

Strength optimization of geopolymer pastes synthesized from high calcium bottom ash and red clay: A study of composition and activation parameters

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ABSTRACT

This study aims to synthesize and enhance the strength of geopolymer pastes using high-calcium bottom ash (BA) and red clay (RC), both abundant and sustainable resources. The physical and chemical properties of the synthesized geopolymer pastes were evaluated by investigating key synthesis parameters, including the BA/RC weight ratio, the $\text{SiO}_2/\text{Al}_2\text{O}_3$ mole ratio, the $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ mole ratio, and the concentration of sodium hydroxide. Various ratios of BA and RC were combined with alkaline activators of sodium hydroxide and sodium silicate to prepare the geopolymer pastes. The optimal conditions for achieving the highest compressive strength were identified as a BA/RC weight ratio of 90/10, a $\text{SiO}_2/\text{Al}_2\text{O}_3$ mole ratio of 6, a $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ mole ratio of 2, and activation with 10 M sodium hydroxide. Compressive strength increased with a higher $\text{SiO}_2/\text{Al}_2\text{O}_3$ mole ratio, while an elevated $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ mole ratio initially reduced strength. Additionally, the dissolution of silica and alumina from the BA and RC precursors significantly improved with increased sodium hydroxide concentration. This research supports environmental sustainability by reducing industrial waste, minimizing reliance on Portland cement, and promoting circular economy principles through the development of high-strength, eco-friendly construction materials.

Keywords: geopolymerization, aluminosilicate, pozzolanic materials, green cement.

INTRODUCTION

Geopolymer production requires low energy and has a very low carbon footprint compared to traditional Portland cement [1, 2]. Geopolymers are green binder materials with an amorphous three-dimensional network structure that can be synthesized from an appropriate aluminosilicate precursor via polymerization of SiO_4 and AlO_4 tetrahedra induced in alkaline activators [3]. Geopolymer polymerization includes three stages: (1) dissolution of the aluminosilicate precursor in alkaline solution to form SiO_4 and AlO_4 tetrahedral units, (2) polycondensation of monomers to form the network structure, and (3) solid-state transformation and hardening to form geopolymers [4, 5].

Pozzolanic materials, primarily composed of SiO_2 and Al_2O_3 , react chemically with calcium hydroxide in the presence of water to form compounds that possess cementitious properties. These materials can be utilized to synthesize geopolymers. Sources of pozzolanic materials include by-products of combustion, such as BA, fly ash (FA), and rice husk ash (RHA) [6–8], as well as calcined clay [9, 10] and natural minerals [11]. Many researchers have focused on obtaining good chemical and mechanical properties in FA-based geopolymers, leading to widespread acceptance of FA as the primary raw material for geopolymer synthesis [12–14]. However, coal combustion still produces BA, which constitutes 20–25% of the total ash. Consequently, the utilization of

BA-based geopolymers has not been studied as extensively as those based on FA, primarily because BA has a larger particle size and tends to agglomerate. Therefore, using BA in geopolymer synthesis requires a particle size reduction process, which may increase production costs compared to using fly ash. Nevertheless, employing BA can help control environmental pollution, reduce industrial waste, and comply with green construction standards.

BA is an inorganic waste material from coal combustion that must be disposed of using an environmentally friendly approach. BA has high silica and alumina contents similar to FA and can react like a pozzolanic substance to replace Portland cement. BA can also be used as a starting material in geopolymer synthesis [2, 15]. The silica and alumina react with an alkaline medium, specifically sodium silicate and sodium hydroxide, to produce sodium aluminosilicate hydrate (N-A-S-H). Additionally, BA containing high calcium content leads to the formation of calcium aluminosilicate hydrate (C-A-S-H) gel. This gel results in strong cementitious compounds that act as a binder in cement [16]. These processes enhance the setting time and strength development, particularly under ambient curing conditions [17].

RC, a naturally occurring clay rich in silica, alumina, and iron [18], is abundant in many regions, such as Phayao province in Thailand, where it is traditionally used in pottery and other household items. Thermal activation of RC significantly enhances its reactivity, making it an ideal precursor for producing durable, high-strength geopolymers [4]. When combined with BA, RC introduces an optimal balance of silica and calcium, facilitating improved microstructural properties and compressive strength in the resulting geopolymer.

This study investigates the effects of BA/RC weight ratio, $\text{SiO}_2/\text{Al}_2\text{O}_3$ mole ratio, $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ mole ratio, and NaOH concentration on the phase development, microstructure, and mechanical properties of geopolymer pastes. Through controlled variations of these parameters, the study aims to elucidate the reaction mechanisms and strength development pathways that govern geopolymer performance. Notably, altering Si/Al and Na/Al ratios has been shown to affect the degree of polycondensation, resulting in different structural densities and mechanical properties. By advancing the understanding of these complex

interactions, this study not only provides insights into geopolymer synthesis from a chemical and engineering perspective but also contributes to the circular economy. It presents a model for sustainable materials engineering that bridges fundamental research with practical applications, offering a promising pathway for the development of eco-friendly construction materials.

MATERIALS AND METHODS

Materials

BA was obtained from the Mae Moh Power Plant (Thailand), ground with a ball mill, and then passed through sieve No.200 to obtain mean particle diameter of 64.3 μm . RC was collected from Phayao Province in Northern Thailand, subjected to calcination in an oxidizing atmosphere at 750 $^\circ\text{C}$ with a heating rate of 5 $^\circ\text{C}/\text{min}$ for 3 h, and then ground to pass through sieve No.200 to obtain mean particle diameter of 56.5 μm . The chemical compositions of BA and RC obtained by X-ray fluorescence (XRF) are presented in Table 1. The major components in BA were CaO , Fe_2O_3 , SiO_2 , and Al_2O_3 while RC mainly comprised SiO_2 , Al_2O_3 , and Fe_2O_3 . BA had high CaO and Fe_2O_3 contents of 31.78 and 28.93 wt%, respectively while RC had high SiO_2 content of 54.77 wt%. Mineralogical analyses of BA and RC observed by an X-ray diffractometer (XRD) are shown in Figure 1(A). BA consisted of amorphous aluminosilicate, quartz (SiO_2), anhydrite (CaSO_4), hematite (Fe_2O_3), calcite (CaCO_3), and crandallite ($\text{Al}_3\text{CaH}_7\text{O}_{14}\text{P}_2$), while RC was mainly composed of quartz (SiO_2) with minor amounts of illite ($\text{Al}_3\text{H}_2\text{K}_{0.5}\text{O}_{12}\text{Si}_4$). BA and RC morphologies observed by a scanning electron microscope (SEM) are shown in Figure 4. The alkaline activators used in the geopolymer synthesis were sodium hydroxide (NaOH) and sodium silicate (Na_2SiO_3) containing 32.39 wt% SiO_2 , 13.44 wt% Na_2O , and 54.17 wt% H_2O .

Synthesis of geopolymer pastes

The initial mole ratios of $\text{SiO}_2/\text{Al}_2\text{O}_3$ obtained from BA and RC were 3.74 and 5.17, respectively. In this study, the $\text{SiO}_2/\text{Al}_2\text{O}_3$ mole ratios of geopolymer gel were adjusted using sodium silicate to fall within the range of 5–7. The geopolymers were synthesized by mixing BA, RC,

Table 1. Chemical compositions (wt%) of the BA and RC starting materials

Chemical compositions (wt%)	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	K ₂ O	TiO ₂	MnO	SO ₃	LOI
BA	19.91	9.06	28.93	31.78	2.20	0.52	0.39	6.69	0.52
RC	54.77	18.00	17.38	1.25	4.90	2.16	0.28	1.05	0.21

and the alkaline activator solution at different compositions, as shown in Table 2, to obtain a homogeneous mixture that was then cast into acrylic molds with 25 × 25 × 25 mm³ internal dimensions. The molds were vibrated for 5 min to remove the trapped air and wrapped in vinyl film to prevent moisture loss. The samples were left in a 25 °C controlled room for 1 h and then cured at 60 °C for 48 h. After cooling to room temperature, the specimens were demolded and stored in plastic bags.

Characterization and compressive strength tests

The phase compositions of the geopolymers were determined by an XRD (Rigaku Miniflex 600) instrument with Cu K α radiation. The microstructure was examined using an SEM (FEI Quanta 250), with elemental analysis conducted by an energy dispersive X-ray spectrometer (EDX, Inca x-act, Oxford Instruments). The chemical bonds were identified using a Fourier transform infrared spectrometer (FTIR, Thermo Scientific Nicolet iS5), with strength tested by a compressive strength testing machine at 7 and 28 days according to ASTM C109 [19].

RESULTS AND DISCUSSION

X-ray diffraction analysis

The synthesized geopolymer pastes with a BA/RC weight ratio of 90/10 were used to study the differences in SiO₂/Al₂O₃ mole ratios of 5, 6, and 7, as shown in Figures 1(b) and 2(a). The X-ray diffraction patterns demonstrated the dissolution of the amorphous and crystalline phases present in BA and RC (Figure 1(a)) to form geopolymers. The XRD result identified the main quartz phase in the geopolymers under all synthesis conditions. After 7 days, the crystalline phase of sodalite demonstrated that the SiO₂/Al₂O₃ mole ratio of 5 transformed into hydroxycancrinite after 28 days. The presence of the zeolite phase (sodalite and hydroxycancrinite) resulted in decreased geopolymer compressive strength [20]. Crystalline phases of supplementary cementitious materials such as α -kermanite and gehlenite were present in SiO₂/Al₂O₃ mole ratios of 6 and 7, respectively thereby increasing geopolymer compressive strength [21]. Increasing concentration of NaOH at 8, 10, and 12 M increased the dissolution of BA and RC, especially quartz minerals in RC. The mineral phases that appeared in the

Table 2. Mixture compositions of BA, RC, and alkaline activators

Sample code	BA/RC	BA	RC	Na ₂ SiO ₃	NaOH	NaOH	SiO ₂ /Al ₂ O ₃	Na ₂ O/Al ₂ O ₃
	(weight ratio)	(g)	(g)	(g)	(mol/L)	(mL)	(mole ratio)	(mole ratio)
100BA6N10A20	100/0	100	0	37.39	10	19.32	6	2.0
100BA6N10A21	100/0	100	0	37.39	10	21.09	6	2.1
100BA6N10A22	100/0	100	0	37.39	10	22.87	6	2.2
90BA6N10A20	90/10	90	10	36.38	10	23.26	6	2.0
90BA5N10A20	90/10	90	10	18.28	10	31.11	5	2.0
90BA7N10A20	90/10	90	10	54.48	10	15.42	7	2.0
90BA6N8A20	90/10	90	10	36.38	8	29.08	6	2.0
90BA6N12A20	90/10	90	10	36.38	12	19.39	6	2.0
90BA6N10A21	90/10	90	10	36.38	10	25.22	6	2.1
90BA6N10A22	90/10	90	10	36.38	10	27.17	6	2.2
80BA6N10A20	80/20	80	20	35.37	10	27.21	6	2.0
70BA6N10A20	70/30	70	30	34.36	10	31.15	6	2.0
60BA6N10A20	60/40	60	40	33.35	10	35.09	6	2.0

geopolymers were the same at all concentrations, as shown in Figure 1(c). After 28 days, the dissolution of the original crystalline phases was similar (Figure 2(b)). The differences in Na₂O/Al₂O₃ mole ratios 2.0, 2.1, and 2.2, as shown in Figures 1(d) and 2(c), indicated that increasing Na₂O/Al₂O₃ mole ratio above 2.0 led to the formation of sodalite in the geopolymer.

Fourier transform infrared spectroscopy

The FTIR spectra of the geopolymer samples with different SiO₂/Al₂O₃ mole ratios, Na₂O/Al₂O₃ mole ratios, and NaOH concentrations at 7 days are presented in Figure 3. All samples showed similar intensities at 604 cm⁻¹ and 962 cm⁻¹, related to Si-O-T (T = Al, Fe or Si) bending vibrations and asymmetrical stretching, respectively in the tetrahedral configurations [22, 23]. The bands at 797 cm⁻¹ were attributed to Fe-O-T (T=Al, Fe or Si) and Si-O-Si bonds, associated with the formation of geopolymer gel [23, 24]. The band at 1412 cm⁻¹ was assigned to stretching vibrations of C-O bonds linked to calcium carbonate [22, 25],

while the bands at 1637 cm⁻¹ and 3300 cm⁻¹ were assigned to bending and stretching vibrations of the O-H group, respectively [25].

Microstructure

Figure 4 shows SEM photomicrographs of the geopolymer samples after 7 days under different synthesis conditions. When BA and RC reacted with an alkaline activator solution, the structure transformed into a dense geopolymer. Almost identical microstructures were observed for all geopolymer samples, indicating that polymerization of the geopolymer gel produced a well-linked glassy phase structure. The BA/RC weight ratio of 90 was most suitable for workability. The XRD results showed that samples 90BA5N10A20, 90BA6N10A21, and 90BA6N10A22 contained zeolite crystals in the geopolymer microstructure. The EDX analysis of the 90BA6N10A21 sample (Figure 5) revealed the presence of a sodalite phase, characterized by aggregated platelet particles with a mole ratio of Si/Al in the range of 1.1–1.2 in the geopolymer sample. Figure 6

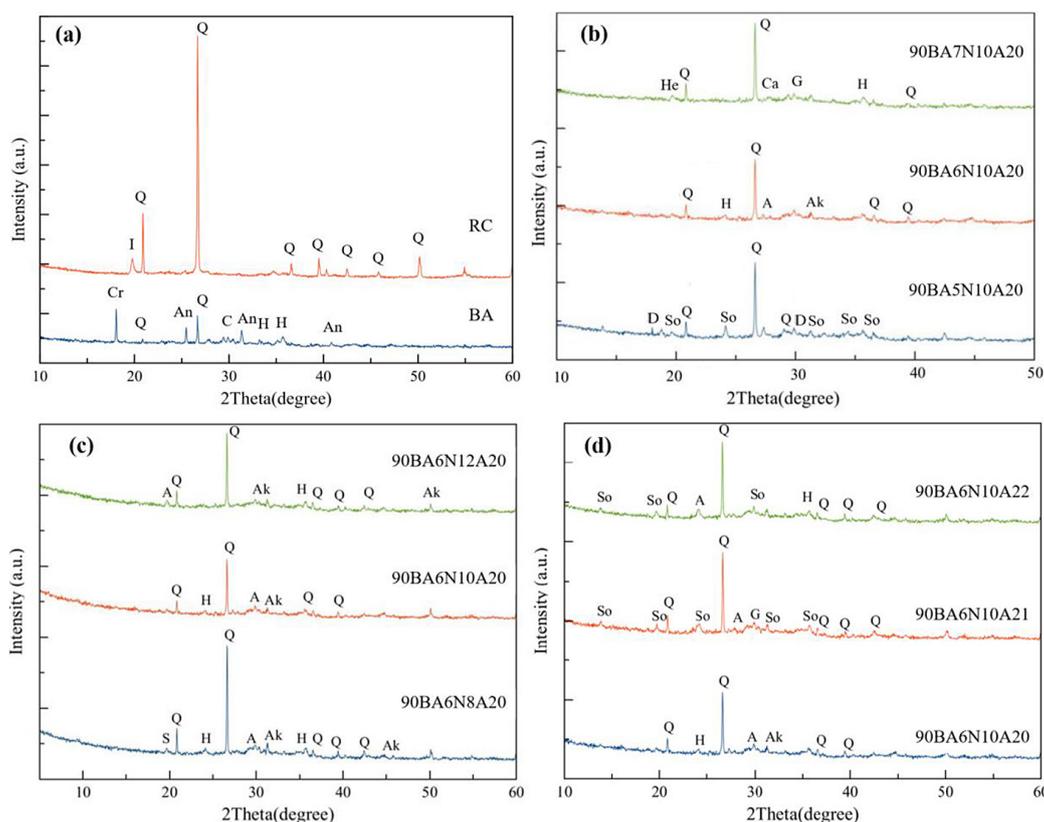


Figure 1. XRD patterns of starting materials (a), and geopolymer samples after 7 days with different SiO₂/Al₂O₃ mole ratios (b), NaOH concentrations (c), and Na₂O/Al₂O₃ mole ratios (d). Q, Quartz; I, Illite; An, Anhydrite; Cr, Crandallite; H, Hematite; C, Calcite; A, Augite; Ak, Åkermanite; G, Gehlenite; Ca, Calcium Aluminium Oxide; He, Hedenbergite; So, Sodalite; D, Diopside; S, Spinel

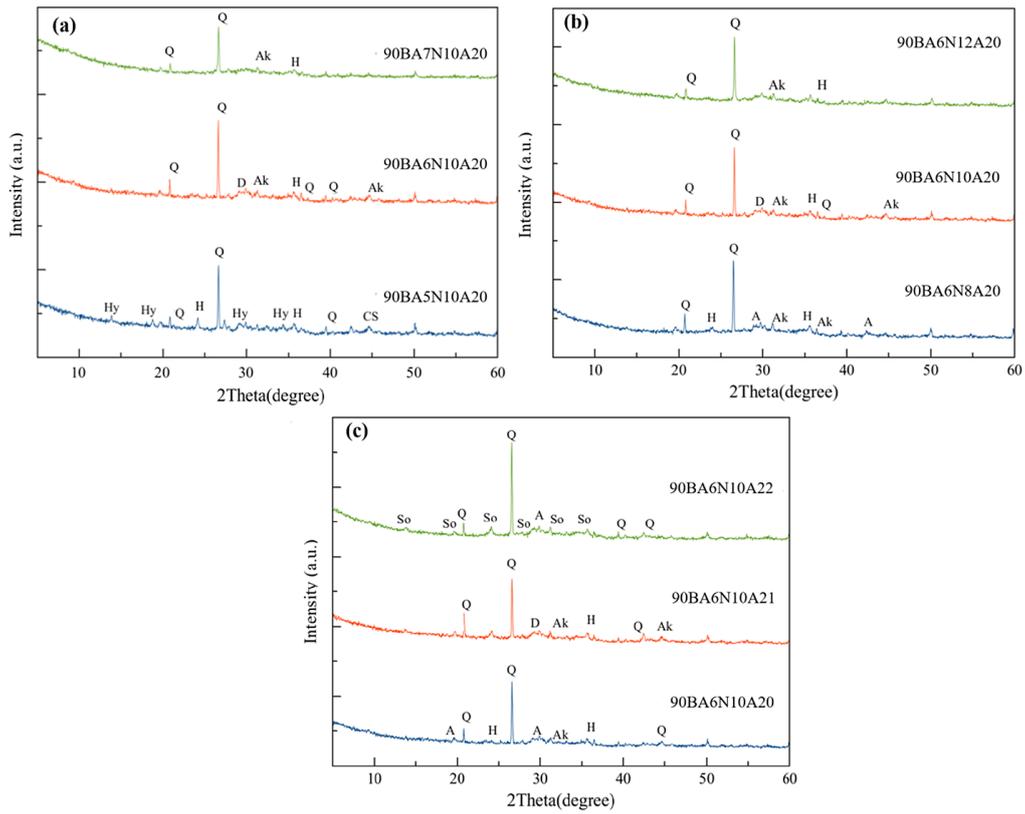


Figure 2. XRD patterns of geopolymer samples after 28 days with different $\text{SiO}_2/\text{Al}_2\text{O}_3$ mole ratios (a), NaOH concentrations (b), and $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ mole ratios (c). Q, Quartz; H, Hematite; Ak, Akermanite; D, Diopside; Hy, Hydroycancrinite; CS, Calcium Silicate; A, Augite; So, Sodalite

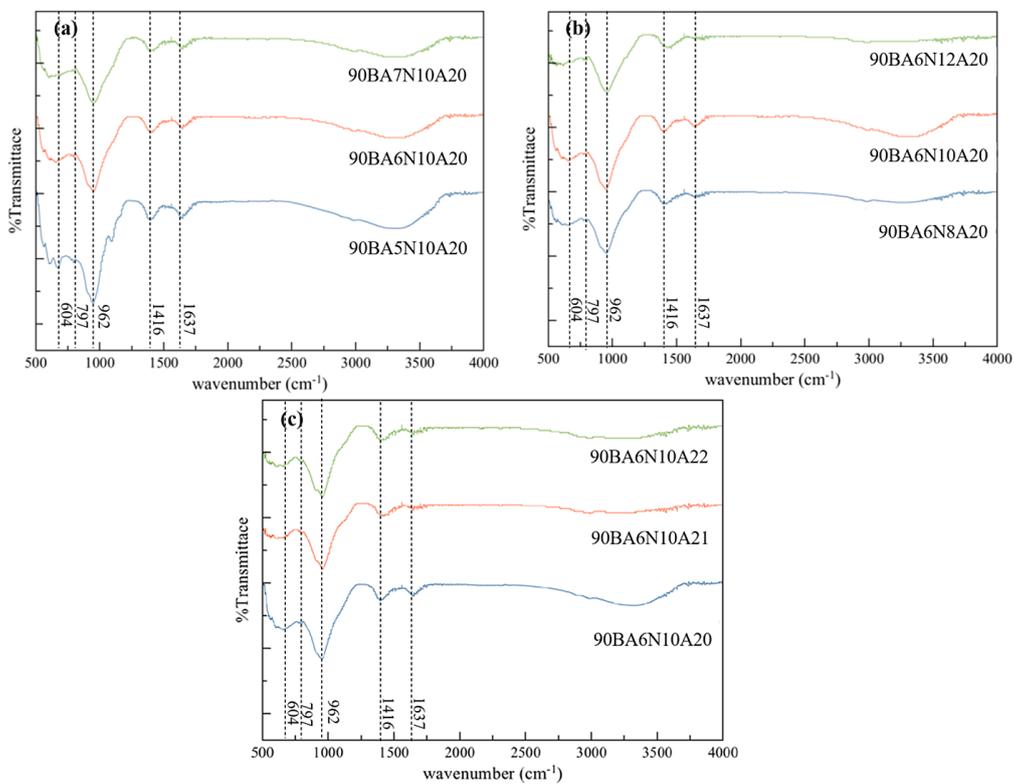


Figure 3. FTIR spectra of geopolymer samples after 7 days with different $\text{SiO}_2/\text{Al}_2\text{O}_3$ mole ratios (A), NaOH concentrations (B), and $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ mole ratios (C)

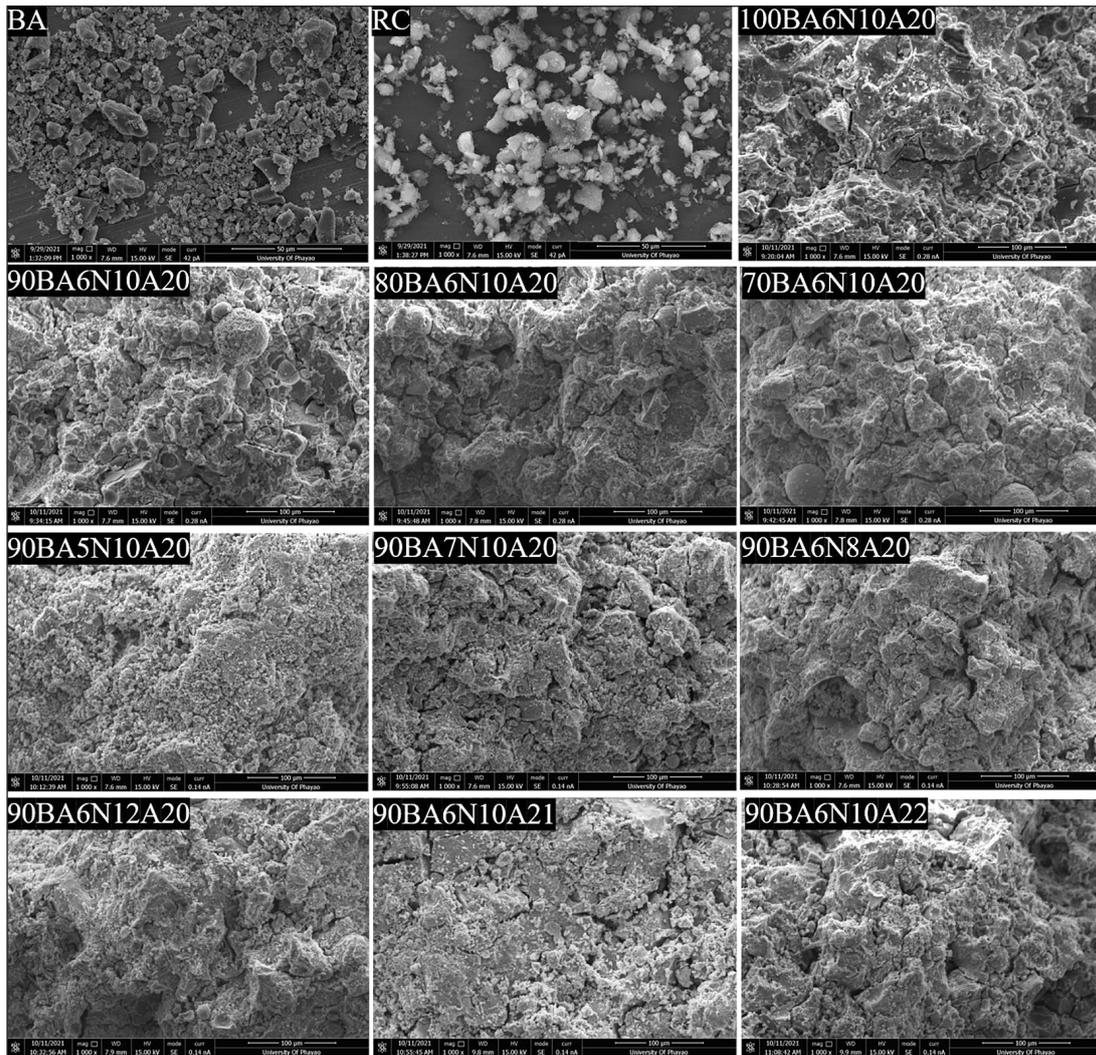


Figure 4. Micrograph of the starting materials and geopolymer samples after 7 days under different synthesis conditions

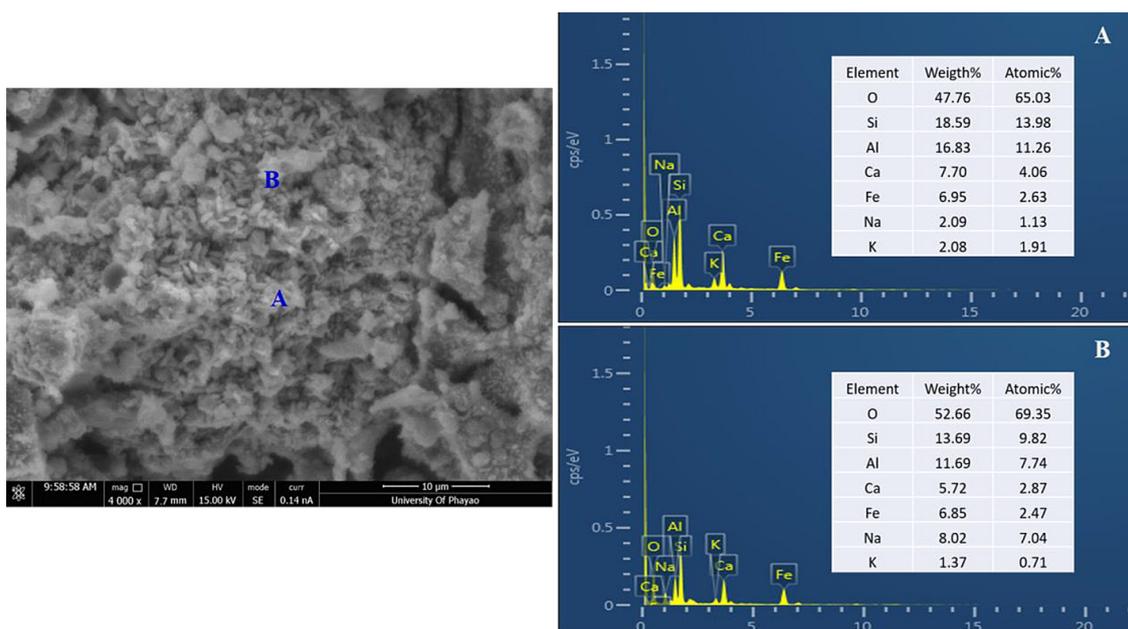


Figure 5. Micrograph showing the EDX result of 90BA6N10A21 after 7 days

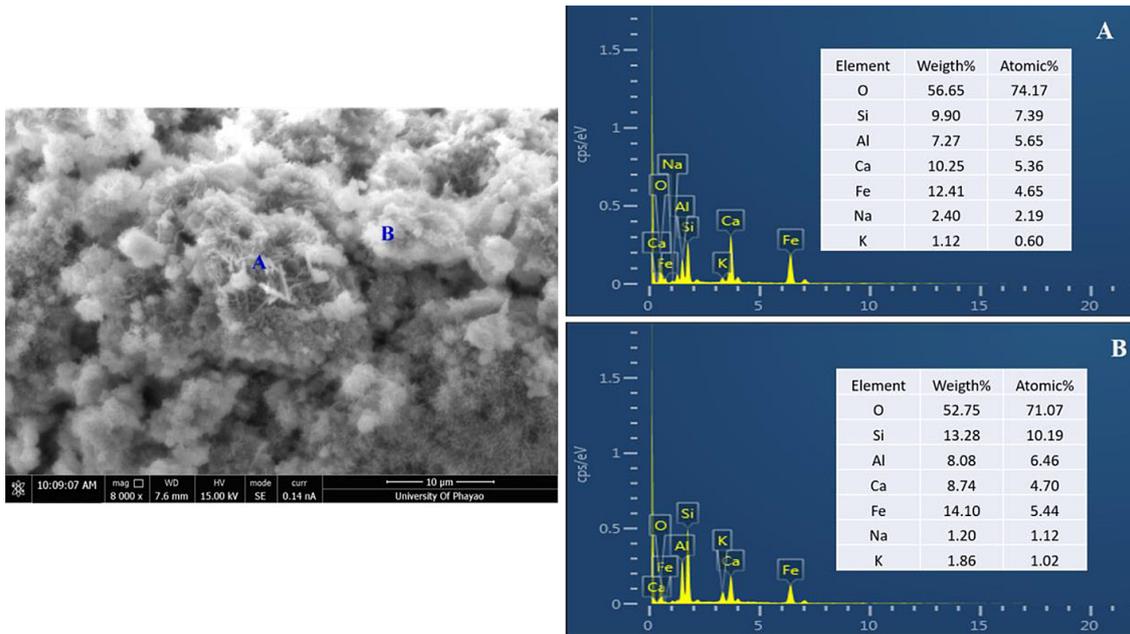


Figure 6. Micrograph showing the EDX result of 90BA6N10A20 after 7 days

presents the EDX analysis of the 90BA6N10A20 sample. The main hydration products identified were sodium aluminosilicate and calcium silicate hydrates, consistent with findings reported by Kaya et al. [25]. Additionally, needle-like iron silicates were observed, as reported by Zailani et al. [26]. These results confirmed that the reaction occurred on the surface from the crystallization of iron oxide and geopolymerization between iron and the alkali activator solution [23, 26].

Factors affecting the compressive strength

Figure 7 shows the compressive strength at 7 and 28 days. Many factors affect the mechanical

properties of geopolymer pastes and these are described in more detail below.

Effect of BA/RC weight ratio

This study found that blends prepared from the 100BA6N10A20 sample (Table 2), using only BA as the starting material for geopolymer synthesis, had insufficient liquid content for initial gel formation; thus, geopolymer could not be produced under this condition. However, gel formation occurred when the $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ mole ratios were increased to 2.1 and 2.2. The results showed that geopolymers with high compressive strength were obtained using BA/RC weight

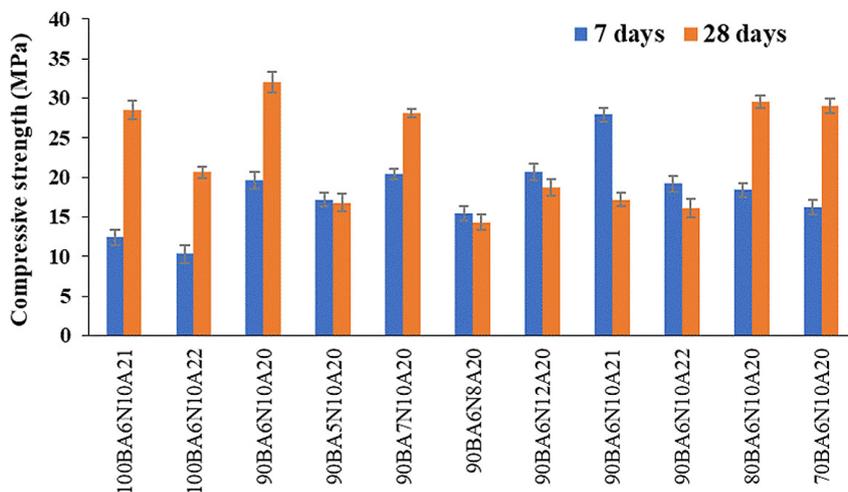


Figure 7. Compressive strength of geopolymer samples after 7 and 28 days under different synthesis conditions

ratios of 90/10, 80/20, and 70/30. Additionally, a BA/RC weight ratio of 60/40 (sample 60BA-6N10A20) resulted in slow geopolymer formation, taking more than 24 hours. Therefore, this condition is not suitable for the workability of the geopolymer. Furthermore, geopolymers prepared with high contents of BA exhibited greater compressive strength, leading to the selection of a BA/RC weight ratio of 90/10 for this study. The moles of SiO₂, Al₂O₃, CaO, and Fe₂O₃ in the sample mixtures using different BA/RC weight ratios are shown in Table 3. It should be noted here that the high CaO content in BA promoted glass microsphere dissolution during the geopolymerization or hydration processes, thereby accelerating the setting time [27]. High Fe₂O₃ in BA also promoted the formation of ferrosilicate covalent bonding derived from Si-O-T (T=Al, Fe or Si) during the geopolymerization process, as indicated by the FTIR analysis, leading to improved geopolymer strength [25]. Consequently, sample 60BA6N10A20, which has the lowest CaO and Fe₂O₃ content, exhibited a longer setting time.

Effect of SiO₂/Al₂O₃ mole ratio

The compressive strength of synthesized geopolymers using SiO₂/Al₂O₃ mole ratios ranging from 5 to 7 was investigated. Highest compressive strength was obtained using a SiO₂/Al₂O₃ mole ratio of 6, and this decreased at a SiO₂/Al₂O₃ mole ratio of 7 and significantly decreased at a SiO₂/Al₂O₃ mole ratio of 5. These results concurred with the XRD analysis, suggesting the occurrence of sodalite in samples obtained at a SiO₂/Al₂O₃ mole ratio of 5, resulting in decreasing strength. A high SiO₂ content formed SiO₄ tetrahedral units in the geopolymer structure that were more stable than AlO₄ tetrahedral units. The network structure of the geopolymer obtained from polycondensation contained more SiO₄ tetrahedral units than AlO₄ tetrahedral units. Thus, a high SiO₂/Al₂O₃ mole ratio promoted hardening,

forming high compressive strength geopolymers [22]. However, using a high SiO₂/Al₂O₃ mole ratio also impacts the defect density resulting from unreacted material, leading to an increase in the number of potential pathways to diminish polymerization efficiency [28]. Thus, the sample obtained using a SiO₂/Al₂O₃ molar ratio of 7 showed decreased compressive strength.

Effect of Na₂O/Al₂O₃ mole ratio

Geopolymers synthesized using a Na₂O/Al₂O₃ mole ratio of 2.0 showed lower compressive strength after 7 days than samples using Na₂O/Al₂O₃ mole ratios of 2.1 and 2.2. After 28 days, the compressive strength of the geopolymer using a Na₂O/Al₂O₃ mole ratio of 2.0 was higher, while using Na₂O/Al₂O₃ molar ratios of 2.1 and 2.2 gave lower compressive strength. Results indicated that an increase in the Na₂O/Al₂O₃ mole ratio enhanced the dissolution of starting materials to form polymerization monomers resulting in increasing the early strength. By contrast, using too high a Na₂O/Al₂O₃ mole ratio resulted in weaker bonding strength in the network structure of the geopolymer, thus causing early and late strength decrease. The anions of SiO₄ and AlO₄ tetrahedral units of geopolymer structures are [SiO₄]⁴⁻ and [AlO₄]⁵⁻. These have oxygen atoms that act as bridge connections during the process of polycondensation, and the Na⁺ ions compensated the negative charge of Al³⁺ ions [9]. Using a high Na₂O/Al₂O₃ mole ratio resulted in a negative charge at the aluminum site with an excess of Na⁺ ions, thus the bonding strength in the network structure was weakened, thereby resulting in decreased geopolymer strength.

Effect of NaOH concentration

The NaOH concentration used to synthesize the geopolymers significantly impacted their compressive strength. Geopolymer synthesis using 10M NaOH showed higher early and

Table 3. Moles of SiO₂, Al₂O₃, CaO and Fe₂O₃ in the sample mixtures using different BA/RC weight ratios

Sample code	SiO ₂	Al ₂ O ₃	CaO	Fe ₂ O ₃
	(mole)	(mole)	(mole)	(mole)
100BA6N10A20	0.533	0.089	0.567	0.185
90BA6N10A20	0.586	0.098	0.512	0.177
80BA6N10A20	0.638	0.106	0.458	0.170
70BA6N10A20	0.691	0.115	0.403	0.163
60BA6N10A20	0.743	0.124	0.349	0.155

late compressive strength than using 8 and 12 M NaOH, while late strength using 8 and 12 M NaOH, was lower than early strength. The NaOH solution affected the dissolution of silica and alumina from the starting materials in the polymeric system, with increasing NaOH molarity leading to increasing detachment of the active raw material elements, thereby promoting the formation of more monomers to form the network structure in the polycondensation process. However, a high concentration of NaOH increased the coagulation of silica, leading to decreasing geopolymer strength [22].

CONCLUSIONS

This study synthesized BA and RC into geopolymer material using mixed NaOH and Na_2SiO_3 solutions as an alkali activator. Factors affecting compressive strength as weight ratio of BA/RC, mole ratio of $\text{SiO}_2/\text{Al}_2\text{O}_3$, mole ratio of $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$, and concentration of NaOH were investigated. The following conclusions were drawn:

1. The BA had high CaO and Fe_2O_3 contents, therefore increasing the BA/RC weight ratio accelerated the setting time and also improved geopolymer strength.
2. Increase in the $\text{SiO}_2/\text{Al}_2\text{O}_3$ mole ratio promoted hardening to form high compressive strength geopolymers, with too high $\text{SiO}_2/\text{Al}_2\text{O}_3$ mole ratios causing increased defect density.
3. Increase in the $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ mole ratio improved the dissolution rate and enhanced the workability. Using too high a $\text{Na}_2\text{O}/\text{Al}_2\text{O}_3$ mole ratio reduced the bonding strength in the network structure causing decreased geopolymer strength.
4. Increasing the NaOH concentration enhanced the dissolution of silica and alumina from BA and RC, thereby increasing the geopolymer gel network, while too high a concentration increased silica coagulation.
5. Geopolymer synthesis using BA and RC was shown to be economically advantageous, offering a valuable option for reducing environmental contamination.

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