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Utilization of Silicon Carbide Sludge as Metakaolin-Based Geopolymer Materials

Kae-Long Lin ¹ , Kang-Wei Lo ^{2,3,*} , Ta-Wui Cheng ³ , Wei-Ting Lin ⁴ and Ya-Wen Lin ³

¹ Department of Environmental Engineering, National Ilan University, Ilan City 260, Taiwan; kllin@niu.edu.tw

² Graduate Institute of Engineering Technology, National Taipei University of Technology, Taipei City 106, Taiwan

³ Institute of Mineral Resources Engineering, National Taipei University of Technology, Taipei City 106, Taiwan; twcheng@ntut.edu.tw (T.-W.C.); urine1001@gmail.com (Y.-W.L.)

⁴ Department of Civil Engineering, National Ilan University, Ilan City 260, Taiwan; wtlin@niu.edu.tw

* Correspondence: dark83054689@gmail.com; Tel.: +886-3935-7579

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Abstract: The recycling of SiC sludge material is crucial for resource reutilization and environmental protection. In the current study, the effect of the mass ratio between the Na₂SiO₃ and sodium hydroxide (NaOH) solutions (NS/SS ratio) and the effect of SiC sludge on metakaolin geopolymers was comprehensively investigated to determine the underlying performance of the geopolymerization system. During thermal evolution, the second exothermic peak of 1.6NS10SCS (NS/SS ratio: 1.6, 10% SiC sludge) showed a heat evolution value of 990.6 W/g, which was the highest among other geopolymers. Additionally, the 1.6NS10SCS sample after 28 days of curing showed the highest flexural strength (6.42 MPa), compared to that of the others, and the DTA/TG (differential thermal analysis/thermogravimetry) results showed that the weight loss percentage increased to 14.62% from 400 to 750 °C. For the ²⁹Si nuclear magnetic resonance deconvolution, 1.6NS10SCS exhibited high fractions of Q⁴(3Al) (33.63%), Q⁴(2Al) (23.92%), and Q⁴(1Al) (9.70%). Thus, the geopolymer with the optimal SiC-sludge replacement level and NS/SS ratio contained more macropores and geopolymer gels, which benefit structural development. The experimental results indicated that SiC-sludge can potentially serve as a partial replacement for metakaolin and exhibited favorable mechanic characteristics.

Keywords: utilization; SiC sludge; alkaline activator solutions; synergistic effect; geopolymer reaction

1. Introduction

The solar cells generated electricity will become the primary global energy source, because the solar power was safe, efficient, nonpolluting and reliable [1]. Recycling silicon is of great importance, because the demand for silicon for solar cells is increasing globally, while concern about the limited silicon supply is emerging [2]. Generally, solar cells use multicrystalline wafers made of silicon [1]. Crystalline silicon cells have laboratory energy conversion efficiencies over 25% for single-crystal cells and over 20% for multicrystalline cells. In 2008, more than 100 GW silicon solar panels were produced based on an energy-intensive Siemens process [3], which cost approximately 400,000 tons of silicon. There were various types of solar cells, whereby the C-Si solar cell dominates 80% of the market globally [1]. Industry mainly uses silicon carbide (SiC) for wafer slicing [4], and a substantial amount of SiC sludge is formed when a silicon ingot is cut into wafers, with a kerf loss of over 50% [5]. The kerf-loss silicon waste is greater than 200,000 tons per year, and this sludge contributes to environmental pollution. Therefore, the recycling of SiC sludge material is crucial for resource reutilization and environmental protection. In addition, the primary composition of SiC sludge is SiO₂

and Al_2O_3 . Therefore, SiC sludge can be used as a raw material in producing silicon carbide sludge metakaolin-based (SCSMB) geopolymers.

Inorganic polymer (geopolymer) is a cementation material that can be used as an alternative to ordinary Portland cement. Geopolymers are mostly formed by mixing silicate aluminum sources (industrial waste such as slag and fly ash) with alkaline silicate solutions or an alkaline metal [6]. The polymerization process produced a three-dimensional (3D) compound comprising of $[\text{AlO}_4]$ and $[\text{SiO}_4]$ tetrahedrons [7,8]. Geopolymerization begins with the generation of -Si-O-Al-O-Si- or -Si-O-Al- monomers (oligomers) in the solution. These monomers join to form a geopolymer. As a structural material, a geopolymer is a potential replacement material for ordinary Portland cement (OPC). The most common alkaline activator solution (AAS) is a combination of Na_2SiO_3 and NaOH. AAS plays a crucial role in the polymerization process, which in turn has a strong dependence on the Si/Al ratio, $\text{M}_2\text{O}-\text{H}_2\text{O}$ ratio (where M = Na and/or K), hydroxide concentration, and alkali cation used [9–12].

Recently, there have been many studies on SiC sludge applied in the field of geopolymer materials. For example, Medri et al. [13] reported that silicon carbide (SiC) foams were developed using a low-temperature process, such as chemical consolidation, that is suitable for replacing the sintering step. The amorphous silica layer covering the surface of SiC particles participates in geopolymerization so that nanoprecipitates easily form on the SiC surface [13]. Rahman et al. [14] reported that silicon carbide whiskers were introduced into sodium-based geopolymers in order to evaluate the response of silicon carbide whiskers to the interfacial bonding and strength of sodium-based geopolymers along with rice husk ashes. Results show that the simultaneous additions of rice husk ashes and silicon carbide whiskers resulted in flexural strength improvement by ~27% and ~97%, respectively. The increase in flexural strength due to simultaneous inclusion of silica and silicon carbide whiskers suggests that silica particles are compatible with the metakaolin-based geopolymers, which is effective in consolidation [14]. Medri et al. [14] reported that refractory paints based on silicon carbide (SiC) were developed using inorganic alkali-aluminosilicate binders. R-SiC coatings applied by brushing on a Si_3N_4 -TiN substrate cannot act as barriers against oxygen penetration due to the presence of large pores [15]. Bai et al. [16] reported that high-strength open-celled SiC/geopolymer foam composites (SFCs) were fabricated by a combined saponification/peroxide route. The optimal sample possessed a total porosity of up to ~84 vol%, an open porosity of ~83 vol%, a thermal conductivity of 0.15 W/mK, and a compression strength of ~1.1 MPa [16]. Jia et al. [17] reported that bulk and porous AlN/SiC-based ceramics were prepared in situ from graphite/geopolymer composites based on a carbon thermal reduction reaction under high temperatures. Moreover, both h-AlN and SiC ceramics derived by this technique show porous features, with porosities of 61% and 68% and considerable compressive strength values of 0.76 MPa and 0.63 MPa, respectively [17]. Although, previous investigations have studied SiC sludge in the field of geopolymer materials, such as silicon carbide (SiC) foams [13], SiCf-reinforced geopolymers [14], refractory paints [15], high-strength open-celled SiC/geopolymer foam composites (SFCs) [16], and porous AlN/SiC-based ceramics [17].

Most of the previous investigations were based on studies on SiC sludge applied in the field of geopolymer materials, which led to knowledge; few reports are available regarding the effect of alkaline-activator solution on the geopolymerization of SiC sludge-based geopolymers. In this study, a new way to the effect of the mass ratio between the Na_2SiO_3 and sodium hydroxide (NaOH) solutions (NS/SS ratio) and the effect of SiC sludge on metakaolin geopolymers was comprehensively investigated to determine the underlying performance of the geopolymerization system. The flexural strengths of the geopolymers were examined in a laboratory, and their microstructures were characterized using an isothermal conduction calorimeter, differential thermal analysis (DTA/TG), and nuclear magnetic resonance (NMR).

2. Materials and Methods

The silicate-aluminum sources were metakaolin (MK) and SiC sludge. The SiC sludge was gained from a plant that makes LED substrates (Taiwan). The major chemical composition of the SiC sludge was 75.4 wt % SiO₂, 23 wt % SiC, and 0.8 wt % Al₂O₃; the major chemical composition of the MK was 51.8 wt % SiO₂ and 43 wt % Al₂O₃, as shown in Table 1. Sodium hydroxide (NaOH, 10 M) and distilled water were mixed with a commercial sodium silicate solution (Na₂SiO₃), Ms (SiO₂ mole/Na₂O mole) = 3.1 (SiO₂ = 28.1%, Na₂O = 9.09%, H₂O = 62.8%). The mass ratio between Na₂SiO₃ and sodium hydroxide (NaOH) solutions (NS/SS ratio) was in the range of 0.8 to 2.0 (0.8, 1.2, 1.6, and 2.0) and analysis of the effect of an alkaline-activator solution on a geopolymer reaction. Calculate the amount of NS/SS ratio as follows.

$$\text{NS/SS ratio} = \frac{\text{SiO}_2 \text{ mole (Sodium silicate solution)}}{[\text{Na}_2\text{O mole (Sodium silicate solution)} + \text{Na}_2\text{O mole (10 M NaOH)}]}$$

Table 1. The major chemical composition of materials.

Composition	SiC Sludge	MK
SiO ₂ (wt %)	75.40	51.80
Al ₂ O ₃ (wt %)	0.80	43.00
Fe ₂ O ₃ (wt %)	0.58	1.30
CaO (wt %)	0.09	0.25
MgO (wt %)	N.D. ¹	N.D. ¹
SO ₃ (wt %)	0.06	N.D. ¹
Na ₂ O (wt %)	N.D. ¹	0.04
K ₂ O (wt %)	0.01	0.32
SiC (wt %)	23.00	N.D. ¹

¹ N.D.: not detected.

Then, MK and 0–40 wt % SiC sludge were added into an alkali activator, and analysis of the effect of a Si/Al mole ratios on a geopolymer reaction. The ratios of material in the mixture of geopolymers are listed in Table 2. Calculate the amount of Si/Al mole ratios as follows. First, the MK, SiC sludge, and alkaline solution were stirred for 60 min, and deionized water (3.8 L) was subsequently added to the blend to generate a suspension by stirring for 30 s. After filtration, the dissolved Si and Al concentrations were measured by atomic absorption spectroscopy (AAS). SiC-sludge metakaolin-based geopolymer samples were synthesized by mixing powder and alkaline activator solution for 5 min, and the paste was poured into a plastic mold for the analysis of microstructure characteristics and mechanical properties. The flexural strength tests were performed after 1, 7, 14, 28, and 56 days using a Hung Ta HT-2402 testing machine (Hung Ta Instruments, Taichung City, Taiwan) with a three-point bending test method, according to ASTM C348. The average strength value of the three specimens is presented, and the coefficient of variation of these results is less than 10%. The mechanical properties of the three specimens were evaluated and expressed. Finally, the samples were analyzed at appropriate curing times by DTA/TG and ²⁹Si MAS NMR analyses. The heat evolution in the geopolymerization reactions, such as dissolution, polymerization, and condensation, were characterized by an isothermal conduction calorimeter (Calmetrix I-CAL 2000 HPC, Boston, MA, USA) conducted at a constant temperature of 30 ± 0.02 °C for 168 h, according to ASTM C1702. The DTA/TG studies were conducted, in which the heating range was from 50 °C to 1000 °C by an STA6000 thermal analyzer. High-resolution ²⁹Si MAS/NMR spectra were recorded at 39.72 MHz on an MSL Bruker MAS/NMR-200 solid-state high-resolution spectrometer using rapid (approximately 3 kHz) sample spinning at the magic angle to the external magnetic field. Analysis method based on reference [18], the coordination of Q⁴(mAl) species (4Al, 3Al, 2Al, 1Al, 0Al) in the SCSMB geopolymer was obtained by applying Seasolve PeakFit™ software, version 4 (Seasolve Software Inc., Framingham, MA, USA).

Table 2. The ratios of material in mixture.

Mix Designation	NS/SS	MK (g)	SiC Sludge (g)	Sodium Silicate Solution (g)	10 M NaOH (g)	Si/Al Mole Ratios
0.8NS0SCS	0.8	500	0	240	260	1.01
0.8NS10SCS		450	50	240	260	1.36
0.8NS20SCS		400	100	240	260	1.24
0.8NS30SCS		350	150	240	260	1.74
0.8NS40SCS		300	200	240	260	2.60
1.2NS0SCS	1.2	500	0	315	185	0.98
1.2NS10SCS		450	50	315	185	1.34
1.2NS20SCS		400	100	315	185	1.22
1.2NS30SCS		350	150	315	185	1.87
1.2NS40SCS		300	200	315	185	2.72
1.6NS0SCS	1.6	500	0	374	126	1.16
1.6NS10SCS		450	50	374	126	1.51
1.6NS20SCS		400	100	374	126	1.43
1.6NS30SCS		350	150	374	126	1.92
1.6NS40SCS		300	200	374	126	2.89
2.0NS0SCS	2.0	500	0	421	79	1.07
2.0NS10SCS		450	50	421	79	1.48
2.0NS20SCS		400	100	421	79	1.23
2.0NS30SCS		350	150	421	79	1.78
2.0NS40SCS		300	200	421	79	2.67

3. Results

3.1. Heat Evolution of Geopolymers with SiC Sludge

Geopolymers comprise a framework containing inorganic T-O (T: Si or Al). Geopolymerization is analogous to the curing process of polymers, which includes gelation, polymerization, and solidification [9]. The heat evolution can be measured by an isothermal conduction calorimeter, which indicates the progress of the geopolymerization reaction. In all cases, the first exothermic peak, which is related to the dissolution of solid materials in the alkaline activator, is quite sharp at the beginning of the preinduction period [19]. The samples were mixed outside the device, and the heat evolution was so fast that there was not sufficient time to achieve thermal equilibrium; thus, the first peak was only partially registered. The second exothermic peak indicated continuous dissolution and polymerization, during which oligomers combined to form a geopolymer network.

Figure 1a–d illustrates the effects of the NS/SS ratio on the heat evolution during geopolymerization reactions. When the NS/SS ratio was 0.8, the heat evolutions of 0.8NS0SCS, 0.8NS10SCS, 0.8NS20SCS, and 0.8NS30SCS were 806.5, 720.3, 415.3, and 445.8 W/g heat, respectively (Figure 1a). The high NaOH concentration prompted the dissolution of the initial solid in the alkaline activator, which caused the first exothermic peaks of 0.8NS0SCS and 0.8NS10SCS to be more intense than those of the others. In addition, the second exothermic peaks of 0.8NS0SCS and 0.8NS10SCS appeared at approximately 6.65 h and 9.98 h, respectively. This indicated that a higher NaOH content promoted the reactivity of the geopolymerization reaction. However, the second exothermic peak of 0.8NS40SCS emerged at 14.82 h. According to Hu et al. [20], the geopolymer gel formed by the rapid reaction of active aluminosilicate under a high concentration of alkali hindered the subsequent dissolution of the FA (Fly ash) [20]. The results indicate that the amounts of dissolved silica and alumina from MK are limited due to precipitations of geopolymer gels around the surface of SCS particles. Therefore, the heat evolution duration of the second exothermal peak clearly increased from 6.65 h to 14.82 h as the SiC sludge-replacement levels increased from 0% to 40%, as shown in Figure 1a. An increase in the NS/SS ratio (1.2) was expected to improve the geopolymerization reaction, and the results of 1.2NS10SCS showed that the second exothermic peak reached 958.3 W/g. A synergistic effect between the SCS and the MK was suggested to contribute to the promotion of the geopolymerization reaction, which caused the heat evolution of 1.2NS10SCS to be higher than that of 1.2NS0SCS (945.2 W/g). Additionally, the heat evolution duration of the second exothermal peak increased from 14.82 to 17.82 h as the NS/SS ratio increased from 0.8 to 1.2 (0.8NS40SCS and 1.2NS40SCS). According to Figure 1b, it can be inferred

that, although the NS/SS ratio increased, the amounts of dissolved silica and alumina from MK were limited due to the precipitations of geopolymer gels around the surface of SCS (SiC sludge) particles.

Figure 1. Effects of NS/SS ($\text{Na}_2\text{SiO}_3/\text{NaOH}$ solutions) ratios on heat evolution rate for geopolymer with SiC sludge: (a) NS/SS ratio = 0.8; (b) NS/SS ratio = 1.2; (c) NS/SS ratio = 1.6; (d) NS/SS ratio = 2.0.

Figure 1c–d shows the heat evolution curves of the SCSMB geopolymer sample with NS/SS values of 1.6 and 2.0, in which two exothermic peaks are classified. The heat evolution (990.6 W/g) of the second exothermic peak of 1.6NS10SCS was higher than those of all geopolymers. This fact implies that the synergistic effect promoted the oligomers to combine and form integral geopolymer gels [20]. However, for 2.0NS10SCS, the heat evolution of the second exothermic peak decreased from 990.6 W/g to 951.5 W/g with an increase in the NS/SS ratio. According to Lo et al. [21], decreasing the concentration of Na_2O caused the decreased of the amount of dissolved Si and Al and restrained the geopolymer synthesis reaction.

3.2. Mechanical Properties

The results of the flexural strength of the geopolymer with SiC sludge cured for 1–56 days are shown in Figure 2. The flexural strength of geopolymers varied at different replacement levels of SiC sludge and was positively correlated with the replacement levels. On the first day of the curing process, the flexural strengths of 0.8NS0SCS, 0.8NS10SCS, 0.8NS20SCS, 0.8NS30SCS, and 0.8NS40SCS were 4.12, 3.97, 3.89, 3.56, and 3.57 MPa, respectively (Figure 2a). The amounts of dissolved silica and alumina from MK are limited due to geopolymer gel precipitations around the surface of SCS particles. This reduction generated an inadequate amount of leading precursors, thus reducing the strength. Hu et al. [20] discussed that the decrease in strength at a longer curing age might be due to

depolymerization or dissolution of aluminosilicates resulting from the low $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio in formulation, which was 2.15 for the G-RM-5N-0FA. In particular, the strength of the G-RM-5N-0FA cured for 7 days was below 5 MPa [20]. The flexural strength for the 28 day SiC sludge geopolymers is 4.05 MPa for 0.8NS30SCS ($\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio of 1.74), as shown in Figure 2a. A low $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio significantly reduced the strength for 0.8NS30SCS, which is in line with the study from Hu et al. for geopolymer materials [20].

Figure 2. Flexural strength of geopolymer with SiC sludge cured for 1–56 days: (a) NS/SS ratio = 0.8; (b) NS/SS ratio = 1.2; (c) NS/SS ratio = 1.6; (d) NS/SS ratio = 2.0.

For the NS/SS ratio of 1.2, the flexural strengths of 1.2NS20SCS, 1.2NS30SCS, and 1.2NS40SCS steadily decreased to 3.64, 3.74, and 3.69 MPa on the first day of curing, respectively (Figure 2b). It is possibly the increase in the NaOH content, which generated a sufficient amount of “OH[−]” in the system and thus led to the increased solubility. This behavior caused unsatisfactory polycondensation and reduced the formation of amorphously structured geopolymer gels. The high Na₂O concentration may block the acceleration effect of geopolymer synthesis, leading to slow strength development, which is in line with previous studies for geopolymer materials [21].

In contrast, the increase in the curing time from 1 to 56 days increased the strengths of 1.6NS10SCS and 2.0NS10SCS from 4.08 to 6.42 MPa and from 1.66 to 5.03 MPa, respectively (Figure 2c). Increasing the NS/SS ratio promoted the dissolution of the initial solid, which accelerated the geopolymerization reaction and led to the rapid formation of geopolymer network structures. Therefore, the strength increased when the amount of amorphously structured geopolymer gel formed in the geopolymerization system increased. Lo et al. [21] reported that MKGP (MK-based geopolymer) with a 10% TLWD (TFT-LCD waste glass) replacement level showed the strength of 6.5 MPa with a S/N ratio of 1.6.

The results indicated that strength increased with the NS/SS ratio. Yaghoubi et al. [22] noted that the geopolymer strength was related to the degree of polycondensation, which was strongly influenced by the soluble SiO_4 and AlO_4 content of the polymer system. A high degree of polycondensation in the Si-O-Al framework structure corresponds to high strength [23]. In addition, the flexural strength of 1.6NS10SCS ($\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio of 1.51) is also very high, as shown in Figure 2c. This increase in strength could be from the increased $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio. The $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio has a definite effect on strength, increasing the strength to 6.37 MPa. In addition, Rahman et al. [14] also noted that the increase in flexural strength due to simultaneous inclusion of silica and silicon carbide whiskers indicates that it is possible that silica particles are compatible with the metakaolin-based geopolymers, which is effective in consolidation [14]. However, the flexural strength of 1.6NS40SCS with a curing age of 28 days decreased to 2.73 MPa ($\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio of 2.89), as shown in Figure 2c. With increasing SiC sludge content in the geopolymer, the $\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio increases, but the amounts of dissolved silica and alumina from MK are limited due to precipitation of geopolymer gels around the surface of SCS particles. A greater amount of SiC sludge (over 20%) may block the synergistic effect of SCS and MK. Hu et al. [20] reported about when the FA (fly ash) content was 100 wt %, and the strength of the FA-based geopolymer with a curing age of 28 days was only 5 MPa ($\text{SiO}_2/\text{Al}_2\text{O}_3$ molar ratio of 4.38).

3.3. DTA/TG of Geopolymers with SiC Sludge

Figure 3 displays the effects of the geopolymers with SiC sludge on the DTA/TG results. According to the weight loss of the samples during heating, all the processes of DTA/TG results are categorized into Stages I, II, and III. The first peak at approximately 230 °C was associated with the loss of carbonate salt. Almost all the weight loss at the second peak, which occurred from 230 °C to 400 °C, was likely due to the dehydration of the macropore structure of the material [21]. The third peak, which occurred from 400 °C to 750 °C, was mainly related to the loss of the amorphous composition and dehydration of the geopolymer gel [21,24]. On the first day of curing, the weight loss percentages of 0.8NS0SCS, 0.8NS10SCS, 0.8NS20SCS, and 0.8NS30SCS were 10.21, 10.20, 10.09, and 10.08 wt %, respectively (Table 3). The amounts of dissolved silica and alumina from MK are limited due to precipitation of geopolymer gels around the surface of SCS particles, thus reducing the macropore structure. Therefore, the weight loss of 0.8NS40SCS was 10.06 wt %. The geopolymers with high replacement levels of SiC sludge generated an inadequate amount of leading precursors, thus limiting the polycondensation process. Therefore, the weight loss percentage of 0.8NS40SCS from 400 to 750 °C decreased to 1.95%, as shown in Table 3.

Figure 3. Cont.

Figure 3. The DTA/TG results of the geopolymers with SiC sludge and NS/SS ratio of 1.6, during heating: (a) replacement level = 0%; (b) replacement level = 10%; (c) replacement level = 20%; (d) replacement level = 30%; (e) replacement level = 40%.

When the NS/SS ratio increased from 0.8 to 1.6, the weight loss percentage of 1.6NS10SCS was 13.42% in a temperature range of 230–400 °C, as depicted in Figure 3b. The high NS/SS ratio prompted the dissolution of the initial solid and consequently promoted the geopolymerization reaction, which caused the rapid formation of 3D silico–aluminate structures. Furthermore, the synergistic effect between the SCS and the MK was assigned to the promotion of the geopolymerization reaction, which caused the oligomers to form integral geopolymer gels. Therefore, the structural strength increased with an increase in the amount of amorphously structured geopolymeric gel in the geopolymerization system. According to the results, the weight loss percentage of 1.6NS10SCS increased to 14.94% after 56 days of curing. Furthermore, low weight loss occurred in the temperature range of 400–750 °C. The weight loss percentages of 1.6NS0SCS, 1.6NS10SCS, 1.6NS20SCS, 1.6NS30SCS, and 1.6NS40SCS were 4.87, 4.51, 4.40, 4.15, and 4.26 wt %, respectively (Figure 3a–e).

Table 3. Weight loss of the geopolymers with SiC sludge during heating from ambient temperature to 1000 °C (NS/SS ratio = 0.8 and 1.2).

Samples	NS/SS	Curing Time (Days)	Temperature (°C)		
			50–230	230–400	400–750
0.8NS0SCS	0.8	1	2.58	10.21	2.61
		28	3.70	11.81	4.24
		56	4.37	12.36	4.68
0.8NS10SCS		1	2.77	10.20	2.35
		28	3.83	11.69	4.24
		56	4.42	12.36	4.62
0.8NS20SCS		1	3.72	10.09	2.33
		28	3.83	11.66	4.03
		56	4.48	12.32	4.24
0.8NS30SCS		1	3.49	10.08	2.22
		28	3.94	11.64	3.68
		56	4.48	12.24	4.03
0.8NS40SCS		1	3.49	10.06	1.95
		28	3.94	11.59	3.52
		56	4.50	12.21	3.68
1.2NS0SCS	1.2	1	3.49	10.97	2.73
		28	3.94	12.51	4.09
		56	4.51	12.96	4.77
1.2NS10SCS		1	3.49	10.96	2.72
		28	4.14	12.49	4.07
		56	4.55	12.93	4.34
1.2NS20SCS		1	3.52	10.93	2.64
		28	4.21	12.46	3.99
		56	4.55	12.86	4.33
1.2NS30SCS		1	3.52	10.92	2.64
		28	4.25	12.42	3.96
		56	4.61	12.75	4.21
1.2NS40SCS		1	3.58	10.88	2.57
		28	4.25	12.39	3.90
		56	4.61	12.65	4.14

3.4. ^{29}Si NMR Spectra and Deconvolutions of Geopolymers with SiC Sludge

To further validate the evolution of chemical reactions in geopolymerization with SiC sludge, ^{29}Si NMR analyses were conducted on the geopolymer samples. The ^{29}Si NMR spectra analysis enables the short-range ordering and molecular structure in the geopolymerization reaction to be determined [25]. In the initial stage, geopolymerization involved the release of the silicate derivatives (Q^1 , Q^2 , and Q^3) in the solution by alkaline activation. Here, the resonances of Q^1 , Q^2 , and Q^3 resonate at approximately -79 , -85 , and -95 ppm, respectively, and the resonance of Q^4 is the same as the $\text{Q}^4(0\text{Al})$ of the geopolymer gel [26]. The detailed reaction mechanism is discussed in the following sections. The resonance of a $\text{Q}^4(\text{mAl})$ ($0 \leq m \leq 4$) center with the replacement of each aluminum by silicon is approximately -5 ppm, with chemical shifts at -84 , -89 , -93 , -99 , and -107 ppm that correspond to $\text{Q}^4(4\text{Al})$, $\text{Q}^4(3\text{Al})$, $\text{Q}^4(2\text{Al})$, $\text{Q}^4(1\text{Al})$, and $\text{Q}^4(0\text{Al})$, respectively [25].

Figure 4 displays the ^{29}Si NMR spectra and deconvolutions of SiC sludge geopolymers with 28 days of curing. After 28 days of curing, the spectra of 0.8NS0SCS revealed a high percentage of $\text{Q}^4(3\text{Al})$, thus indicating a high initial level of aluminosilicate. For the ^{29}Si NMR deconvolution of 0.8NS0SCS, the percentages of $\text{Q}^4(3\text{Al})$, $\text{Q}^4(2\text{Al})$, and $\text{Q}^4(1\text{Al})$ were 34.32%, 16.06%, and 7.06%, respectively. However, geopolymers with high replacement levels of SiC sludge generated an inadequate amount of leading precursors, thus limiting the polycondensation process. For example, for 0.8NS40SCS, the percentages of $\text{Q}^4(3\text{Al})$, $\text{Q}^4(2\text{Al})$, and $\text{Q}^4(1\text{Al})$ were 29.59%, 24.91%, and 20.05%, respectively. On the 28th day of curing, the spectra of 0.8NS40SCS revealed a high percentage of $\text{Q}^4(0\text{Al})$, thus indicating the formation

of sodium silicate glass from the unreacted Na_2SiO_3 activator [27]. This result indicates that SiC sludge inhibits geopolymerization reactions.

Figure 4. The ^{29}Si NMR spectra and deconvolutions of SiC sludge geopolymers at Curing Time = 28 day, and NS/SS ratio of 0.8: (a) replacement level = 0%; (b) replacement level = 10%; (c) replacement level = 20%; (d) replacement level = 30%; (e) replacement level = 40%.

Figure 5 depicts the ^{29}Si NMR spectra deconvolution obtained for the geopolymers with SiC sludge over 28 days of curing. After the 28th day of curing for 1.2NS10SCS, the percentages of $\text{Q}^4(3\text{Al})$, $\text{Q}^4(2\text{Al})$, and $\text{Q}^4(1\text{Al})$ were 33.71%, 22.47%, and 9.03%, respectively. The results revealed that when the NS/SS ratio was relatively low, an increase in the NaOH content caused the generation of a

sufficient amount of OH^- in the system to increase the solubility, thus limiting the polycondensation process. This behavior caused a decrease in the formation of amorphously structured geopolymer gels. Therefore, the $\text{Q}^4(3\text{Al})$, $\text{Q}^4(2\text{Al})$, and $\text{Q}^4(1\text{Al})$ contents of 1.2NS10SCS slowly increased, as displayed in Figure 5b. When the NS/SS ratio increased to 1.6, 1.6NS10SCS exhibited high percentages of $\text{Q}^4(3\text{Al})$ (33.63%), $\text{Q}^4(2\text{Al})$ (23.92%), and $\text{Q}^4(1\text{Al})$ (9.70%), as illustrated in Figure 6b. Because of a synergistic effect between silicon carbide sludge and metakaolin, the silica and alumina of metakaolin were dominant in the geopolymerization reaction, and the addition of silicon carbide sludge, which had silica was provided more reaction paths. Wan et al. [28] indicated that geopolymers with high compressive strengths have high percentages of $\text{Q}^4(3\text{Al})$, $\text{Q}^4(2\text{Al})$, and $\text{Q}^4(1\text{Al})$, and were possibly N–A–S–H gels [28]. The percentages of $\text{Q}^4(3\text{Al})$, $\text{Q}^4(2\text{Al})$, and $\text{Q}^4(1\text{Al})$ silicon centers in the ^{29}Si NMR spectra increased, thus indicating the formation of an increased number of tetrahedral aluminum-linking sites with tetrahedral silicon (Figure 6).

Figure 5. The ^{29}Si NMR spectra and deconvolutions of SiC sludge geopolymers at Curing Time = 28 day, and NS/SS ratio of 1.2: (a) replacement level = 0%; (b) replacement level = 10%; (c) replacement level = 20%; (d) replacement level = 30%; (e) replacement level = 40%.

Figure 6. The ^{29}Si NMR spectra and deconvolutions of SiC sludge geopolymers at Curing Time = 28 day, and NS/SS ratio of 1.6: (a) replacement level = 0%; (b) replacement level = 10%; (c) replacement level = 20%; (d) replacement level = 30%; (e) replacement level = 40%.

4. Discussion

The geopolymerization process of geopolymers with SiC sludge is illustrated in Figure 7. In steps 1 and 2, the active components of metakaolin and SiC sludge particles were dissolved to form monosilicates (Q^0) that were linked to the end groups (Q^1), middle chain groups (Q^2), layers and branching sites (Q^3), and $[\text{SiO}_4]$ and $[\text{AlO}_4]$ tetrahedrons in the AASs. In step 3, primary particles of oligomeric gel were formed by the oligomers through the polycondensation reaction in the AASs. In step 4, the oligomers

were polycondensed to form geopolymerization gels. In step 5, the polymers were polycondensed and accumulated around the unreacted SiC sludge surface to form 3D networks (Q^4).

Figure 7. The geopolymerization process of geopolymers with SiC sludge.

At an early curing time, for 0.8NS0SCS, the sum of the silicate derivatives (Q^1 , Q^2 , and Q^3) was 13.59% (Table 4). After 28 days of curing, the percentage of silicate derivatives increased to 19.23%, as shown in Table 4. The results revealed that when the NS/SS ratio was relatively low, an increase in the NaOH content caused the generation of sufficient OH^- in the system to increase the solubility. This solubility increase caused hindrance in polycondensation, as indicated by the heat evolution rate and DTA/TG results (Figure 1 and Table 3). The inhibition of polycondensation reduced the mechanical strength (Figure 2). An increase in the NS/SS ratio promoted the dissolution of the initial solid, thus accelerating the geopolymerization reaction and leading to the rapid formation of geopolymer network structures. During the geopolymerization reaction, silicate derivatives were transformed into geopolymer gels. Therefore, the weight percentage of the silicate derivatives of 1.6NS0SCS decreased to 10.06% after 28 days of curing. Furthermore, the synergistic effect between the SCS and the MK was assigned to the promotion of the geopolymerization reaction, which caused the flexural strength of 1.6NS10SCS to increase to 6.42 MPa after 28 days of curing. First, the silica and alumina of metakaolin were dominant in the geopolymerization reaction, and the addition of silicon carbide sludge, which had silica, was provided more reaction paths. In addition, Rahman et al. [14] also noted that given the increase in flexural strength due to simultaneous inclusion of silica and silicon carbide whiskers, it is possible that silica particles are compatible with the metakaolin-based geopolymers, which is effective in consolidation [14]. According to the DTA/TG results, the weight loss percentage of 1.6NS10SCS increased to 14.62% from 400 to 750 °C, as shown in Figure 3b. This fact implies that the synergistic effect promoted the oligomers to combine and form integral geopolymer gels [20].

However, geopolymers with high replacement levels of SiC sludge generated an inadequate amount of leading precursors, thus limiting the polycondensation process. For example, for 1.6NS30SCS and 1.6NS40SCS, the weight percentages of silicate derivatives (Q^1 , Q^2 , and Q^3) were 9.81% and 9.13%, respectively, as presented in Table 4. In steps 1 and 2, SiC sludge dissolves to release Al^{3+} and Si^{4+} , which are hydrolyzed into $[AlO_4]$ and $[SiO_4]$ tetrahedrons, respectively, immediately after contact with the AASs. The amounts of dissolved silica and alumina from MK are limited due to precipitations of geopolymer gels around the surface of SCS particles, which caused the number of $[SiO_4]$ and $[AlO_4]$ tetrahedrons in the system to be low. Thus, the amount of oligomeric gel was low. Because the N-A-S-H gels activated by the sodium silicate solution required an increased amount of oligomeric gel, the rate of the geopolymerization reaction decreased. Therefore, the heat evolution duration of the second exothermic peak increased from 26.15 to 33.32 h as the replacement levels of SiC sludge increased from 30% to 40% (1.6NS30SCS and 1.6NS40SCS), as shown in Figure 1c. Additionally, the flexural strengths of 1.6NS30SCS and 1.6NS40SCS were 5.31 MPa and 2.73 MPa, respectively (Figure 2c). This result indicated that a relatively high SiC sludge content (more than 20%) might block the synergistic effect between SCS and MK, which is confirmed by the heat evolution rate, DTA/TG results, and ^{29}Si MAS NMR analyses. This is consistent with the SEM images results of previous studies [29] that the

amorphous gel products had gradually filled the pores of the SiC sludge-based geopolymer, resulting in an increase in density and compressive strength of the geopolymer structure [29]. The synergistic effect of the SiC sludge and MK promoted the reaction progression, which caused increasing amounts of amorphously structured geopolymeric gels in the geopolymerized system [29]. Then, the unreacted SiC sludge was in the form of uniform plate particles [29]. In summary, this study demonstrated that this renders SiC sludge as promising additives for the production of metakaolin-based geopolymers. As a structural material, the SiC sludge-based geopolymer is a potential replacement material for OPC.

Table 4. ^{29}Si MAS NMR spectral deconvolution of the silicate derivatives (Curing Time = 1 day and 28 days).

Samples	Curing Time	Silicate Derivatives (%)			Deconvoluted Fractions of Silicon Centers (%)				
		Q ¹	Q ²	Q ³	Q ⁴ (0Al)	Q ⁴ (1Al)	Q ⁴ (2Al)	Q ⁴ (3Al)	Q ⁴ (4Al)
0.8NS0SCS	1 day	10.07	1.41	2.11	0.66	5.62	16.17	35.85	28.11
0.8NS10SCS		8.72	2.53	2.30	1.30	7.59	18.64	33.82	25.10
0.8NS20SCS		9.22	2.16	2.55	2.85	5.89	19.23	34.15	23.95
0.8NS30SCS		4.36	3.11	2.08	4.12	10.79	21.79	34.24	19.51
0.8NS40SCS		1.56	3.31	4.28	3.43	17.66	25.46	26.38	17.92
0.8NS0SCS		10.22	4.62	4.39	0.46	7.06	16.06	34.32	22.87
0.8NS10SCS	28 days	10.58	5.06	2.99	0.43	3.45	14.26	36.79	26.44
0.8NS20SCS		11.26	4.77	2.29	1.74	4.96	16.22	34.53	24.23
0.8NS30SCS		10.42	4.59	2.08	0.58	5.84	18.13	36.27	22.09
0.8NS40SCS		4.13	3.90	4.82	4.24	20.05	24.91	29.59	8.36
1.2NS0SCS		7.74	3.61	3.10	2.45	9.81	20.39	33.55	19.35
1.2NS10SCS		8.05	4.14	2.76	1.73	8.05	20.25	34.98	20.04
1.2NS20SCS	1 day	7.17	4.01	3.59	3.57	9.50	20.68	34.18	17.30
1.2NS30SCS		3.94	5.03	4.81	3.94	10.36	24.02	32.26	15.64
1.2NS40SCS		3.08	3.74	4.17	6.18	21.53	25.71	26.37	9.22
1.2NS0SCS		6.44	5.77	4.89	2.94	10.88	19.99	34.43	14.66
1.2NS10SCS		6.17	3.97	3.08	3.50	9.03	22.47	33.71	18.07
1.2NS20SCS		2.41	6.03	5.07	2.72	11.10	21.00	35.72	15.95
1.2NS30SCS	28 days	5.59	3.30	4.94	2.05	14.21	22.30	34.11	13.50
1.2NS40SCS		0.84	4.60	5.85	5.73	21.95	25.71	27.38	7.94
1.6NS0SCS		5.00	3.81	3.81	3.40	13.58	25.66	30.69	14.05
1.6NS10SCS		5.87	4.51	4.96	4.81	14.46	23.80	31.50	10.09
1.6NS20SCS		4.91	5.40	3.93	6.62	16.00	27.18	29.37	6.59
1.6NS30SCS		2.23	3.57	4.01	6.64	18.05	27.85	29.63	8.02
1.6NS40SCS	1 day	0.89	2.23	6.01	9.19	27.82	28.93	17.81	7.12
1.6NS0SCS		5.03	1.76	3.27	4.94	12.63	24.54	30.97	16.86
1.6NS10SCS		5.13	3.50	3.27	2.28	9.70	23.92	33.63	18.57
1.6NS20SCS		2.41	3.07	3.07	2.32	11.78	24.30	33.87	19.18
1.6NS30SCS		0.92	4.16	4.16	5.46	18.26	27.51	29.13	10.40
1.6NS40SCS		1.69	3.74	4.12	9.75	26.59	26.78	21.53	5.80

5. Conclusions

The present study investigated the effects of different SiC sludge contents and alkaline activator solutions on the geopolymer reaction and microstructures of metakaolin-based geopolymers subjected to different curing times. The geopolymer with the optimal SiC sludge replacement level and NS/SS ratio contained a high heat evolution value (990.6 W/g), excellent mechanical properties (6.42 MPa), and high deconvolution percentages of Q⁴(3Al, 2Al, and 1Al). The conclusions can be summarized as follows. The high NaOH concentration prompted the dissolution of the initial solid in the alkaline activator, which caused the first exothermal peaks to be more intense. The 1.6NS10SCS (SiO₂/Al₂O₃ molar ratio of 1.51) had favorable mechanical strength due to an increasing SiO₂/Al₂O₃ molar ratio and a synergistic effect between the SiC sludge and the metakaolin. The geopolymers with high replacement levels of SiC sludge not only contained dissolved silica and alumina from metakaolin but also underwent polycondensation on a geopolymer reaction of SiC sludge-based geopolymers. A greater amount of SiC sludge (over 20%) may block the synergistic effect between the SiC sludge and metakaolin. The results of this study have shown that a geopolymer with an optimal SiC-sludge

replacement level and NS/SS ratio expresses a synergistic effect that allows an improvement of both the geopolymer reaction and the strength of metakaolin-based geopolymers. This renders SiC sludge as promising additives for the production of metakaolin-based geopolymers. For future work, we expect to analyze other important properties of SiC sludge-based geopolymers by thermal conductivity, and fire performance, which was a promising alternative as fire resistance materials in the construction industry.

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