Optimizing the Mechanical Performance and Microstructure of Alkali-Activated Soda Residue-Slag Composite Cementing Materials by Various Curing Methods

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Abstract: Aiming to promote further the application of alkali-activated soda residue-ground granulated blast furnace slag (SR-GGBS) cementing materials, this study explored the optimal curing method for enhancing mechanical performance. The optimal curing method was determined based on the development of compressive strengths at different curing periods and microstructural examination by XRD, FTIR, SEM, and TG-DTG. The results show that the strength of cementing materials after room-temperature (RT) dry curing was the poorest, with the slow development of mechanical performance. The 7d and 28d compressive strengths were only 14.62 and 20.99 MPa, respectively. Compared with the values after RT dry curing, the samples’ 7d and 28d compressive strengths after RT water curing, standard curing, and RT sealed curing were enhanced by 16.35%/24.06%, 30.98%/23.77%, and 38.24%/37.97%, respectively. High-temperature (HT) curing can significantly improve the early strength of the prepared cementing materials. Curing at 60°C for 12 h was the optimal HT curing method. Curing at 60°C for 12 h enhanced the 3d strength by 100.84% compared with standard curing. This is because HT curing promoted the decomposition and aggregation of GGBS, and more C-A-S-H gel and crystal hydration products, including ettringite and calcium chloroaluminate hydrate, were produced and filled the inner pores, thereby enhancing both the overall compactness and mechanical performance. However, curing at too high temperatures for too long can reduce the material’s overall mechanical performance. After excess HT curing, many shrinkage cracks were produced in the sample. Different thermal expansion coefficients of different materials led to a decline in strength. The present study can provide a theoretical foundation for extensive engineering applications of alkali-activated SR-GGBS composite cementing materials.

Keywords: composite cementing materials; soda residue; ground granulated blast furnace slag; mechanical performance; curing method

1. Introduction

Alkali-activated cementing materials are hydraulic cementing materials prepared under the action of alkali activators based on catalysis principles [1]. Over the past two decades, alkali-activated cementing materials attracted extensive interest worldwide. Compared with Portland cement, alkali-activated cementing materials possess several advantages, including low energy consumption, high intensity, and good durability in production and performance [2,3]. The main hydration products of Portland cement are hydrated calcium silicate and calcium hydroxide. In contrast, alkali-containing aluminosilicate gels with poor crystallinity act as the main products of alkali-activated cementing materials, which can also account for their excellent durability [4]. In addition to industrial byproducts, alkali-activated materials can also be used for waste disposal and environmental protection [5].

Many cementing materials, including ground granulated blast furnace slag (GGBS), steel slag [6], and fly ash [7], can be used for preparing alkali-activated materials. Alkali-activated GGBS cementing materials have received the most extensive research. Nedenuri...
et al., investigated the mechanical and chemical structural changes of alkali-activated GGBS cementing materials in the early stage [8]. It was found that the setting time of the cementing material drops with the increasing addition of GGBS, accompanied by an increase in hydration products and hydration heat. As the Moore modulus and the mixing amount of activator increase, the setting time can be shortened. The rapid setting of the alkali-activated GGBS mixture refers to the rapid setting of the gel network accompanied by the formation of hydration products. Lv et al., investigated the applicability of seawater to the preparation of GGBS-fly ash alkali-activated materials. They found that adding seawater can accelerate the alkali activation process, shorten the setting time and enhance both early and long-term strength [9]. According to their results, the 3d and 1-year compressive strengths exceeded 25 and 73 MPa, respectively. Moreover, adding seawater negatively affected rebars and expanded the application range of alkali-activated materials. Adesanya et al. used desulfurization dust (DeS-dust) to replace sodium hydroxide as an alkali activator to activate GGBS cementing materials [10]. Using DeS-dust as an alkali activator, the 28d compressive strength can reach up to 33 MPa; in contrast, the 28d compressive strength of the materials after the addition of sodium hydroxide was only 25 MPa. Based on the micromeasurement results, the microstructures after the addition of the two activators are comparable, and the cementing materials with DeS-dust exceeded those with sodium hydroxide in performance.

At normal temperature, because of the high activity of GGBS, alkali-activated cementing materials undergo hydration under an alkali activator with a specific strength [11,12]. However, to save cost, many solid wastes, such as calcium carbide slag [13] and gypsum [14], have been added to alkali-activated GGBS cementing materials in recent years. Considering the low activity of wastes at normal temperatures, the curing of wastes should be optimized to achieve better performance. Guo et al. [15] used calcium carbide slag slurry and soda residue (SR) slurry to activate GGBS and prepare a new kind of cementing material; according to their experimental results, the 3d and 28d compressive strengths of the prepared samples can be enhanced by 50.0 and 34.7%, respectively, compared with the samples produced with the addition of powders. It was concluded that curing at 60 °C for 12 h proved to be optimal. Compared with the samples after water curing, the 3d compressive strength of the sample after curing at 60 °C for 12 h can be enhanced by 66.7%. This is because high temperature (HT) promotes the decomposition and aggregation of GGBS, thereby accelerating the generation of C-S-H gel and calcium chloroaluminate hydrate crystals. However, after curing at too high a temperature for a long time, the long-term strength of the prepared sample drops, which can be attributed to the formation of drying shrinkage cracks in the sample. By adding NaOH, Na₂SiO₃, and silicon manganese dust (SMF) to slag (BFS), Nasir et al. [16] prepared a kind of alkali-activated cementing material and determined the optimal curing conditions in the oven (at 60 °C for 6 h); after curing at 60 °C for 6 h, the 3d, 7d, and 28d compressive strengths were 38, 41, and 45.2 MPa, respectively. As the crystallinity degree of the C-(A)-S-H and quartz phases increased, strätlingite and gehlenite were formed to fill the internal pores and form special phases, such as C-Mn-S-H and K-A-S-H.

SR is a kind of alkali waste residue discharged during the production of sodium carbonate with the ammonia-soda process. SR is characterized by strong alkalinity, with a pH value of approximately 11–12, and mainly consists of CaCO₃, Ca(OH)₂, CaCl₂, and CaSO₄ [17–19]. In particular, China has a vast production scale of sodium carbonate, with an annual SR discharge of over 1000 t. Approximately 0.3 t SR should be discharged for preparing 1 t sodium carbonate [20]. SR accumulation can bring about high processing costs for enterprises and occupy a large land area. The harmful ingredients are easily permeated into the soil, which can cause land salinization and lower soil quality. Moreover, SR powder particles are quite fine and can be easily inhaled by people, thereby endangering health [21]. In recent years, some scholars have conducted an increasing number of studies on preparing a novel kind of alkali-activated cementing material based on the characteristics of SR, which can solve the secondary pollution problems caused by SR. An et al. used
gypsum powder-SR-GGBS to prepare a novel cementing material, in which the mixing ratio of gypsum powder was 0, 5, and 10%, the mixing ratios of SR were 0, 70, and 80%, and the rest was GGBS [22]. The results show that the strength dropped with the increasing addition of SR. The optimal mixing ratios of gypsum powder and SR were determined to be 5% and 60%, respectively. Under the optimal conditions, the 28d unconfined compressive strength was 9310 kPa. Because of the activation of SR, C-S-H gel was produced, and AFT acted as the main crystal product and provided the primary source of strength. Lin et al. employed strong alkaline SR to activate GGBS and measured the prepared mortar samples’ fluidity and compressive strength. According to their results, under the condition (with a water-to-binder ratio of 0.5, SR and GGBS mixing ratios of 16–24% and 76–84%, respectively. The 28d compressive strength was 32.3–35.4 MPa, and the fluidity ranged from 181 to 195 mm. The main hydration products were ettringite, Friedel's salt, and C-S-H gels.

Finally, based on previous research results [3] and a literature review, this study aimed to optimize the curing method of alkali-activated SR-GGBS cementing materials. The most appropriate curing methods can be obtained by observing the strength development and microformation process of the different samples. The present research results can contribute to promoting and popularizing the SR-GGBS system.

2. Experimental Section

2.1. Materials

The raw materials used in this study mainly include SR, GGBS, NaOH, standard sand, and water. The chemical components, micromorphology, and mineral composition of SR and GGBS were measured via XRF, SEM, and XRD, and the results are illustrated in Figures 1 and 2, as well as tabulated in Table 1.

Figure 1. SEM images of (a) SR and (b) GGBS.

Table 1. Chemical composition of GGBS and SR.

| Material | CaO  | SiO₂ | Al₂O₃ | Fe₂O₃ | MgO  | TiO₂ | K₂O  | SO₃  | MnO  | Na₂O  | Cl⁻ | LOI * |
|----------|------|------|-------|-------|------|------|------|------|------|-------|-----|------|
| GGBS     | 33.70| 32.60| 17.10 | 1.18  | 7.96 | 2.54 | 0.57 | 3.21 | 0.31 | 0.56  | -   | 2.14 |
| SR       | 43.20| 9.87 | 3.25  | 0.91  | 9.77 | 0.12 | 0.29 | 5.57 | -    | 3.93  | 23.00| 2.86 |

* LOI = loss on ignition.
The particle sizes of the two raw materials were analyzed with a BT-9300H Baxter laser particle sizer, and the results are shown in Figure 3. The specific surface area was measured via the gas absorption BET method, and the density was measured using Lee’s bottle method, as shown in Table 2.

![XRD spectra of SR and GGBS](image)

**Figure 2.** XRD spectra of SR and GGBS.

The specific surface area was measured via the gas absorption BET method, and the density was measured using Lee’s bottle method, as shown in Figure 3. The specific surface area was measured via the gas absorption BET method, and the density was measured using Lee’s bottle method, as shown in Table 2.

![Particle size distributions of SR and GGBS](image)

**Figure 3.** Particle size distributions of SR and GGBS.

**Table 2.** Physical properties of GGBS and SR.

| Material | Specific Gravity (g/cm²) | Specific Surface Area (m²/kg) |
|----------|--------------------------|-------------------------------|
| GGBS     | 2.742                    | 419.5                         |
| SR       | 2.351                    | 261.2                         |

This study used SR manufactured by Tangshan Sanyou Chlor-Alkali Co., Ltd., Hebei, China. The initial moisture content of the SR mortar exceeded 90%. Before use, the slurry was allowed to stand still for 2–8 days, and the upper clear liquid was removed until the slurry was separated. The lower slurry with a moisture content of 50–60% was dried for
24 h and ground to SR powder with a particle size below 0.16 mm. As shown in Figure 1a, SR particles could be easily aggregated with many pores. The SR mineral phases were mainly calcium chloride hydroxide, gypsum, halite, and calcite. According to the XRF measurement results, SR is primarily composed of CaO (43.2%) and Cl\(^{-}\) (23.0%), with small fractions of SiO\(_2\) (9.87%) and Al\(_2\)O\(_3\). The density, specific surface area, and mean particle size of SR were 2742 kg/m\(^3\), 419.5 m\(^2\)/kg, and 20.31 µm, respectively. GGBS, from Hebei, China, was ground with S95 GGBS. As shown in Figure 1b, GGBS was composed of irregular blocks in terms of micromorphology. The main mineral phase of GGBS was quartz. Based on XRF measurement results, GGBS under study was composed of CaO, SiO\(_2\), and Al\(_2\)O\(_3\) in terms of chemical components, with proportions of 33.7, 32.6, and 17.1%, respectively. The density, specific surface area, and mean particle size of SR were 2742 kg/m\(^3\), 419.5 m\(^2\)/kg, and 14.6 µm, respectively. The sand used in this study satisfied the GB/T17671 standard, with a particle size range of 0.08–2.0 mm and a sediment content below 0.2%. Tap water in the laboratory was also used in this study.

2.2. Mixing Ratios

Based on previous research results, the SR-to-GGBS ratio was fixed at 4:6. This study focused on the effect of different curing methods (including room temperature (RT) water curing, RT dry curing, standard curing, RT sealed curing, and HT curing) on the strength and microstructure of prepared novel gel samples. Table 3 lists the detailed mixing ratios and curing methods.

| Sample ID | Dosage of Dry Basis/g | Curing Method |
|-----------|-----------------------|---------------|
| SR        | GGBS                  |               |
| F1        | 180                   | RT *, Water curing |
| F2        | 270                   | RT *, Dry curing |
| F3        | 1350                  | Standard curing |
| F4        | 0.5                   | RT *, Sealed curing |
| F5        |                       | 60 °C @ 6 h    |
| F6        |                       | 60 °C @ 12 h   |
| F7        |                       | 60 °C @ 24 h   |
| F8        |                       | 40 °C @ 12 h   |
| F9        |                       | 50 °C @ 12 h   |
| F10       |                       | 70 °C @ 12 h   |
| F11       |                       | 80 °C @ 12 h   |

* RT = room temperature, 20 ± 2 °C.

2.3. Sample Preparation

Before the test, water usage was first calculated based on the preset water-to-binder ratio. NaOH particles were first dissolved in water and stood still for 24 h. The detailed preparation procedures are described below. First, NaOH solution was poured into the agitating pan, and the mixture of SR powder and GGBS was added. The pan was fixed on the frame and lifted to the operating position. Second, the machine started for 1 min of stirring at low speed. The neat paste remained on the vanes, and the pan wall was spaded into the pan for 2 min of high-speed stirring to prepare neat paste samples. Third, the samples were stirred at low speed for 30 s, and standard sand was added. Then, the samples were stirred at high speed for 1.5 min to prepare the mortar samples. Finally, after adequate stirring, the samples were hierarchically put into test molds with exact sizes of 40 × 40 × 160 mm and then vibrated for 120 s. After the test mold was taken down, the sample was leveled and numbered.

2.4. Curing Procedures

The samples were demolded 24 h after pouring and then cured by methods listed in Table 3. The F1 sample was placed into the curing tank at RT (20 ± 2 °C) and cured
to the preset period. The F2 sample was cured at RT to the preset period. The F3 sample was put into the standard curing box at a temperature of \((20 \pm 2\, ^\circ\text{C})\) and humidity of over 90% and cured for the specified period. The F4 sample was wrapped with plastic film and cured at RT to the preset period. Samples F5–F11 were wrapped into plastic film, placed into an oven for HT curing, then transferred to RT for further sealed curing until the specified period.

2.5. Testing Procedure

The compressive strengths were measured according to the GB/T17671 standard. The test periods were set as 3, 7, and 28 days. At each age, the samples were measured six times for averaging. For each sample, the mean and standard deviation were used for characterization. The load was uniformly applied to the sample during the test at a loading rate of 2400 N/s ± 200 N/s until failure.

Paste samples with a cubic size of 50 mm × 50 mm × 50 mm were used in this study. After curing to the preset period according to the methods described in Table 3, the sample was loaded to failure. The central region without carbonization was taken and soaked in isopropanol for 3–7 days to avoid further hydration. Finally, the small blocks were removed and placed into a vacuum-drying oven to remove the residual isopropanol. Small samples with dry and smooth surfaces and maximum side lengths below 10 mm were selected for the SEM test. Before XRD, FTIR, and TD-DTG tests, small dry samples were removed and ground into a powder with a particle size below 0.075 mm by an agate mortar. The powder sample was then placed into a vacuum bag for standby application. During the present XRD test, a Cu target was used; the tube voltage and current were set as 40 kV and 40 mA, respectively; the scanning angle ranged from 5 to 65°, with the scanning rate preset at 2°/min. The wavenumber in the present FTIR test varied within a range of 4000–400 cm\(^{-1}\) at a resolution of 2 cm\(^{-1}\). During the TG-DTG test, the temperature varied from RT to 1000 °C at a heating rate of 10 °C/min, and Ar acted as the protective gas. XRD, FTIR, SEM, and TG-DTG were tested using a D/MAX-2500/PC X-ray diffractometer, BRUKER TENSOR II infrared spectrometer, TESCAN-VEGA3, and STA449C/6/G integrated thermal analyzer, respectively.

3. Results and Discussion

3.1. Compressive Strength

Figure 4 displays the measured results of the samples’ 3d, 7d, and 28d compressive strengths after different curing methods, curing temperatures, and curing times.

3.1.1. Effect of the Curing Method on the Compressive Strength

Figure 4a depicts the effect of different curing methods on compressive strength. The compressive strength increased with the prolonging of the curing period. The 3d compressive strengths of the samples after water curing, RT dry curing, standard curing, and RT sealed curing were 10.62, 11.03, 9.55, and 9.83 MPa, respectively, with slight differences. After drying at 60 °C for 12 h (first curing at 60 °C for 12 h and then under standard curing conditions to the specified period), the 3d compressive strength could reach up to 19.18 MPa. This might be because appropriate HT curing accelerated the hydration process, generating more hydration products, and enhancing structural compactness, thereby improving the sample’s early strength. As the curing period increased to 7 and 28 days, the sample after RT dry curing showed the poorest performance and slow mechanical performance development. For the sample after RT dry curing, the 7d and 28d compressive strengths were 14.62 and 20.09 MPa, respectively. The reason was that water could be rapidly evaporated in a dry environment, leading to insufficient water consumption for hydration and incomplete hydration. Therefore, the sample has low strength [15]. In contrast to samples after RT dry curing, the samples’ 7d and 28d compressive strengths after water curing, standard curing, and RT sealed curing increased significantly, amounting to 17.01/26.04 MPa, 19.15/25.98 MPa, and 20.21/28.96 MPa, respectively. It can be seen
that the strength of RT sealed curing in the 3 and 7 days is slightly different from the
standard curing, and it is only slightly higher than the standard curing in the 28 days,
which indicates that the water supply of this new type of binder under sealed curing can
meet the long-term hydration demand, and does not need high humidity, which may also
be an advantage of this binder. The 7d and 28d compressive strengths of the mortar sample
after HT curing were the highest, reaching 25.37 and 30.11 MPa, respectively.

![Compressive Strength Graphs](image)

**Figure 4.** Test results on the compressive strength: (a) effect of curing methods; (b) effect of HT curing temperature; (c) effect of HT curing time.

### 3.1.2. Effects of High-Temperature Curing Temperature on Compressive Strength

Figure 4b shows the effect of the curing temperature on the compressive strength.
After curing at high temperatures (40, 50, 60, 70, and 80 °C) for 12 h, the samples were
cured to the specified periods under standard curing conditions. It can be found that
different curing temperatures impose different effects on the development of compressive
strength. For sealed curing from RT to 60 °C, the compressive strength was positively
correlated with the curing temperature. With increasing temperature, the 3d, 7d, and
28d compressive strengths increased from 9.83, 20.21, and 28.96 MPa to 17.44, 25.37, and
30.11 MPa, respectively. As the curing temperature increased, the compressive strength
was negatively correlated with the curing temperature, accompanied by decreases in the
3d, 7d, and 28d compressive strengths from 18.68, 24.86, and 28.24 MPa to 17.94, 23.98,
and 27.44 MPa. Accordingly, the optimal curing temperature was determined to be 60 °C. Previous experimental studies have reported similar findings [15,16]. In particular, Guo et al. revealed that curing at 60 °C enhanced the early strength of alkali-activated GGBS [15]. The 3d compressive strength after curing at 60 °C was 19.81 MPa. The 3d compressive strengths of the samples after curing at RT, 40, 50, 70, and 80°C were 51.25, 76.38, 97.39, and 93.53% of the values after curing at 60 °C. The 7d and 28d compressive strengths after curing at 60 °C were 25.37 and 30.11 MPa, respectively. Similarly, compared with the values after curing at 60 °C, the samples’ 7d and 28d compressive strengths after curing at room temperature, 40, 50, 70, and 80 °C were 79.66%/96.18%, 79.19%/95.75%, 88.49%/94.85%, 97.98%/93.79%, and 94.52%/91.13%, respectively. This suggests that curing at an appropriate high temperature can enhance the early strength and impose no adverse effect on the long-term strength.

3.1.3. Effect of High-Temperature Curing Time on Compressive Strength

Figure 4c illustrates the effect of different HT curing times on compressive strength. The samples were first cured at 60 °C for 6, 12, and 24 h, then placed at RT and cured for the specified periods. As the curing time increased from 6 to 12 and 24 h, the 3d compressive strength increased steadily from 15.98 and 19.18 MPa to 19.48 MPa. The 7d and 28d compressive strengths increased and then dropped, as shown in Figure 4. By considering the strength and energy consumption cost, curing at 60 °C for 12 h was the optimal curing time. As the curing time increased to 24 h, the strength dropped, confirming the adverse effect of long-term HT curing on the strength. This is consistent with previous research results. After heating at a high temperature for a long time, cracks were quickly produced. This can be attributed to different thermal expansion coefficients among different raw materials. Long-term HT curing can lead to the formation of cracks, thereby deteriorating the strength.

3.2. X-ray Diffraction (XRD) Analysis

The produced hydration products were analyzed via XRD based on macromechanical test results to investigate further the difference in mechanical performance under different curing conditions. The 3d and 28d XRD patterns under different curing conditions (RT sealed curing, curing at 60 °C for 12 h, curing at 60 °C for 24 h, and curing at 80 °C for 12 h) are displayed in Figure 5 for comparison. As expected, the sample after HT curing showed a noticeable change.

![Figure 5. XRD patterns of samples: (a) 3 days; (b) 28 days. Hereinafter, the following designations are used: A = hydrotalcite, F = calcium chloroaluminate hydrate, C = calcite, H = halite, Q = quartz, E = ettringite.](image-url)
According to Figure 5a, the 3d crystal hydration products after the RT sealed curing were ettringite (E) and calcium chloroaluminate hydrate (F). Specifically, quartz (Q) was sourced from GGBS, and calcite (C) was sourced from SR. The F-phase and E-phase were enhanced by comparing the patterns of the F4 and F6 samples, while the quartz peak dropped. This suggests that curing at an appropriate temperature can promote the hydration of GGBS and the production of C-A-S-H gel, as well as E and F crystals, thereby enhancing the early strength.

By comparing the patterns of the F6 and F7 samples, the peaks of the E and F crystals showed no changes with prolonged HT curing time. The characteristic peak of quartz dropped, suggesting constant hydration of GGBS. By comparing the XRD patterns of the F6 and F11 samples, the peak of E disappeared with increased curing temperature. This is because E is greatly affected by temperature. At too high a temperature, E may be decomposed into AFm. Accordingly, no diffraction peak of E was observed at 80 °C [23,24]. The decrease in the F and A peaks suggests that too high a temperature was unfavorable for forming F and A.

As shown in Figure 5b, compared with the 3d XRD patterns of the samples, F and E of the F4, F6, and F7 samples were enhanced, while the peak of quartz dropped. This suggests that hydration of GGBS occurred steadily. F, A, and E were gradually produced during the hydration process. No E peaks appeared in the 28d XRD pattern of the F11 sample, indicating the thorough decomposition of E. In addition, humps can be observed in the XRD patterns of all samples at 25–35°, suggesting the production of amorphous C-A-S-H gel in the hydration products [25]. For the samples after curing at 60 °C for 12 and 24 h, both the height and width of the hump were maximal, suggesting that an appropriate curing temperature can promote the generation of gel and the development of strength.

Overall, curing at an appropriate high temperature can promote the dissolution of GGBS in an alkaline environment and generate more hydration products for gap filling. Accordingly, the sample’s mechanical performance can be enhanced. This finding is consistent with the measured compressive strength results.

3.3. FTIR Analysis

Figure 6 displays the FTIR spectra of the F4, F6, F7, and F11 samples after curing periods of 3 and 28 days.

![FTIR curves](image1.png)

**Figure 6.** FTIR curves of samples after curing periods of 3d (a) and 28d (b).
The peak at 583 cm\(^{-1}\) can be attributed to the asymmetric stretching vibration of Si-O-Al, and the peaks at 797 and 974 cm\(^{-1}\) to the stretching vibration of Si-O. All these absorption peaks are induced by forming a C-A-S-H gel \([26,27]\). The peak at 663 cm\(^{-1}\) can be attributed to the vibration of Mg-O and Al-O \([25]\). The absorption peak at 1103 cm\(^{-1}\) can be attributed to the stretching vibration of SO\(_4^{2-}\) \([28]\). The peaks at 713, 875, and 1452 cm\(^{-1}\) correspond to the asymmetric structures of CO\(_3^{2-}\) \([29,30]\). The peaks at 1640 and 3641 cm\(^{-1}\) can be attributed to the bending vibration and stretching vibration of the H-O-H bond in crystal water. The peak at 3440 cm\(^{-1}\) corresponds to the stretching vibration of Al-OH stretching vibration in the octahedral structure of [Al(OH)\(_6\)]\(^{3-}\), implying the formation of F \([31,32]\).

By comparing the FTIR patterns of the F4 and F6 samples, the peaks at 583, 797, and 974 cm\(^{-1}\) increased after curing at 60 °C for 12 h, suggesting that appropriate curing can promote the formation of C-A-S-H. The peaks at 663, 1103, and 3440 cm\(^{-1}\) were enhanced, indicating that HT curing could accelerate the formation of E, F, and A. This is consistent with the present XRD measured results. As the curing time increased from 12 to 24 h, the peaks corresponding to C-A-S-H increased, but the peaks corresponding to A, F, and E varied slightly. Accordingly, it can be concluded that curing time can promote gel formation but imposes a slight effect on the production of crystal hydration products. However, based on the research results by Guo et al., a curing time that is too long may lead to the formation of fractures \([15]\). As the curing temperature increased, the peak at 1103 cm\(^{-1}\) disappeared, indicating the decomposition of F at high temperatures. This fits well with the XRD measurement results that the peaks of the C-A-S-H gel, F, and A crystals varied slightly.

As the curing period increased to 28 days, the characteristic peaks at 583, 663, 797, 974, 1103, and 3440 cm\(^{-1}\) depicted in Figure 6b were enhanced compared with the peaks in the FTIR curves of 3d samples in Figure 6a. Accordingly, increasing amounts of A, F, and E were generated, suggesting that hydration was a sustainable development process. The observations from FTIR curves fit well with the XRD results.

3.4. SEM Analysis

To characterize the effects of different curing methods, temperatures, and times on the sample’s microstructure, SEM images of the F4, F6, F7, and F11 samples at a curing period of 3 days are displayed in Figure 7. Under RT sealed curing (as shown in Figure 7a), unreacted GGBS, SR, and a few C-A-S-H gels can be observed. The produced gel wrapped unreacted GGBS. The needle-shaped E was interwoven into a network. Overall, the structure was relatively loose with low compactness. Many pores can be observed, which can be attributed to the slow reaction of alkali-activated GGBS. Figure 7b depicts the results of the samples after curing at 60 °C for 12 h. The microstructure can be improved to a certain degree, and only a few unreacted particles were observed, while an increasing amount of E was produced. A large amount of C-A-S-H gel was produced and filled the pores, thereby improving structural compactness. This suggests that appropriate HT curing can accelerate the activity of hydroxide ions in alkali solution, accelerating the solution rate of aluminum silicate precursors \([33]\).

This can also account for the favorable mechanical properties of alkali-activated GGBS after curing at 60 °C. After curing at 60 °C from 12 to 24 h, many fractures can be observed on the surface despite the generation of many gels. This is consistent with the research results by Nasir \([16]\). As the curing temperature increases to 80 °C, almost no E can be observed, suggesting that E has almost been decomposed. This is consistent with the above-described XRD results. At excessively high temperatures, the surface of the raw materials was wrapped by the produced hydration products, which can inhibit the reaction with the alkali solution, thereby reducing the long-term strength. According to the present experimental results, 60 °C was determined as the optimal curing temperature.
3.5. Thermogravimetry/Derivative Thermogravimetry Analysis (TG-DTG)

Thermogravimetry/derivative thermogravimetry (TG-DTG) curves of the F4, F6, F7, and F11 samples after a curing period of 3 days are plotted in Figure 8, while Figure 9 presents the statistics on weight losses of four samples in a temperature range from 50 to 400 °C. In Figure 8, three weight loss peaks can be observed, which are located at 50-200 °C, 250-400 °C, and 400-600 °C.

![Figure 7. SEM images of the samples after a 3d curing period: (a) F4, (b) F6, (c) F7, and (d) F11.](image)

![Figure 8. DTG curves of 3d-cured specimens.](image)
weight loss ratio dropped. Thus, an excessively high curing temperature has an adverse effect on the production of crystal hydration products, including A and F. As the curing temperature further increased, the weight loss ratio varied slightly, suggesting that curing at too high temperatures can reduce the production of crystal hydration products. On the one hand, high temperature can promote the decomposition of E, reducing its further formation. On the other hand, the surface of raw materials was wrapped by the hydration products rapidly generated at high temperatures, hindering further reaction and thereby reducing the strength [35].

The peak at 50–200 °C can be attributed to the water loss-induced decomposition of the C-A-S-H gel and E. Specifically, the peak at 85–110 °C corresponds to E [34]. Compared with the condition under RT curing, the water loss peak was enhanced after heating, accompanied by an increase in the weight loss ratio from 6.21 to 7.72%. Accordingly, it can be concluded that curing at an appropriate temperature can promote the formation of E and C-A-S-H gels. The generated hydration products filled the pores. Therefore, the sample can obtain higher mechanical performance, which is consistent with the measured results of compressive strength. With prolonged curing time, the water loss ratio increased steadily from 7.22 to 7.85%, suggesting the constant formation of hydration products. As the curing temperature rose to 80 °C, the characteristic peak can be attributed to the formation of the C-A-S-H gel because of the disappearance of E. Meanwhile, the water loss ratio was reduced to 6.84%. This suggests that curing at too high temperatures can reduce the production of hydration products. On the one hand, high temperature can promote the decomposition of E, reducing its further formation. On the other hand, the surface of raw materials was wrapped by the hydration products rapidly generated at high temperatures, hindering further reaction and thereby reducing the strength [35].

The peak at 250–400 °C was induced by A and F [36,37]. For the four samples, the weight loss ratios were 4.42, 5.35, 5.38, and 4.87%, respectively. As the curing temperature increased from RT to 60 °C, the weight loss ratio increased, suggesting that curing at an appropriate temperature can promote the formation of crystal hydration products such as A and F. As the curing time increased, the weight loss ratio varied slightly, suggesting that curing at too high temperature imposed almost no effect on the production of crystal hydration products such as A and F. As the curing temperature further increased, the weight loss ratio dropped. Thus, an excessively high curing temperature has an adverse effect on the production of crystal hydration products, including A and F.

The peak at 400–600 °C seems to be mainly related to water loss of Ca(OH)₂ [16]. However, the XRD and FTIR analyses revealed no apparent changes in Ca(OH)₂ under different curing conditions, which may be due to the less amount of Ca(OH)₂ generated.

4. Conclusions

This study analyzed the effects of different curing methods on the mechanical performance and microstructure of alkali-activated SR-GGBS cementing materials. The experimental results obtained made it possible to draw the following conclusions:

1. For alkali-activated SR-GGBS cementing samples under different curing conditions, the sample after RT sealed curing showed optimal mechanical performance. The compressive strength increased from 9.83 MPa at 3d to 28.96 MPa at 28d. However,
under all curing conditions under study, samples exhibited low early strength and developed early in terms of long-term strength.

(2) In contrast to the above four curing methods, HT curing at an appropriate temperature can remarkably enhance the early strength of the prepared alkali-activated SR-GGBS cementing materials without harming their long-term strength. This is because the hydration of GGBS is promoted at HT, generating more C-A-S-H gel and crystal hydration products, which fill the internal pores and improve the material integrity.

(3) HT curing at 60 °C for 12 h proved to be the optimal method among the investigated ones. Noteworthy is that excessive HT curing deteriorates long-term strength, producing shrinkage cracks and hindering further strength development.

(4) HT curing at 60 °C for 12 h is the most appropriate for preparing samples with high early strength in rapid construction. For the samples with a low requirement on early strength, RT sealed curing is suitable for reduced energy consumption and convenient operation advantages.

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