

Geopolymerization: A Review on Physico-chemical Factors Influence to the Reaction Process

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Abstract

A geopolymer is one of materials belonging to an inorganic polymer group based on aluminosilicate structural networks. The material is a product of reactions among the activated aluminosilicate resources and solutions of alkaline activators in various conditions. There have been many investigations conducted to produce geopolymer using different raw materials and methods. The chemical factors have a decisive role in the reaction process and the formation of the geopolymeric structural networks. The physical factors significantly influence on engineering properties, microstructural characteristics as well as the effectiveness of the reactions. This study is a review on evaluation of relationships among physico-chemical factors in the reaction process. In which, the chemical factors were considered both the aluminosilicate raw materials and alkaline activators. The physical factors were evaluated on the various conditions known as mixing time, curing time and curing temperature.

Keywords: Aluminosilicate structural networks, Geopolymerization, inorganic polymer, physico-chemical factors, solutions of alkaline activators

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INTRODUCTION

A geopolymer is the term coined by Joseph Davidovits in the 1970s for the new inorganic polymer materials he developed [1]. Geopolymers consist mainly of amorphous or semi-crystalline phases from the initial geopolymeric reactions and the formation of polymeric $-\text{Si}-\text{O}-\text{Al}-\text{O}-$ bonds [2,3]. Davidovits' first studies were carried out between aluminosilicate kaolinite and NaOH at 100–150°C resulting into the formation of atecto-aluminosilicate with structure [1] and bonds as shown in Figure 1.

Studies have been done to understand further the geopolymerization reaction [1–8]. Geopolymerization was then understood as a chain of reactions between different aluminosilicate oxides with silicates in high-alkali environment.

Although Jaarsveld et al. [4] proposed that geopolymerization can proceed from any aluminosilicate sources, more studies have been reported indicating that the geopolymerization only proceeds with amorphous or semi-crystalline Al-Si materials [1, 5–8].

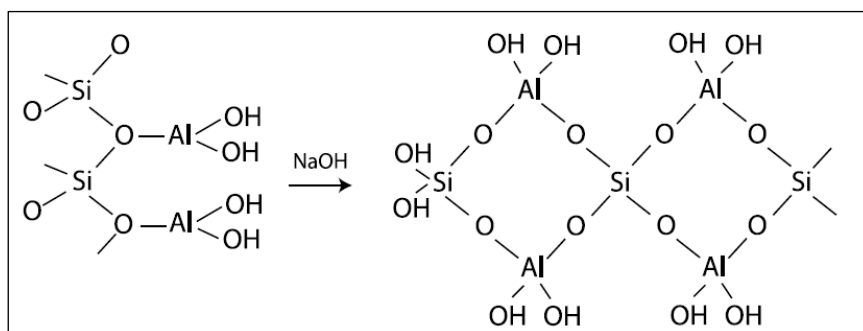
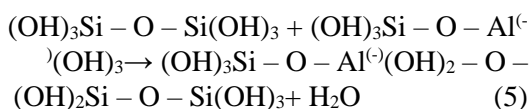
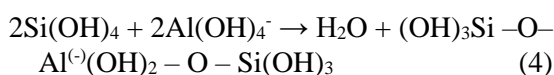
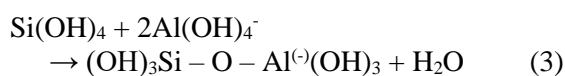
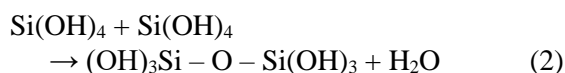
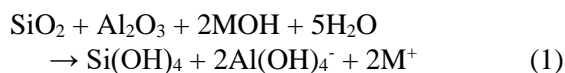


Fig. 1: Structure of First Geopolymeric Generation in Alkali Condition [1].

Reactions were described by the formation of every oligomer until the oligomer species combine together into a final geopolymeric product. These detailed reactions were illustrated as follows [9, 10], where M is either sodium (Na) or potassium (K).



First, alumino-silicates (SiO_2 and Al_2O_3) are dissolved in alkaline solution and allowed to react, forming tetra-silicate (silicic acid, $\text{Si}(\text{OH})_4$) and tetra-aluminate $[\text{Al}(\text{OH})_4]^-$ products (reaction (1)). Further reaction of these product forms oligomer precursors such as sialate ($-\text{Si}-\text{O}-\text{Al}-\text{O}-$) (reaction (3)), sialate-siloxo ($-\text{Si}-\text{O}-\text{Al}-\text{O}-\text{Si}-\text{O}-$) (reaction (4)) and sialate-disiloxo ($-\text{Si}-\text{O}-\text{Al}-\text{O}-\text{Si}-\text{O}-\text{Si}-\text{O}-$) (reaction (5)). Combinations of these oligomer precursors form alumino-silicate geopolymer networks [1, 5, 9–11].

Davidovits [12] showed that the products from geopolymerization can be classified into five systems. This classification is based on various $\frac{\text{Si}}{\text{Al}}$ ratios as summarized in Table 1.

EFFECTS OF RAW MATERIALS TO FORMATION OF POLYMERIC NETWORKS

Davidovits [1] reported that the required materials for geopolymerization must be high silica and aluminum in amorphous forms, which form polymeric networks under alkaline condition as illustrated in the following Figures 2 and 3.

In the research done by Kumar et al., calcium was determined that it also reacts to form geopolymeric structure similar to mortar and concrete structures in cement hydration process such as C–S–H, A–S–H, C–A–S–H (with C = CaO, S = SiO_2 , A = Al_2O_3 and H = H_2O) [13, 14]. These materials are named in high calcium alkaline activated materials which have the ratio of Ca/Si in the range of 0.85 to 1.8 for formation of C-S-H and C-A-S-H gels, and the ratio of Na/Si over 0.25 for formation of C-N-S-H and C-N-A-S-H gels [5]. All of these gel structures change the mechanical strength of geopolymer product. Hence, the various ratios between oxides shown in Table 1 will make different structures and affect the technical properties of the products.

Particle size distribution (PSD) of raw materials influences the rate and efficiency of geopolymerization. Similar to theories of ordinary chemical reactions, the finer the powder is, the more rapid the rate of reaction is [7]. This is clearly shown through strength values of product samples after finishing the whole reaction process. The finer the powder is, the higher compressive strength values are. In most of the studies in geopolymer, particle sizes of raw materials are set similar to cement

Table 1: Various Ratios between Si and Al, and their Applications [12, 16,17].

The mole ratios of $\frac{\text{Si}}{\text{Al}}$	Structures	Applications
20:1 < Si:AlSi:Al < 35:1	2D cross-link	Fire resistant and heat resistant fiberComposites
3:1 < Si:Al	Polymeric character	Sealant for industry (200°C < use < 600°C, tools for aeronautics SPF aluminum
Si:Al = 3:1		Fire protection, fiberglass composite, foundry equipment, heat resistant composites (200°C < use < 1000°C)
Si:Al = 2:1	3D network	Low CO ₂ , cements and concretes, radioactive and toxic waste, encapsulation
Si:Al = 1:1		Bricks, ceramics, fire protection

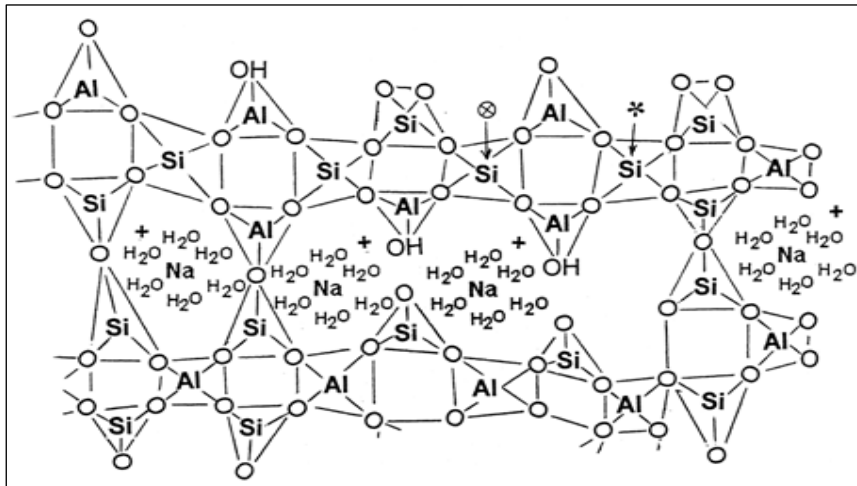


Fig. 2: Structure of Geopolymer in Na-geopolymer System with Semi-schematic [14].

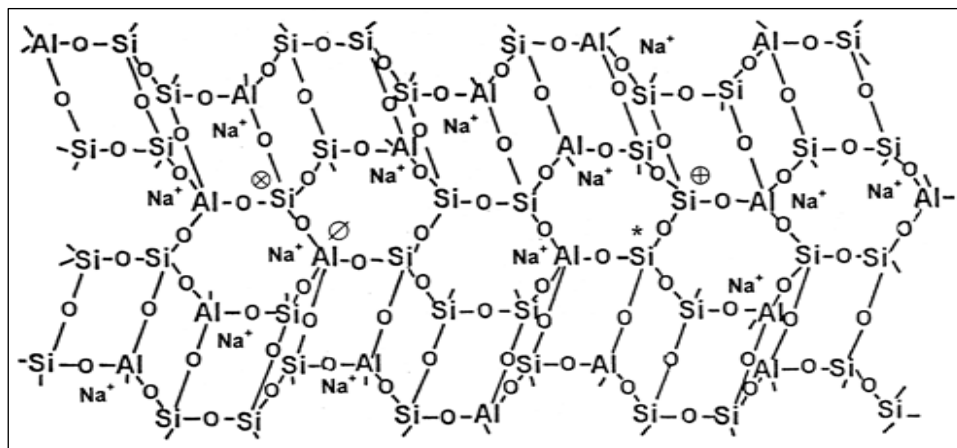


Fig. 3: Structure of Geopolymer in Na-geopolymer System with Three-dimensional Framework [14].

standards to ensure complete hydration reaction [15]. For ASTM C115 [15], cement PSD must have 100% particles under 80 μm and over 90% particles under 50 μm , with minimum PSD of 7.5 μm [15]. In related research, Allahverdi et al. [18] showed the mean pozzolan PSD of 18.5 μm , while Zhang and He [7] average PSD was under 38 μm for most silicate materials. These values were similar to Kumar et al. [13].

EFFECTS OF ALKALINE ACTIVATORS TO GEOPOLYMERIZATION REACTIONS

Alkaline activators are compounds formed between anions ($[\text{OH}]^-$, $[\text{SiO}_3]^{2-}$, $[\text{CO}_3]^{2-}$, $[\text{SO}_4]^{2-}$) and cations of alkali metals (Li^+ , Na^+ , K^+ , Rb^+ , Cs^+ , Fr^+), and two commonly used cations are sodium (Na^+) and potassium (K^+)

[1, 5, 16]. Normally, the alkaline activators increase the pH value when they are dissolved into solutions. And at high pH (>12), aluminosilicates (SiO_2 and Al_2O_3) are easily dissolved and reacted to form tetra-aluminate ($[\text{Al}(\text{OH})_4]^-$) and tetra-silicate ($\text{Si}(\text{OH})_4$ or H_4SiO_4 -acid silicic) [1, 5, 11, 19, 20].

Based on the anions, Provis and Deventer [5] classified the alkaline activators into four groups: alkali hydroxides, alkali silicates, alkali carbonates, and alkali sulfates. The alkali hydroxides and alkali silicates are commonly used in geopolymeric research because they are cheaper than others and produced in a large amount all over the world [1,5]. For the cations, there has been no study carried out to form the aluminosilicate geopolymeric networks from alkali activators of rubidium (Rb^+), cesium (Cs^+), and francium (Fr^+) because these compounds

are very expensive and scarce. Hence, this study only focused on discussion to the alkali activators of lithium (Li^+), sodium (Na^+), and potassium (K^+).

The alkali hydroxides include LiOH , NaOH , KOH , RbOH , and CsOH (FrOH is not reported because of its scarcity). The LiOH has the lowest solubility in comparison with the others (at 25°C , under $5.4 \text{ mol LiOH/kg H}_2\text{O}$, over 20 mol NaOH or $\text{KOH/kg H}_2\text{O}$ [21]). The low solubility of LiOH decreases dissolution of SiO_2 and Al_2O_3 in solution and cause low effectiveness of geopolymerization reactions [1, 5]. Moreover, LiOH as well as RbOH and CsOH are of limited large-scale production because of the cost and their scarcity. Both NaOH and KOH are produced by electrolysis of chlor-alkali solutions in industrial process which consumed a large amount of electricity. Both the productions of alkali hydroxides and electricity impact strongly and negatively to environment related to greenhouse emissions of Cl_2 and CO_2 [5]. There are two biggest undesired problems, the high temperature of the solution and efflorescence, when high concentration of alkali hydroxides is dissolved in solution. For high temperature solution, it is reported that 10 mol of NaOH dissolved in a liter of water can release 90% of heat (around 400 kJ) and increase the temperature of the solution over 90°C [22, 23]. The high temperature of solution in geopolymeric mixtures causes hydrothermal reactions to form zeolite crystals (zeolite crystals are formed over 50°C [24] as well as microcracks and macrocracks due to thermal stress gradient. Both phenomena are undesirable to engineering properties of copolymer products, which result in low compressive strength and high water absorption [1, 5, 24,25]. For the efflorescence, this is related to the formation of white carbonate or bicarbonate crystals due to the reaction between the alkali and atmospheric CO_2 on the sample surface. This phenomenon can be harmful to the geopolymeric structures in a long time, which is similar to the carbonation in cement-based materials [1, 5]. Moreover, geopolymers from the alkali hydroxides have lower heat and fire resistance than the geopolymers from the alkali silicates [1, 13].

The alkali silicates include sodium and potassium silicates, which have the greatest amount of industrial production as alkaline activators [5]. Lithium silicate with low solubility as well as rubidium and cesium silicates are high costs and the limited industrial production. Therefore, these compounds are not used commonly in research and industry. The sodium and potassium silicates are produced from calcination of carbonate salts and silica and then dissolved in water with the desired ratios. This process also consumes the energy and emitted CO_2 but total energy consumption and CO_2 emission are much lower than Portland cement production [5, 26–28].

Figure 4 showed a portion of the $\text{Na}_2\text{O}.\text{SiO}_2.x\text{H}_2\text{O}$ composition space with crystallization isotherms at 25°C . It showed a more detailed, complex phase relationship at lower Si content, which illustrated the potential formation of more siliceous phases observed in hydrothermal geological deposits. In high alkali solution, the silica (SiO_2) is dissolved and reacted to form silicate oligomer that is monomeric silica, $\text{Si}(\text{OH})_4$, which is also known as “orthosilicic or silicic acid” [5]. Therefore, the alkali silicate solutions have lower pH value ($12\text{--}13.5$) than the alkali hydroxides but an amount of silicate oligomer precursor in alkali silicate solutions will rapidly dissolve and react the solid alumino-silicate resources (Al_2O_3 and SiO_2) in the geopolymer mixtures [1, 5, 11, 16].

Alkali carbonates are compounds of sodium and potassium with carbonate ($[\text{CO}_3]^{2-}$) and hydro-carbonate ($[\text{HCO}_3]^-$) that were not commonly used because the alkali carbonate solutions cause low pH value for geopolymer mixtures, high CO_2 emission that directly affects the structure of the geopolymer (high porosity but high water absorption and low compressive strength), and carbonation problems. The other alkali carbonates from lithium, rubidium and cesium are rarely reported due to high cost and scarcity of these compounds. The geopolymer products are also known for having low heat and low fire resistance at high temperature [1, 5, 13].

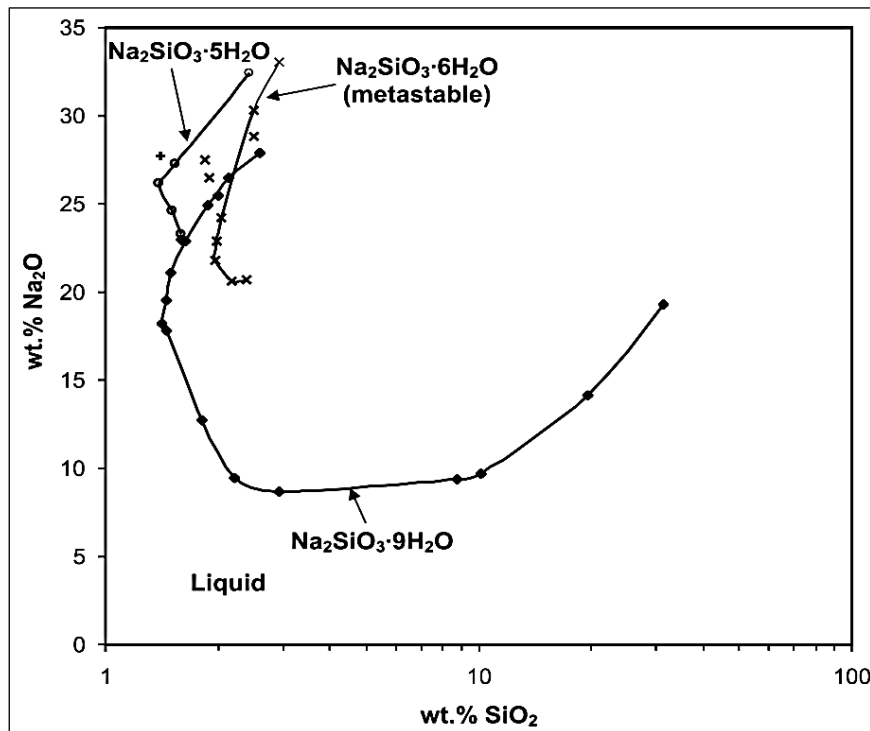


Fig. 4: Crystallisation Isotherms for Hydrated Sodium Metasilicate Phases at 25°C [5].

Alkali sulfates are composed of sodium or potassium and sulfate ($[\text{SO}_4]^{2-}$) that are used in geopolymerization but limited in research application because the alkali sulfate solutions cannot bring a high pH value for geopolymer mixtures. There are many complex reactions related to formation and deformation of anhydrous salts (mirabilite- $\text{Na}_2\text{SO}_4 \cdot 10\text{H}_2\text{O}$, thernadite- Na_2SO_4 , metastable heptahydrate- $\text{Na}_2\text{SO}_4 \cdot 7\text{H}_2\text{O}$) among alkali (Na^+ , K^+), sulfate ($[\text{SO}_4]^{2-}$), and aluminosilicate resources (Al_2O_3 and SiO_2) in the geopolymer mixtures [5].

Among the alkali silicates, the sodium silicate solution or water glass solution has more advantages than potassium silicate solution. The sodium silicate solution is more commonly produced and used than the potassium silicate solution [5]. When the potassium silicate is dissolved in solution, formation of KOH causes higher standard enthalpy dissolution (around 60 kJ/mol) than dissolution of the sodium silicate (around 45 kJ/mol) [5, 22, 23]. Therefore, the use of high concentration sodium silicate solution could increase the temperature of the geopolymer mixtures, which negatively affects the geopolymerization reactions as well as the

hardening process of geopolymer products similarly to increase temperature of solution when used to high concentration of the alkali hydroxides [1, 5].

EFFECTS OF THE RATIOS AMONG H_2O AND M_2O , Na_2O AND SiO_2

The foundation of geopolymerization method emphasized the role of SiO_2 and Al_2O_3 in the formation of aluminosilicate networks. However, Davidovits [1, 12] mentioned that alkali and water are also two necessary factors that directly affect to the geopolymeric reactions. Many investigations focused on these problems to develop and find new solutions for this research field.

Komnitsas and Zaharaki [9] summarized previous investigations and listed in their study about the range of values between oxides ratios used to synthesize geopolymers. The results were used for the production of geopolymeric cements, concretes and ceramics, and they were entirely based on the previous studies as well as the geopolymerization principles of Joseph Davidovits [5, 9, 10, 12, 16]. The ratios are shown as the Table 2.

Table 2: Ratios of Oxides for Geopolymerization [8, 17, 19,29–31].

Mole Ratios	Range of Value
SiO ₂ : Al ₂ O ₃	3.30–4.50
M ₂ O : SiO ₂	0.20–0.48
M ₂ O : Al ₂ O ₃	0.80–1.60
H ₂ O : M ₂ O	10–25

Davidovits as well as Hardjito and Rangan and Provis and Deventer [1, 5, 29] suggested the best mole ratios between oxides to synthesize geopolymer systems like as mortar and concrete are limited in a range of values as follows:

$$0.2 < \frac{\text{Na}_2\text{O}}{\text{SiO}_2} < 0.28,$$

$$\text{and } 15 < \frac{\text{H}_2\text{O}}{\text{Na}_2\text{O}} < 17.5$$

$$\text{or } 10 < \frac{\text{H}_2\text{O}}{\text{Na}_2\text{O}} < 25$$

The amount of alkali and water added to the mixture increases the pH value, and all investigations showed that this value should be over 12 [1, 5, 16].

EFFECTS OF MIXING TIME AND CURING REGIMES TO PROPERTIES OF GEOPOLYMER

This section presents effects of mixing time and curing regimes to properties of geopolymer.

Effects of Mixing Time

Mixing process affects the distribution of reacted materials in mixture statistically. If the particles of the reactants are distributed evenly and alternately, reactive component diffusion and reaction will occur rapidly and there will be high effect for properties of product in whole geopolymerization process. The longer the mixing time is; the higher strength value of product is [29]. Hardjito and Rangan studied mixing time in two ways: continuous mixing and discontinuous mixing, results of compressive strength of 21-day samples increased from 37 MPa to 51 MPa when mixing time of the mixture was changed from 2 minutes up to 16

minutes, respectively in the case of discontinuous mixing. For continuous mixing, 3-day curing samples, there is an increase of compressive strength for mixing time of 4 and 16 minutes ranging from 40 to 55 MPa, respectively [29].

Effects of Curing Time

According to Davidovits [1], curing require time for reactions proceed completely in geopolymeric mixture. For hydration reaction, curing time is a long time; it can be 1 hour, one day, one month or even all year. For curing time considerations, there are standards in material engineering, ASTM C109/C109M–99 [32]. Samples must be tested for mechanical strength after 24 hours, 3, 7, 28 and 90days. Mostly, research for geopolymers apply this standard to check strength of samples.

However, curing time can be shortened by interactions with a reactive environment such as controlling temperature, pressure and humidity. The research of Yunsheng [33] is an example with results shown in Figure 5. Although curing regimes such as at high temperature (around 100°C), in an autoclave, and in steam condition can obtain the results of the higher strength values, these conditions require more energy consumption or the geopolymerization process to take place.

Effects of Curing Temperature

The temperature of the processes significantly affects geopolymerization reactions. The geopolymer cured in high-temperature condition will obtain better strength in comparison with reference samples. Hardjito and Rangan [29] researched about the effects of curing temperature to compressive strength of 7-day samples. When studied, temperature increased from 25–90°C linearly, compressive strength values also raised from 20 to 70 MPa [29]. There have been so many related investigations affirming the role of curing temperature on geopolymeric properties [1, 12, 15, 31–36].

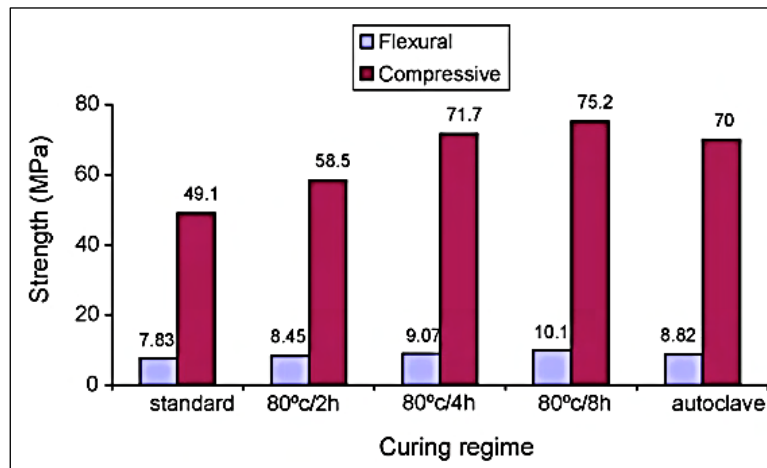


Fig. 5: Effect of different Curing Regime to Strength [33].

CONCLUSION

The investigations of geopolymer materials have brought many useful solutions for the production process in the future. Because the inorganic polymer materials have a lot of outstanding engineering properties compared to the same traditional products. More specially, the raw materials with high activated aluminosilicate resources can be reused from industrial solid wastes or agricultural residues.

The evaluation of the factors influencing the reaction conditions as well as the product specification will bring various aspects to the geopolymer production efficiency. More importantly, the reaction conditions need to be selected to not only synthesize the high quality geopolymer but also bring about energy saving efficiency as well as simplifying the process of technology.

Nomenclature

PSD : Particle Size Distribution
 ASTM : American Society for Testing and Materials
 v : Velocity

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