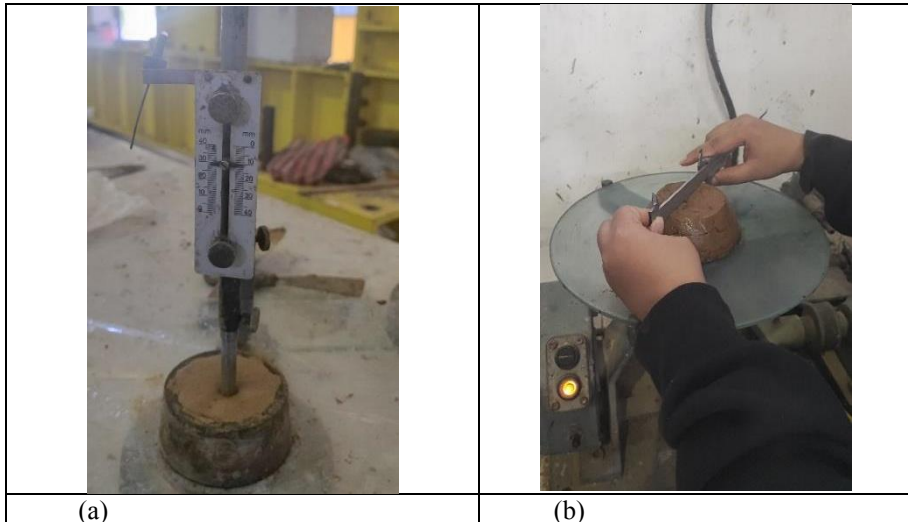


Fig 3. (a) Normal Consistency Test and (b) Flow Test

The 10 M diatom paste has a higher water content and a larger flow diameter compared to the 12 M paste due to differences in the molarity concentration of the alkaline activators, NaOH and Na₂SiO₃. The lower molarity in the 10 M paste (compared to the 12 M paste) means that less alkali is used, so more water is needed to achieve optimal viscosity and workability. The flow test results indicate that alkali activator molarity significantly influences the workability of diatomaceous earth-based geopolymer paste. Experimentally, the 10 M paste, with a water content of 25% by binder weight, achieved a larger flow diameter of 12 cm, whereas the 12 M paste, containing 24% water, exhibited a slightly lower flow diameter of 11.8 cm. This reduction in flowability at higher molarity is attributed to the increased concentration of alkaline ions, which accelerates the dissolution of silica and alumina and promotes early gel formation, thereby increasing paste viscosity. The highly porous nature of diatomaceous earth further intensifies water absorption, reducing free water available for lubrication and flow. Similar observations regarding reduced workability at higher alkali molarity in alkali-activated and geopolymer systems have been reported by [5-6]. Despite the reduced flow diameter, the 12 M paste still exhibited adequate workability for casting, indicating that the selected molarity range provides a practical balance between flowability and early geopolymerization activity.

3.2 Setting Time

The setting time data shows that the 12 M paste hardens faster than the 10 M paste, achieving a significantly higher level of hardness in just 45 minutes. This indicates that the higher molarity of the alkali activator in the 12 M paste accelerates the geopolymerization process, leading to faster development of mechanical strength and structural stability. The setting time test for 10 M and 12 M can be seen in Figure 4a.

Figure 4b shows the needle penetration depth at different time intervals for 10 M and 12 M pastes. At 15 minutes, the needle penetration depth for the 10 M paste is 40 mm, while for the 12 M paste it is 22 mm. At 30 minutes, the needle penetration depth decreases to 21 mm

for the 10 M paste and 16 mm for the 12 M paste. Finally, at 45 minutes, the needle penetration depth for the 10 M paste reaches 15 mm, while for the 12 M paste it reaches 10 mm. The decrease in needle penetration depth indicates that the geopolymer paste has started to harden at 45 minutes. The differences in setting time and early-age compressive strength observed in this study are strongly governed by geopolymerization mechanisms associated with alkali activator molarity. Experimentally, the 12 M geopolymer paste exhibited a faster setting behavior than the 10 M paste, as indicated by a lower needle penetration depth of 10 mm at 45 minutes compared to 15 mm for the 10 M paste. This accelerated stiffening is attributed to the higher concentration of hydroxide ions at 12 M, which enhances the dissolution of silica and alumina from the diatomaceous earth and promotes rapid formation of aluminosilicate gel networks. Similar effects of alkali concentration on reaction kinetics and setting behavior have been reported by [4-5].

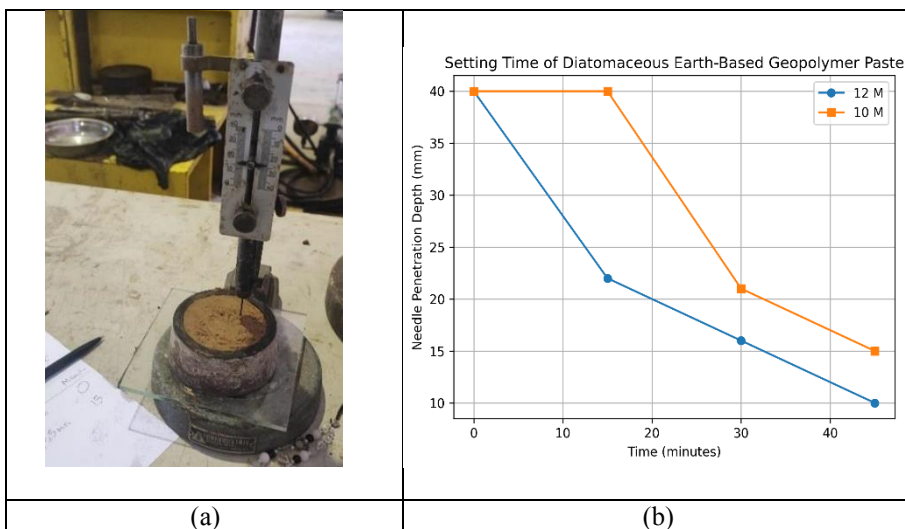


Fig 4. (a) Setting Time Test and (b) Setting Time Test Results

The difference in setting times between the 10 M and 12 M pastes can be explained by the variation in the concentration of the alkaline activators, namely NaOH and Na₂SiO₃. In the 12 M paste, the higher molarity means there are more alkaline ions available to trigger the geopolymerization reaction. These alkaline ions accelerate the hydration process and the formation of bonds between silica and alkali, which in turn speeds up the hardening of the paste. In contrast, the 10 M paste, with its lower molarity, has fewer available alkaline ions, causing the reaction to proceed more slowly and the paste to take longer to set. This process is reflected in the needle penetration test, where the 12 M paste shows a faster decrease in penetration depth, indicating that this paste hardens more quickly compared to the 10 M paste.

3.3 Compressive Strength and Density

In Table 2, the results of the compressive strength and bulk density tests of 3-day diatomaceous earth paste show that the paste with a 10 M concentration has a compressive strength of 19.91 MPa, while the paste with a 12 M concentration has a compressive strength of 20.02 MPa. Table 1 also presents the density data of the paste, recorded as 1.235 g/cm³ for the 10 M paste and 1.292 g/cm³ for the 12 M paste, indicating differences in composition and material structure between the two types of paste.

Table 2. Compressive Strength and Density of 3-Day Diatomaceous Earth Paste

Property	Pasta 10 M	Pasta 12 M
Compressive Strength (3 days)	19.91 MPa	20.02 MPa
Density	1.235 g/cm ³	1.292 g/cm ³

The difference of compressive strength and density in the 12 M diatomaceous earth paste compared to the 10 M paste is likely caused by the higher concentration's effect on the bonding between particles in the paste. A higher concentration may increase the material's density, resulting in stronger particle bonds, which in turn enhances compressive strength. Additionally, the higher concentration may influence particle distribution and porosity, leading to a more compact structure that is more resistant to pressure. In terms of mechanical performance, the 3-day compressive strength of the 12 M paste reached 20.02 MPa, slightly higher than the 19.91 MPa recorded for the 10 M paste. This improvement is consistent with the higher bulk density measured for the 12 M paste (1.292 g/cm³) compared to the 10 M paste (1.235 g/cm³), indicating a denser and more compact geopolymer matrix. The increased density reflects improved particle packing and gel formation, which enhances load transfer and early-age strength development. These findings align with previous studies reporting that higher alkali molarity contributes to matrix densification and improved early strength in alkali-activated materials [14-15].

4. Conclusion

Based on the research, the following conclusions can be drawn:

- The 10 M diatom paste, with the addition of 25% water by the weight of the binder, has a more fluid consistency, making it flow more easily with a flow diameter of 12 cm. This indicates that the 10 M paste is easier to apply in the geopolymerization process because it requires more water to reach optimal viscosity.
- The 12 M diatom paste, which contains 24% water, is thicker with a flow diameter of 11.8 cm. Although less water is needed to achieve the desired consistency, the 12 M paste still has sufficient workability for the geopolymerization process, as the higher molarity of the alkali activator makes the paste more dense.
- The 12 M paste hardens faster compared to the 10 M paste, reaching a higher hardness level in just 45 minutes, indicating that the higher molarity of the alkali activator accelerates the geopolymerization process and the development of mechanical strength.
- The difference in setting rates between the 10 M and 12 M pastes can be explained by the difference in alkali activator concentration, where the 12 M paste, with its higher molarity, shows a faster reduction in needle penetration depth, indicating a faster hardening process compared to the 10 M paste.
- The diatomaceous earth paste with a 12 M concentration has higher compressive strength and density compared to the 10 M paste, indicating that an increase in alkali activator concentration can strengthen the bonds between particles and improve the material's quality.

- f) The increase in compressive strength and density in the 12 M paste is likely due to the higher concentration's effect on the material structure, which results in stronger bonds and a denser particle distribution, thereby enhancing resistance to pressure.

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