Regular Article

Chengfang Yuan, Di Wang*, Haris Setiawan and Yiran Wei

Effect and mechanism of different excitation modes on the activities of the recycled brick micropowder

https://doi.org/10.1515/secm-2021-0062
Received August 15, 2021; accepted November 07, 2021

Abstract: The recycled brick micropowder was formed after crushing and ball milling and waste clay bricks have great potential activity. It can replace some percentage of cement in the preparation of concrete through activation effects of excitation, and thus, reutilization of waste products is the objective of this study. This study analyzed the activation effects of mechanical excitation, chemical excitation, and high-temperature excitation on the activity of regenerated brick powder. The correlation between different excitation methods and the activity index was fitted, and the microstructure and mechanism of action of the material were analyzed and revealed using SEM and XRD techniques. The results show that after using mechanical excitation, chemical excitation, and high-temperature excitation to activate the brick powder, the activity index of the material was improved to varying degrees, and there is a good correlation between different excitation modes and the activity index. The study shows that the 45 min mechanical excitation of ball milling and the high-temperature excitation of 800°C have better effects, with the highest activity index reaching 71%, and the highest activity index in the chemical excitation mode is 65%. Considering the excitation effect, energy consumption, economy, and practical operation feasibility, it is recommended to use the ball milling 45 min excitation method as the best activation method for the recycled brick micropowder.

Keywords: recycled brick powder, activity index, mechanical excitation, chemical excitation, high-temperature excitation

1 Introduction

In recent years, China’s cities and the new rural area construction have developed rapidly, and the construction waste generated by demolition and renovation of buildings has been increasing. In addition, sudden natural disasters such as earthquakes will also bring a lot of construction waste [1,2]. Most of China’s construction waste has been stacked or landfilled, and the comprehensive utilization rate is less than 5%, which is much lower than that European Union, Japan, South Korea, and other developed countries and regions [3]. The extensive treatment method not only occupies a large amount of land, but also brings a series of problems such as dust, dust flying, and alkaline waste slag that make the soil “inactivate,” which seriously damages the ecological environment and affects the social and economic life. With the increase of people’s awareness of saving resources and protecting the environment, research on the utilization of construction waste as a resource continues to increase.

In the past few decades, due to China’s traditional building habits and production technology, sintered clay bricks have been widely produced and used in the construction field, which will lead to waste clay bricks becoming the main construction waste in the next few decades. People continue to study and explore how to recycle waste clay bricks. In some cities, waste clay bricks are crushed and used as fillers and cushions in engineering [4]. Beddu [5] studied the possibility of using waste bricks to replace cement clinker to prepare cement mortar, and found that the 90 days mechanical properties of cement mortar prepared by replacing cement clinker with 10% waste bricks were almost the same as those of...
the benchmark cement mortar. In the process of crushing the construction waste, a large amount of fine powder with a particle size of less than 0.16 mm will be produced, which accounts for about 15% of the total mass of construction waste, namely recycled brick powder [6]. Sintered clay bricks in the construction waste contain a large amount of silicon dioxide (SiO₂) and aluminum oxide (Al₂O₃), as well as a small amount of calcium peroxide (CaO₂). Recycled brick powder prepared from waste clay bricks has potential pozzolanic activity, and the activity increases with the increase of hydration age [7–10]. In the study of Ahmad et al. [11], a comparative test of recycled brick micropowder and mineral materials to asphalt mixtures was applied, and they found that the recycled brick micropowder has better mechanical properties on asphalt mixtures than the limestone powder. Kartini et al. [12] prepared composite cementation materials by partially replacing cement with recycled brick powder and limestone powder, and found that increasing the content of recycled brick powder is beneficial to increase the strength of the material later. Chen [13] found that mechanical properties of concrete decrease by addition of 15% recycled brick powder over replacement of cement. In order to better exert the potential activity of the regenerated brick micropowder, in recent years, researchers have carried out activation effects of excitation on the regenerated brick micropowder to explore the influence of different excitation methods on the activity of the regeneration of brick micropowder. Commonly used excitation methods for regenerating micropowder include mechanical excitation, chemical excitation, and thermal excitation. Xiaoxiao et al. [14] compared and analyzed the effects of different mechanical force grinding methods on the particle size distribution, phase configuration, microscopic morphology, and mechanical properties of the recycled brick micropowder and found that the recycled brick micropowder pulverized by the jet mill has regular particle shapes, good filling effect, and higher activity. Studies [15,16] compared the influence of different fineness of recycled brick powder on the strength of mortar and found that the recycled brick powder with higher fineness obtained by ball milling has stronger activity. Chen [17] replaced certain proportion of cement with recycled brick micropowder in cement mortar in the result optimal mechanical properties of cement mortar was achieved. Dong et al. [18], studied the effects of lime and gypsum as activators on the properties of brick powder foam concrete with different cementitious material systems. The test results show that when the cementing material system contains a cement/brick powder ratio of 70:30, 25% lime or 20% gypsum can be used to obtain the foam concrete with higher strength and lower water absorption. Feng et al. [19] used recycled brick powder instead of cement to study the influence of the brick powder and the activation method on the anti-carbonization performance of concrete. The results show that the addition of 10% recycled brick micropowder can improve the anti-carbonization performance of concrete. The addition of an appropriate amount of sodium metasilicate nonahydrate (Na₂SiO₃·9H₂O) activator or grinding and refining the powder can stimulate the activity of the regenerated fine powder and further improve the anti-carbonization performance of the concrete. Letelier et al. [20] stimulated the heat treatment activity of recycled brick micropowder and studied the effect of the recycled brick powder on the work performance, strength and anti-chloride ion permeability of concrete after being partially replaced by cement. The results showed that the activity of the recycled brick micropowder increased with the increase of temperature. When the replacement rate of the regenerated brick micropowder is 10%, its resistance to chloride ion penetration is enhanced.

Although the three activation treatment technologies of mechanical refinement, heat treatment, and chemical activation have related research reports, there is still a lack of systematic comparative experimental research, and the effect and mechanism of the above methods are also worthy of further exploration. In this article, the three methods – mechanical excitation, chemical excitation, and high-temperature excitation – were used to activate the recycled brick micropowder. The activation effect was evaluated according to the activity index scanning electron microscopy (SEM), and X-ray diffraction (XRD) testing techniques were used to explore the mechanism of action of different excitation methods. This study proposed the optimal excitation method for recycled brick powder. The research results can provide the basic experimental basis and theoretical guidance for the resource utilization of construction waste of brick to recycle in the form of brick micropowder.

2 Experimental setup

2.1 Raw materials

The ordinary Portland cement (P·O42.5) produced by Henan Mengdian Group Cement Co., Ltd. is used in this study. The main technical indicators are shown in Table S1. The
mineral admixture adopts Grade 1 fly ash produced by Henan Hennuo Filter Material Co., Ltd., and silica fume of the Qinghai Yuanheng Brand; the material composition is shown in Table S2. The waste bricks are first crushed with a jaw crusher and then sieved to obtain a particle size of less than 0.16 mm recycled brick powder. The preparation process is shown in Figure 1, and the material composition is shown in Table S2; the aggregate is the standard sand of Xiamen Aisiou Standard Sand Co., Ltd., and the natural medium sand with a fineness modulus of 2.6; steel fiber is a straight steel fiber with a length of 12 mm, a diameter of 0.2 mm, and a tensile strength >2,000 MPa; the water reducing agent adopts the CQJ-JSS polycarboxylic acid superplasticizer produced by Shanghai Chenqi Chemical Technology Co., Ltd., with a water reduction rate of 26.5%; the test water is ordinary tap water that meets the national standards.

### 2.2 Test design

Three excitation methods – mechanical excitation, chemical excitation, and high-temperature excitation – are used. The activity test of the recycled micropowder after excitation is carried out according to ref. [21], and the activity index is taken as the evaluation index and the experimental design is shown in Table 1.

In Table 1, Group “A” is a pure cement mortar group without the recycled brick powder; Group “B” is a mortar group prepared by replacing cement with 30% of the nonexcited recycled brick powder; Groups “C” are mechanically activated groups; SM500 × 500 ball mill is used as shown in Figure S1 to ball mill the recycled brick powder for 15, 30, 45, 60 min, and then replaced the cement with 30% to prepare mortar test pieces, and used the specific surface area to characterize the fineness of the recycled brick micropowder after ball milling; Groups D and E are chemically activated groups. About 2.5–4.5% calcium hydroxide Ca(OH)₂ or 3.5–5.5% sodium metasilicate nonahydrate Na₂SiO₃·9H₂O of the quality of recycled brick powder are added, and then replaced the cement with 30% to prepare mortar specimens; Group F is a high-temperature excitation group. Considering energy consumption, the maximum temperature of the high-temperature excitation group is set to 800°C. The regenerated brick powder is put into the SRJX-4-13 box-type resistance furnace as shown in Figure S2 and heated from 200 to 800°C. It is kept for 2 h and then replaced with cement to prepare the mortar test piece.

### 2.3 Specimen production and maintenance

Each group was made of three 40 mm × 40 mm × 160 mm test blocks, i.e., a total of 60 blocks in 20 groups. The molding method of the test block was according to the specification [22]. First, water is added to the pot and then cement and brick powder are mixed in water. Then, the pot is put on a fixed rack and raised to a fixed position. The mixture is stirred at a low speed for 30 s and standard sand is added evenly at the same time and stirred for another 30 s. The machine is turned to high speed and mixed for another 30 s. Mixing is stopped for 90 s, and a rubber scraper is used to scrape the sand on the blade and pot wall into the middle of the pot within the first 15 s. Stirring

![Figure 1: Preparation process of the recycled brick micropowder.](image-url)
is continued at a high speed for 60 s. The mixed mortar is put evenly into the test mold, and the vibrating table is vibrated for 120 s. The mortar is scraped higher than the test mold with a flat ruler and smoothened. Then, the test piece number is marked on the test mold. It is put in a standard curing box for 24 h and demolded; after demolding, it is put into a standard curing box to cure for 28 days.

### 2.4 Experiment method

The activity index calculation formula [21] is as follows, and the result is retained to 1%:

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

where $H$ is the strength activity index (%); $R$ (=28 days) is the compressive strength of the test mortar (MPa); $R_0$ (=28 days) is the compressive strength of the pure cement mortar (Group A) (MPa).

The compressive and flexural strength tests of the specimens are carried out. The test equipment adopts the YAW-300C cement flexural and compressive integrated testing machine. Before testing for flexural strength, the surface moisture of the test block is wiped, placed on the fixture, adjusted the system parameters, and loaded. The loading rate is $50 \pm 10$ N/s. When the specimen is ruptured, the load value is recorded.

The flexural strength $R_f$ (MPa) is calculated according to formula (2) [21] and the final data is accurate to 0.1 MPa:

$$R_f = \frac{1.5F_L}{b^3},$$

where $F_f$ is the failure load (N); $L$ is the distance between two fulcrums (mm); and $b$ is the side length of the square section of the prism (mm).

The compressive strength test was carried out on the prism body that was broken after the flexural test. The two sides of the specimen were selected as the compression surface, and the compression area was $40 \text{ mm} \times 40 \text{ mm}$. After placing it, adjust the parameters of the control end and start loading, the loading rate is $2,400 \pm 200$ N/s, load uniformly until it fails, and record the load value. The compressive strength $R_c$ (MPa) is calculated according to formula (3) [21], and the final data are accurate to 0.1 MPa:

$$R_c = \frac{F_c}{A},$$

where $F_c$ is the ultimate load (N) and $A$ is the compression area of the test piece ($\text{mm}^2$).
3 Test results and discussion

3.1 The effect of mechanical excitation on the activity of recycled micropowder

Table 2 shows the test results of the activity index of the recycled brick micropowder after mechanical ball milling. Regression analysis was performed on the correlation between ball milling time and activity index, and the relationship curve is shown in Figure 2. It can be seen from Figure 2 that there is a good quadratic function relationship between the milling time and the activity index, and the correlation coefficient is 0.968.

It can be seen from Table 2 that the activity index of the mortar groups (Groups B, C1, C2, C3, and C4) in which cement is replaced by 30% recycled brick powder is significantly lower than that of Group A. With the extension of the ball milling time, the activity index of the recycled brick powder continues to increase. The activity index of Groups C1, C2, C3, and C4 increased by 14.6, 19.3, 22.0, and 21.7%, respectively, compared with Group B. Ball milling effectively stimulated the recycled brick micropowder active. When the ball milling time is 45 min, the activity index of the recycled brick powder reaches the maximum value of group C, and the 28 days flexural strength of the mortar specimen also reaches the maximum value of 6.2 MPa, which reaches 92.5% of group A, and then the activity index does not increase with the extension of the milling time. The activity index of the recycled brick powder after ball milling for 60 min is basically the same as that of 45 min. The optimal ball milling time for the recycled brick micropowder under the conditions of this experimental study is 45 min.

The reasons were analyzed: on the one hand, with the extension of the ball milling time, the particle size of the recycled brick powder continues to decrease. When the particle size of the recycled brick powder is smaller than the pore diameter in the mortar specimen, the pores can be physically filled to make the material microstructure. It is more dense and solid, thereby improving the strength of the mortar specimen. When the ball milling reaches a certain period of time, the particle size of the recycled brick micropowder basically does not change, and the filling effect on the pores cannot be further strengthened.

On the other hand, when the ball milling time is short, the specific surface area of the recycled brick micropowder is smaller, and the recycled brick powder is combined with the cement hydration products. The contact area is also small, and the pozzolanic reaction is relatively weak. The resulting calcium metasilicate $\text{Ca}_2\text{SiO}_4\cdot2\text{H}_2\text{O}$, tricalcium aluminate $\text{Ca}_3\text{Al}_2\text{O}_6\cdot\text{xH}_2\text{O}$ and ettringite are less, and most of the recycled brick powders only play a role of physical filling, which has a limited effect on improving the strength of mortar specimens, so the activity index is low. With the extension of the ball milling time, the particle size of the regenerable brick powder decreases, the specific surface area increases, the contact area with cement hydration products increases, and more $\text{Ca}_2\text{SiO}_4\cdot2\text{H}_2\text{O}$, $\text{Ca}_3\text{Al}_2\text{O}_6\cdot\text{xH}_2\text{O}$ and ettringite can significantly increase the strength of mortar specimens. However, with the extension of the ball milling time, the particle size of the recycled brick powder decreases, the specific surface area increases, and the water absorption will gradually increase, resulting in a decrease in the fluidity of the mortar mixture. The influence of strength is not obvious. When the fluidity is reduced extensivly, the structure of the mortar specimen is not dense and the strength is reduced. Therefore, under
the comprehensive influence of multiple factors, the activity index increases with the extension of the milling time, and finally, remains unchanged.

The micro morphology of each group of mortar samples is shown in Figure 3. It can be seen from the figure that the structure of the samples of Group A is the densest, while the samples of Group B have relatively porous and loose structures. The samples of C1 and C2 groups after ball milling have fewer holes and compact structures. The structures of the C3 and C4 samples are more compact. It can be seen that ball milling reduces the particle size of the recycled brick powder, fully fills the pores of the structure, and makes the structure more compact. The hydration products are mainly Ca(OH)$_2$ crystals and the C–S–H gel. The Ca(OH)$_2$ crystals are distributed in a lamellar shape, and the C–S–H gel is distributed in a network shape. Compared with Group A, it is mixed with recycled micropowder mortar. The Ca(OH)$_2$ crystals in the sample are obviously reduced. After ball milling, the degree of polymerization of the silicon-oxygen tetrahedrons in the recycled micropowder is continuously reduced and converted into monomers, which can react with the cement hydration product Ca(OH)$_2$ to further generate the C–S–H gel.

Figure 3: Microscopic morphology of the mortar specimen for each group.
The XRD test results of each group of mortar samples are shown in Figure 4. It can be seen from the figure that the samples are mainly composed of four crystals of silicon dioxide $\text{SiO}_2$, calcium hydroxide $\text{Ca(OH)}_2$, calcium carbonate $\text{CaCO}_3$, and calcium metasilicate $\text{Ca}_2\text{SiO}_4\cdot2\text{H}_2\text{O}$. The diffraction peaks of $\text{Ca}_3\text{Al}_2\text{O}_6$ crystals are only found.

**Figure 4:** XRD diffraction pattern of mortar samples (Groups A, B, and C1–C4).
in Group B, which may be due to the fact that the recycled micropowder of Group B is not ball milled and contains part of the cement mortar. Ca$_3$Al$_2$O$_6$ crystals may be secondary hydration products. Comparing the test results of the recycled micropowder mortar samples in each group, it is not difficult to find that the diffraction patterns of the mortar samples are not much different at different ball milling times, and there is no obvious difference in the crystals contained.

The diffraction results of Group C3 and Group C4 are very close. The diffraction patterns of Groups B and C1–C4 all have obvious SiO$_2$ diffraction peaks. Among them, Group B has the strongest SiO$_2$ diffraction peak, followed by Group C1, and Group A is the weakest. The Ca(OH)$_2$ diffraction peak is the strongest in Group A, followed by Group B, and Group C3 is the weakest. This is because Group A is not mixed with the recycled micropowder, while the SiO$_2$ in the mortar sample of the ball milling group mainly comes from the sand and recycled micropowder in the aggregate, and the Ca(OH)$_2$ mainly comes from the cement hydration. This shows that after mechanical ball milling, the SiO$_2$ in the partially recycled micropowder is activated, and the pozzolanic reaction occurs with the cement hydration product Ca(OH)$_2$ to form Ca$_2$SiO$_4$·2H$_2$O.

### 3.2 Influence of chemical activator on the activity of recycled micropowder

The test results of the activity index of the recycled micropowder after chemical excitation are shown in Table 3. Regression analysis was performed on the correlation between calcium hydroxide Ca(OH)$_2$, sodium silicate pentahydrate Na$_2$SiO$_3$·9H$_2$O content, and activity index, and the relationship curves are shown in Figure 5. It can be seen from the figure that Ca(OH)$_2$ and the activity index satisfy a good Gaussian function relationship, with a correlation coefficient of 0.999, and the content of Na$_2$SiO$_3$·9H$_2$O satisfies a good cubic function relationship with the activity index, with a correlation coefficient of 0.968.

It can be seen from Table 3 that after 30% of recycled micropowder replaces cement, the material activity index is significantly lower than that of Group A, with a decrease of 52–65%, indicating that the activity of the recycled micropowder is lower than that of cement. After adding a suitable amount of the stimulant Ca(OH)$_2$ or Na$_2$SiO$_3$·9H$_2$O, the activity index of the recycled micropowder increased, and the activity index of Groups D1–D5 increased by 7.9, 10.6, 5.9, 4.7, and 4.3%, respectively; compared to Group B, indicating that the findings are consistent with the results reported by Lu Jing et al. [23].

The activity index of Groups E1–E3 increased by 3.5, 9.8, and 1.6%, respectively, compared with Group B, indicating that the stimulant effectively stimulated the activity of the recycled micropowder. However, the activity index of Groups E4 and E5 decreased by 4.3 and 11.0%, respectively, compared to Group B, indicating that adding excessive Na$_2$SiO$_3$·9H$_2$O would cause the activity of the recycled micropowder to decrease. When 3.0% Ca(OH)$_2$ was added, the activity index of the recycled micropowder reached the maximum value of group D, and the 28 days flexural strength of mortar specimens reached 8.1 MPa, which was 20.9 and 76.1% higher than that of group A and group B, respectively. Then, the activity index of the recycled micropowder decreased with the increase of the Ca(OH)$_2$

### Table 3: Influence of chemical stimulants on the activity index

<table>
<thead>
<tr>
<th>Group</th>
<th>Stimulant</th>
<th>Activator dosage (%)</th>
<th>28 days flexural strength (MPa)</th>
<th>28 days compressive strength (MPa)</th>
<th>Activity index (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>—</td>
<td>—</td>
<td>6.7</td>
<td>43.4</td>
<td>100</td>
</tr>
<tr>
<td>B</td>
<td>—</td>
<td>—</td>
<td>4.6</td>
<td>25.4</td>
<td>59</td>
</tr>
<tr>
<td>D1</td>
<td>Ca(OH)$_2$</td>
<td>2.5</td>
<td>6.0</td>
<td>27.4</td>
<td>63</td>
</tr>
<tr>
<td>D2</td>
<td>Ca(OH)$_2$</td>
<td>3.0</td>
<td>8.1</td>
<td>28.1</td>
<td>65</td>
</tr>
<tr>
<td>D3</td>
<td>Ca(OH)$_2$</td>
<td>3.5</td>
<td>7.7</td>
<td>26.9</td>
<td>62</td>
</tr>
<tr>
<td>D4</td>
<td>Ca(OH)$_2$</td>
<td>4.0</td>
<td>6.3</td>
<td>26.6</td>
<td>61</td>
</tr>
<tr>
<td>D5</td>
<td>Ca(OH)$_2$</td>
<td>4.5</td>
<td>5.4</td>
<td>26.5</td>
<td>61</td>
</tr>
<tr>
<td>E1</td>
<td>Na$_2$SiO$_3$·9H$_2$O</td>
<td>3.5</td>
<td>5.5</td>
<td>26.3</td>
<td>61</td>
</tr>
<tr>
<td>E2</td>
<td>Na$_2$SiO$_3$·9H$_2$O</td>
<td>4.0</td>
<td>8.3</td>
<td>27.9</td>
<td>64</td>
</tr>
<tr>
<td>E3</td>
<td>Na$_2$SiO$_3$·9H$_2$O</td>
<td>4.5</td>
<td>7.0</td>
<td>25.8</td>
<td>60</td>
</tr>
<tr>
<td>E4</td>
<td>Na$_2$SiO$_3$·9H$_2$O</td>
<td>5.0</td>
<td>6.8</td>
<td>24.3</td>
<td>56</td>
</tr>
<tr>
<td>E5</td>
<td>Na$_2$SiO3·9H2O</td>
<td>5.5</td>
<td>5.0</td>
<td>22.6</td>
<td>52</td>
</tr>
</tbody>
</table>
content. When 4.0% Na$_2$SiO$_3$·9H$_2$O was added, the activity index of the recycled micropowder reached the maximum of Group E. At this time, the 28 days flexural strength of the mortar specimen reached 8.3 MPa, which was 23.9 and 80.4% higher than that of group A and group B, respectively. Then, the activity index of the recycled micropowder decreased with the increase of the Na$_2$SiO$_3$·9H$_2$O content. The best chemical stimulant for recycled micropowder and Ca(OH)$_2$ with a content of 3.0% under the experimental conditions in this article.

On analyzing the reason, it was found that the addition of an alkaline stimulant can increase the liquid phase alkalinity of the material and the liquid phase pH value was maintained in a suitable range. The increase of liquid phase alkalinity will promote the breakage of the chemical bonds of SiO$_2$ and Al$_2$O$_3$ on the surface of the recycled micropowder, and it will be easier to react with active ingredients, such as pozzolan reaction with the hydration product Ca(OH)$_2$ of cement to increase Ca$_2$SiO$_4$·2H$_2$O, Ca$_3$Al$_2$O$_6$·xH$_2$O, and ettringite and other hydration products; thereby ultimately improving the early hydration reaction of the gelling material speed to achieve the purpose of stimulating the activity of the recycled micropowder. Although the degree of rupture of the SiO$_2$ and Al$_2$O$_3$ chemical bonds is directly related to alkalinity, excessive strong alkali may cause the active ingredients in the aggregate (such as SiO$_2$) to react chemically and form alkali-silicate gel on the surface of the aggregate, namely alkali-aggregate reaction. The volume of the alkali-silicic acid gel after water absorption is much larger than the volume of the solid before the reaction.

A large amount of alkali-silicic acid gel accumulates and swells in the aggregate interface area, resulting in uneven expansion inside the mortar sample and the decrease of the internal structure compactness, which affects the strength of the mortar specimen. Therefore, the amount of the stimulating agent should be controlled when using alkaline excitation. When Na$_2$SiO$_3$·9H$_2$O is used as an activator, Na$_2$SiO$_3$·9H$_2$O acts as a skeleton network in the material system, and the Ca$_3$SiO$_4$·2H$_2$O and Ca$_3$Al$_2$O$_6$·xH$_2$O gels formed by the hydration of the recycled micropowder play an inlay and the filling effect makes the material system denser in the microscopic view, and the strength of the mortar specimen is improved in the macroscopic view. However, excessive Na$_2$SiO$_3$·9H$_2$O may reduce the contact area between the recycled powder and the cement clinker, thereby affecting the hydration reaction and ultimately reducing the strength of the mortar.

The micromorphologies of D2 and E2 samples are shown in Figure 6. It can be seen from the figure that ettringite is formed in Groups B, D2, and E2, and there are a lot of holes and loose structures in the test of Group B. The samples of Group E2 that were excited by the chemical activator had fewer holes, and the microstructure was denser than that of Group B. The microstructure of the samples in Group D2 is denser than that in Group E2, indicating that the activation effect of the recycled micropowder in Group D2 is the best in the chemical excitation group.

The XRD diffraction patterns of the Group D2 and E2 samples are shown in Figure 7. It can be seen from the figure that the samples are mainly composed of four
crystals: SiO$_2$, Ca(OH)$_2$, CaCO$_3$, and Ca$_2$SiO$_4$·2H$_2$O. Combined with the XRD analysis chart of Group B in Figure 3, it can be seen that the diffraction peak of Ca(OH)$_2$ in Group D2 is the strongest, Group B is the second, and Group E2 is the weakest. The SiO$_2$ diffraction peaks in Group B are the strongest, and the SiO$_2$ diffraction peaks in Group D2 and Group E2 are almost the same. This is because the mortar specimens of Group D2 are mixed with Ca(OH)$_2$ from the outside, and the Ca(OH)$_2$ of the other groups of specimens mainly comes from cement hydration, and the SiO$_2$ mainly comes from the sand in the aggregate and the recycled brick micro-powder. It shows that after chemical activation, the SiO$_2$ in part of the recycled micropowder is activated, and the pozzolanic reaction occurs with the cement hydration product Ca(OH)$_2$ to form Ca$_2$SiO$_4$·2H$_2$O.

3.3 Effect of high temperature on the activity of the recycled micropowder

Table 4 shows the test results of the activity index of the recycled micropowder after high-temperature excitation. Regression analysis was performed on the correlation between temperature and the activity index, and the relationship curve is shown in Figure 8. It can be seen from the figure that the temperature and the activity index satisfy a good quadratic function relationship, and the correlation coefficient is 0.982.

It can be seen from Table 4 that the activity index of the mortar groups (Groups B, F1, and F2) that replaced cement with 30% recycled brick powder was significantly lower than that of Group A, which were 59, 67, and 71% of Group A, respectively. As the temperature increases, the
activity index of the recycled micropowder increased. The activity index of Groups F1 and F2 increased by 18.1 and 20.9%, respectively, compared to Group B. When the temperature is 800°C, the activity index of the recycled micropowder reaches the maximum, and the 28 days flexural strength of the mortar specimen reaches 7.7 MPa, which is 14.9 and 67.4% higher than that of group A and group B, respectively. Therefore, the optimal high-temperature excitation temperature of the recycled micropowder under the experimental conditions in this article is 800°C. This is because high temperature can decompose the original hydration products in the recycled micropowder, and “reduce” them to the original material with higher activity. Especially when the temperature reaches 800°C, the unstable hydration products are completely decomposed. Studies have pointed out [24] that high temperature also increases the content of oxides such as SiO₂, CaO, Fe₂O₃, and Al₂O₃ in the recycled micropowder, which are also the main components of a cement.

The micromorphologies of the F1 and F2 samples are shown in Figure 9. It can be seen from the figure that after high-temperature excitation, there are a small number of holes and microcracks in the mortar specimen, but there are interlaced net-like and needle-like hydration products in the holes, and its microstructure is relatively dense. High-temperature excitation can break the chemical bonds of active components such as SiO₂ and Al₂O₃ in the recycled micropowder, and cause a part of the recycled micropowder that is not originally involved in hydration to react and generate a large amount of the network C–S–H gel. As the temperature increases, the active components in the recycled micropowder are further stimulated, and more C–S–H gel is also generated. After the high temperature is reached, the original hydration products in the recycled micropowder are decomposed but the newly generated hydration products effectively fill the holes and microcracks, making the microstructure of the mortar sample compact and improving the compressive strength of the mortar specimen.

The XRD diffraction patterns of F1 and F2 samples are shown in Figure 10. It can be seen from the figure that the

<table>
<thead>
<tr>
<th>Group</th>
<th>Temperature (°C)</th>
<th>28 days flexural strength (MPa)</th>
<th>28 days compressive strength (MPa)</th>
<th>Activity index (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>A</td>
<td>—</td>
<td>6.7</td>
<td>43.4</td>
<td>100</td>
</tr>
<tr>
<td>B</td>
<td>—</td>
<td>4.6</td>
<td>25.4</td>
<td>59</td>
</tr>
<tr>
<td>F1</td>
<td>200</td>
<td>5.1</td>
<td>26.7</td>
<td>62</td>
</tr>
<tr>
<td>F2</td>
<td>400</td>
<td>5.9</td>
<td>28.0</td>
<td>65</td>
</tr>
<tr>
<td>F3</td>
<td>600</td>
<td>6.6</td>
<td>30.0</td>
<td>67</td>
</tr>
<tr>
<td>F4</td>
<td>800</td>
<td>7.7</td>
<td>30.7</td>
<td>71</td>
</tr>
</tbody>
</table>

Table 4: Effect of high temperature on the activity index of the recycled micropowder

Figure 8: The fitting curve of the correlation between temperature and the activity index.

Figure 9: SEM images of Groups F1 and F2.
material is mainly composed of four crystals: SiO₂, Ca(OH)₂, CaCO₃, and CaₓSiO₄·2H₂O. Combined with the XRD analysis chart of Group B in Figure 3 (right panel), it can be seen that the diffraction peaks of Ca(OH)₂ and SiO₂ in Group B are the strongest, followed by Group F₁, and Group F₂ the weakest. In the high-temperature mortar specimens, Ca(OH)₂ mainly comes from cement hydration, and SiO₂ mainly comes from the sand in aggregate and recycled fine powder. It shows that after high-temperature excitation, the SiO₂ in part of the recycled micropowder is activated, and the pozzolanic reaction occurs with Ca(OH)₂, the hydration product of cement to form CaₓSiO₄·2H₂O. The fact that the CaₓSiO₄·2H₂O peak in Figure 4 (right) is higher than the CaₓSiO₄·2H₂O peak in Figure 4 (left) can also illustrate this point.

4 Conclusion

Three kinds of excitation methods, mechanical excitation, chemical excitation, and high-temperature excitation were used to activate the regenerated brick powder. Through the cement mortar strength test, the law of the influence of different excitation methods on the activity of the regenerated powder was grasped. Using material microscopic testing technology, the mechanism of the effect of the excitation method on the activity of the regenerated micropowder was explored. The main conclusions of the research are as follows: after mechanical excitation, the activity index of recycled micropowder increased first and then remained flat with the increase of the ball milling time, which was greater than that of the unexcited recycled micropowder group (Group B), but all were lower than the pure cement mortar group (Group A). When the ball milling time was 45 min, the activity index of the recycled micropowder reaches a maximum value of 71% in this group.

There is a good quadratic function correlation between the milling time and the activity index of the recycled micropowder, and the correlation coefficient is 0.968. After using chemical excitation, the activity index of the recycled micropowder showed a trend of first increasing and then decreasing with the increase of Ca(OH)₂ and Na₂SiO₃·9H₂O content, which were all smaller than that in Group A. It is worth noting that adding too much Na₂SiO₃·9H₂O (5.0–5.5%) will reduce the activity index of the recycled micropowder, even lower than that of Group B, while the activity indexes of other groups were greater than that in Group B. The excitation effect of Na₂SiO₃·9H₂O was slightly worse than that of Ca(OH)₂. When 3.0% of Ca(OH)₂ was added, the activity index of the recycled powder reaches a maximum of 65% in this group. The contents of Ca(OH)₂ and Na₂SiO₃·9H₂O, and the activity index of the regenerated powder have a good correlation between the Gaussian function and cubic function, and the correlation coefficients are 0.999 and 0.968, respectively. By using high-temperature excitation, the activity index of the recycled micropowder showed an upward trend with the increase of high temperature, which was all higher than that in Group B, but all lower than that in Group A. When the high temperature was 800°C, the activity index of the recycled micropowder reaches 71% of the maximum value of this group. There is a good quadratic function correlation between the temperature and the activity index of the regenerated micropowder, and the
correlation coefficient is up to 0.982. According to the analysis of test results of each group, the high-temperature excitation effect at 800°C and the mechanical excitation effect at 45 min of ball milling are the best. Considering the excitation effect, energy consumption, economy, and practical operation difficulty in the project, the ball milling 45 min excitation method can be preferred.

Conflict of interest: There is no conflict of interest among the authors.

References


