

Article

Experimental Study on the Activation Mechanism of Residual Slag Micro Powder After Recycled Aggregate of Waste Concrete

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Abstract

This study investigated sustainable activation strategies for residual slag micro powder derived from recycled waste concrete aggregates, aiming to advance circular economy principles in construction materials. An experimental study was carried out to explore the activation mechanisms of slag micro powder from recycled waste concrete aggregates to enhance its utility in building materials. Three methods—mechanical grinding, high-temperature calcination, and mechanical grinding–thermal activation—were evaluated comparatively. The results showed high-temperature calcination at 750 °C for 10 min proved most effective, achieving a 95.85% activity index. High-temperature calcination may contribute to the release of active SiO₂ and Al₂O₃ substances of slag micro powder, thereby improving the hydration performance of slag micro powder and its cement mortar's compressive strength. The flexural strength of cement mortar after different activation treatments was also analyzed. Mechanical grinding alone showed limited benefits, only achieving a less than 65.59% activity index, while the combined method negatively impacted the mechanical properties of cement mortar samples. An SEM (scanning electron microscope) and EDS (energy dispersive X-ray spectrometer) microstructural analysis supported these findings, highlighting enhanced hydration product formation after calcination at 750 °C for 10 min. This work may contribute to sustainable construction practices through the resource-efficient utilization of industrial by-products.

Keywords: waste concrete; sustainability; slag micro powder; activation mechanism; mechanical properties



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1. Introduction

The exponential increase in construction and demolition waste poses a serious threat to global environmental sustainability, with concrete waste dominating this growing burden [1–3]. Recycled aggregate technology provides a promising solution by crushing and screening waste concrete into usable materials, thereby reducing a reliance on raw natural aggregates and mitigating the damage to habitats of quarrying. However, this process inevitably produces residual slag micro powder (SMP), which is a low value by-product with a complex composition and inert mineral phases [4–6]. If not managed, SMP will exacerbate resource waste and environmental risks, including the contamination of soil/leachate by unhydrated cement particles and heavy metals. Therefore, sustainably stabilizing SMP is crucial for achieving closed-loop

material cycling in buildings. Revealing the activation mechanism of SMP, improving its activity, and achieving effective resource utilization have important practical significance.

The residual micro powder of recycled aggregate in waste concrete mainly comes from the attachments on the surface of cement and sand in the waste concrete, as well as the small particles generated during the crushing process [7]. Its chemical composition is complex, mainly including silicon dioxide (SiO_2), aluminum oxide (Al_2O_3), calcium oxide (CaO), etc., as well as a certain amount of unhydrated cement particles, impurities, and harmful substances [8,9]. In addition, the mineral composition of slag micro powder is mainly composed of inert minerals, with a relatively low content of active minerals, resulting in poor volcanic ash activity and limited application in building materials [10,11].

Due to the low activity of slag micro powder, if directly mixed into building materials such as concrete, it will reduce the strength, durability, and other properties of the material. However, the potential active components (such as SiO_2 , Al_2O_3 , etc.) contained in the micro powder of slag can be excited under certain conditions to produce volcanic ash reaction, generating hydration products with gelling properties, thereby improving the performance of the material [12–15]. Through activation treatment, the potential activity of slag micro powder can be stimulated, enabling it to play a greater role in building materials, achieving an efficient utilization of waste concrete resources, reducing dependence on natural resources, and reducing environmental pollution caused by construction waste, which is in line with the concept of sustainable development.

Mechanical activation is the most commonly-used method [16–18]. Mechanical activation is the process of grinding slag micro powder through the use of mechanical forces (such as ball milling, vibration milling, etc.) to reduce its particle size and increase its specific surface area. On the one hand, the reduction in particle size increases the contact area between the slag micro powder particles and water, promoting their chemical reaction with water. On the other hand, a mechanical force can cause a distortion of the crystal structure and an increase in defects on the surface of the slag micro powder particles, generating more active sites and thus enhancing their reactivity [17]. Peng et al. [18] prepared the micro-slag cement composite and found that the mechanical grinding process could improve the particle morphology, thereby enhancing the interaction between the particles and the strength and workability of the concrete.

Chemical activation is also widely used for the treatment of slag micro powder. Chemical activation is the use of chemical activators to bring about chemical reactions with slag micro powders, changing their chemical composition and structure, thereby stimulating their activity. Common chemical activators include alkaline activators (such as sodium hydroxide, sodium silicate, etc.), sulfate activators (such as sodium sulfate, calcium sulfate, etc.), and composite activators [19–21]. Thermal activation is the treatment of slag micro powder using high-temperature calcination. At high temperatures, the mineral components in the micro powder of slag may undergo decomposition, phase transformation, and other reactions, generating amorphous phases with high activity [22]. However, the thermal activation process requires a large amount of energy consumption, and high-temperature calcination may cause certain corrosion to the equipment. Therefore, in practical applications, energy consumption and cost issues need to be comprehensively considered.

To best of our knowledge, there is little research on the effect of the mechanical grinding–thermal activation method on the activity of slag micro powder. Although previous studies [23–28] have separately explored mechanical, chemical, and thermal activation methods, this study innovatively compared three activation strategies—mechanical grinding, high-temperature calcination, and mixed mechanical thermal processes—to determine the synergistic effects and trade-offs in energy efficiency, reactivity enhancement, and sludge replacement potential. This tripartite approach addressed a key knowledge gap:

optimizing SMP activation for industrial scalability while minimizing energy consumption and environmental impact. An experimental study was conducted on the activation mechanism of residual slag micro powder from recycled aggregates of waste concrete in this article. The slag micro powder required for the experiment is activated through three methods: mechanical grinding, high-temperature calcination, and mechanical grinding–thermal activation. Subsequently, the activated slag micro powder was replaced with cement at mass fractions of 15% to prepare cement sand specimens. Finally, the activity index was compared and analyzed to compare the activation mechanisms of different activation methods. The slag micro powder in this article is the residual slag micro powder from the recycled aggregates of demolished construction waste. This study would be beneficial for promoting the further high-value recycling of such slag micro powders.

2. Experimental Section

2.1. Raw Material

As shown in Figure 1, the raw materials used in this experiment are from Jiangsu Kangju Renewable Resources Technology Co., Ltd., Suqian, China. After crushing, screening, and washing, they are obtained in powder form, mainly concrete micro powder. Table 1 presents the basic performance indicators of slag micro powder.



Figure 1. The material of slag micro powder is sourced from Jiangsu Kangju Renewable Resources Technology Co., Ltd., Suqian, China.

Table 1. Basic performance indicators of slag micro powder provided by Jiangsu Kangju Renewable Resources Technology Co., Ltd., Suqian, China.

Slag Micro Powder	Color	Fineness (cm^2/g)	Clay Content (wt.%)	Loss on Ignition (wt.%)
performance index	off-white	1593	15%	2.5%

2.2. Experimental Design for Activation of Slag Micro Powder

As shown in Figure 2, the slag micro powder required for the experiment was activated through three methods: mechanical grinding, high-temperature calcination, and mechanical grinding–thermal activation. For the mechanical activation, a planetary grinder was used to grind the regenerated micro powder separately for 30 min, 60 min, and 120 min. The planetary ball mill operated at a rotational speed of 300 rpm, utilizing a combination of 5 mm, 10 mm, and 15 mm stainless steel grinding balls to achieve the optimal particle size reduction and surface activation of the slag micro powder. After screening, slag-regenerated micro powders of different fineness were obtained. For the thermal activation, the resistance furnace was used to activate the slag micro powder at high temperatures. The slag micro powder was calcined at 750 °C for 10 min, 20 min, and 30 min. For the

mechanical grinding–thermal activation, first the resistance furnace was used to heat the slag-regenerated micro powder to 300 °C, 500 °C, and 750 °C, with a heating time of 20 min, and then it was ground again for 60 min using the planetary grinder. Various types of cement mortar samples were prepared by a commonly-used cement sand mixer. According to the standard ISO Method for Testing the Strength of Cement Mortar GB/T17671-1999; Method of testing cement mortar strength; China Standards Press: Beijing, China, 1999 [29]), three 40 mm × 40 mm × 160 mm mortar specimens were prepared for each group. The test pieces were placed in the YH-40B standard constant temperature and humidity curing box for curing. After 24 h, they were demolded and placed in the standard curing box for complete curing. The curing temperature was (20 ± 2) °C and the relative humidity was 95%. Tables 2–4 present the three different activation methods in detail.

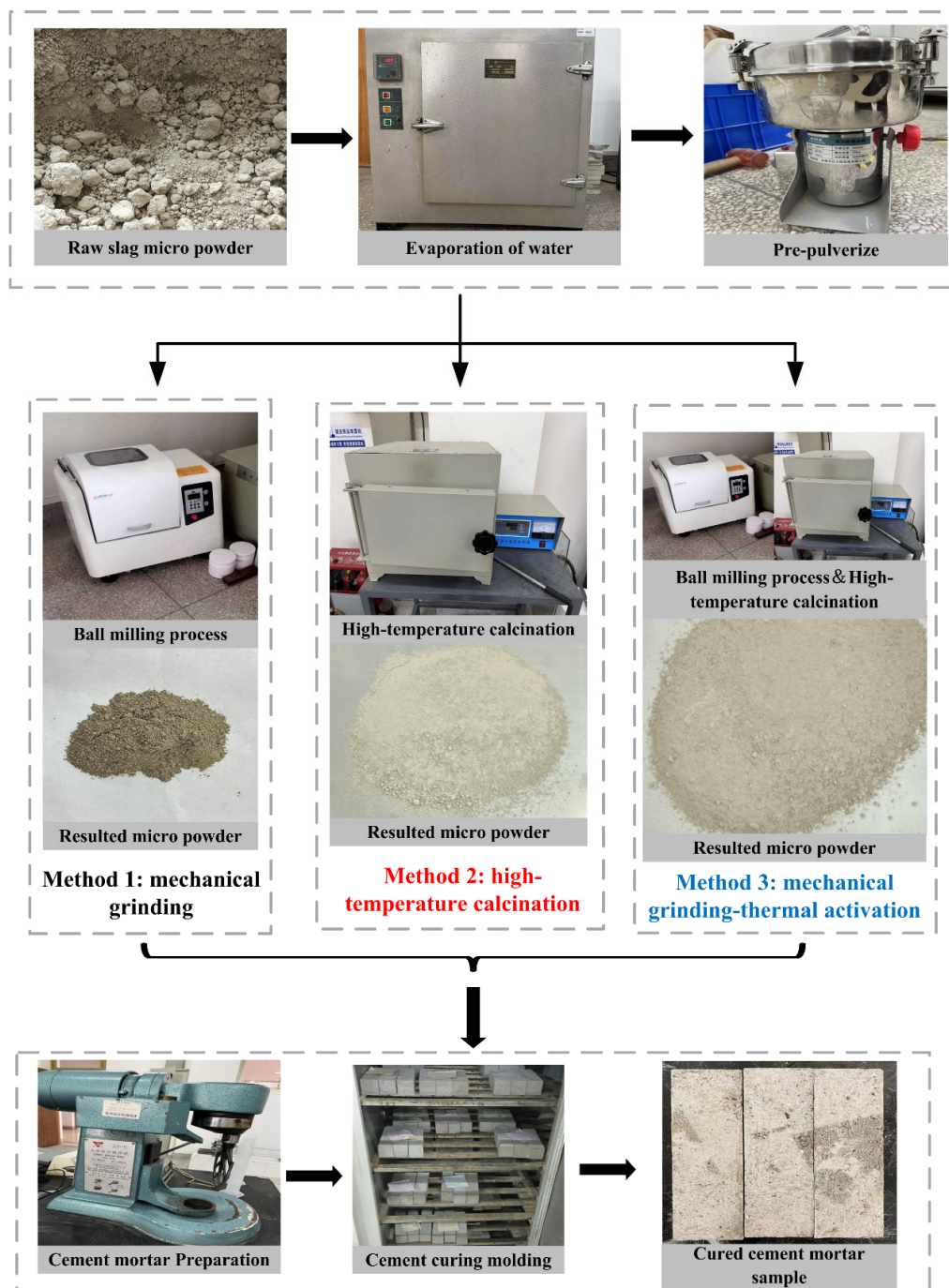


Figure 2. Experimental design for the activation of slag micro powder.

Table 2. Mechanical grinding method.

Sample Type	Grinding Time/min	Content of Slag Micro Powder	Cement Weight/g	Standard Sand Weight/g	Water Weight/g
Unfilled	-	-	450	1350	225
A	-	15%	380	1350	225
B	30	15%	380	1350	225
C	60	15%	380	1350	225
D	120	15%	380	1350	225

Table 3. High-temperature calcination method.

Sample Type	High-Temperature Calcination Time/min	Temperature/°C	Content of Slag Micro Powder	Cement Weight/g	Standard Sand Weight/g	Water Weight/g
E	10	750	15%	380	1350	225
F	20	750	15%	380	1350	225
G	30	750	15%	380	1350	225

Table 4. Mechanical grinding–thermal method.

Sample Type	Grinding Time/min	High-Temperature Calcination Time/min	Temperature/°C	Content of Slag Micro Powder	Cement Weight/g	Standard Sand Weight/g	Water Weight/g
H	60	20	300	15%	380	1350	225
I	60	20	500	15%	380	1350	225
J	60	20	750	15%	380	1350	225

2.3. Grain Size Analysis

In order to study the influence of the particle size distribution of slag micro powder on the mechanical behavior of cement mortar samples, the MS3000 laser particle size analyzer from the UK was used, and the parameter results of regenerated micro powder particles treated with different activation methods were tested using the wet method. The basic information of the instrument measurement includes the dispersant name being Water and the dispersant refractive index being 1.330; the particle absorption index is 0.100 and the particle refractive index is 1.710.

2.4. Determination of Activity Index

As shown in Figure 3, according to Test Method for Strength of Cement Mortar (ISO Method) [29], the mechanical properties of the prepared cement mortar samples were tested using the HYE-300B microelectromechanical hydraulic servo pressure testing machine (with a grade of 0.5 and a maximum test force of 300 kN/10 kN, produced by Hebei Sanyu Testing Machine Co., Ltd., Cangzhou, China). A three-point bending test was carried out first to check the flexural strength of the specimens. After the bending test, the compressive strength of the broken halves of the mortar specimens were measured, and two sides were selected as the compression surface.

The activity index (H) is calculated as the ratio of compressive strength between cement mortar specimens containing slag micro powder (as a cement substitute) and reference specimens made with pure cement mortar.

$$H = \frac{R}{R_0} \times 100\%$$

In the above equation, H , R , and R_0 represent the activity index, the compressive strength of the test sample containing the added slag micro powder, and the compressive strength of the pure cement mortar, respectively.

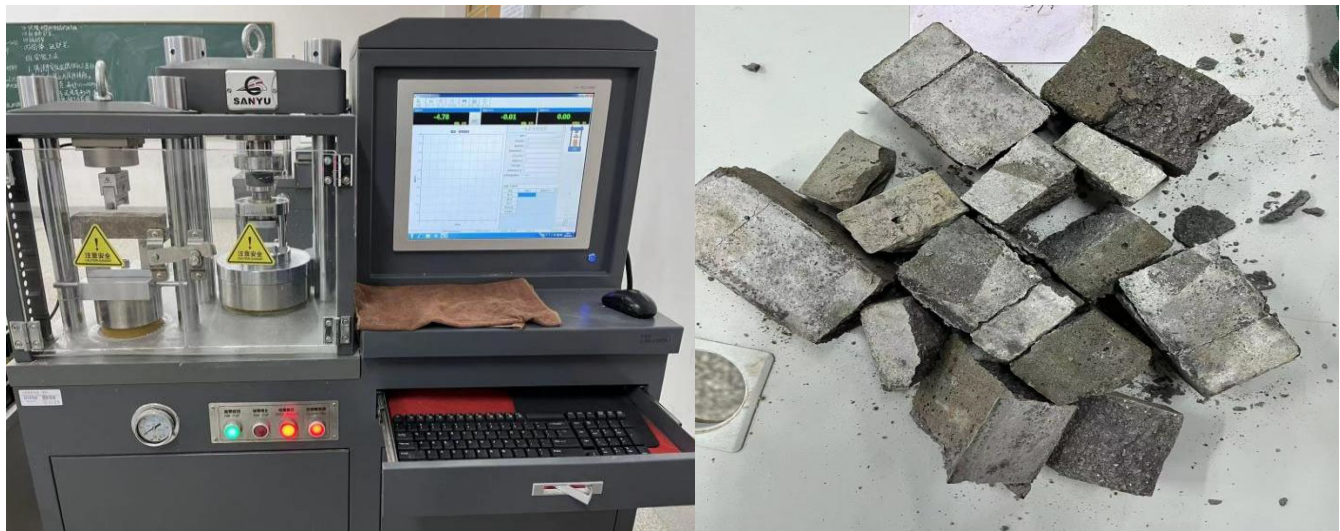


Figure 3. Compression and bending tests of cement mortar samples based on universal Instron testing machine.

2.5. SEM and EDS

After measuring the strength of cement mortar specimens with the high activity index, a thin strip of mortar specimen with a diameter of about 2 cm and a thickness of about 1 cm was selected, soaked in anhydrous ethanol with a content of not less than 99.7% and a density of 0.789–0.791 g/mL for 24 h, then removed and its surface wiped clean. In total, 10 mg of each powder sample was selected. Following the prescribed steps, SEM images and EDS spectra were obtained using a Nova NanoSEM 450 field emission scanning electron microscope produced by FEI Corporation in the United States. The microstructure, pores, structure, hydration products, and other changes in the activated slag micro powder were observed and analyzed.

3. Results and Discussion

3.1. Particle Characteristics of Activated Slag Micro Powder

The morphologies of regenerated micro powder samples varies greatly, with high porosity and diverse inner surfaces [30]. The distribution characteristics of particle size has a significant impact on the physical and chemical properties of the cementitious specimens. Therefore, the British MS3000 laser particle size analyzer was used to scientifically detect the particle distribution of activated micro powder in slag. Figure 4 shows the cumulative percentage of slag micro powder particles under different activation methods. Table 5 presents the detailed values of various particle size ranges in the distribution of slag micro powder. Table 6 presents the characteristic particle size parameters of slag micro powder. D10 represents the fine telomere diameter, which is the particle size value corresponding to the cumulative distribution of powder particles reaching 10%. D50 and D90 represent the median particle size and coarse telomere size, respectively. R80 is a sieve residue of 80 μm .

Sample A represented raw slag micro powder, while B, C, and D were slag samples mechanically ground for 30 min, 60 min, and 120 min, respectively. E, F and G denoted slag samples treated by heating to 750 $^{\circ}\text{C}$ for 10 min, 20 min, and 30 min, respectively. Finally, H, I, and J were slag samples under mechanical grinding–thermal activation at temperatures of 300 $^{\circ}\text{C}$, 500 $^{\circ}\text{C}$, and 750 $^{\circ}\text{C}$, respectively. As shown in Figure 4, except for the E sample, the

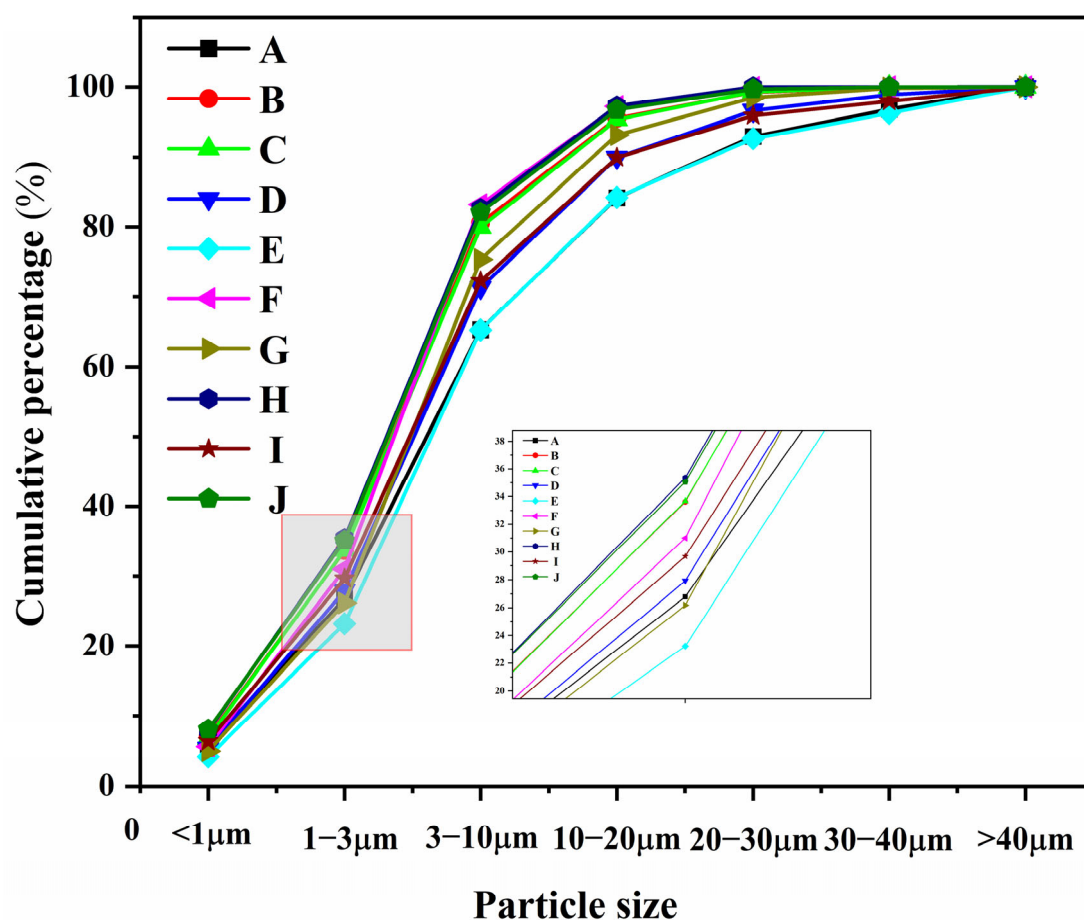
overall particle size of the slag micro powder decreased obviously after various activation methods. This indicated that only 10 min of heating time at a temperature of 750 °C was not sufficient to significantly alter the particle size distribution of the slag micro powder. As presented in Table 5, after grinding for 30 min and 60 min, the content of slag micro powder below 10 µm increased from 65.24% (Sample A: raw material) to 80.57% (Sample B), and 79.93% (Sample C), respectively. However, when the grinding time was further increased to 120 min (Sample D), the content of slag micro powder below 10 µm actually decreased to 71.29%. This may be because the original slag micro powder contained more large particles, and proper grinding could reduce the content of large particles. However, during the grinding process, if the grinding time was too long, small particles may agglomerate and become larger due to excessive grinding, resulting in an overall coarsening of the particle size [31]. The data in Table 6 also confirms this speculation. The specific surface areas of Sample B and Sample C are 1923 m²·kg^{−1} and 1912 m²·kg^{−1}, respectively. However, when the grinding time is 120 min, the specific surface area of the slag micro powder decreases to 1642 m²·kg^{−1}. Different from the mechanical grinding method, the mechanism of the high-temperature calcination method was very complicated. There are many minerals in the slag micro powder, which accumulate in a specific structure at room temperature and have relatively large particles. During high-temperature calcination, the original structure of the slag powder may become unstable and begin to “collapse” and reorganize [32]. In the newly formed structure, the grains may become smaller than before, and when numerous small grains gather together, the overall particle size would appear smaller. In addition, there are many small pores inside the particles of slag micro powder. During high-temperature calcination, the substances in the particles may change; some components, for example, may decompose, evaporate, flow, and rearrange [33,34]. As shown in Figure 4, for sample F, the content of slag micro powder below 10 µm was 83.24%, which was 27.59% higher than that of the raw material (Sample A). However, similar to sample D, a longer calcination time resulted in an increase in the particle size of the slag. Finally, for the mechanical grinding–thermal activation method, the particle sizes of the sample H, I, and J were significantly reduced compared to the untreated sample. However, this method could not further significantly reduce the particle size of the slag micro powder.

Table 5. Particle content of various particle size ranges in the distribution of slag micro powder particles under different activation methods.

Sample Type	<1 µm	1–3 µm	3–10 µm	10–20 µm	20–30 µm	30–40 µm	>40 µm	Cumulative Percentage (%)
A	5.8	20.99	38.45	18.94	8.78	3.91	3.13	100
B	7.01	26.61	46.95	15.01	3.68	0.68	0.05	99.99
C	6.83	26.84	46.26	15.48	3.91	0.65	0	99.97
D	5.36	22.57	43.36	18.62	6.76	2.25	1.06	99.98
E	4.16	19.06	42	19.02	8.42	3.63	3.69	99.98
F	5.64	25.36	52.24	14.03	2.65	0.08	0	100
G	4.99	21.19	49.13	17.89	5.32	1.31	0.18	100.01
H	7.88	27.47	47.34	14.65	2.67	0.02	0	100.03
I	6.38	23.29	42.57	17.69	6.02	2.04	2	99.99
J	8.02	27.02	47.08	14.69	2.9	0.29	0	100

Table 6. Characteristic particle size of regenerated micro powder particles.

Sample Type	BET Surface Area/m ² ·kg ^{−1}	D10/μm	D50/μm	D90/μm	R80/μm
A	1595	1.52	6.82	26.9	0
B	1923	1.36	4.74	15.9	0
C	1912	1.37	4.76	16.1	0
D	1642	1.55	5.96	21.3	0.14
E	1431	1.79	7.05	27.1	0
F	1814	1.51	4.83	14.3	0
G	1617	1.65	5.8	18.4	0
H	2022	1.28	4.53	14.5	0
I	1741	1.43	5.76	21.3	0
J	2016	1.27	4.62	14.8	0

**Figure 4.** Cumulative percentage of slag micro powder particles under different activation methods.

3.2. Effects of Different Activation Methods on the Mechanical Properties of Cement Mortar Filled with Slag Micro Powder

Figure 5 showed the effects of the mechanical grinding activation method on the compressive and flexural strengths of cement mortar filled with slag micro powder under different curing days. ‘Unfilled sample’ denoted the pure cement mortar. It was obvious that after adding slag micro powder, the compression and bending strength of the cement mortar was significantly reduced. This was because the strength of the cement mortar mainly came from the bonding effect of cement hydration products [35]. Highly active materials could undergo secondary hydration reactions with cement hydration products, generating more cementitious substances and enhancing the structure of the binder [36].

However, the activity of the slag micro powder was usually low, making it difficult for it to fully react with cement hydration products, unable to effectively fill the pores in the mortar, and unable to form sufficient bonding strength. After mechanical grinding activation, sample B exhibited higher compressive strength compared with sample A. This was because the cement mortar would form a porous structure during the hardening process. The particle size of the ground slag micro powder decreased, which could better fill the pores between cement particles, reduce the porosity inside the mortar, and make the mortar structure more compact, resulting in higher compressive strength. Strangely, the bending strength of sample B (after 28 days of curing) did not increase after the mechanical grinding activation. This may be because after mechanical grinding, the particle sizes of the slag micro powder became small, the specific surface area was large, and the surface energy was high, which would make it easy to agglomerate in the mortar. Agglomerations could form larger stress concentration points in the mortar. Moreover, under bending load, the testing sample was more sensitive to stress concentration, and cracks were prone to occur around the aggregates, which rapidly propagated and led to a decrease in the bending strength of the materials [37]. Importantly, simply activating through mechanical grinding could not significantly improve the strength of the cement mortar. Compared with unfilled cement mortar, the compressive strengths of the samples were largely reduced after filling with 15% slag micro powder, which was unfavorable for the engineering application of slag micro powder.

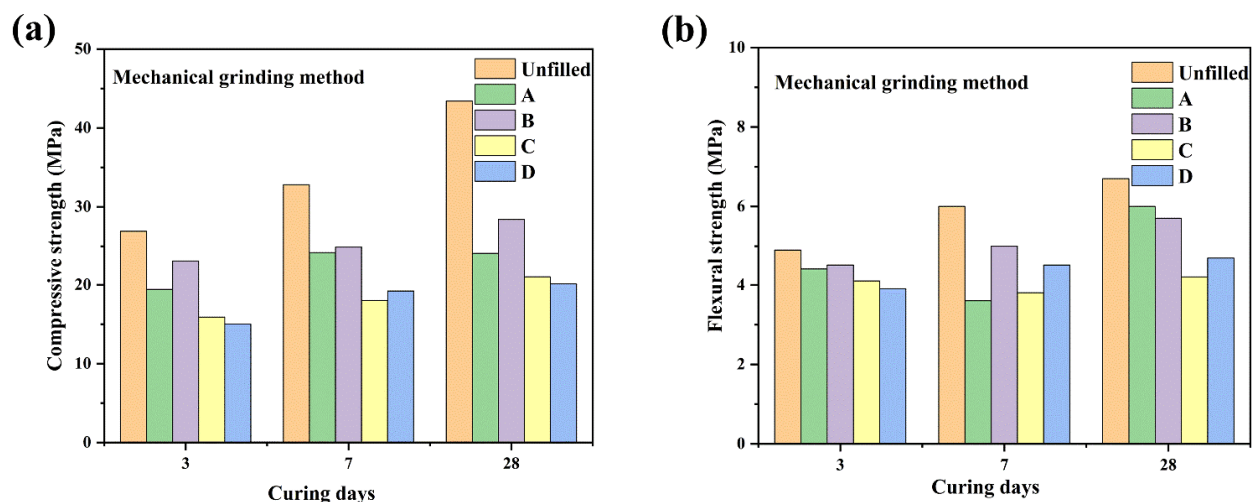


Figure 5. Effect of mechanical grinding activation method on the compressive (a) and flexural strengths (b) of cement mortar filled with slag micro powder under different curing days.

Fortunately, as shown in Figure 6, the high-temperature calcination method exhibited totally different experimental phenomena. The high-temperature calcination activation method significantly improved the compressive strength of the cement mortar. For example, the compressive strength of sample E increased by 72% compared to sample A. In addition, as discussed above, the particle size range of sample G was between sample E and sample F. However, the compressive strength of sample G was much lower than that of samples E and F. At the same time, there was a significant difference in particle size between samples E and F, but their mechanical behavior was very similar. These results indicated that there was no significant correlation between the mechanical properties of cement mortar and the particle size of the slag micro powder. Similarly, in mechanical grinding, regardless of the grinding time or the difference in particle size of the slag micro powder, the mechanical strength of the cement mortar was significantly reduced. This further proved that simply changing the particle size of the slag micro powder did not significantly improve the

strength of the cement mortar. After high-temperature calcination, the cement mortar exhibited an excellent residual mechanical property (the ratio of compressive strength between samples filled with micro powder and samples without micro powder). This indicated that high-temperature calcination could significantly enhance the activity of the slag micro powder. Under high-temperature heat treatment conditions, the slag powder may undergo thermally-induced phase transformation and amorphous transformation, and its mineral components were decomposed and reconstructed to form highly active amorphous phases [38]. This process may release active SiO_2 and Al_2O_3 components through the deconstruction of clay minerals, significantly enhancing their volcanic ash reactivity [39]. At the same time, it was accompanied by organic impurity pyrolysis and crystal water removal, achieving mineral surface purification and pore structure optimization, ultimately leading to a comprehensive improvement in cementitious activity and hydration reactivity. However, when the calcination time was 30 min, the strength of the cement sand sample filled with slag micro powder decreased significantly. This may be because during the long-time high-temperature calcination process, active SiO_2 and Al_2O_3 may undergo excessive sintering due to a prolonged exposure to high temperatures, reducing active sites such as unsaturated bonds and defects, thereby weakening the mineral composition's ability to react with cement hydration products and reducing the strength of the binder.

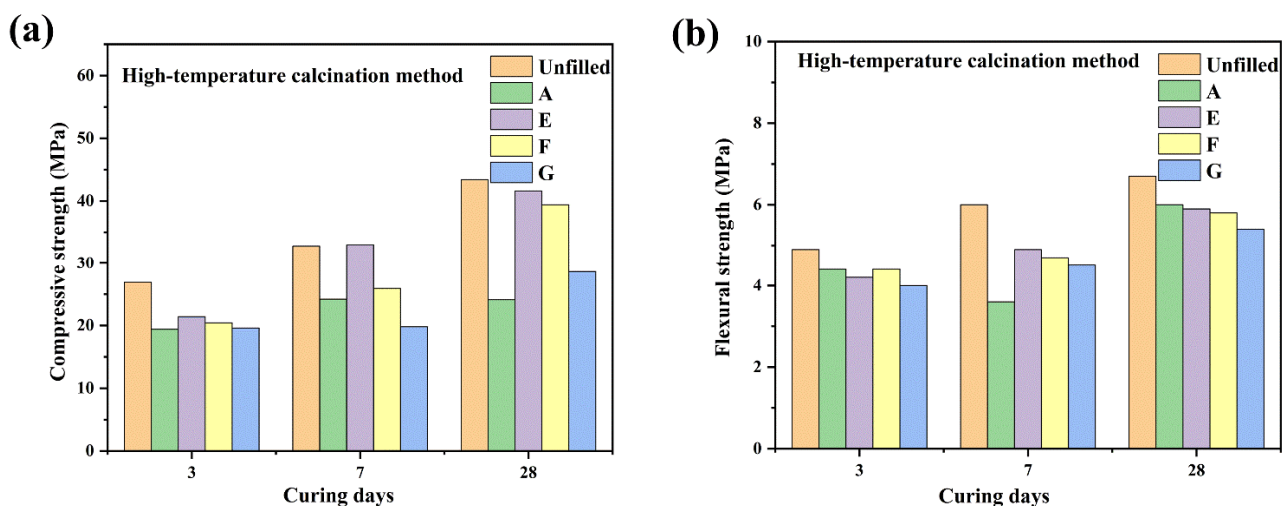


Figure 6. Effect of high-temperature calcination activation method on the compressive (a) and flexural strengths (b) of cement mortar filled with slag micro powder under different curing days.

In order to further improve the activity index of slag micro powder, this article adopted the third activation method (mechanical grinding–thermal activation method). Unfortunately, from Figure 7, it can be observed that this activation method did not further enhance the strength of the cement mortar, but instead produced negative synergistic effects. The reasons for this phenomenon may be very complex. There were essential differences in the impact pathways of the different processing methods on the physical structure, chemical composition, and reactivity of micro powders. The composite effect of the mechanical grinding and high-temperature treatment may be not simply a superposition, but involve a complex interaction of physical structure evolution, chemical phase transition, and thermodynamic equilibrium [40,41]. Further research was needed to reveal its microscopic mechanism.

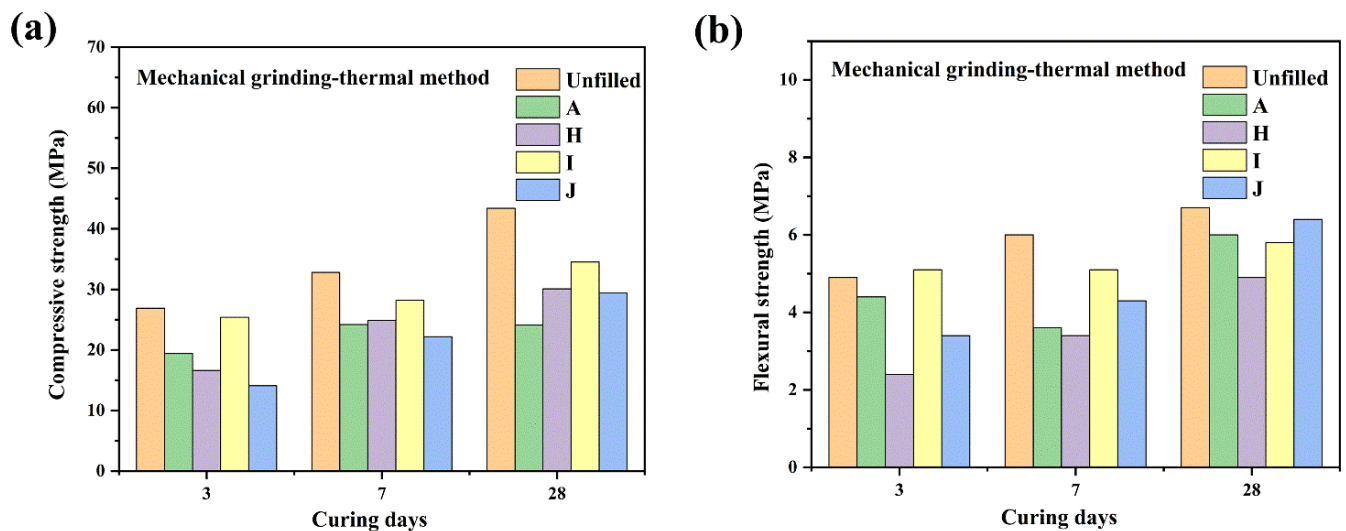


Figure 7. Effect of the mechanical grinding–thermal activation method on the compressive (a) and flexural strengths (b) of cement mortar filled with slag micro powder under different curing days.

Table 7 and Figure 8 present the mechanical properties of slag micro powder-filled cement sand composite materials under different activation methods. In the activation index determination, bending strength was not a specified indicator; its test results were greatly affected by the sample size and span, and the data dispersion may be higher than that of the compressive strength [42]. Therefore, this paper used the compressive strength to define the activation index. In addition, the activation index aimed to evaluate the long-term activity effect of slag soil, and the strength after complete solidification (28 day strength) more accurately reflected the degree of promotion of slag soil on the cement hydration reaction. Except for samples E and F, the strength of the cement mortar decreased by more than 15% after other activation methods. Due to the fact that the filling amount of slag micro powder was only 15%, a strength attenuation exceeding 15% was unacceptable in engineering application. Sample E not only had a high activity index, but also required relatively lower energy compared with high-temperature calcination for a longer period of time; therefore, the activation method of sample E exhibited potential in terms of engineering application.

Table 7. Mechanical properties of slag micro powder-filled cement sand composite materials under different activation methods.

Sample Type	Compressive Strength (MPa)	Flexural Strength (MPa)	Activity Index <i>H</i>
Unfilled	43.4 ± 3.9	6.7 ± 0.5	100%
A	24.1 ± 2.5	6.0 ± 0.2	55.50%
B	28.4 ± 3.1	5.7 ± 0.4	65.59%
C	21.0 ± 2.4	4.2 ± 0.5	48.39%
D	20.1 ± 2.2	4.7 ± 0.6	46.31%
E	41.6 ± 3.2	5.9 ± 0.3	95.85%
F	39.4 ± 3.1	5.8 ± 0.3	90.78%
G	28.6 ± 2.4	5.4 ± 0.4	65.90%
H	30.1 ± 2.7	4.9 ± 0.5	69.35%
I	34.5 ± 2.9	5.8 ± 0.6	79.49%
J	29.4 ± 2.3	6.4 ± 0.7	67.74%

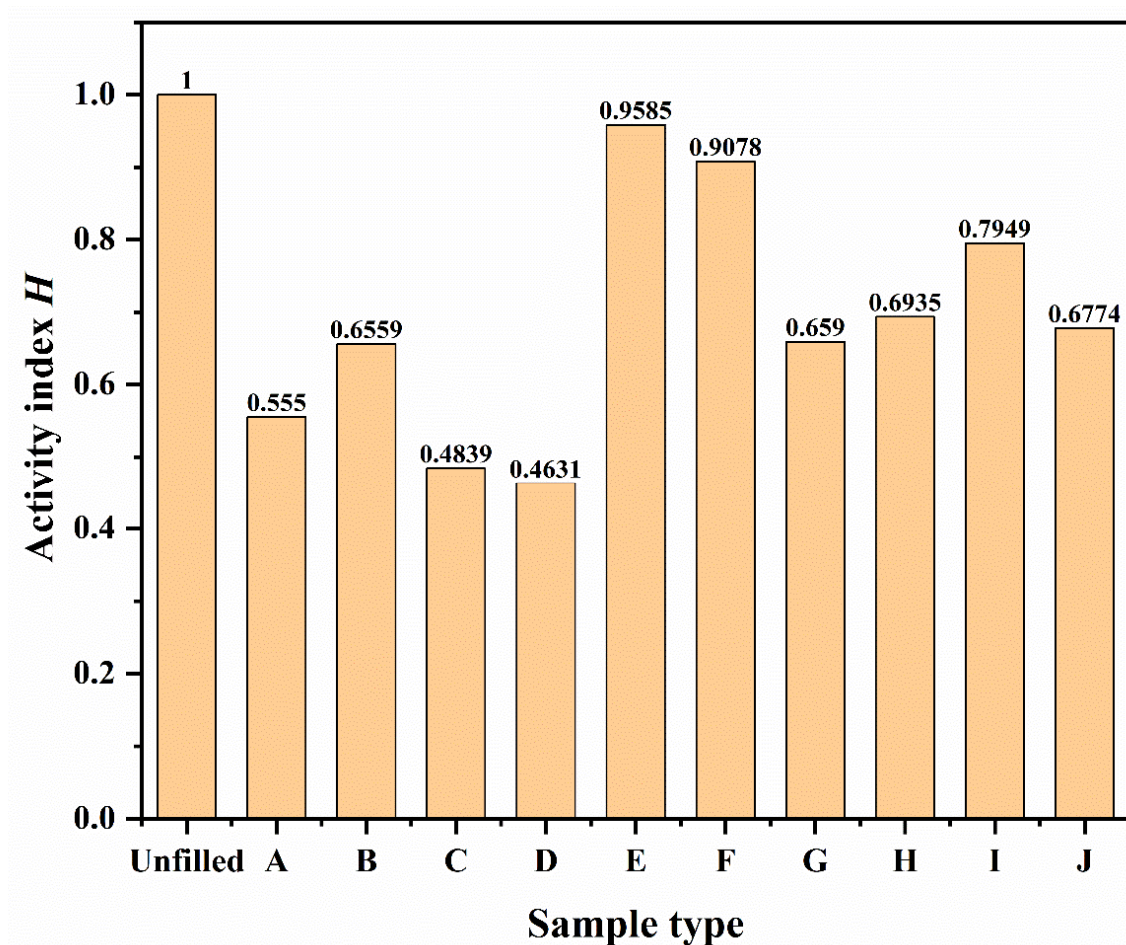


Figure 8. Effect of different activation methods on the activity index H.

3.3. Micro-Scale Mechanism Analysis

In order to further verify that the high-temperature calcination method could enhance the activity of slag micro powder, this paper comparatively analyzed the microstructures of cement mortar samples under different activation methods using SEM–EDS equipment. Figure 9a shows the original morphology of the untreated slag micro powder, and it can be seen that the particle size of the slag was uneven. In addition, needle-shaped substances could also be observed, indicating the possible presence of some impurities in the slag micro powder [43]. Figure 9b–d showed the microscopic morphologies of untreated slag micro powder-filled cement mortar after curing for 3, 7, and 28 days, respectively. After 3 days of hydration, Figure 9b clearly showed the unhydrated slag micro powder. After 7 days of hydration, crushed stone-like slag micro powders could be observed, indicating that no hydration substance had formed on the surface of the slag micro powder. After 28 days of hydration, still no obvious hydration products were found in the test sample. These conclusions were in good agreement with the mechanical experimental results presented in the previous section. The activation index of sample A was very low, only 55.50%. This indicated that directly filling untreated slag micro powder into cement would significantly reduce the strength of the cement mortar. Untreated slag micro powder may not be used as a filling material in cement mortar.

Figure 10 shows the microstructures of the sample with the highest activation index (sample E). Figure 10a–c shows the microscopic morphologies of sample E after curing for 3, 7, and 28 days, respectively. It can be clearly seen that with the increase of hydration days, more and more hydration substances were produced on the surface of the slag micro powder after the high-temperature treatment. After 3 days of hydration, although the

morphology of the slag particles could still be observed, their surface had become relatively smooth, indicating that a small amount of hydration substances had been generated on the surface of the slag micro powder. After 7 days of hydration, cotton-like hydration products could be found. By 28 days, the surface of the slag micro powder had been completely covered by hydration products. These results indicated that high-temperature calcination at 750 °C for 10 min could release the active ingredients in the slag micro powder. The hydration reaction of these active substances could improve the interfacial strength between the slag micro powder and the cement matrix, thereby enhancing the strength of the cement mortar. The EDS curves in Figure 10d further confirmed that the slag micro powder generated hydrated calcium silicate substances during the curing process. This indicated that high-temperature treatment may release reactive SiO_2 and Al_2O_3 components that participated in volcanic ash reactions, forming hydration products (e.g., C-S-H gel) and significantly improving the compressive strength of the cement mortar. However, it should be noted that SEM–EDS can only provide qualitative insights into the elemental composition and microstructure. Although EDS spectra confirm the presence of Si and Al (which are crucial for the reactivity of volcanic ash), they cannot directly verify the formation of amorphous phases or the dissolution of inert minerals in slag micro powders.

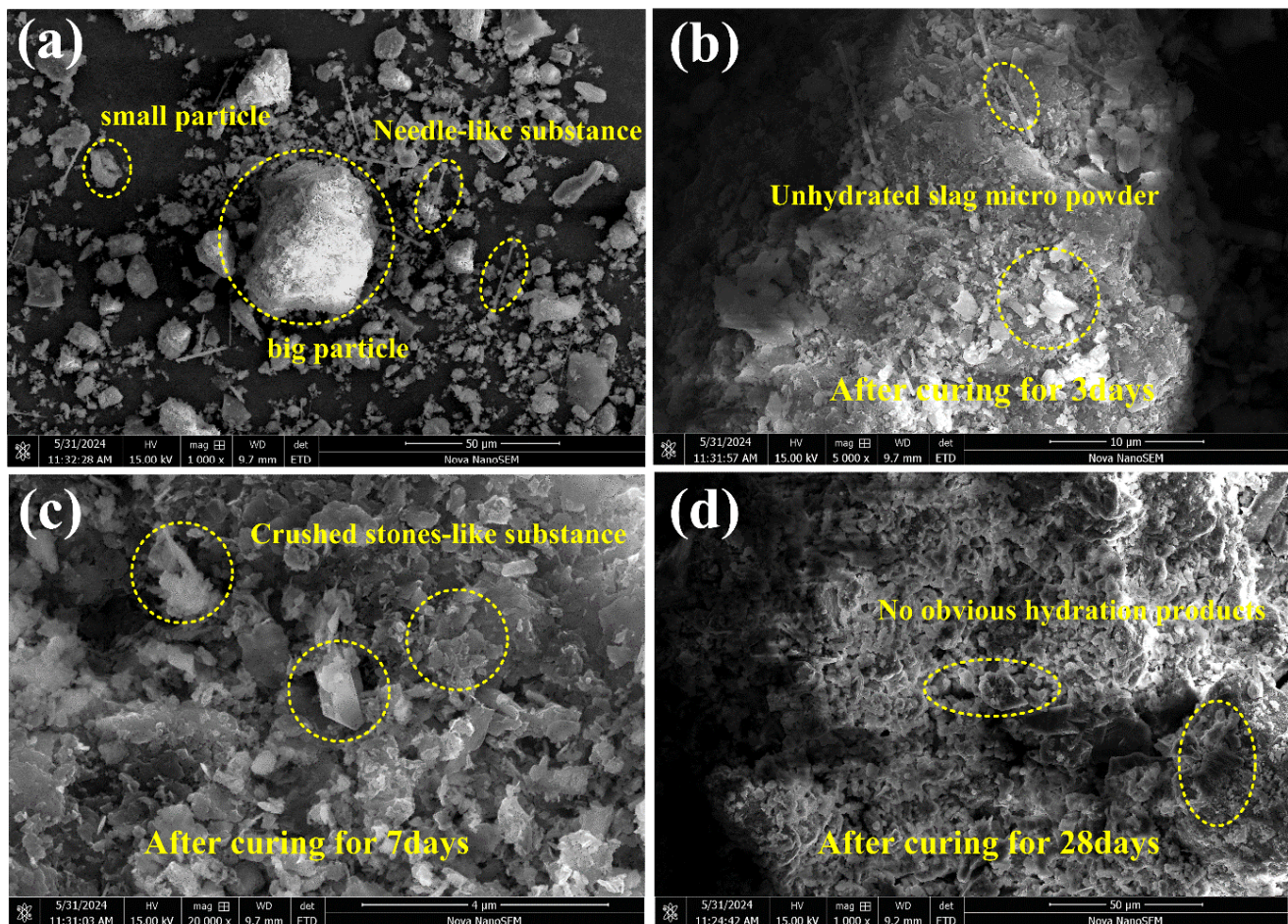


Figure 9. Microscopic morphologies of untreated slag micro powder (a) filled cement mortar after curing at 3 (b), 7 (c), and 28 (d) days, respectively.

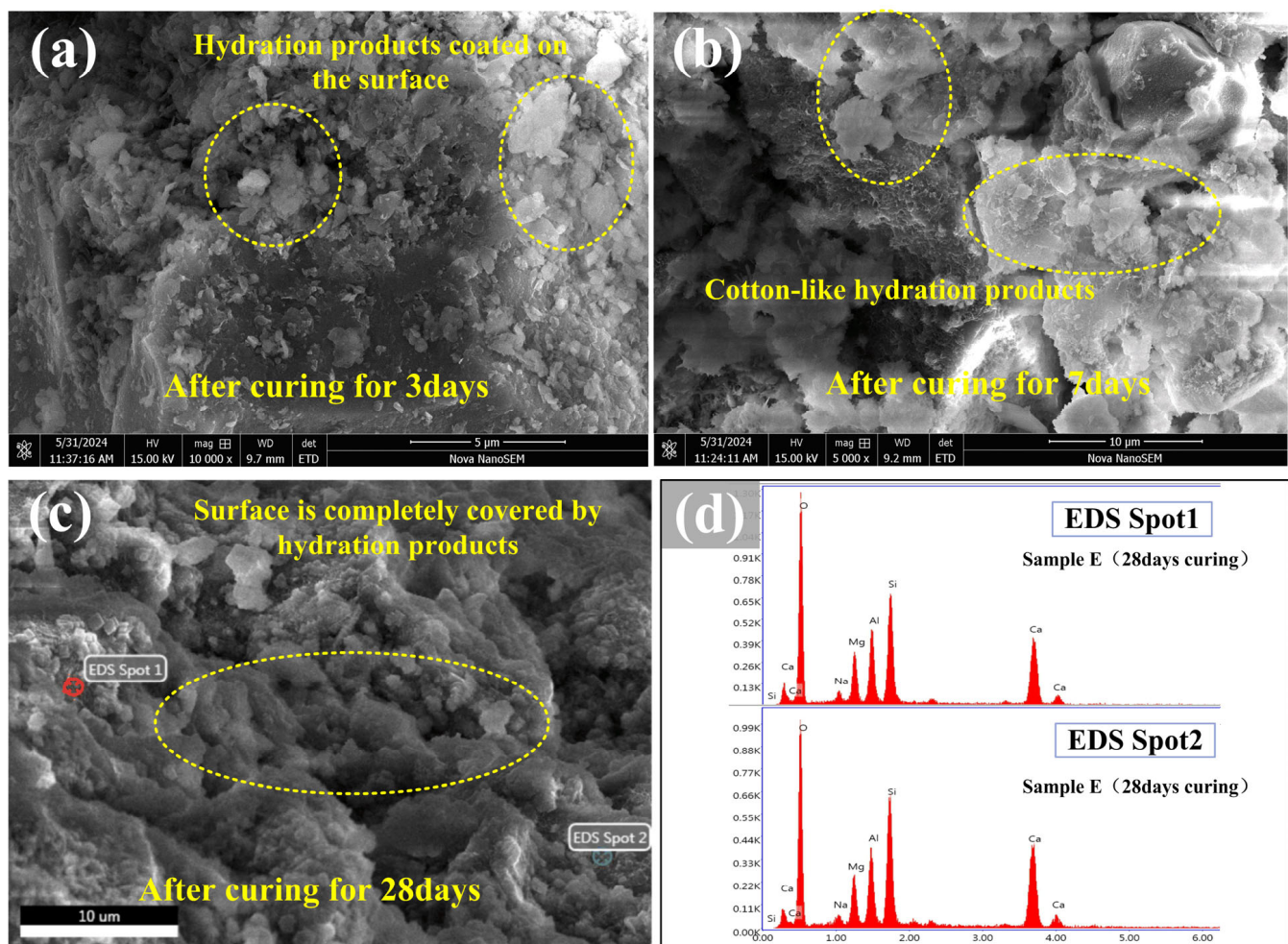


Figure 10. Microscopic morphologies of high-temperature calcination treated slag micro powder-filled cement mortar (Sample E) after curing at 3 (a), 7 (b), and 28 (d) days, respectively (Figure 10 (d) was related to the EDS spots of Figure 10 (c)).

4. Conclusions

This study systematically evaluated the activation mechanisms of residual slag micro powder derived from recycled waste concrete aggregates using three methods: mechanical grinding, high-temperature calcination, and mechanical grinding–thermal activation. The key findings are as follows:

1. High-temperature calcination emerged as the most effective activation method, achieving a 95.85% activity index by calcining slag micro powder at 750 °C for 10 min. This treatment may release reactive SiO_2 and Al_2O_3 components that participated in volcanic ash reactions, forming hydration products (e.g., C-S-H gel) and significantly improving the compressive strength of cement mortar.
2. Mechanical grinding alone demonstrated limited benefits. While it reduced particle size and increased the specific surface area, thereby promoting partial pozzolanic reactivity, the improvement in compressive strength was marginal, and flexural strength even decreased due to particle agglomeration and stress concentration. However, compared with untreated slag, as long as the cost loss of mechanical grinding is low enough, this method can still be easily used to improve the activity of slag micro powder.
3. The combined mechanical grinding–thermal activation method produced negative synergistic effects, likely due to over-densification of the particle structure or conflicting activation pathways, which reduced the reactivity of the slag micro powder. In

the future, different grinding times and temperatures can be attempted for activation treatment, which may further enhance the activity of slag micro powder.

4. A microstructural analysis via SEM–EDS confirmed that calcined slag micro powder exhibited dense hydration product coverage after 28 days of curing, in contrast to the inert state of untreated slag micro powder. This highlights the critical role of thermal activation in enhancing slag micro powder’s reactivity and cementitious performance.

In conclusion, high-temperature calcination provided a viable route for transforming low-activity residual slag micro powder into a valuable supplementary cementitious material, contributing to sustainable construction practices by reducing waste, conserving natural resources, and lowering the environmental impact of concrete production.

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