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Clay-Based Geopolymer Material: A Review

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Abstract

This paper provides an in-depth review of the fundamentals of geopolymers, with a focus on clay materials. It covers the chemistry, structure, and reaction mechanisms involved in geopolymer formation, highlighting key materials such as aluminosilicates and alkali reactants. The review delves into the characterization of geopolymers, addressing microstructural, crystallographic, and functional aspects. It critically examines important factors affecting geopolymer properties, including alkali concentration, mixing parameters, curing conditions, and water content. Understanding these parameters ensures the effective synthesis of high-quality geopolymers. Furthermore, the paper explores past successful applications of geopolymers and outlines potential future research areas and applications.

Keywords: Clay, Geopolymers, Aluminosilicate, Alkaline Activators.

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1. Introduction

This review indicates the properties of geopolymer materials and structures derived from clay sources. Geopolymers are inorganic polymers synthesized through the reaction of aluminosilicate with a concentrated alkali hydroxide or alkaline silicate solution. The mixture is then subjected to a curing process at room temperature or slightly elevated temperatures ranging from 20 to 100°C. The process through which geopolymers form is known as the geopolymerization reaction. In essence, the formation of geopolymers involves combining solid and liquid components Fig. 1 shows this formation. Solid constituents comprise powdered aluminosilicate materials, which possess a suitable proportion of highly reactive silica and alumina. Simultaneously, the solution exhibits a significant concentration of alkaline properties.

2. Terminology and Structure of Geopolymers

Davidovits introduced the geopolymer word for the first time. The prefix "geo" denotes the aluminosilicate originating from geological sources, that undergoes a polycondensation reaction with an alkaline solution which produce a binder; geopolymer structures can be divided into three main types—poly (sialate), poly (sialate-siloxo), and poly (sialate-disiloxo)—based on their Si/Al ratios, depicted in Fig. 2[1].

Geopolymers exhibit a predominantly amorphous to semi-crystalline architecture defined by intricate three-dimensional Si-O-Al polymeric frameworks. The SiO_4 and AlO_4 tetrahedra are alternately connected by subscription of all oxygen atoms, with aluminum in a four-fold (IV) harmony [2]. This configuration results in a negative charge on Al in IV coordination, which is equilibriumed by cations such as Na^+, K^+, Li^+, Ca^{2+}, Ba^{2+}, NH^{4+}, and H_3O^+. Being of these cations is essential for maintaining the neutrality of the structure [3]. Furthermore, it is asserted that the inclusion of cations is crucial not only for charge equilibrium but also for the structural integrity of the resultant product. Na^+ ions result in how fragile geopolymers are Saidi et al.[4].

The geopolymer formula is represented as follows:

$$M_n\{-(SiO_2)_z\text{-}AlO_2\}.\ wH_2O \qquad 2.1$$

Where M represents a cation like K^+, Na^+, or $Ca^{2+;}$ n denotes the level of polycondensation; z can be 1, 2, or 3; and w indicates the quantity of bound water.

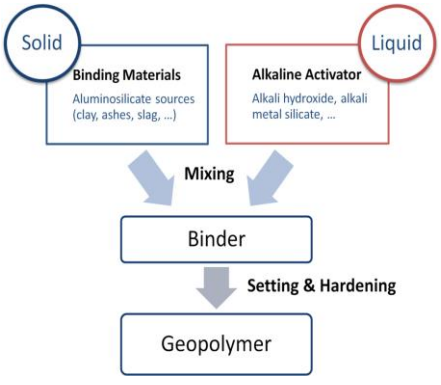


Fig. 1. Schematic diagram of geopolymer formation.[5]

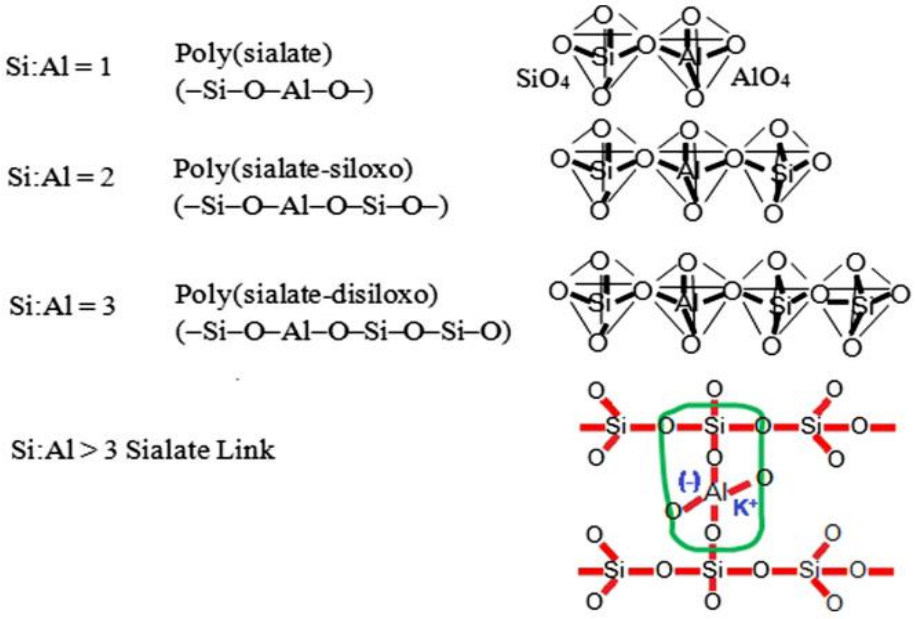


Fig. 2. Different geopolymer systems based on the number of siloxo SiAO units[1].

3. Aluminosilicate sources

Geopolymers have been synthesized using a diverse range of raw materials, beginning with kaolinite in early research [4], [6], [7], and later incorporating calcined clays [8], [9], industrial byproducts such as ashes [10], [11], [12], slag [13], [14], waste glass [15], copper mine tailings [16]. Along with various natural and synthetic silicoaluminates including zeolite [17], pure Al_2O_3–$2SiO_2$ powder [18], and magnesium-rich minerals.

Primary raw materials for producing geopolymers are aluminosilicates, which are abundant in alumina (Al_2O_3) and silica (SiO_2) and are widely available on the earth. These materials are crucial for geopolymer formation, providing essential Al^{3+} and Si^{4+} ions for the binding system. Ideally, the Al_2O_3 and SiO_2 compounds in the source material should be in a reactive amorphous phase [19] and constitute more than 70% of the total composition. This paper deeply reviewed the role of clay and clay minerals in the synthesis of geopolymers, offering a detailed analysis of their structural characteristics and intrinsic properties. It explores how these materials contribute to the overall formation and performance of geopolymers.

4. Structure of clay minerals

Kaolinite is the most commonly utilized clay mineral in the synthesis of geopolymers. Kaolinite features a 1:1 uncharged dioctahedral layer structure Fig. 3a with the chemical formula $Al_2O_3 \cdot 2SiO_2 \cdot 2H_2O$. This structure consists of a $(Si_2O_5)_n^{2-}$ sheet and an $Al(OH)_3$ sheet, which are interconnected with shared oxygen atoms. The sheets are bound by van der Waals forces and hydrogen bonds, creating a layered configuration illustrated in Fig. 3b, characterized by negligible charge and limited elemental substitution due to their stacking. Therefore, kaolinite exhibits a reduced surface area for the geopolymerization

procedure relative to fly ash, which is characterized by its spherical particle morphology. The constrained surface area of kaolinite induces reduced dissolution by the alkaline reactant, consequently diminishing strength. [20]. Xu and van Deventer [21] explored the use of 16 natural aluminosilicate minerals precursor materials for geopolymer formation (including almandine, andalusite, pumpellyite, augite, lepidolite, illite, spodumene, sodalite, hydroxyapophyllite, stilbite, kyanite, heulandites, celsian, grossular, anorthite, and sillimanite,).To achieve the desired strength parameters, certain clays require the addition of a supplementary material, which may be kaolinite. Among all minerals analyzed, stilbite exhibited the greatest strength at 18 MPa in compression; furthermore, Van Jaarsveld et al [22] discovered, the inclusion of kaolinite (41%) by weight in fly ash-based geopolymers adversely affected the final result. Calcinid kaolinite leads to an enhancement in compressive strength Table 1.

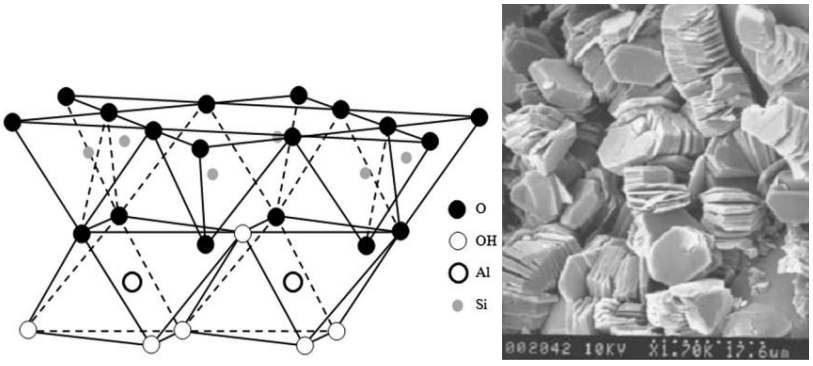


Fig. 3. (a) Structure of kaolinite and (b) microstructure of kaolinite [23].

Table 1. Calcination conditions and its corresponding compressive strength of fly ash-kaolinite geopolymers with a clay content of 14 wt%, water/fly ash mass ratio of 0.31, and K2O/SiO2 ratio of 0.57 [22].

Time (h)	Temperature (C)						
	300	400	500	600	700	800	900
1	14	14	30	54	17	7	8
6	11	31	13	9	15	7	15
12	12	47	28	25	14	3	15
24	6	32	31	18	6	15	12

5. Pre-treatment of clay minerals

The characteristics of geopolymers are greatly affected by the clay's pre-treatment, as evidenced by the distinctive traits of geopolymers derived from thermally treated halloysite, whereas those produced from mechanochemically treated halloysite exhibited a less complete geopolymerization process [24]. Chemically treated halloysite in acidic conditions yielded suboptimal geopolymers, whereas alkaline treatment exceeding three hours fostered crystalline zeolite formation that strengthened during geopolymer synthesis; generally, geopolymers derived from thermally activated precursors like metakaolin, fly ash, and blast furnace slag demonstrate enhanced mechanical properties

[21], [25], [26]. Upon dehydroxylation of kaolinite, the tightly bound hydroxyl ions in the aluminum-rich layer release water at temperatures between 550 and 800°C, while maintaining a layered structure, as illustrated in Fig. 4. The calcination temperature for kaolinite typically lies between 600 and 900°C, [27] with an optimal temperature reported at 900°C; concurrently, XRD analyses by Zuhua [23], reveal that increasing temperature weakens kaolinite's crystalline peaks, signifying structural degradation Fig. 5. Ferone et al. [28] demonstrated that thermal treatment of clay sediments at 400°C and 750°C enhances dissolution, with the latter yielding greater compressive strength, thus corroborating the assertion that elevated temperatures increase surface area and facilitate subsequent geopolymerization reactions.

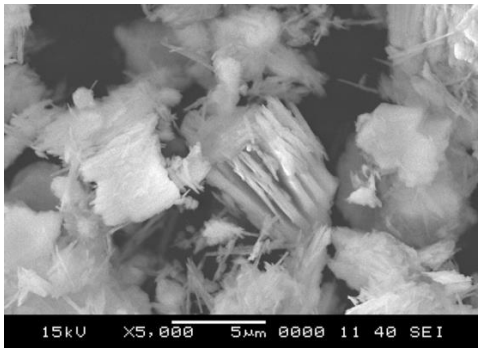


Fig. 4. SEM micrograph of metakaolin calcined at 800 C for 2 h at magnification 5000 [29].

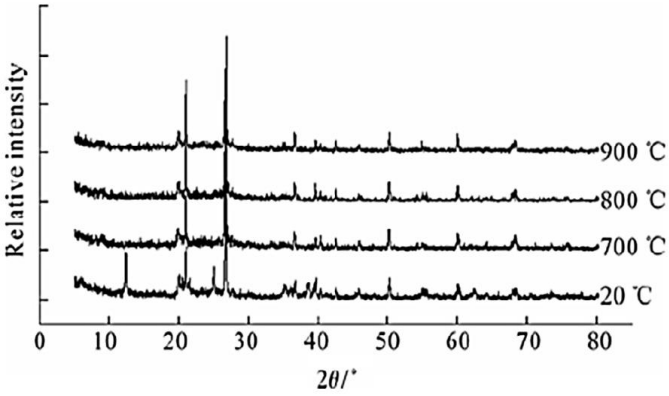


Fig. 5. XRD diffractograms of kaolinite and its calcined product at 700 C, 800 C, and 900 C [23]

6. Clay-based geopolymers

Clay is a popular raw material for geopolymer synthesis due to its straightforward composition, primarily consisting of alumina (Al_2O_3) and silica (SiO_2) in a range of 70-90% Table 2. This simplicity simplifies the interpretation of results compared to more complex materials like fly ash and slag, which often contain impurities. The specific composition of clay can vary widely based on its geological origin and formation. Extensive research has been conducted on metakaolin geopolymers, investigating the influence of various factors such as Si/Al and Na/Al molar ratios, specific surface area, clay composition, alkali concentration and modulus of the alkali silicate solution, and curing conditions [30], [31].

To further diversify geopolymer compositions, researchers have explored blending metakaolin with other source materials such as calcium hydroxide, slag, and ashes. These additional components serve as both binders and fillers within the geopolymer matrix. For example, Alonso and Palomo [32], [33] incorporated calcium hydroxide into metakaolin-based geopolymers without compromising the final product's quality. Similarly, Yunsheng et al. [34] observed enhanced strength development in metakaolin geopolymers containing 30% slag. However, exceeding 50% slag content led to a decrease in strength.

Table 2. Chemical composition of clays from different origin

Clay mineral	SiO2	Al2O3	Fe2O3	TiO2	MgO	P2O5	Na2O	CaO	K2O	MnO	SO3	LOI
Metakaolin [35]	51.35	44.24	0.98	0.9	0.48	0.45	0.16	0.13	0.08	0.01		0.72
Metakaolin [36]	52.1	43.0	0.7	-	0.3	-	0.12	-	2.5	-	-	1
Metakaolin [37]	59.7	34.1	0.9	-	-	-	0.2	0.1	-	-	-	1.2
Clay sediment from Occhito reservoir, Italy [28]	47.5	15.6	6.7	-	2.4	-	0.3	10.2	1.9	-	0.1	15.4
Clay sediment from Sabetta reservoir, Italy [28]	50.0	15.9	5.7	-	1.9	-	0.3	6.9	1.7	-	-	17.5
Kaolinite [38]	49.35	36.03	0.2	0.02	0.02	-	0.04	0.02	2.29	-	2.92	11.94
Kaolinite [38]	40.86	39.87	0.39	0.46	0.12	-	0.01	0.12	0.17	-	-	17.91
Kaolinite [38]	42.66	40.92	1.12	0.45	0.04	-	0.14	0.14	0.09	-	-	14.13
Halloysite [38]	48.12	36.33	0.33	0.16	-	-	0.05	0.04	0.03	-	-	14.8

7. Alkali reactant

The alkali reactant, a soluble metal compound, is commonly derived from sodium or potassium. This category encompasses alkali silicates, hydroxides, carbonates, as well as other materials like sodium aluminates or cement kiln dust. NaOH or KOH solutions are commonly employed to provide the necessary alkalinity for geopolymer synthesis. The leaching capacity of various aluminosilicate sources in these alkaline solutions has been extensively studied. Generally, the dissolution of aluminosilicate sources increases with increasing alkali solution concentration, which is often linked to the ultimate strength of the resulting geopolymers [21]. However, it is widely recognized that aluminosilicate sources tend to dissolve more readily in NaOH solutions compared to KOH solutions.

Panagiotopoulou et al. [39] investigated the leaching behavior of aluminosilicates in separate 10 M NaOH and KOH solutions. As expected, the aluminosilicates exhibited a higher dissolution rate in NaOH. The leaching ability decreased in the following order: Metakaolin > zeolite > slag > fly ash > pozzolana > kaolin Fig. 6. Xu and van Deventer [21] proposed that smaller Na+ ions more easily pair with silicate anions to form smaller oligomers, while larger K+ ions tend to form larger oligomers with silicate anions. Consequently, K-based geopolymers typically exhibit lower compressive strength than Na-based geopolymers, a trend also observed in kaolinitic residue [19]. Furthermore, larger K+ ions can enhance the setting rate of geopolymers.

Fig. 7 illustrates the microstructure of geopolymers prepared using K and Na-based alkali solutions. Morphologically, Na-based geopolymers exhibited a more discrete geopolymer matrix with globular units and some unreacted metakaolin particles (layered structure). In contrast, K-based geopolymers displayed a finer texture and denser structure. Interestingly, despite the apparent denser microstructure of K-based geopolymers, Na-based geopolymers exhibited higher compressive strength [40].

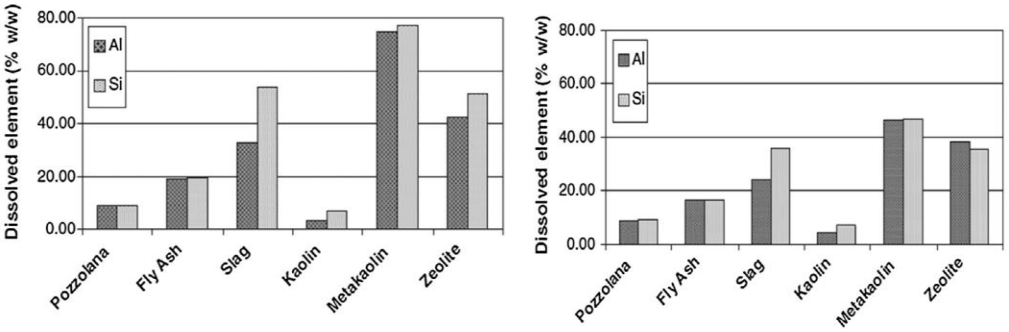


Fig. 6. Dissolved Al and Si in 10 M of (a) NaOH solution and (b) KOH solution after leaching for 24 h [39]

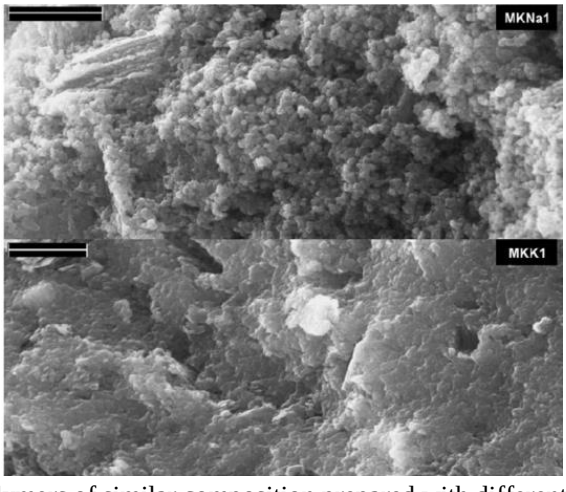


Fig. 7. Metakaolin geopolymers of similar composition prepared with different alkali cations (MKNa1 – geopolymer prepared with Na-based alkali solution and MKK1 – geopolymer prepared with K-based alkali solution)[41].

8. A mixture of alkali hydroxide and alkali silicate solutions

Alkali silicate functions as a binder, alkali reactant, dispersant, and plasticizer while alkali hydroxide dissolves aluminosilicates [42]. As an alternative or supplement to metal silicate, silica fumes can also be used. Silica fume can sometimes be used as a substitute for metal silicate and is occasionally added to alter the silica content in the mixture, promoting gel formation and precipitation. Several laboratory studies have been conducted to produce geopolymers using only hydroxide, as well as a combination of hydroxide and silicate solution. Results have shown that the addition of sodium hydroxide and metal silicate improves the microstructure and strength properties of the geopolymer. The silicate solution introduces a specific amount of SiO2 into the geopolymer, leading to the formation of monomers, dimers, and oligomers[43].

Mohsen and Mostafa [44] reported that a kaolin-based geopolymer activated with NaOH achieved a compressive strength of less than 20 MPa, whereas the geopolymer synthesized using sodium hydroxide and sodium silicate attained a significantly higher strength, reaching approximately 60 MPa. The microstructure shown in Fig. 8 reveals that the kaolin-based geopolymer prepared with a combination of sodium hydroxide and sodium silicate solution developed a denser structure, which is primarily attributed to the effect of the sodium silicate solution.

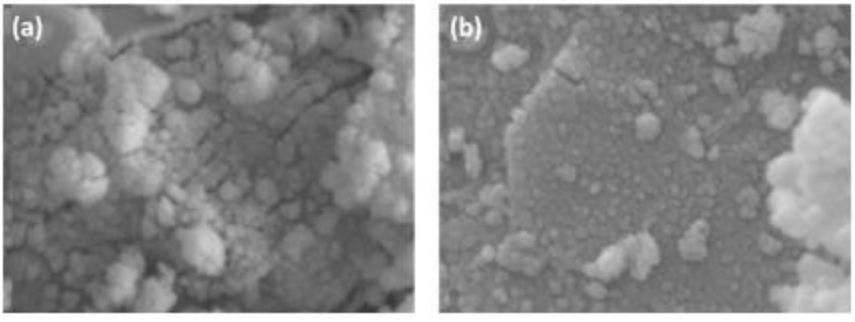


Fig. 8. SEM micrographs of kaolin prepared with (a) NaOH solution and (b) alkaline sodium silicate solution at magnification 8000 [44].

9. Factors influenced in geopolymer.

Various factors influence the formation of geopolymers, including the solid-to-liquid ratio, particle size of the materials and reactive phases, chemical composition of aluminosilicates, type of metal silicates, concentration of sodium hydroxide solution, curing conditions, amount of filler materials, additives, and water content to ensure proper workability.

9.1 Alkali concentration

The concentration of sodium hydroxide (NaOH) solution influences the physical and mechanical properties of geopolymers. This solution enhances the dissolution of aluminosilicates, accelerating the geopolymerization reaction [30]. It should be noted that a NaOH concentration range of 4 to 12 M has shown optimal strength for metakaolin-based geopolymers. Additionally, XRD analysis has indicated that, within this concentration range, the amount of amorphous phases is increased Fig. 9 [45].

Na^+ and OH^- ions combine during the reaction. In geopolymers with low sodium content, there will not be enough OH^- to fully dissolve Si^{4+} and Al^{3+}, and the Na^+ concentration, which is essential for geopolymerization, will also be insufficient. As a result, the compressive strength of these geopolymer samples will not be satisfactory and will remain low [31].

As previously mentioned, NaOH concentration increases the strength of geopolymers, but it also raises the alkalinity. Several researchers have noted that excessive alkalinity can have a negative effect on geopolymer strength. It should be noted that NaOH concentration enhances geopolymer strength up to a certain optimal level, beyond which strength begins to decrease. For example, Zuhua et al. [46] prepared metakaolin-based geopolymers with various NaOH concentrations and recommended an optimal concentration of 9 Molar. Higher NaOH concentrations result in excessive viscosity of the solution, disrupting the leaching of silicon and aluminum ions from the aluminosilicate source. This causes geopolymer gels to precipitate prematurely, leaving unreacted precursor materials, which ultimately reduces the mechanical strength of the final product [47].

Alonso and Palomo [32] investigated the NaOH concentrations in the range of 10 to 18 molar, a significantly different range compared to previous studies. They reported that excessive concentration delays the polymerization process [3],[32]. Similarly, Singh et al. do not recommend highly alkaline environments with Na_2O content exceeding 30 mol%, as their observations were consistent with previous research findings [48].

The rate of geopolymerization is directly related to the setting time of geopolymers. This was examined by Steveson and Sagoe-Crentsil[41] for metakaolin-based geopolymers, who noted that metakaolin in a 6 M NaOH solution does not set, while its setting time becomes very rapid in a 13 M solution. The smoothness of the geopolymer surface increases with alkali concentration from 7 to 12 M. Higher concentrations reduce the dissolution time of reactive materials, leaving some unreacted materials in the final product. Other studies have shown that contrary to expectations, increasing NaOH concentration can lengthen the setting time. For example, geopolymers made from blast furnace slag activated by KOH solutions experience delayed setting time [14].

Another important aspect of geopolymers is their workability. High alkali concentration affects the workability of geopolymers, leading to faster setting rates and consequently reduced workability. The impact of high alkali concentration on the workability of metakaolin-based geopolymers has been studied to a limited extent; however, studies indicate that for fly ash-based geopolymers, an increase in alkali concentration leads to reduced workability [49], [50].

The compressive strength of geopolymers is influenced by both low and high alkali concentrations, and geopolymers made from fly ash and slag exhibit a similar trend compared to metakaoline base geopolymers [14],[51]. High concentrations lead to the formation of sodium carbonate, which is considered a detrimental factor, and thus the alkali concentration should be kept at an optimal level [52].

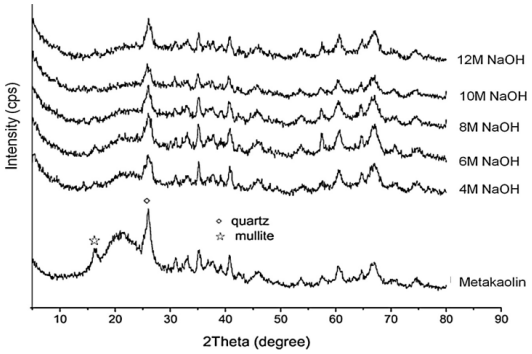


Fig. 9. XRD diffractograms of metakaolin and the corresponding geopolymers synthesized with various NaOH concentrations and cured at room temperature (20^0C) for 60 min and at 65^0C for another 10 [45].

9.2. Solids/liquid ratios

During geopolymer formation, the solid content refers to the aluminosilicates, while the liquid content is the alkali reactant. The solid-to-liquid (S/L) ratio is considered one of the key factors in geopolymer preparation. This ratio directly influences the workability, solubility, geopolymerization reaction, and strength of the final product. For instance, an S/L ratio of 0.8 has been recommended for metakaolin-based geopolymers [8],[46],[53]. The workability of geopolymer is directly influenced by the S/L ratio. As this ratio increases, workability tends to decrease. Xiao et al. [45] examined this ratio for metakaolin-based geopolymers and found that ratios above 2 reduce workability and simultaneously delay the geopolymerization reaction.

Fernandez-Jimenez and Palomo [54] studied the workability of geopolymers made from fly ash and reported similar results. They also noted that this type of geopolymer can tolerate higher S/L ratios. A higher liquid content increases the spacing between the particles in the constituent materials, thereby enhancing workability [20].

The fineness of materials requires a higher amount of activating liquid, which affects the S/L ratio and also impacts workability. The S/L ratio differs between fly ash-based and metakaolin-based geopolymers. Moreover, particle shape also influences this ratio; metakaolin, with its layered structure, requires more liquid, while the spherical structure of fly ash particles reduces the need for additional liquid [8].

Zuhua et al. noted that the solubility of aluminosilicates increases at lower S/L ratios [46]. Some reports have documented S/L ratios exceeding 3 [48], and some suggest that an optimal range lies between 1 and 5 [46].

9.3. Alkali reactant ratios

In geopolymer synthesis, the ratio of NaOH solution to Na_2SiO_3 solution plays an important role. The NaOH solution primarily acts as a solvent, while the Na_2SiO_3 solution functions as the binding agent.

Numerous studies have investigated the Na_2SiO_3/NaOH ratios, with reported ranges from 0.24 to 2.2. Wang et al. [45] suggested a ratio of 0.24 for metakaolin-based geopolymer production, achieving a compressive strength of 59 MPa in samples prepared with this ratio. Pelisser et al. [55] examined metakaolin-based geopolymers with Na_2SiO_3/NaOH ratios of 1.0, 1.6, and 2.2, analyzing their compressive strengths. They found that samples

with a ratio of 1.6 exhibited the highest 7-day compressive strength of 64 MPa, while the lowest strength was observed at a ratio of 1. These findings were confirmed by Poowancum and Horpibulsuk [56], as illustrated in Fig. 10.

Increasing the Na_2SiO_3/NaOH ratio enhances the strength of geopolymers, thereby reinforcing the geopolymerization process [25]. Higher Na_2SiO_3/NaOH ratios accelerate the geopolymerization reaction rate [40]. However, excessively high ratios not only improve strength but can also lead to a reduction in strength and limit the workability of the paste.

Pinto noted that a ratio lower than 0.85 would not be suitable for the production of metakaolin-based geopolymers, as it makes the mixing process difficult [57]. The reason for this is the viscosity of the Na_2SiO_3 solution, which causes the geopolymer paste to become sticky [20]. The compressive strength of geopolymers made with zeolite increases with the Na_2SiO_3/NaOH ratio up to 1.5 [17]. The ratios of alkaline activators vary for different types of soils, and typically, for clay-based soils, this ratio falls within a narrow range. The Na_2SiO_3/NaOH ratio for geopolymers made with fly ash has been studied in the range from 0.05 to 3, and from 0.4 to 2.5 utilized [54], [58], [59]. Fly ash, which contains spherical particles, reduces the frictional surface area between particles, resulting in better workability, and the aforementioned ratios support this finding [60].

Increasing the ratio of alkaline activators in geopolymer production results in an unsuitable geopolymer formation, with a higher presence of inactive and unreacted particles. This claim was confirmed by Duxson et al.[61].

Finally, it should be noted that NaOH pellets are cheaper than the liquid of Na2SiO3 solution, and the lower this ratio, the more cost-effective the produced geopolymer will be. However, the focus should solely be on the performance and final strength of the product.

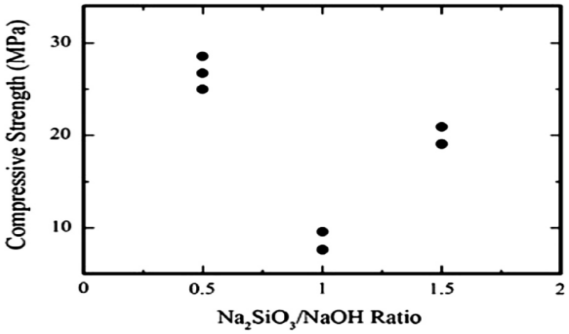


Fig. 10. Compressive strength of calcined sedimentary clay geopolymers at various alkali reactant ratios[62].

9.4. Molar ratios (Si, Al, Na, and H2O contents)

The amount of Na in a geopolymer system is sourced from various materials, including NaOH solution and Na2SiO3 solution. The amount of Si is obtained from aluminosilicate sources and Na2SiO3 solution, while the amount of Al is derived solely from aluminosilicates. The amount of H_2O in a geopolymer system is also derived from various

sources, including the NaOH solution, Na_2SiO_3 solution, and additional water used in the geopolymer preparation. It is noteworthy that the geopolymer system is influenced by the aforementioned parameters, which include NaOH concentration, the solid-to-liquid (S/L) ratio, and the Na_2SiO_3/NaOH ratio.

The conversion between amorphous and crystalline phases is directly dependent on the initial concentrations of Si, Al, and Na [9], [63], [64]. However, the Si/Al and Na/Al ratios are the most critical. Davidovits stated that to achieve a highly durable geopolymer, these ratios should fall within the range shown in Table 3. He also identified optimal ratios, suggesting that the Na_2O/Al_2O_3 and SiO_2/Al_2O_3 ratios should be approximately between 1 and 4, respectively. The Si/Al ratio also plays a significant role in geopolymers, and based on conducted research, the optimal SiO_2/Al_2O_3 ratio is between 3 and 3.8, which is slightly lower than the range reported by Davidovits.

The SiO_2/Al_2O_3 ratio is a key factor in geopolymer synthesis, influencing mechanical properties. Increasing this ratio enhances mechanical characteristics [65], as silica contributes to strength improvement while alumina affects setting time [30]. Higher Si/Al and Na/Al ratios promote uniform structure within geopolymers [31].

Table 3. Recommended oxide molar ratios of geopolymers [66].

Oxide molar ratios	Range
SiO_2/Al_2O_3	3.50–4.50
Na_2O/Al_2O_3	0.80–1.20
H_2O/Na_2O	15–17.50
Na_2O/SiO_2	0.20–0.28

Research by De Silva et al. [63] demonstrated that low Si/Al ratios lead to the formation of poly(sialate) structures.

Specimens prepared by Stevenson and Sagoe-Crentsil [41], with SiO2/Al2O3 ratios between 3 and 3.9 achieved a compressive strength of 48 MPa, which was nearly double the compressive strength reported by De Silva et al [63]. Fig. 11 shows that the geopolymer attained a denser and more interconnected microstructure, resulting in a continuous increase in compressive strength. Lizcano et al. [67] reported a compressive strength of 34 MPa with a SiO2/Al2O3 ratio of 3 while keeping the H2O/Na2O and Na2O/Al2O3 ratios constant at 10 and 1, respectively. In most of the studies conducted, the SiO2/Al2O3 ratios show a very different range, which researchers have used, and it significantly differs from the recommended range in Table 3. The geopolymerization process is influenced by the molar ratios of SiO_2/Na_2O. Additionally, increasing the content of K_2O or Na_2O induces significant changes in the geopolymer, such as increasing the setting speed, enhancing strength, and reducing cracking [68]

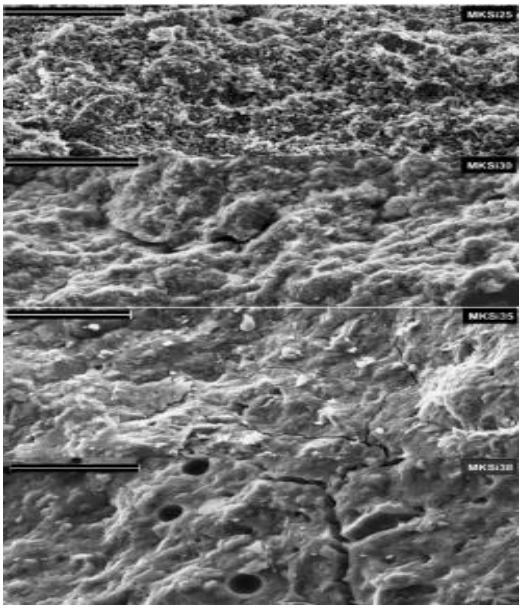


Fig. 11. SEM micrographs of metakaolin geopolymers with different silica content (MKSi25 – SiO_2/Al_2O_3 = 2.5; MKSi30 – SiO_2/Al_2O_3 = 3.0; MKSi35 – SiO_2/ Al_2O_3 = 3.5 and MKSi38 – SiO_2/Al_2O_3 = 3.8) [41].

The Na_2O/H_2O ratio represents the alkalinity of the activating solution, and this factor also plays a crucial role [68]. In general, in clay-based soils, increasing the Na_2O/H_2O ratio improves solubility and also enhances compressive strength [21]. However, contrary to this claim, Latella et al. [69] reported that a molar ratio of H_2O/Na less than 5.5 leads to cracking and aging, while an increase in this ratio to 6 results in the formation of porosity. These results are in line with the findings of Steveson and Sagoe-Crentsil [41], who reported that increasing the H_2O/Al_2O_3 ratio from 12 to 16 leads to an increase in porosity. However, a certain amount of water is considered essential for geopolymer production.

Kamalloo et al.[70] reported the highest compressive strength of a geopolymer at 80 MPa, achieved with SiO_2/Al_2O_3, M_2O/Al_2O_3, and H_2O/M_2O ratios, where M represents an alkali metal. Additionally, the K_2O/Na_2O ratio was within the ranges of 3.6–3.8, 1–1.2, 10–11, and 0.6–1, respectively Fig 12.

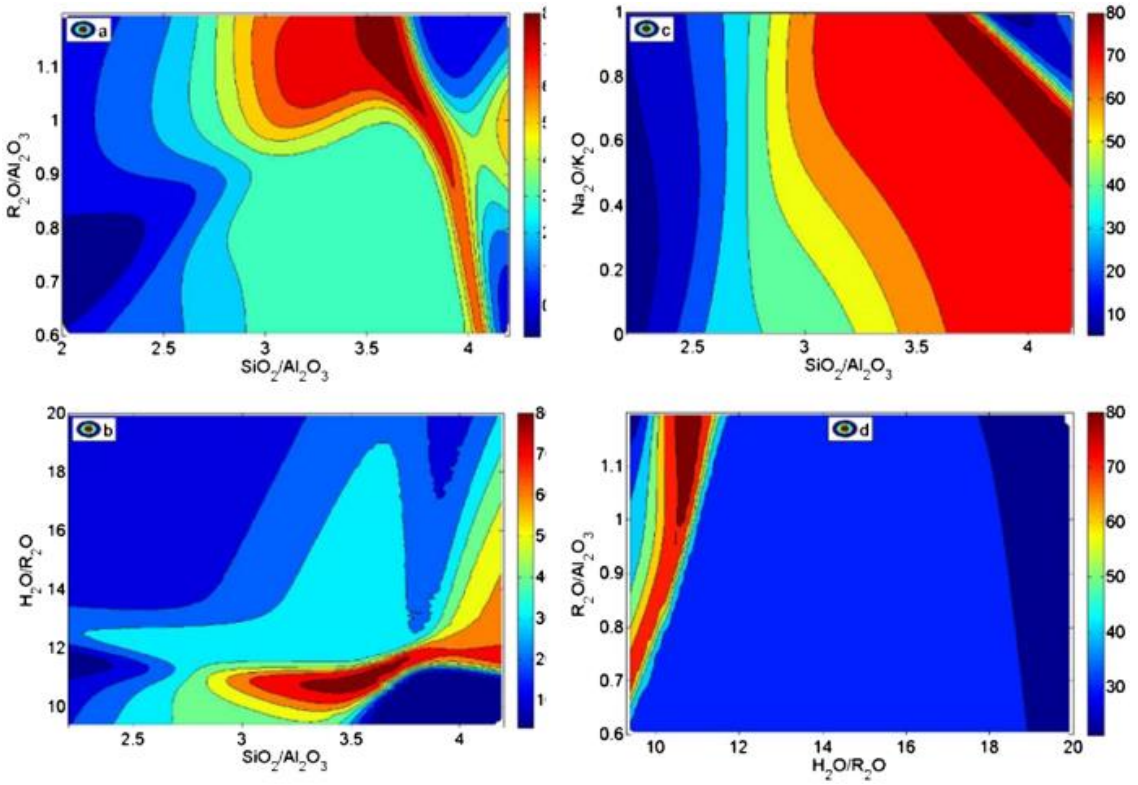


Fig. 12. Filled contours plot of compressive strength showing the effect of various molar ratios (M_2O/Al_2O_3, SiO_2/Al_2O_3, Na_2O/K_2O, and H_2O/M_2O) on the compressive strength of metakaolin geopolymers. The contours are in units of MPa [70].

9.5. Water content

Water is a fundamental component in the synthesis of geopolymers, playing a crucial role in their formation, structure, and properties. The presence of water facilitates the dissolution of materials, and the transport of dissolved ions, and enables the hydrolysis and polycondensation of oligomers.

The presence of water in geopolymers also enhances their flowability and workability, making ion transport within the system easier [9]. However, excess water introduces certain issues and concerns, such as reducing alkalinity and displacing reactive ions away from the reaction zone [7]. Excessive water can inhibit the geopolymerization process, which is one of the major challenges in geopolymer production [71].

Rahier et al. stated that an unconventional amount of water in geopolymers has a significant impact on the rate of geopolymerization, where both low and high water content reduces the reaction rate [40].

In a geopolymeric system, the required amount of water is directly related to the composition of the geopolymer. For instance, with a lower Na/Al ratio, more water is needed, which can increase the likelihood of cracking during curing [72]. Excess water also leads to additional issues, such as increased density and porosity [69].

Water that resides in the pores of the material and is not chemically bound, leaving the material prior to shrinkage due to drying, is referred to as *free water*. In contrast, water

that remains within the geopolymer matrix and plays a role in the shrinkage process is termed *structural water* [73]. To maintain the strength of the geopolymer, the presence of non-evaporable water within its structure is considered essential Fig. 13.
The amount of water required in the geopolymer structure is influenced by various parameters such as the S/L ratio, alkali concentration, and alkaline activators. The water content should be adjusted accordingly, taking these factors into account.

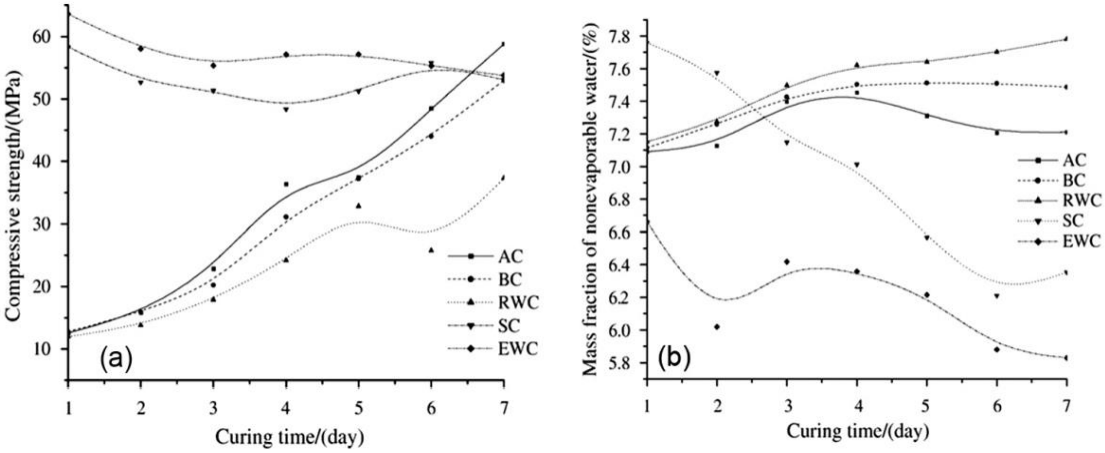


Fig. 13. Variations of (a) compressive strength and (b) non-evaporable water by mass fraction of metakaolin geopolymers under different curing conditions. [AC = in air (20±2°C), BC = in sealed bag (20±2°C), RWC = room temperature water (20±2 °C), SC = in steam (80°C) and EWC = in elevated temperature water (80°C)] [46].

9.6. Curing regime

The curing of geopolymers is typically conducted in two ways: either at room temperature or at elevated temperatures. The maximum curing temperature agreed upon by most researchers is 100°C. Davidovits suggested an optimal curing temperature range for geopolymers between 60°C and 95°C. Curing geopolymers at 75°C enables the samples to achieve acceptable compressive strength, reaching up to 39.8 MPa [3].
To achieve optimal mechanical properties and durability in geopolymers, curing is considered a critical factor [42]. The dissolution of silica and alumina species from aluminosilicate sources accelerates significantly in the presence of heat, which leads to the hardening of the geopolymer matrix [32],[47], [73]. As mentioned, curing temperature enhances the compressive strength of geopolymers. However, excessive temperatures or prolonged exposure to high heat can lead to a reduction in both the strength and durability of the geopolymer[37].
The reaction rate of the geopolymerization process increases significantly at curing temperatures between 20 to 50°C, as demonstrated by Xiao et al. [30]. Curing at ambient temperatures proceeds more gradually, whereas curing in a warm environment at 50°C accelerates the process considerably. However, it should be noted that high-temperature curing does not necessarily enhance the final strength [46]. Based on research findings, the optimal curing temperature for geopolymers has been identified as 35°C.

Various studies have investigated high-temperature curing, yielding mixed results. High-temperature curing tends to create larger pores, which significantly impacts the strength of geopolymers; this claim has been noted by Rovenanik [74]. Additionally, curing at temperatures between 60 to 80°C results in better early-age strength for samples, but strength tends to decrease after 28 days [74], as illustrated in Fig. 14. Findings by Juha et al. indicate that curing geopolymers at 20 to 40°C promotes a continuous strength gain from day 1 to day 28 without any reduction [46]. On the other hand, curing geopolymer samples in water at 20°C results in lower compressive strength.

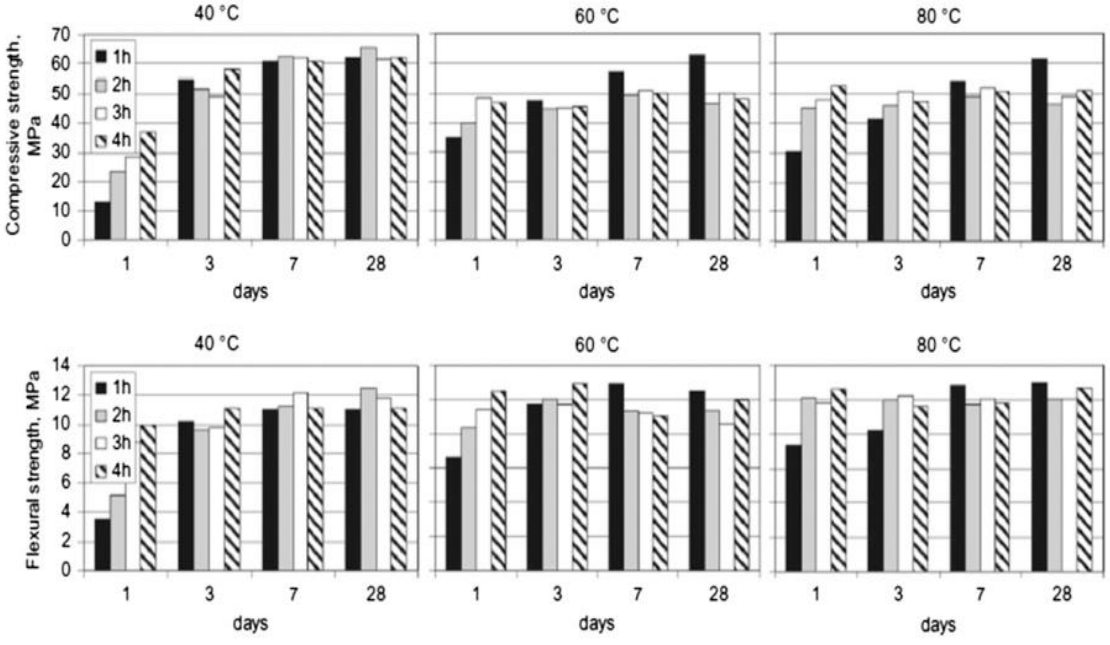


Fig. 14. Effect of curing time and temperatures on compressive and flexural strengths of metakaolin geopolymers [74]

The duration of curing has a significant impact on the microstructure of geopolymers. As shown in Fig. 15, geopolymer samples were cured at 65°C for durations of 1 hour, 4 hours, 48 hours, and 72 hours, with results indicating that the optimal curing time was 72 hours. During this period, an amorphous, dense geopolymer matrix developed, creating a suitable microstructure [75]. It is noteworthy that after a certain curing period, geopolymers reach the completion of geopolymerization, beyond which no further strength gain occurs. For example, red mud- and rice husk ash-based geopolymers achieved a stable strength of 11.7 MPa after a 35-day curing period [47].

In some cases, geopolymers derived from various aluminosilicate sources, long curing times are not only ineffective but can also have a negative impact on their mechanical strength. For example, geopolymers made from fly ash [54] and zeolite [17], when exposed to prolonged heat, experience a reduction in mechanical properties. In contrast, clay-based geopolymers typically require a long curing period to achieve optimal strength. Therefore, it can be concluded that the need for curing depends precisely on the

aluminosilicate sources and their compositions. Choosing an appropriate curing regime is crucial to ensure that silica and alumina species are densely arranged together.
A very important point in the synthesis of geopolymers is the relationship between curing temperature and the concentration of alkaline solutions. This relationship has a significant impact on the development of geopolymer strength, and it must be examined with great precision.

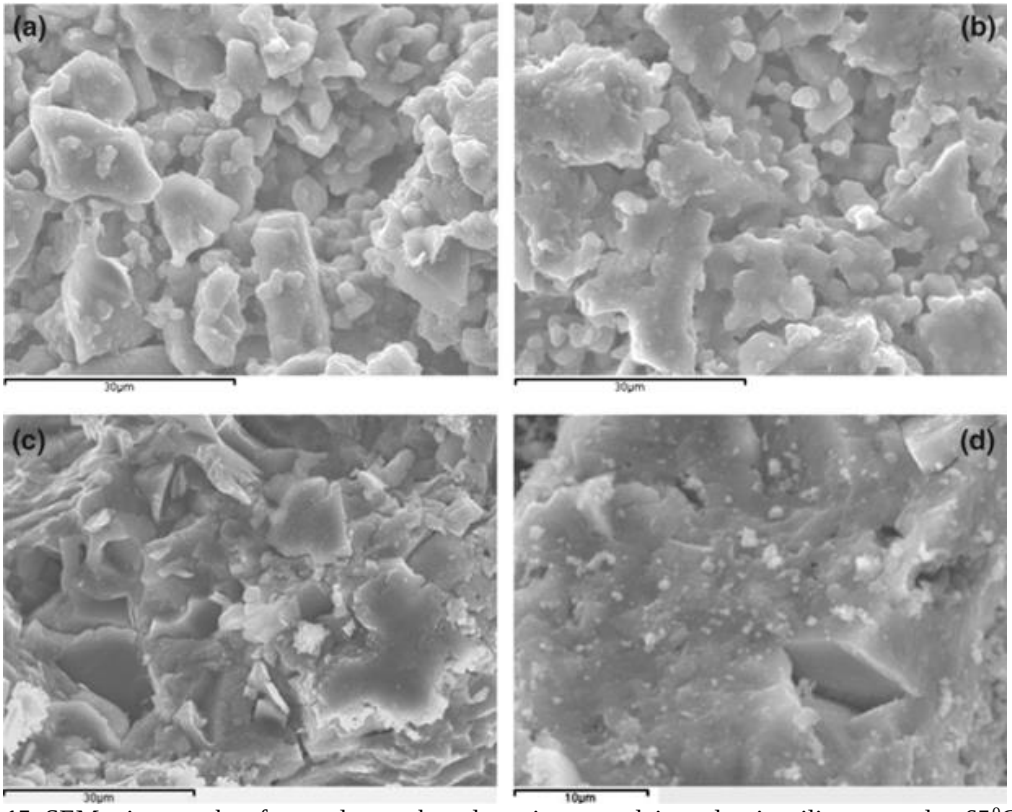


Fig. 15. SEM micrographs of geopolymers based on vitreous calcium aluminosilicate cured at 65^0C for (a) 1 h, (b) 7 h, (c) 48 h, and (d) 72 h [75].

10. Conclusion

The formation and performance of clay-based geopolymers are significantly influenced by several key parameters, including the aluminosilicate sources, alkali reactants, and their concentrations. Key factors such as the Si/Al and Na/Al molar ratios, solid-to-liquid ratios, and alkali reactant ratios play critical roles in determining the properties of the final geopolymer. Optimal values for these parameters must be carefully balanced to achieve the desired strength and durability. Specifically, Si/Al ratios impact the mechanical properties, with higher ratios generally leading to improved strength, while Na/Al ratios and alkali concentrations influence the dissolution and reaction rates. Additionally, the appropriate use of alkali hydroxides and silicates, along with careful consideration of the water content and curing conditions, is essential for producing high-quality geopolymers. Ongoing research into these variables and their interactions is crucial for advancing geopolymer technology and optimizing its applications in various fields.

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