Effect of Physical Excitation on the Properties of Recycled Fine Powder

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Abstract. Using the ball mill to grind the recycled fine powder at different times to study the effect of physical excitation on the properties of the recycled fine powder. The results show that the use of physical excitation can make the particle size of the recycled fine powder continuously decrease, and the particle size distribution tends to be reasonable. With the prolongation of the grinding time, the particle content of <20 µm increases continuously, especially the content of particles with <4 µm increases greatly. The mortars reclaimed with 30 minutes of grinding time have the strongest compressive strength and flexural strength at various ages at various substitution rates, indicating that the effective physical excitation of recycled fine powders can improve their activity.

1. Introduction
In recent years, the technology of crushing, cleaning and grading construction waste that has been dismantled and destroyed in natural disasters, and reused as concrete aggregate (recycled aggregate) to prepare new concrete materials (recycled concrete) has caused domestic and overseas external attention[1-2]. In the process of crushing and grinding of recycled aggregates, fine powder with a particle size of ~0.16 mm, which is about 5% to 10% of the total weight, will be produced, that is, recycled fine powder. Recycled fine powder has certain activity. If it is not recycled, it will cause serious environmental pollution and also cause a lot of waste of resources. This does not meet the requirements for full recycling of green concrete and waste concrete. At present, the recycled fine powder is more used as a mineral admixture instead of cement added to the mortar or concrete[3-7], but due to the low activity of the recycled fine powder, the strength of the mortar or concrete will be affected after the incorporation, the application of recycled fine powder is largely limited. Therefore, it is necessary to conduct research on the performance activation of recycled fine powder, further stimulate its activity and increase its utilization efficiency.

At present, there are many method to activate the performance of recycled fine powder such as physical excitation, chemical excitation, heat treatment, etc. The physical excitation refers to the use of mechanical force to stimulate the activity of the recycled fine powder by grinding. The stable crystalline α-SiO₂ in the powder first transforms into the more stable β-SiO₂ under mechanical force, and finally transforms into amorphous SiO₂ [8]. At the same time, under the action of mechanical force, the original regular hydration products of Ca(OH)₂ and C-S-H crystals in hydraulic cement paste are constantly distorted and eventually become amorphous substances. Therefore, the mechanical force makes the activity of SiO₂ and hydraulic cement paste increase, and the activity of recycled fine powder increases.
For this reason, the ball mill was used to reground the recycled fine powder for 0 min, 10 min, 20 min, and 30 min to stimulate the activity of the recycled fine powder. The particle size distribution, phase composition, and microstructure of the recycled fine powder under physical excitation were studied. The mortar was prepared with different rates of recycle fine powder by grinding at different times, and the effects of physical excitation on the strength and microstructure of the recycled mortar were examined.

2. Materials and methods

2.1. Materials

Cement: P•O 42.5 ordinary Portland cement in Qinghai Datong Cement Plant, its chemical composition is shown in Table 1;

Standard sand: ISO standard sand produced by Xiamen Aishou Standard Sand Co., Ltd.;

Recycled fine powder: The fine particles with a particle size of less than 0.15 mm are collected during the process of crushing and recycling the construction waste. Its chemical composition and performance indicators are shown in Table 2.

2.2. Testing methods

2.2.1. Physical excitation of the recycled fine powder. The recycled fine powder was pulverized in a PM2L planetary ball mill for 0 min, 10 min, 20 min, and 30 min, respectively, to obtain four fine powders of different fineness, labeled as M0, M1, M2, and M3, and passed through a laser particle size analyzer, XRD, and SEM. The particle size distribution, phase composition and microstructure of the regenerated fine powder were characterized.

2.2.2. Mortar strength test. The recycled fine powder (RFP) obtained by the above different excitation methods was used as a cementitious material to replace the cement in proportions of 0%, 10%, 20%, 30%, and 40%. According to the national standard GB/T 17671-1999, the cement mortar sand sample was prepared as water-binder ratio was 0.5 and the lime-sand ratio was 1:3. After curing in water for 3 days, 7 days and 28 days, the compressive strength and flexural strength were measured, and the internal structure of the gel after hardening was observed. Mortar mix ratio is shown in Table 3.

Table 1. Cement chemical composition (%)

| chemical composition | SiO₂ | CaO | Al₂O₃ | MgO | Fe₂O₃ | SO₃ |
|----------------------|------|-----|-------|-----|-------|-----|
| content              | 19.8 | 40.4| 7.67  | 2.06| 2.66  | 2.31|

Table 2. Recycled fine powder chemical composition (%)

| chemical composition | SiO₂ | CaO | Al₂O₃ | MgO | Fe₂O₃ | SO₃ |
|----------------------|------|-----|-------|-----|-------|-----|
| content              | 33.3 | 27.4| 8.1   | 2.66| 3.61  | 1.19|

Table 3. Mortar mix ratio

| No. | cement(g) | RFP replacement rate(%) | M0(g) | M1(g) | M2(g) | M3(g) | sand(g) |
|-----|-----------|-------------------------|-------|-------|-------|-------|---------|
| S1  | 405       | 10%                     | 45    | 0     | 0     | 0     | 1350    |
| S2  | 405       | 10%                     | 0     | 45    | 0     | 0     | 1350    |
| S3  | 405       | 10%                     | 0     | 0     | 45    | 0     | 1350    |
| S4  | 405       | 10%                     | 0     | 0     | 0     | 45    | 1350    |
| S5  | 360       | 20%                     | 90    | 0     | 0     | 0     | 1350    |
| S6  | 360       | 20%                     | 0     | 90    | 0     | 0     | 1350    |
| S7  | 360       | 20%                     | 0     | 0     | 90    | 0     | 1350    |
2.2.3. Cement gel microstructure test. The microstructure and hydrated state of hydration products of the cement gel that was hydrated up to 28 days were tested by JSM-5610LV/INCA scanning electron microscope. The cement mortar specimens that were hydrated to 28 days were crushed and sampled. After sampling and before scanning electron microscope observations, the sample was soaked with absolute alcohol to stop hydration of the cement.

3. Results and discussion

3.1. Particle size distribution of recycled fine powder

![Figure 1. Particle size distribution of recycled fine powder](image)

Figure 1 shows the particle size distribution of recycled fine powders pulverized by vibrating ball mill for 0 minutes, 10 minutes, 20 minutes, and 30 minutes. As can be seen from the figure, the non-grinding recycled fine powder M0 has a non-uniform particle size distribution with <10 μm particles are less. With the increase of ball milling time, the particle size of the recycled fine powder decreases, and the particle size distribution continuously narrows. The peak of the particle size distribution shifts to the left as the milling time increases, and the distribution curve tends to be in a normal distribution. It can be seen from Table 4 that with the prolongation of the grinding time, the particle content of <20 μm increases continuously, especially the content of particles of <4 μm increases greatly. However, the difference in particle distribution between the grinding time of 20 min and 30 min is not significant, indicating that the grinding efficiency is not high when the grinding time exceeds 20 min. After more than 20 min, the content of fine particles (<10 μm) decreased due to agglomeration.

| Milling time | <1μm | <2μm | <4μm | <10μm | <20μm | <64μm | <80μm | <100μm | <200μm |
|--------------|------|------|------|-------|-------|-------|-------|--------|--------|
| M0           | 2.36 | 3.84 | 6.32 | 12.68 | 21    | 47.05 | 55.74 | 65.07  | 94.79  |
| M1           | 5.22 | 8.38 | 13.55| 25.33 | 38.18 | 69.35 | 77.66 | 85.59  | 96.57  |
| M2           | 9.12 | 14.65| 22.67| 38.15 | 50.77 | 82.36 | 89.73 | 94.15  | 99.95  |
| M3           | 8.98 | 15.47| 23.95| 35.02 | 51.61 | 78.64 | 85.12 | 90.71  | 99.96  |
3.2. Phase composition of recycled fine powder

![XRD patterns of recycled fine powder](image)

Figure 2. Effect of physical excitation on phase composition of recycled fine powder

Figure 2 (a) shows the XRD pattern of the non-grinding recycled fine powder. The figure shows that the recycled fine powder is mainly composed of calcite (CaCO₃), quartz (α-SiO₂), dolomite [CaMg(CO₃)₂], and a small amount of cement gel such as cement hydration product C₂S. The partial crystallization peak of the recycled fine powder after the ball mill grinding for 10 min is lower than that of the untreated recycled fine powder. For example, the crystallization peak of SiO₂ around 35° is obviously weakened, which is due to the mechanical force of SiO₂ (α-SiO₂), the tetrahedral structure has been distorted and changed into an amorphous state. The amorphousness of SiO₂ has increased the activity of recycled fine powder. The crystallization peak of Ca(OH)₂ at 30° is obviously weakened, and Ca(OH)₂ is exposed to the air. The reaction with CO₂ leads to the increase of the crystallization peak of CaCO₃. CaCO₃ can promotes the hydration of the tricalcium silicate ((3CaO·SiO₂, C₃S) and the tricalcium aluminate (3CaO·Al₂O₃, C₃A), thus promotes the hydration degree of the cementitious material and thus promotes the strength development of the mortar[9]. Some of the crystallization peaks are intensified, such as about 28° in dicalcium silicate (2CaO·SiO₂, simplified C₂S) and about 30°CaCO₃. This is due to the fact that mechanical grinding causes the C₂S and Ca(OH)₂ to be entrapped from the hydrated components. The release of C₂S and Ca(OH)₂ increased the crystallization peak of C₂S. C₂S was the main component of the hydration reaction at the later stage of the cementitious material. Adding it into the mortar played an important role in the later development of the strength.

With the continuous increase of milling time, the phase composition of the recycled fine powder does not change much, and only part of the SiO₂ crystallization peaks will change. For example, after grinding for 20 minutes, C₂S of about 33° and 42° appears, indicating that machinery force grinding results in the further release of the unhydrated component C₂S contained in the recycled fine powder.

3.3. Microstructure of recycled fine powder

![SEM images of recycled fine powder](image)

Figure 3. Effect of physical excitation on microstructure of recycled fine powder

Figure 3 is an SEM image of the recycled fine powder after activated for 0 min, 10 min, 20 min, and 30 min. As can be seen from Figure 3 (a), the non-grinding recycled fine powder has a non-uniform particle size distribution, a large number of coarse particles, and many polygonal shapes, and many fine particles adhere to the surface. With the increase of the grinding time, the thicker and more granular particles were significantly reduced, and the particle size distribution gradually became
uniform. However, when the powder was ground for 30 minutes, the number of large particles adhered with fine particles on the surface was increased, indicating that the agglomeration of fine particles began to be apparent, which was consistent with the results of particle size distribution.

3.4. Strength of recycled mortar

From Figure 4 to Figure 7, it can be seen that the mortar with recycled fine powder grinded for 30 minutes has the strongest compressive strength and flexural strength at all ages at various substitution rates, demonstrating that the physical excitation can improve the activity of recycled fine powder. It can be seen from Figure 4 that when the amount of recycled fine powder is 10%, the compressive strength of the mortar shows a gradually increasing trend as the milling time of the fine powder increases, and the bending strength basically conforms to this rule in the early stage. By 28 days, the mortar strength of grinding for 10 minutes exceeded the mortar strength of grinding for 20 minutes.

![Figure 4. Mortar strength change with the content of recycled fine powder is 10%](image1)

From Figure 5 and Figure 6, it can be seen that when the content of recycled fine powder is 20% and 30%, the mortar of grinding for 10 minutes is very close to the mortar of grinding for 20 minutes, and even its 28-day bending strength exceeds the latter.

![Figure 5. Mortar strength change with the content of recycled fine powder is 20%](image2)

![Figure 6. Mortar strength change with the content of recycled fine powder is 30%](image3)
From Figure 7, it can be seen that when the content of recycled fine powder is 40%, the mortar strengths of grinding for 20 minutes and grinding for 30 minutes are higher than those for mortar that has not been ground or ground for 10 minutes, which means that as the amount of regenerated fine powder increases, the physical strength increases. The advantages of physical excitation can be better played.

**Figure 7.** Mortar strength change with the content of recycled fine powder is 40%

3.5. Microstructure of recycled mortar

It can be seen from Figure 8 that at the time of hydration for 3 days, there are more pores in the recycled mortar, and the cracks are wider and the surrounding hydration products are less. This is because the surface of the recycled fine powder is rough, irregular in geometry, poor in particle size, and there are a large number of connected pores in the interior, resulting in a large water absorption of the recycled fine powder. This will have an adverse effect on the early hydration of the surrounding cement, making the hydration rate slow, the degree of hydration reduced, and the amount of hydration products reduced. The structure of the recycled mortar in Figure 8 (b) is more compact than Figure 8 (a). This is because the recycled mortar in this group was pulverized for 30 min. The particles are finer and the particle size distribution tends to be relatively reasonable. The good filling in the internal pores of the recycled mortar improves the compactness of the mortar, thereby increasing the strength of the mortar.

From Figure 8 (c) and Figure 8 (d), it can be seen that with the growth of curing age, there are a large number of hydrous calcium silicate gels, Ca(OH)₂ and calcium aluminate crystals, etc. The hydration products are produced. They are closely packed and filled in the hydraulic cement paste to refine the pores in the mortar. The internal structure of the entire recycled mortar is gradually dense, showing that the layered, plate-like and flocular structures are overlapped and tightly combined. Macroscopically, the strength of recycled mortar is continuously increasing.

**Figure 8.** Effect of physical excitation on microstructure of recycled mortar

4. Conclusions

(1) The use of physical excitation can make the particle size of the recycled fine powder continuously decrease, and the particle size distribution tends to be reasonable. With the prolongation of the grinding time, the particle content of <20μm increases continuously, especially the content of particles with <4μm increases greatly. However, the difference in particle distribution between the grinding
time of 20 min and 30 min is not significant, indicating that the grinding efficiency is not high when the grinding time exceeds 20 min.

(2) For mortars with recycled fine powder grinded for 30 minutes has the strongest compressive strength and flexural strength at all ages at various substitution rates, indicating that the effective physical excitation of recycled fine powder can improve its activity.

(3) After grinding for 30 minutes, the fine particles are finer and the particle size distribution is relatively reasonable, which can be better filled in the internal pores of the recycled mortar, and the compactness of the mortar can be increased, thereby increasing the strength of the mortar.

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References
[1] Jianzhuang Xiao. Recycled concrete[M]. Beijing: China Building Industry Press, 2008.
[2] D. Gastaldi, F. Canonico, L. Capelli, L. Buzzi, et al. An investigation on the recycling of hydrated cement from concretedemolition waste[J]. Cement & Concrete Composites, 2016, 61: 29–35.
[3] Xinqi Mao, Wenjun Qi, Peng Zhu. Current Research Status of Construction Waste ReclaimedMicropowder[J]. CHINA Concrete and Cemnet Products , 2015, (8): 89–92
[4] Feng Tai, Geng Ou, Zhao Guiyun. Experimental research on carbonation-resistance property of recycled powder concrete[J]. Industrial Construction, 2015, 45(11): 126–129.
[5] Xiuqin Zhang, Quyi Li, Gongbing Yue, et al. Effect of regeneration powder on cement mortar performance[J]. Concrete, 2015, (8): 120–126.
[6] Shengcai Zhang, Ou Geng, Guiyun Zhao. Compressive strength and active provocation of recycled micro-powder concrete[J]. Concrete, 2015, (11): 49–52.
[7] Ricardo Serpell, Mauricio Lopez. Properties of mortars produced with reactivated cementitious materials[J]. Cement & Concrete Composites, 2015, 64: 16–26.
[8] Xiao-xiao Yu, Ru-yan Li, Xiang Dong, et al. Effect of mechanical force grinding on the properties of recycled powder[J]. Journal of Artificial Crystals, 2017, 46(4): 688–692.
[9] Xiao-xiao Yu. Study on activation and function of recycled powder of waste concrete[D]. Kunming University of Science and Technology, 2017.