Experimental study on chemical activation of recycled powder as a cementitious material in mine paste backfilling

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ABSTRACT
To improve the utilization rate of construction waste as mine backfilling materials, this paper investigated the feasibility of using recycled powder as mine paste backfilling cementitious material, and studied the pozzolanic activity of recycled construction waste powder. In this study, alkali-calcium-sulfur served as the activation principle and an orthogonal test plan was performed to analyze the impact of the early strength agent, quick lime, and gypsum on the pozzolanic activity of the recycled powder. Our results indicated that in descending order, early strength agent > quick lime > gypsum affected the strength of the backfilling paste with recycled powder as a cementitious material during early phases. The strength during late phases was affected by, in descending order, quick lime > gypsum > early strength agent. Using setting time and early compressive strength as an analysis index as well as an extreme difference analysis, it was found that the optimal ratio of recycled powder cementitious material for mine paste backfilling was recycled powder:quick lime:grdenearly strength agent at 78%:10%:8%. X-ray diffraction analysis and scanning electron microscope were used to show that the hydration products of recycled powder cementitious material at the initial stages were mainly CH and ettringite. As hydration time increased, more and more recycled powder was activated. It mainly became calcium silicate hydrate, calcium aluminate hydrate, etc. In summary, recycled powder exhibited potential pozzolanic activities. When activated, it could replace cementitious materials to be used in mine backfill.

Keywords: Chemical activation, Mine paste backfill, Pozzolanic activity, Recycled powder

1. Introduction

As China urbanizes, new construction areas reach up to 2.0 billion m², which is close to half of the global annual construction area [1, 2]. Large scale construction produces a great deal of construction waste. Its storage and backfill consumes significant soil resources. This also brings severe pollution of soil, air and water. Construction waste reuse is thus an urgent research topic.

In the mining and engineering fields, mining subsidence problems must be solved for safe and high efficient mining of China's coal. Technology that makes use of construction waste as a coal mine paste backfilling material is being studied and applied [3-5]. During crushing the construction waste and sieving backfilling aggregate will produce about 10% recycled powder with particle sizes below 0.16 mm. This recycled powder must be removed by sieving or it will affect the quality of the recycled aggregate [6]. These recycled powders can also result in secondary pollution.

Through chemical analysis, the main constituents of the recycled powder were found to be SiO₂, CaO, Al₂O₃ and other active substances; therefore, exploring how to effectively stimulate the cementitious activation of recycled powder so as to meet the requirement of mine on backfilling material has significance in replacing conventional high cost backfilling cementitious materials.

Recycled powder from construction waste is under intense study in terms of its microscopic material properties [7]. Liu et al. [8] utilized scanning electron microscope (SEM), atomic force microscopy and other tools to investigate the microscopic chemistry compositions of mixed powder in construction waste. Wang et al. [9] analyzed the chemical composition and tested the activation strength of recycled powder using SEM, diffractometry and other instruments. Turanli et al. [10] investigated the pozzolanic activity of recycled brick powders and the properties of reduced alkali aggregate reactions. Chen et al. [11] studied the mechanical property assessment of an asphalt mixture using recycled brick powder as
admixture. Yong et al. [12] investigated the Blaine fineness, particle size and other physical properties.

In terms of activation of recycled powder, the current mechanical activation method increases its activity by improving the fineness of waste concrete garbage powder. Wu et al. [13] investigated prepared recycled powder admixtures by grinding waste concrete. They then analyzed the impact of different grinding times on the fineness, specific surface area, density, particle distribution and other physical properties as well as the final activity.

Chemical activation accelerates the hydration and hardening of recycled concrete waste powder concrete by adding various chemical activators. The common activators include sulfate, carbonate, plaster, lime, etc. [14-16]. Yan et al. [17] investigated recycled powder from concrete modified with Na$_2$SiO$_3$ solution, hydrochloric acid, and water boiling. Jiang [18] studied the mechanical property, contract property and chemical resistance property of slag cementitious materials under alkali activation.

The use of recycled powder alternative for cement, plaster and other construction materials not only save cost, but also decrease environmental pollution and recycle waste. Yong et al. [12] conducted experiments and concluded that when the content of the waste powder concrete was less than 15%, then the recycled powder could effectively substitute ordinary Portland cement (OPC). Chen et al. [19] used experiments to show that as more recycled powder was substituted into the cement, the mortar usage increased, but the compressive strength decreased. When the mixture content of recycled powder was 14%, the energy efficiency coefficient reached a maximum value. Ge et al. [20] investigated the impact of the mechanical properties (compressive strength, elastic modulus and flexural strength) of recycled brick powder on concrete. Their study indicated that recycled brick powder could substitute for cement in concrete. Zhu et al. [21] validated showed that as the powder mixture ratio increased, the concrete strength decreased. Li and Lv et al. [22, 23] investigated the impact of the admixture content of recycled powder on the compressive strength and workability of various grades of concrete. They observed the mechanical property, durability and volume stability of concrete prepared with recycled powder admixture and justified that recycled powder from construction waste could be used as a mineral admixture to prepare various common grades of concrete. Gutiérrez-González et al. [24] studied waste polyester powder to prepare light gypsum material, and their experiments indicated that as the polyester powder increased, the density and mechanical properties of the gypsum decreased while improving its permeability and heat resistance.

In summary, research on recycled powder from construction waste is mostly focused on the preparation of concrete in the construction industry; however, the current requirements of mining paste backfilling on cementitious material differ significantly from those in the construction field. The high efficiency of coal mine production implied that the strength of paste backfilling materials must reach 0.1 MPa within 8 to 10 h and above 3.0 MPa in 28 d. The initial setting time of the filler must quickly control roof settling [25]. All of these requirements differ from those of concrete preparation on cementitious material. Thus, this study first analyzed the physicochemical properties of the recycled powder, and then investigated the pozzolanic activity of recycled powder with various activators and additive amounts under condition compliance with backfilling of coal mine paste. This explored the possibility of 100% reuse of construction waste in mine backfilling.

2. Physicochemical Properties Analysis of Recycled Powder

After processing with a jaw crusher and the removal of coarse and fine aggregates with particle sizes over 0.16 mm, the collected waste concrete was used to prepare recycled powder (Fig. 1). The microscopic particle morphology of recycled powder via SEM was shown in Fig. 2. This indicates that the surface of the powder had some joint planes with some attached minute particles. This implied that the inner recycled powder included some easy cleavage of mineral constituents.

Fig. 1. The remaining fine powder material after screening.

Fig. 2. SEM graph of recycled powder (magnified 200 times).
Recycled powder has a packing density of 903 kg/m³ and an apparent density of 2,515 kg/m³. The particle size distribution graph measured by laser particle size analyzer (BT-9300Z, Bettersize Instruments Ltd., China) is shown in Fig. 3, and its specific surface area is stabilized at 4,500-7,500 cm²/g.

The major constituents of the recycled powder include cement mortar powder, cement rock particles and fine lime particles. The X-ray fluorescence analysis highlights the chemical constituents of the recycled powder (Table 1). The chemical constituents of the recycled powder have a higher calcium rate and relatively applicable silica rate of which CaO and SiO₂ were the major constituents of the cement. Therefore, recycled powder substituents of the cement act as a cementitious material. The chlorine ion content of the recycled powder meets the cement (GB175-2007, Qingdao Qian-kun-xing Industry and Trade Co., Ltd., China) requirements.

3. Pozzolanic Activity Test of Recycled Powder

3.1. Active Mechanism of Recycled Powder Cementitious Material from Chemical Activation

This study used recycled powder as a cementitious material for a hydration preparation of cement. When the active SiO₂ and Al₂O₃ in the recycled powder meet water, they react with Ca(OH)₂ to produce cementitious substances such as calcium silicate hydrate (C-S-H), calcium aluminate hydrate (C-A-H), calcium alumininate silicate hydrate (C-A-S-H), and ettringite (AFt) [26].

The preparation of the early strength agent in this experiment considered the following aspects: Most importantly, the early strength agent itself could quickly dissolve into water to produce a high crystal type of cementitious substance. Second, by simulating cement hydration environment, the early strength agent reacted with gypsum and active SiO₂ and Al₂O₃ in cycled powder to produce substances that could provide early strength such as C-S-H or AFt.

3.2. Introduction of Raw Materials

The recycled powder comes from broken concrete in construction waste. After aggregates over 0.16 mm were removed, the remaining material is the recycled powder. Its main components are described in the part 2 of this paper.

The main component of quick lime is calcium oxide (CaO), which comes from natural rock. The rock is calcined under high temperature and then decomposed into carbon dioxide and CaO. Gypsum (CaSO₄·2H₂O) is collected from the industrial waste of the electric power plant in the Huangdao district of Qingdao City, Shandong Province. The complex and fast solidification agent is obtained from the laboratory preparation, and its main components are sodium salt and silicate.

3.3. Experimental Scheme

We used an orthogonal design in this research. Recycled powder was the major raw material. The activators mainly consisted of quick lime, gypsum, compound early strength agent, etc. These experiments utilized alkali-calcium-sulfur as the basic activation principle. The compound early strength agent prepared was added to quickly produce early strength minerals at an early stage that

| Chemical constituents | SiO₂  | CaO   | Fe₂O₃ | Al₂O₃ | MgO | Cl  | Ignition loss |
|-----------------------|-------|-------|-------|-------|-----|-----|---------------|
| Content/%             | 47.86 | 17.48 | 3.27  | 6.58  | 1.53 | 0.04| 10.15         |

| Level | A: Quicklime | B: Gypsum | C: Compound early strength agent |
|-------|--------------|------------|-----------------------------|
| 1     | 5%           | 4%         | 2%                          |
| 2     | 10%          | 8%         | 4%                          |
| 3     | 15%          | 12%        | 6%                          |

Note: the admixture content of various factors was the percentage of entire cementitious system.
provided early strength for the recycled powder cementitious system. On the basis of multiple single-factor test at an early stage, three major factors were determined in the contents of quick lime A, gypsum B and compound early strength agent C. Three levels for each factor consisted of L9 (3^3). An orthogonal table was used for experimental design as selected in Table 3.

Water consumption was accurately measured in accordance with the Chinese national standard during cementation. Tests showed normal consistency, setting time and soundness of the Portland cements (GB1346-2001, Qingdao Qian-kun-xing Industry and Trade Co., Ltd., China).

### 3.4. Introduction to the Uniaxial Compression Test

According to the mixture ratio above, the materials including the recycled powder, quick lime, gypsum, and complex fast solidification agents were added to the mixer. The mixing process lasted 180 s. The mixer was then shut down, and purified water was added. The mixing process proceeded for 300 s. The mixed materials were taken out and put into a 70.7 mm × 70.7 mm × 70.7 mm die. The materials were shaped in a shaking table. After one day of curing in the die, the formed materials were placed in a standard curing box under 95% humidity and 20°C. The same three specimens were produced for each material ratio and tested under the uniaxial compression. The test procedures were as follows. First, every side length of the specimen was measured so that its section area could be calculated. Second, the specimen was located in the center of the bearing plate. A spherical seat was adjusted to make specimens under the uniformly distributed load with a loading rate of 0.5-1.0 MPa/s. The expression of axial stress was shown as follows:

\[
\sigma = \frac{P}{A}
\]

Here, \(P\) is the axial load on the specimens with the unit of N; \(\sigma\) is the axial stress with the unit of MPa; and \(A\) is the vertical cross-section of the specimen with units of mm².

### 3.5. Experimental Results and Analysis

The range of each factor was calculated to analyze its influence degree and effect on each examination index. This was used to determine the optimal experimental scheme. The range calculation results are shown in Table 5.

Table 5 shows that the early strength of the cement agent, especially the 8 h-strength, was very low to an extent that some formwork could not be removed. This exhibited a large plastic deformation with axial strain ranging from 6% to 8%. Fig. 4 presents the stress-strain curve for J8. When the sample reached its peak value, its axial strain was close to 8%. This indicated that the cement agent with this ratio could not achieve the desired strength and was actually disadvantageous to the final application. As the hydration time increased, its strength significantly increased, and the plastic deformation was lower (Fig. 5).
After analyzing the calculation results, the primary and secondary influence level of various factors on various examination indexes are presented as follows:

(1) Conclusions can be drawn about the range of analysis in the early cement strength including 1-d, 3-d and 7-d compressive strengths. The range of the various factors on the cement compressive strength in descending order was early strength agent, quick lime and gypsum. The range of the 3-d compressive strength of gypsum was only 23% greater than that of quick lime. The effect of quick lime was greater than that of gypsum from the standpoint of overall trend. The early strength agent is alkaline, and the alkalinity is critical for activation of recycled powder. Theoretically, higher alkalinity systems have more OH\(^{-}\), and thus the ability to damage the Si-O, Al-O, and Si-Al bond is stronger. This will produce strong depolymerization against the minerals of recycled powder to dismantle its structure. Meanwhile, the lower the alkalinity, the damage effect will be smaller, and the activation effect will be worse.

(2) Range analysis on the late strength of cement (28-d compressive strength) showed that a range of various factors affect cement compressive strength. In descending order, these were quick lime, gypsum and early strength agent. Adding quick lime was an approach for alkaline activation. The OH\(^{-}\) fostered a rupture of the Si-O and Al-O bonding so as to form free, active unsaturated bonds on the surface that could readily react with Ca(OH)\(_2\) to produce cementitious products such as C-S-H and C-A-H. The alkaline activation would also improve the early hydration reaction rate of the recycled powder. Sulfate activation mainly meant that Ca\(^{2+}\) and SO\(_4^{2-}\) reacted with active Al\(_2\)O\(_3\) dissolved in the solution to produce stable AFt. The data showed that the production of hydrated products similar to Portland cement from recycled powder required activation of the recycled powder with CaO or Ca(OH)\(_2\). Sulfate was sufficient for this activation, which only occurred in the presence of lime.

It can be concluded from above analysis that:

(1) Early strength agents played an important role in activating the early activity of the recycled powder. It not only markedly shortened the initial setting time, but also increased the early strength. Through range analysis, early strength agents played the most important role on 1-d, 3-d and 7-d strength. However, through this long-term hydration process of recycled powder, the activation function of quick lime and gypsum clearly increased.

(2) The ideal ratio for recycled powder cementitious materials is 78%:10%:8%:4% for recycled powder:quick lime:gypsum:early strength agent.

### 4. Hydration Mechanism Analysis on Recycled Powder Cementitious Material under the Optimal Mixture Ratio

The X-ray diffraction (XRD) analysis and SEM data reported the optimal ratio of recycled powder cementitious materials under

| Table 5. Orthogonal Test Range Analysis Table |
|----------------------------------------------|
| No. | 1 d Compressive strength/MPa | 3 d Compressive strength/MPa | 7 d Compressive strength/MPa | 28 d Compressive strength/MPa |
| A   | B   | C   | A   | B   | C   | A   | B   | C   | A   | B   | C   |
| K1  | 0.86 | 0.97 | 0.80 | 2.58 | 3.13 | 2.51 | 3.47 | 4.44 | 3.53 | 6.85 | 7.73 | 7.9  |
| K2  | 0.94 | 0.96 | 0.95 | 2.86 | 2.57 | 2.78 | 4.64 | 4.09 | 4.36 | 9.55 | 10   | 10.68 |
| K3  | 0.99 | 0.86 | 1.04 | 3.05 | 2.79 | 3.2  | 4.71 | 4.29 | 4.88 | 12.45| 11.12| 10.27|
| k1  | 0.287| 0.323| 0.267| 0.86 | 1.043| 0.837| 1.157| 1.48 | 1.177| 2.283| 2.577| 2.633|
| k2  | 0.313| 0.32 | 0.317| 0.953| 0.857| 0.927| 1.547| 1.363| 1.453| 3.183| 3.333| 3.56 |
| k3  | 0.33 | 0.287| 0.347| 1.017| 0.93 | 1.067| 1.57 | 1.43 | 1.627| 4.15 | 3.707| 3.423|
| R   | 0.13 | 0.11 | 0.24 | 0.47 | 0.56 | 0.69 | 1.24 | 0.35 | 1.35 | 5.6  | 3.39 | 2.78 |
various ages. Further study was done to investigate the hydration mechanism of activation for recycled powder cementitious materials under chemical activation.

4.1. XRD Analysis

Diffraction occurs when X-rays pass through the crystal. When a beam of X-ray was incident on the crystal of the powder from the specimens above, X-ray interference occurred. The distance among the atoms of the crystal and the wavelength of the X-ray are similar. The test methods for XRD included: (1) grinding the recycled powder; (2) placing the powder on a sample plate; (3) loading the sample plate in a sample holder; (4) setting up the parameters of the numerical control system on the X-ray diffractometer (D/Max2500PC, Rigaku Corporation, Japan); (5) collecting data; and (6) processing the data.

Fig. 6 presented the XRD achieved at various curing ages for mortar specimens prepared by recycled powder cementitious material.

The following major conclusions can be drawn from Fig. 6:

(1) Recycled powder cementitious material produced AFt at the early stage of hydration reaction. This indicated that a little recycled powder in the raw material began a hydration reaction. This supported the early strength of the cementitious system. Meanwhile, other crystal phases included SiO2, Ca(OH)2, and CaSO4·2H2O. There is not a lot of C-S-H.

(2) As the hydration process is ongoing, the diffraction peak of the AFt basically remained unchanged. Most SiO2 and Ca(OH)2 diffraction peaks significantly decreased. These substances participated in a hydration reaction to produce cementitious products such as C-S-H and C-A-H. This indicated that the activator reacted effectively with recycled powder. This jointly supported the strength of the specimens.

(3) During the 28-d hydration, the hydration reaction fully proceeded. The diffraction peaks of SiO2 and Ca(OH)2 further decreased, but the diffraction peaks of the cementitious substances such as C-S-H and C-A-H increased markedly. This meant that under the application of the activator, the volcanic ash activity of recycled powder was released to the maximum extent. This meant that the entire specimen met certain strength requirements.

4.2. SEM Analysis

Based on the interaction between electrons and materials, the powder from the specimens above was scanned by the high energy electron to get the physical signals of the powder. Then, signals were received, amplified and shown so that the image of surface morphology on the recycled powder could be obtained.

The test procedures for the XRD included: (1) locating the scattered powder from the specimens on a copper strip, and drying the powder; (2) sticking the copper strip on the conductive tape of the specimen holder of electron microscope, or vapor plating for forming a conducting layer if the specimen had poor conductivity; (3) locating the sample in the sample room of the electron microscope; (4) setting up the parameters of the numerical control

![XRD spectrum of recycled powder cementitious material at various ages.](image-url)
hydration 8 h

hydration 1 d

hydration 7 d
system on the SEM, observing images, slowly scanning and saving images.

Fig. 7 shows SEM data at various curing ages for mortar specimens prepared by recycled powder cementitious materials.

The following major conclusions were drawn from Fig. 7:

1. When the hydration reaction proceeded for 8 h, the specimen exhibited loose structures and larger gaps. There were complete spherical particles and flaking of Ca(OH)$_2$; the cementitious system just began the hydration reaction, and the resulting substance included some needle-type AFt.

2. From 1-d and 7-d hydration graphs, clustered C-S-H cementitious substances that gradually filled into the specimen skeleton were noted. Certain gaps still existed as observed from the 1-d hydration graph. On the seventh day, the recycled powder particles had been corroded significantly. Recycled powder particles were no longer isolated, but connected together through clustered, needle type, or flake-like cementitious substances. The hydration process was still on-going.

3. It was observed from 28-d hydration graph that a great deal of clustered C-S-H, partial needle type of AFt and some amorphous cementitious substances are closely arranged together and cross-coupled to form strong specimens.

5. Conclusions

1. Recycled powder has potential pozzolanic activity. It becomes a better substitute cementitious material for mine paste backfill when it is activated by activators.

2. Recycled powder made from waste concrete through crushing and sieving was chemically activated following A-C-S activation. The data indicated that the primary and secondary relation influenced the early strength of the recycled cementitious materials. The ranking was early strength agent > quick lime > gypsum. The primary and secondary relation influencing the late strength was quick lime > gypsum > early strength agent.

3. The optimal mixture ratio of recycled powder cementitious material for mine paste backfilling achieved through range analysis method was 78%:10%:8%:4% for recycled powder:quick lime:gypsum:early strength agent.

4. The XRD and SEM data showed that the initial hydration production of compound cementitious materials under the optimal mixture ratio was predominated by CH and AFt. With increasing hydration time, more and more recycled powder was activated. This was especially dominated by C-S-H, C-A-H and other cementitious materials.

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