

Article

The Effect of Red Mud Content on the Compressive Strength of Geopolymers under Different Curing Systems

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Abstract: To maximize the utilization of red mud in geopolymers, a red mud–metakaolin (RM-MK) geopolymer and red mud–fly ash (RM-FA) geopolymer were prepared, respectively. The effects of red mud content on the compressive strength and microstructure of the geopolymers were investigated under three different curing conditions. The results showed that the strength of the geopolymer decreased linearly with an increase in the red mud content, whether curing at room temperature or 80 °C. Surprisingly, curing in an autoclave, the appropriate amount of red mud had a favorable impact on the mechanical properties of the geopolymers. When the amount of red mud was 50%, the strength of the RM-MK geopolymer reached its highest compressive strength, 36.3 MPa, and the strength of the RM-FA geopolymer reached its highest at 31.7 MPa. Compared with curing at low temperature, curing the red mud-based geopolymers under a higher temperature and higher pressure can maximize the use of red mud. XRD analysis indicated that zeolite minerals formed. The SEM results showed that the geopolymers cured in an autoclave had a dense structure.



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1. Introduction

Red mud is an alkaline solid waste residue generated from the alumina-refining process of bauxite. On average, about 1 to 1.5 tons of red mud are generated from 1 ton of alumina production [1], while its utilization is extremely low [2]. Due to its high content of alkalinity and heavy metals [3], the accumulation of large areas of red mud not only occupies an enormous land area but also poses a serious threat to the surrounding soil, air, water, and human health [4]. Therefore, it urgently necessary to properly treat red mud and realize the utilization of this resource. The currently reported methods for treating red mud include [4–8] (1) recovery of valuable elements in red mud, such as Fe, Al, Ca, Ti, etc.; (2) modified red mud is used as an adsorbent to absorb pollutants in air and water and improve the soil; and (3) red mud is rich in silicate components, such as Si, Al, Fe, and Ca, which can be used to prepare building materials, including cement, glass-ceramics, concrete, and brick. Typically, application in building materials can increase red mud use to the greatest extent. Much research on the application of red mud in building materials have been conducted at home and abroad. Red mud contains a large amount of iron, which has the potential to prepare high-iron aluminum cement. Zhao et al. [9] prepared sulfur aluminate cement with Bayer red mud and fly ash. The use of red mud can reduce the firing temperature of the clinker. In addition, a proper amount of red mud was beneficial to the formation of C_4A_3S and the improvement of compressive strength. Qi et al. [10] successfully prepared a new red mud road base material with excellent performance. Compared with traditional semi-rigid base materials, the red mud base material had a higher strength and resilience modulus, which was suitable for the construction of asphalt pavement bases for highways and expressways. Yang et al. [11] successfully developed a glass-ceramic $CaO-SiO_2-Al_2O_3$ system mostly with red mud

and fly ash. The proportion of the two industrial wastes, red mud and fly ash, was up to 85 wt%, which has significant economic and environmental benefits. Furthermore, some researchers have used red mud and solid wastes to prepare non-fired bricks and sintered bricks that met the standards [12–14]. Although the above research has good application prospects, the existence of alkaline substances and heavy metal elements in red mud limit the large-scale utilization of red mud in traditional buildings, compared to cement [7,15]. Moreover, the current dealkalization technology is not mature and the economic cost is too high [16].

A geopolymers is a new type of green gelling material formed by the reaction of high-silica-alumina natural minerals or solid wastes with alkaline activators. At room temperature or low temperature, the Si–O bond and Al–O bond in the raw material are broken under the action of the alkaline catalysts and then reorganized [17–19]. The reaction mechanism of the geopolymers is extremely complicated, and the reaction process can be summarized as three processes: dissolution–recombination–solidification. The specific reaction steps are [20–22] (1) $[\text{SiO}_4]$ and $[\text{AlO}_4]$ from the aluminosilicate raw material are dissolved in alkaline solutions; (2) under the action of an alkali activator, the monomers are dehydrated and condensed into aluminosilicate oligomers, and the gel is formed; and (3) excess water in the gel phase is drained and an aluminosilicate polymer gel is formed. With the advantages of abundant raw materials, a simple preparation process, energy-saving, environmental protection, excellent mechanical properties, and durability [23,24], geopolymers are expected to become green cementitious material to replace Portland cement.

Red mud contains a great deal of silicon and aluminum compounds, which can be used as raw materials for geopolymers [25]; in addition, the alkalinity in red mud can be used effectively to reduce the cost. Moreover, the toxic metals in the red mud are sealed because of the special three-dimensional network structure of the geopolymers [26]. Therefore, the preparation of a geopolymers from industrial waste red mud not only follows the concept of environmental protection but also has certain application value, which is a highly effective way to realize its resource utilization.

The polymerization reaction of a geopolymers is a complicated process that is affected by many factors, such as the performance of the raw materials (e.g., reactivity, Si/Al ratio, and the quantity and characteristics of the unreacted phases), the concentration of alkaline solutions, and the curing conditions (e.g., temperature, humidity, and pressure) [27–31]. All of the factors ultimately determine the microstructure and mechanical properties of the geopolymers. To improve the activation reaction rate and increase the strength of the geopolymers, red mud is often combined with fly ash, metakaolin, ground blast furnace slag, rice husk ash, and other highly active materials. Kumar et al. [32] investigated the effect of 0–40% red mud addition on the properties of fly ash geopolymers. The results showed that the strength of the geopolymers improved with the addition of red mud but the improvement in strength was only for 5–20% of the red mud content. When the red mud content was 10%, the more intense reaction, structural rearrangement, and better mechanical properties of the geopolymers were mainly attributed to the dense structure caused by the optimal sodium hydroxide concentration and available silicon and aluminum. He et al. [17] prepared a red mud–rice husk ash geopolymers with a compressive strength of 20.5 MPa from two industrial wastes, red mud and rice husk ash, which was comparable to most Portland cement.

The curing system plays an important role in the synthesis and strength development of a geopolymers. Kani et al. [33] studied the effect of curing time and temperature on the strength development of a natural pozzolan-based geopolymers. They concluded that the increase in curing temperature and time had a positive effect on the strength of the geopolymers. Before heating, a long pre-curing time in a humid environment was beneficial to the strength development of the natural pozzolan-based geopolymers. Zhang et al. [34] investigated the reaction kinetics of red mud–fly ash-based geopolymers, and mainly analyzed the effects of curing temperature on the formation of geopolymers gels, porosity,

and mechanical properties. He pointed out that mechanical strength is largely affected by the development of geopolymers and porosity, both of which were affected by the curing temperature. Studies indicated that the SiO_2 in red mud was insoluble in alkaline solutions and did not participate in geological polymerization at atmospheric pressure. Therefore, the higher the red mud content was, the lower the geopolymers strength was [35]. It was found that the SiO_2 in red mud can be effectively utilized to participate in the reaction by solidifying the red mud-based polymer under high temperature and high pressure in an autoclave. Hoang et al. [36] investigated the effect of the curing regime on the synthesis and properties of alkali-activated red mud materials. The SiO_2 in the red mud began to dissolve. Additionally, the dissolution rate of the silica in the red mud and the mechanical properties of the materials were improved by increasing the pressure, time, and alkali liquor concentration in the autoclave. It was predicted that red mud after autoclave curing can participate in the synthesis reaction similar to fly ash under high-temperature curing, which was derived from the result that in the same concentration sodium hydroxide solution, the dissolution rate of silicon dioxide in red mud after high-temperature curing reached and exceeded that of silicon dioxide in fly ash. The solubility of Al_2O_3 both in red mud and fly ash improved with the increase of pressure, temperature, and alkali liquor concentration.

The abovementioned research studies provide a theoretical basis for the polymerization process and strength development of the red mud-based polymer. However, previous research focused on how to improve the strength of the red mud-based polymer, and there are very few studies on improving the utilization rate of red mud while increasing its strength. This article aims to increase the amount of red mud as much as possible in the process of preparing high-performance red mud-based polymers. Different proportions of a red mud-metakaolin geopolymers and red mud-fly ash geopolymers were prepared. The influence of the red mud content on the strength of the geopolymers under different curing systems was explored. To further clarify the relationship between the geopolymers synthesis conditions and mechanical properties, the phase composition and microstructure of the geopolymers under different curing systems were characterized by XRD and SEM.

2. Materials and Methods

2.1. Materials

Red mud (RM), metakaolin (MK), and fly ash (FA) were used as raw materials to provide aluminosilicate. The red mud used in the experiment was obtained by the Bayer process. The metakaolin was prepared by calcining kaolin at $750\text{ }^\circ\text{C}$ for 4 h. Class F fly ash was used in this experiment. The main chemical composition of each raw material in Table 1 is obtained by XRF test. It is seen from Table 1 that the content of Al_2O_3 in the red mud is higher than that of SiO_2 . The content of Fe_2O_3 is 34.5%, which determines that the red mud appears red in appearance. The high sodium hydroxide content is the reason for the high alkalinity of red mud. Metakaolin contains a great number of aluminates, with SiO_2 and Al_2O_3 accounting for more than 95%. The essential components of fly ash are SiO_2 , Al_2O_3 , CaO , and Fe_2O_3 . Figure 1 shows the XRD pattern of red mud. The main mineral composition of red mud is hematite (Fe_2O_3), sodalite ($\text{Na}_8(\text{Al}_6\text{Si}_6\text{O}_{24})\text{Cl}_2$), calcite (CaCO_3), anatase (TiO_2), and cancrinite ($\text{Na}_8(\text{Al}_6\text{Si}_6\text{O}_{24})((\text{CaCO}_3)_2)$). An alkaline activator was made up of sodium silicate, 95% pure sodium hydroxide, and deionized water in a certain proportion.

Table 1. The main chemical composition of the raw materials (%).

Chemical Constituent	SiO_2	Al_2O_3	CaO	Fe_2O_3	Na_2O	K_2O	TiO_2	SO_3
RM	16.2	22.9	1.8	34.5	8.7	—	8.4	—
MK	51.9	45.45	0.142	0.93	0.05	0.445	—	—
FA	43	30.8	3.05	2.23	0.08	0.7	—	0.98

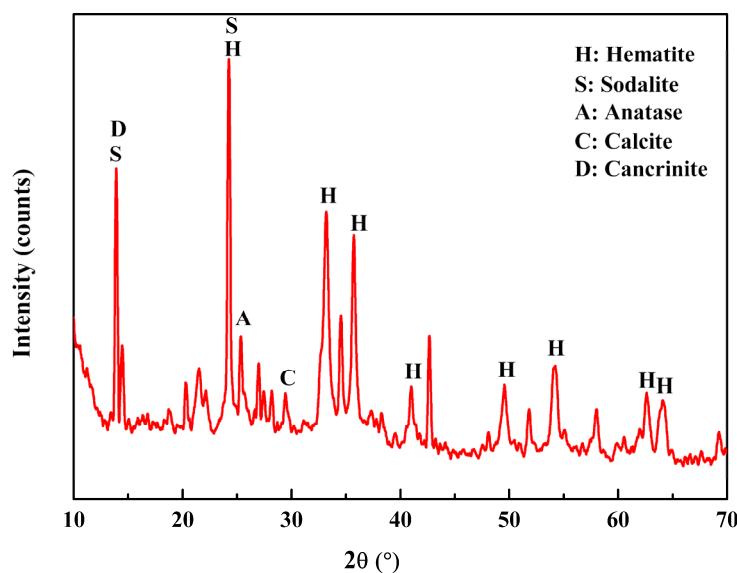


Figure 1. XRD pattern of the Bayer red mud.

2.2. Geopolymer Synthesis

According to the optimal value of the ratio of a high-strength geopolymer reported previously [19,37], in this study, the Si/Al molar ratio and Na/Al ratio of the geopolymers prepared were designed as 2 and 1, respectively. According to a water–solid ratio of 0.5, the alkaline activator was made by mixing 8 mol/L sodium hydroxide, sodium silicate, and deionized water in a certain ratio. Red mud and metakaolin (fly ash) were mixed with an alkaline activator to prepare the geopolymer precursor in a slurry state. A total of 5~6% distilled water was added as a regulator to ensure that the silicoaluminate raw material was fully dissolved in the alkaline solution. The mixed geopolymer slurry was poured into a plastic mold with an inner diameter of 3.6 cm. The height of the geopolymer sample was 3.6 cm. Subsequently, the samples were cured and molded after vibration exhaust. Compressive strength was examined by a universal testing machine. The specific experimental process is recorded in Figure 2.

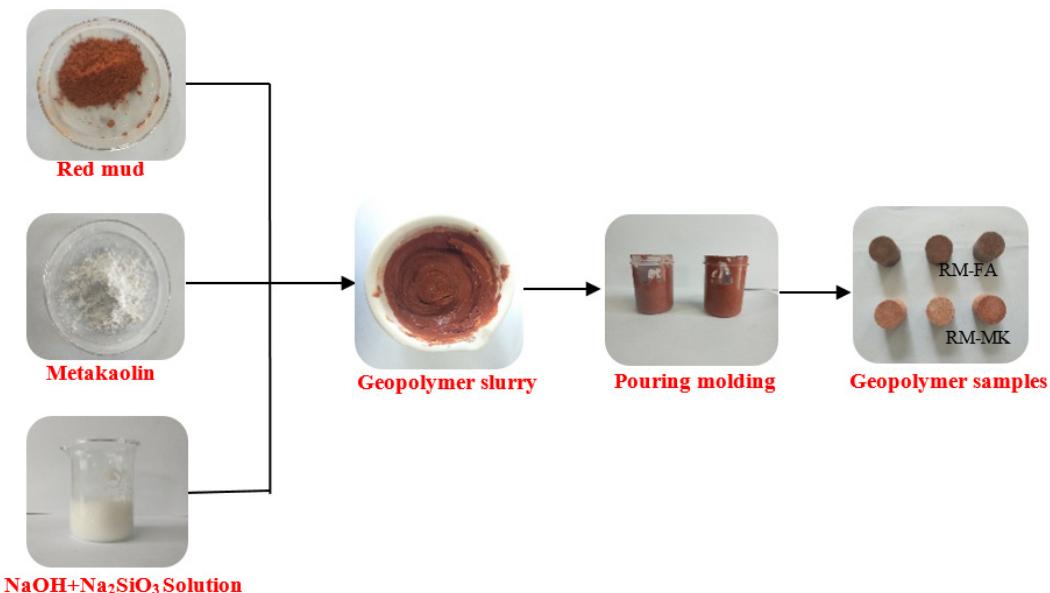


Figure 2. The preparation process of the geopolymer.

To study the effect of curing conditions on the development of the mechanical properties of the geopolymer, three different curing methods were employed, including curing at room temperature for 7 d, curing at 80 °C for 24 h, and curing at 220 °C in the autoclave for 8 h. One should seal the sample at 80 °C and in the autoclave to prevent the moisture from evaporating too fast. To study the influence of red mud content on the mechanical properties of the geopolymers under different curing systems, the mass ratios of the red mud and metakaolin were set as 1:2, 1:1, 2:1, and 3:1, respectively, with other factors unchanged. To ensure reproducibility, three duplicate samples were prepared for each proportion to obtain an average value.

2.3. Characterization Methods

2.3.1. Compressive Strength Test

The mechanical properties of the geopolymer were performed by an unconfined compression test. The diameter of the bottom surface of the cylinder sample was 3.6 cm, measured by a vernier caliper, and the compression area (S) was 10.2 cm². Before the test, both ends of each sample were polished with sandpaper to ensure that the surface of both ends of the samples was smooth, to prevent the experiment error caused by uneven force. The sample was placed horizontally on the compressive strength testing machine for the test of compressive strength. The loading speed was 1 kN/s. The maximum load F (kN) when the specimen was damaged was recorded.

The calculation formula of compressive strength F_{cu} (MPa) is as follows:

$$F_{cu} = F / S \quad (1)$$

where F is the maximum load when the sample was damaged, kN; and S is the compression area of the sample, m².

2.3.2. Structural Characterization of the Geopolymers

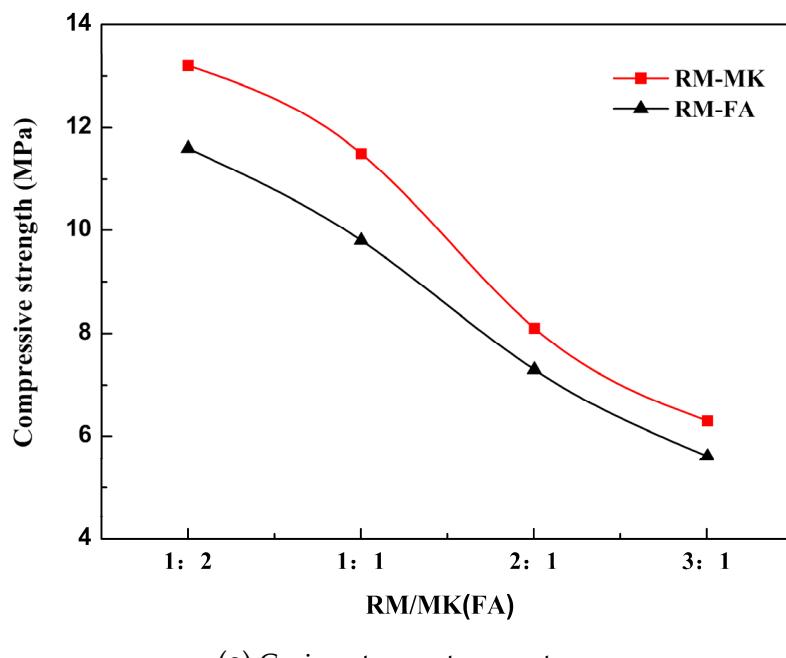
X-ray Diffraction (XRD) was used to analyze the phase composition of the geopolymers quantitatively and qualitatively. The sample was dried in a vacuum drying oven at 60 °C for 2 h and then ground into a powder. The mineral composition of the geopolymer powder was characterized by X-ray powder diffraction (XRD) (Brooke AXS D8 ADVANCE, Germany). XRD scans used Cu K α radiation, a scanning speed of 10°/min, and a scan range of 10–80° 2 θ (diffraction angle). Scanning electron microscopy (SEM) was used to observe the microstructure of the reaction products. Flat and representative cross-sections were elected and sprayed with gold. A Hitachi S4800 scanning electron microscope (Hitachi, Japan) was used to observe the surface morphology and structural changes of the geopolymers at a 10 kV scanning voltage.

3. Results and Discussion

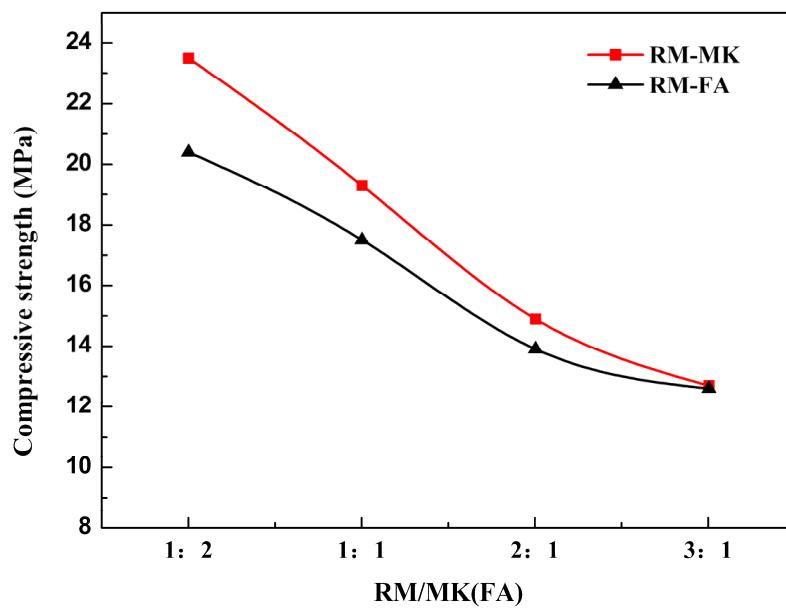
3.1. Compressive Strength

The relationship between the content of the red mud and the compressive strength of the geopolymers under different curing systems is shown in Figure 3. It can be found that the changing trend in compressive strength of the red mud–metakaolin geopolymer and red mud–fly ash geopolymer with the content of red mud is similar. Apparently, the strength of the RM-MK geopolymer is higher than that of the RM-FA geopolymer. This result is due to the large amount of soluble aluminosilicate in metakaolin. As is shown in Figure 3a,b, curing at room temperature and 80 °C under atmospheric pressure, the improvement in the proportion of red mud is not conducive to the development of geopolymer strength. The strength of the geopolymer decreases as the proportion of red mud increases. On the one hand, at atmospheric pressure, the insoluble silicon and low solubility aluminum in red mud cannot be used as the major source of aluminosilicate during the polymerization process, but only as inactive fillers [38]. On the other hand, the high aluminum content in red mud is another reason for the strength reduction. The Al–O bond breaks faster than the Si–O bond [39]. The Al–O bond is rapidly dissolved in the alkali solution and the raw

material particles are wrapped by the generated aluminum-rich gel phase, which prevents the further dissolution process of aluminosilicate [25,30], leading to a decrease in strength.

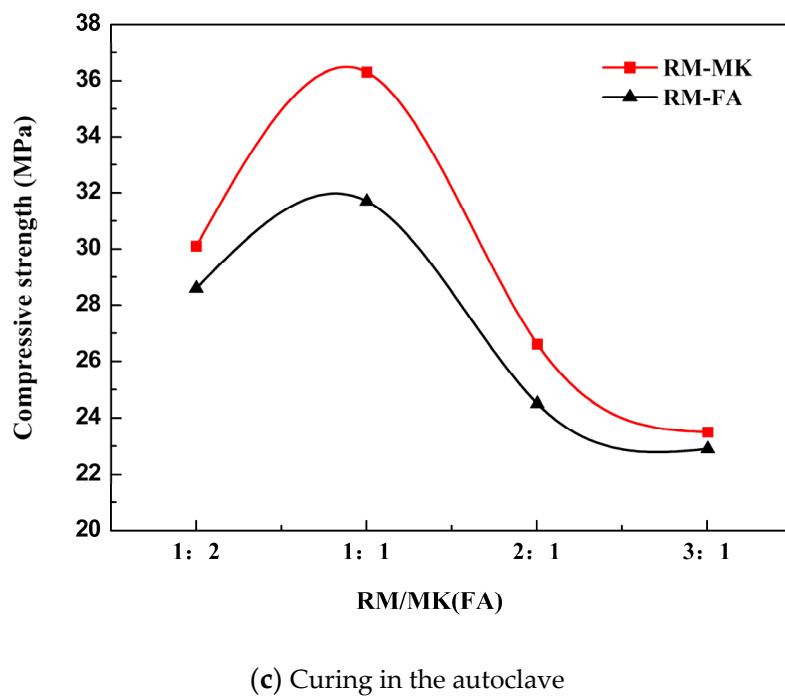


(a) Curing at room temperature



(b) Curing at 80 °C

Figure 3. Cont.



(c) Curing in the autoclave

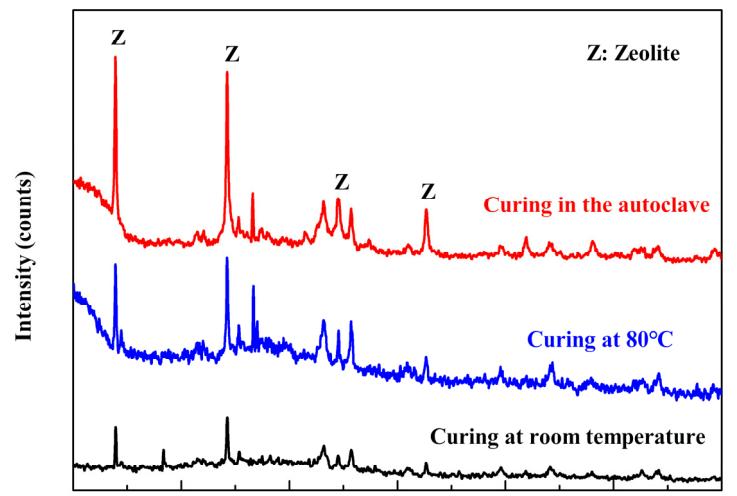
Figure 3. Variation in geopolymer compressive strength with red mud content.

Amazingly, curing in the autoclave, the compressive strength increases first and then decreases with an increase in the red mud proportion. When the red mud content is 50%, the strength of the geopolymer reaches its maximum value, for the RM-KM geopolymer this being 36.3 MPa and for the RM-FA geopolymer 31.7 MPa. This is different from the results obtained under the previous two curing methods. It has been stated that the SiO_2 in the red mud began to dissolve and participated in polymerization under a high-pressure condition in previous work. The Si–O bond broke and then recombined, and stronger Si–O–Si bonds and Si–O–Al bonds were formed. Thus, a stable three-dimensional network polymer structure was framed. In addition, the residual alkali in the red mud accelerates the dissolution of the raw materials and promotes the synthesis of the geopolymer [40]. However, the dissolution rate of the raw material and the subsequent polymerization reaction rate are limited as the red mud content increases. As a result, the intensity goes down. From the above results, the first step of geological polymerization (the dissolution of silicoaluminate raw material in alkaline solution) plays a very critical role in the whole polymerization process. When the synthesis condition was changed, the role of the SiO_2 in the red mud changed from an inactive filler to active substance. Therefore, a high-strength geopolymer with 50% red mud content can be obtained when curing under high temperature and high pressure in an autoclave. High red mud usage, given the excellent mechanical properties of its products, provides a possibility for its extensive application in the field of building materials.

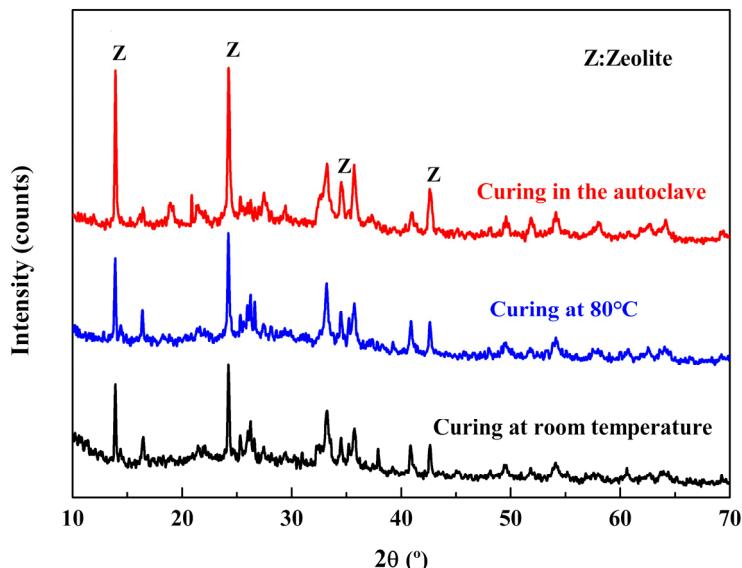
In previous studies, the strength of the geopolymer decreased with an increase in red mud content. Generally, the best dosage of red mud was 20–40% [32,40]. In this study, the effect of curing methods on the compressive strength of the red mud geopolymers was studied, and the microstructure of the geopolymers was characterized. Compared with previous studies, the preparation of a geopolymer with red mud in the autoclave not only can obtain a high-strength geopolymer but also ensures the best use of red mud, up to 50%, which is something we did not find before.

3.2. X-ray Diffraction (XRD) Analysis

Figure 4 shows the XRD patterns of the geopolymers with a 50% red mud content solidified under different conditions. The XRD patterns of the red mud–metakaolin geopolymer and red mud–fly ash geopolymer have similar changes in phase structure.



(a) RM-MK geopolymers



(b) RM-FA geopolymers

Figure 4. XRD pattern of the geopolymers with a 50% red mud content.

In the XRD pattern, the zeolite phases can be clearly observed. The diffraction peak intensity of the zeolite phases increases gradually with an increase in curing temperature and pressure. A large number of zeolite minerals in the geopolymer cured in the autoclave indicate that the degree of polymerization of the $[\text{SiO}_4]$ and $[\text{AlO}_4]$ monomers is maximized [41].

3.3. Scanning Electron Microscopy (SEM) Analysis

Figure 5 shows the microstructure of the geopolymers with a red mud content of 50% cured under different systems. The SEM of the RM-MK geopolymer is shown in

Figure 5. Curing at room temperature (Figure 5a), the geopolymer is made up of porous, loose gel particles. As the curing temperature increases (curing at 80 °C) (Figure 5b), the porosity between the cementing products decreases and the density improves. Curing in the autoclave (Figure 5c), the geopolymer shows continuous and denser gels, indicating that the degree of polymerization of the geopolymer is further improved. A small part of the unreacted raw materials and the zeolite crystal structure is embedded in the geopolymer gel phase to form a uniform and dense structure, bringing about a high strength and stiffness of the geopolymer. The same conclusion can be obtained in the microstructure of the RM-FA geopolymer in Figure 6.

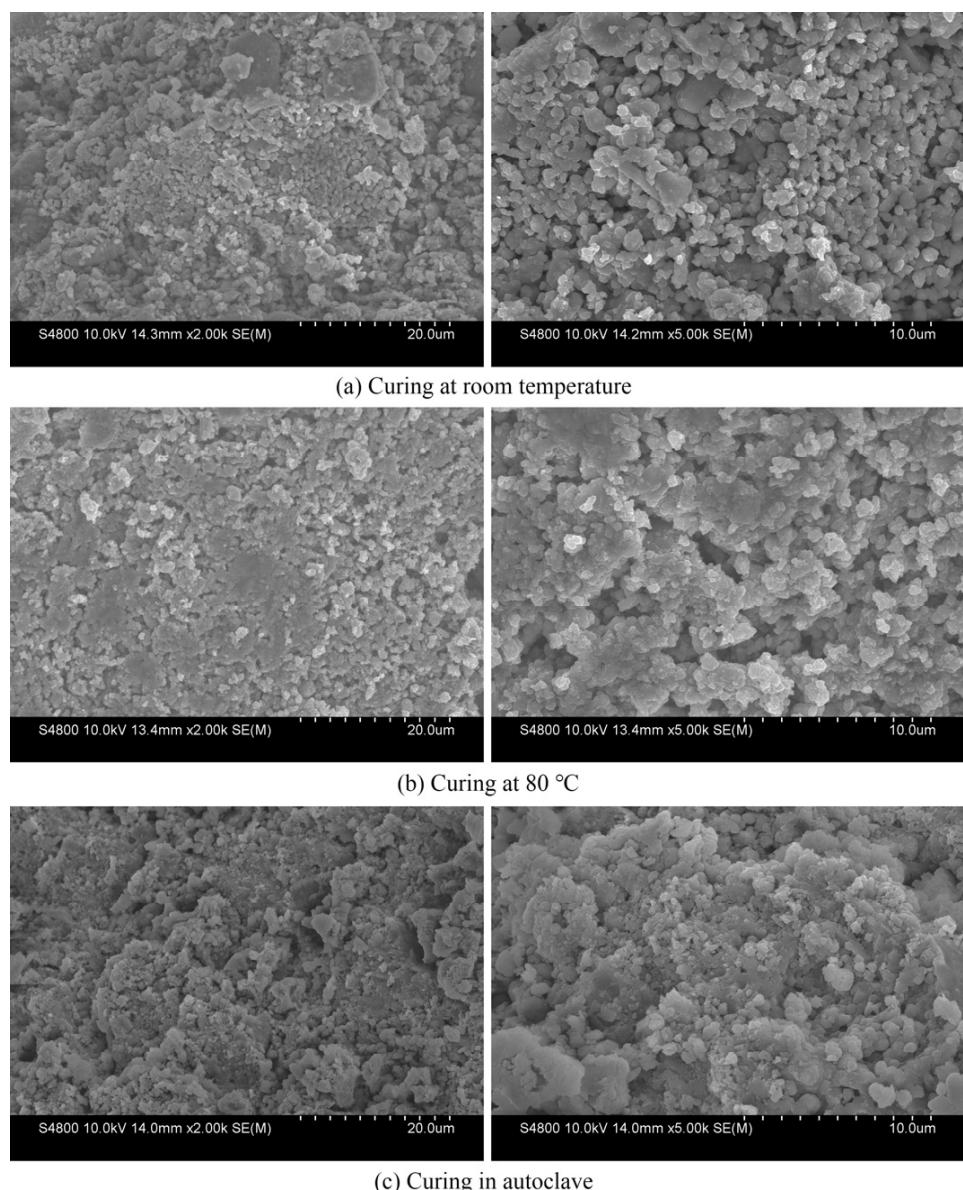


Figure 5. SEM micrographs of the RM-MK geopolymers with a 50% red mud content.

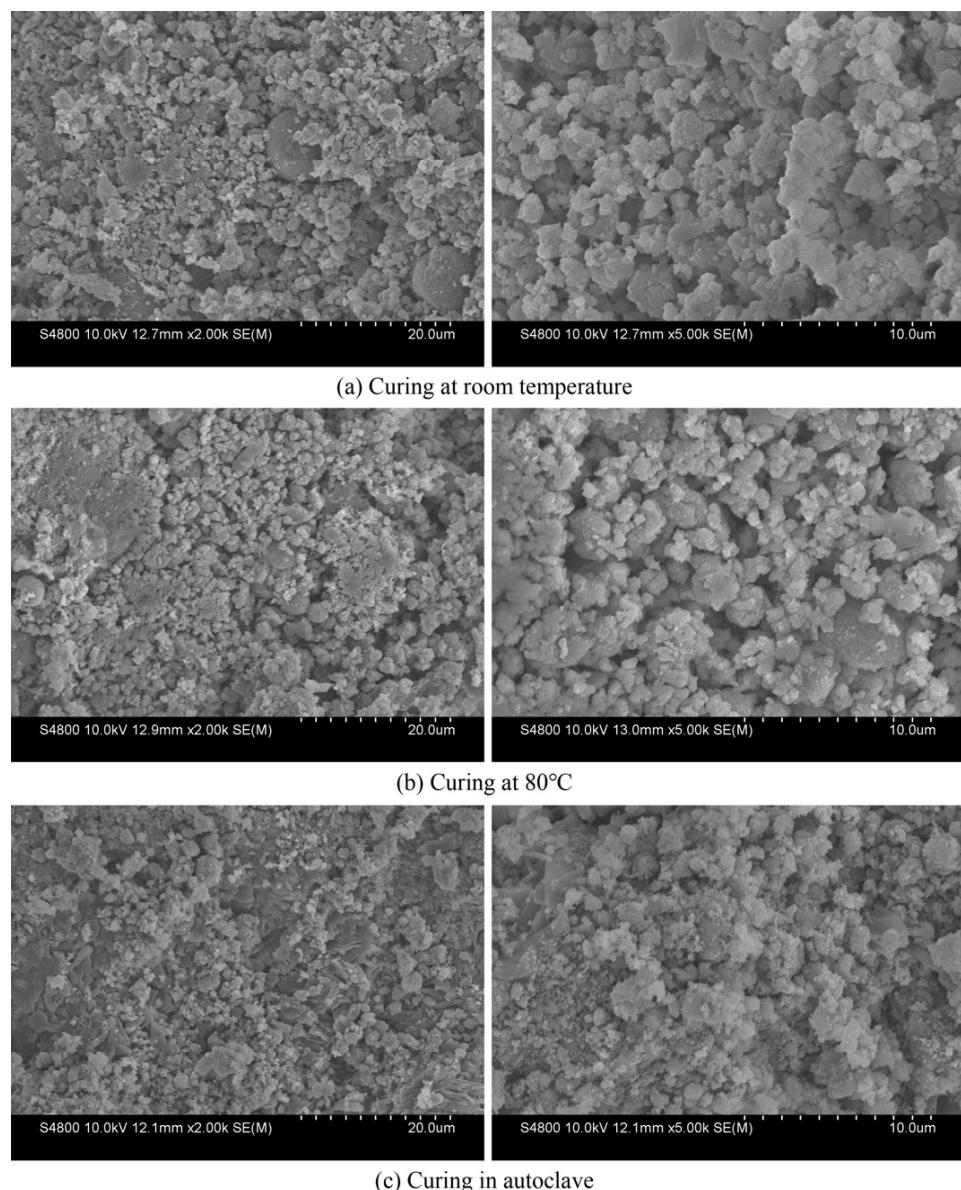


Figure 6. SEM photography of the RM-FA geopolymers with a 50% red mud content.

4. Conclusions

In this paper, three curing methods were studied, namely, room temperature, 80 °C, and high pressure in an autoclave. The mechanical properties and microstructure of the geopolymers were examined by changing the proportion of red mud. The crucial conclusions obtained from the experimental results are as follows:

1. The curing system affects the synthesis and mechanical properties of the geopolymers tremendously. Curing at room temperature and 80 °C, the higher the proportion of red mud, the lower the strength of the geopolymer. Yet, curing at high pressure, the higher red mud content improves the strength. A 50% red mud content is the optimal value at which the geopolymers have their highest compressive strength.
2. The curing method affects the first step of the polymerization of the red mud-based polymer: dissolution of aluminosilicate in an alkali solution. Compared with curing under atmospheric pressure, autoclave solidification can make it possible for the silica in the red mud to dissolve in an alkaline solution, and a more silica-rich gel phase is produced. Furthermore, the alkali in the red mud accelerates the dissolution of the aluminosilicate under high pressure.

3. The results of the XRD and SEM analyses show that more amorphous silicate gel products and zeolite structures are generated at a high pressure. This result illustrates that the geopolymer reaction is more sufficient. With the decrease in porosity and the densification of the structure, the geopolymers show good mechanical properties.

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Conflicts of Interest: The authors declare no conflict of interest.

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