Experimental Study on the Characteristics of Ultrafine Grinding of Potassium-bearing Shale and the Feasibility of Roasting Pre-cracking

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Experimental study on the characteristics of ultrafine grinding of potassium-bearing shale and the feasibility of roasting pre-cracking

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Highlights:

1. The particle size distributions of both the raw potassium-bearing shale and the ultrafine crushed product by high energy ball milling conformed to the RRB particle size distribution model.
2. The optimal solutions obtained are different in terms of $d_{\max}$ and fractal dimension of the particle size distribution, respectively.
3. AHP-fuzzy comprehensive evaluation of the three indicators of product quality and quantity was used, which showed roasting pre-cracking for 2 h to have a facilitating effect but at greater cost that is not suitable for industrial application.
4. Microwave pretreatment for 5 min and 15 min did not improve the quality of ultrafine potassium-containing shale powder.

Graphical abstract

Abstract: As potassium-bearing shale is still being developed as a potential alternative to potash, the first step to improve its utilisation is to reduce its particle size. This paper explores whether roasting pre-cracked potassium-bearing shale can improve the quality of ultra-fine crushing products. By analysing the particle size distribution and the fractal dimension of the particle size distribution, the results for 1 h and 2 h roasting pre-cracking experiments were found to be contradictory. AHP-fuzzy comprehensive evaluation of the three indicators of product quality and quantity was used, resulting in a unique indicator. Roasting pre-cracking for 2 h was found to have a facilitating effect, with the average of the three fuzzy comprehensive evaluation values increasing

from 0.71 to 0.78. The great difference in cost outlay suggests that the technique is not suitable for industrial applications. Subsequently, two groups of microwave pretreatment experiments were carried out but led to unsatisfactory results in which microwave pretreatment was not conducive to the ultrafine grinding of potassium-bearing shale.

Keywords: Potassium-bearing shale; Ultrafine grinding; Pre-cracking; AHP-Fuzzy comprehensive evaluation

1. Introduction

Potassium-bearing shale is a type of sedimentary rock in the form of layers and is an exploitable deposit of water-insoluble potassium-bearing rocks. According to data, there are large sources of potassium-bearing shale in some parts of China, such as Henan, Guizhou, Sichuan, Hubei and Hunan province ¹. Potassium-bearing shale can be prepared into potassium sulphate ², as well as improve cement varieties and produce low alkali cement when blended with fly ash as alternatives to clay ingredients ³. As a largely agricultural country, potassium-bearing shale is a potential supplier of potash fertiliser until large quantities of soluble potash are found, and there is a need to enhance the integrated use of potash-bearing rocks. Many researchers have shown acid leaching by activated roasting when extracting potassium from potassium-bearing shale ⁴⁵. The use of potassium-bearing shale for the preparation of potash fertilisers has been documented ⁶, with experimental research showing that the smaller the particle size, the greater the fertility.

The microcrack theory proposed by Griffiths suggests that there are many defects and cracks in a material ⁷, which, under the action of external forces, produce a stress concentration. After a certain point, the cracks begin to expand and lead to fracture, or the internal microcracks "grow" and create many new cracks simultaneously.

Internal structural changes of the rock can be attributed to a variety of factors, such as differences in the expansion coefficients of the mineral components, structural pre-cracking that occurs during the roasting treatment process, growth of microscopic internal fractures, pores and micropores, residual microscopic defects after water loss, and uneven stresses caused by structural thermal stresses ⁸. Roasting treatment can reduce the strength of the rock as well as induce grain boundary fractures, causing local stress concentrations that lead to comminution. Since the 20th century, thermally-assisted crushing studies of a variety of geological minerals have shown exceptional results ⁹, with some literature indicating that potassium-bearing shale roasting leads to acid leaching during potassium extraction ¹⁰¹¹. To date, researchers have seldom reported the roasting pre-cracking effect on potassium-bearing shales.

The geometrical characteristics of the evolution of many materials from microscopic damage to macroscopic crushing and the mathematical characteristics of the evolution of mechanical and physical quantities have good statistical self-similarity ¹². This self-similar behaviour leads to fractal characteristics of the particle size distribution of the crushed powder. The fractal dimension has been demonstrated in many papers to characterise the width of the particle size distribution ¹³¹⁴¹⁵, for which the larger the fractal dimension is, the larger the distribution width will be, and vice versa.

The AHP-fuzzy comprehensive evaluation method uses the Analytic Hierarchy Process (AHP) to obtain the weight vector of each indicator ¹⁶ and fuzzy mathematics to calculate the comprehensive evaluation index, transforming the multi-indicator decision problem into a single indicator decision problem ¹⁷. It adopts the membership degree theory to assess its level with the characteristics of clear results and a strong system, providing a powerful basis for the combination

of qualitative and quantitative decision-making¹⁸.

2. Experimental

2.1 Materials

The raw potassium-bearing shale was collected from a site in Jiangsu Province, and the specific surface area was measured by a digital blaine permeability surface area analyser (Wuxi Jian Yi Instrument Co., Ltd., model: SBT-127) at 141.9 m²/Kg with a moisture content of 0.5%. The content of K₂O is 9.07%. The laser particle size analyser (Dandong Baxter Instruments Co., Ltd., model: BT-9300H) was used to detect the particle size distribution. The raw potassium-bearing shale was mixed well in the medium of water from which three samples were taken and tested and for which the results are shown in Figure 1. The crystal structure was analysed by an X-ray diffractometer (Rigku, Japan, model: Smartlab SE) with a scanning range of 2Theta = 10°~70°. The operating current was 30.0 mA, the operating voltage was 40.0 kV, the maximum power was 12 kW, the scanning speed was 5°/min in continuous mode and Cu K α was used as the X-ray source. As shown in Figure 2, the main mineral phases of the potassium-bearing shale are diopside, quartz, orthoclase and microcline feldspar. The sharpness of each peak indicates that the mineral phases are crystalline and have small grains with good crystallinity.

Figure 1. Particle size distribution of raw materials.

Figure 2. XRD of raw materials.

Electronic Scale, manufactured by Shanghai Jiayi Electronic Technology Co., Ltd, model: TCS.

Intelligent Fibre Chamber Resistance Furnace, manufactured by Tianjin Zhonghuan Experimental Electric Furnace Co Ltd, model: SX3-5-12A, rated at 4000 W, rated temperature at 1200 °C. ¥4300 RMB, 15 years of service life.

Ball Mill; manufactured by Changsha Tianchuang Powder Technology Co., Ltd, model: QM-5, rated power 1000W. Stainless steel tank; inner diameter of 230 mm, inner height of 260 mm, volume of 10.8 L, critical speed of 88.4 RPM. Grinding media is tungsten carbide high energy balls, with a total weight of 18.52 kg (15 mm media ball mass is 10.61 kg and 10.5 mm media ball mass is 8.06 kg). The specific surface area of the media is 2.486 m²/kg. ¥5,000 RMB, 15 years of service life.

2.2 Experimental method

0.957 kg of water and 1.445 kg of potassium-bearing shale at 60% concentration were mixed, to which 2.89 g of NP-45 dispersant at 0.2% by mass was added. The tank speed was set at 80% of the critical speed, i.e. 70.72 RPM. The roasting pre-cracking method was carried out as follows: 1.445 kg of potassium-bearing shale was placed in a tray and placed in a resistance furnace at 500°C. After roasting for 1 h or 2 h, the potassium-bearing shale was taken out and poured directly into a ball mill jar containing an equal amount of water and grinding media, where the weight of the water includes the water evaporated from the potassium-bearing shale, i.e. 0.964 kg. Natural cooling and addition of grinding aid was followed. The material was then ground into an ultrafine product in the high-energy ball mill. Samples were taken at three points on the slurry surface of the ball mill jar and the particle size distribution checked by the laser particle size analyser.

3. Results and discussion

3.1 Effect of roasting pre-crushing on particle size

The change in the maximum particle size of the product is shown in Figure 3. It can be seen that the product particle size is partially uneven when roasted for 2 h and then crushed. Individual indicators, however, do not accurately reflect the overall quality of the product.

Figure 3. $d_{\max}$ of the ultrafine product by different processing modes.

The crystal structures of shelf-structured potassium feldspar and layered mica-like minerals are relatively stable. Roasting increases the internal porosity of the potassium-bearing shale but also increases the strength of the particles¹⁹. More pores are of benefit to the comminution, while a higher strength is not. Roasting pre-cracking for 1 h may increase the pores less, but the particle strength increases, making the ultra-fine product size become larger. Roasting pre-cracking for 2 h increases pores while $d_{\max}$ becomes smaller.

The Rosin-Rammler-Bennett distribution (RRB) is one of the most widely applied distribution patterns^{20,21}, given by

$$R = 100 - 100 \cdot \exp \left[- \left(\frac{d}{d_e} \right)^n \right] \quad (1)$$

where d is the particle size (μm), R is the negative accumulation of relative mass under the sieve for size d (%), d_e is the characteristic particle size corresponding to the particle size with an oversize percentage of $100-100e^{-1}$, i.e. 63.21% (μm) and n is the uniformity factor (dimensionless). Both the raw potassium-bearing shale and ultrafine product particle size distributions conform to the RRB model, as shown in Figure 4.

Figure 4. Particle size distribution of different products.

The RRB model of the raw potassium-bearing shale is $R = 100 - 100\exp(-(d/23.03)^{0.96})$ with a correlation coefficient of 1.0.

The RRB model of the product in the blank test is $R = 100 - 100\exp(-(d/3.73)^{0.93})$ with a correlation coefficient of 0.99.

The RRB model of the product by roasting for 1 h is $R = 100 - 100\exp(-(d/5.68)^{1.34})$ with a correlation coefficient of 0.99.

The RRB model of the product by roasting for 2 h is $R = 100 - 100\exp(-(d/3.95)^{1.12})$ with a correlation coefficient of 1.0.

These results show that the particle size with an oversize percentage of 63.21% in the roasting pre-cracking test is larger than the black test. In particular, the size of the crushed product becomes larger in roasting pre-cracking for 1 h, which further indicates that roasting for 1 h is not conducive to ultrafine comminution.

3.2 Effect of roasting pre-cracking on fractal dimension

Provided that the particle size distribution conforms to the RRB model, previous work has summarised the formulae for calculating the fractal dimension of the particle size distribution based on the particle size distribution¹³, which is given by

$$S(d) = k \cdot \left(\frac{d}{d_{max}}\right)^{3-D} \quad (2)$$

where $S(d)$ is the percentage of mass under the sieve smaller than the particle size d (%), D is the fractal dimension of the particle size distribution of the particle population (dimensionless), k is a constant, d is the particle size (μm) and d_{max} is the maximum particle size of the particle population (μm). Figure 4 shows the particle size at which the cumulative content first reaches 100%.

The fractal dimension of the particle size distribution of the potassium-bearing shale feedstock

was calculated in Origin 2018 by fitting the data with equation 2. The fractal dimension of the feedstock size distribution is $D = 2.62$ with a correlation coefficient of 0.97 as shown in Figure 5.

Figure 5. Calculation of the fractal dimension of the particle size distribution

By analogy, the fractal dimensions of the particle size distribution of other products are calculated and the variation of the fractal dimension of the particle size distribution of the ultrafine pulverised products after different roasting pre-cracking treatments are shown in Figure 6.

Figure 6. The fractal dimension of the particle size distribution of different ultrafine products.

As can be seen from Figure 6, the fractal dimensions of the particle size distribution of the ultrafine pulverised products are smaller than that of the raw material. The smallest particle size distribution is obtained by roasting pre-cracking for 1 h.

3.3 Influence of roasting pre-cracking on product by fuzzy comprehensive evaluation

Three indicators were selected to reflect the product's comprehensive characteristics. In terms of yield, the weight of the -10 μm product was chosen as the measure. In terms of product quality, the fractal dimension of the particle size distribution (which indicates the width of the particle size distribution) and 97% by particle size (d_{97}) were chosen as measures. Since only a few parameters were selected for "yield" and "quality" characterisation, a comprehensive evaluation system was constructed.

3.3.1 Calculation of the weight vectors of the evaluation indicators

The three indicators to be evaluated are the "yield of qualified product" (W1), "number of particle size distribution components" (W2) and "97% of the powder passing through particle size, d_{97} " (W3), where the weight vector is denoted as W .

An expert in the powder industry (Prof. Gai Guosheng from Tsinghua University) compared the three indicators to be evaluated according to the relevant diagram ⁸. The results of the two comparisons provided by the experts are: (i) the yield of qualified products is slightly but not significantly more important than the particle size distribution fractional dimension, which is significantly more important than the powder 97% through particle size d_{97} , and (ii) the particle size distribution fractional dimension is equally as important as the powder 97% through particle size d_{97} .

The descriptions of the combined comparison of the indicators given by the experts can be written into the descriptive matrix of importance, and then transformed into a judgment matrix, $A_{3 \times 3}$, given by

$$A = (a_{ij})_{3 \times 3} = \begin{bmatrix} 1 & 5 & 4 \\ 0.2 & 1 & 1 \\ 0.25 & 1 & 1 \end{bmatrix}$$

The weight vector is calculated as $W = (0.69, 0.149, 0.161)^T$.

3.3.2 Establishment of the fuzzy comprehensive evaluation system

(1) Determining the evaluation set of test results

For the blank test, the three measurement results of the product after 3 h of ultrafine grinding are shown in Table 1.

Table 1. Three times parallel values of product indicators in the blank test.

No.	W1 (kg)	W2 (-)	W3 (μm)
1	1.31	2.54	14.27
2	1.29	2.52	16.83
3	1.36	2.58	12.69

The evaluation sets of the three measurement results are

$$U = \{W1, W2, W3\} = \{1.31, 2.54, 14.27\}, \{1.29, 2.52, 16.83\}, \{1.36, 2.58, 12.69\}.$$

(2) Determining the value range of W1, W2 and W3

The total mass of the feed was 1.445, so the range of values for the -10 μm powder mass, W1, is [0,1.445].

From geometry, we know that the dimension of a straight line is one, the dimension of a flat figure such as a square, circle or ellipse is two, and the dimension of a three-dimensional figure such as a cube or ball is three. For everyday objects, the dimension is between one and three, so the value range of the particle size distribution fractional dimension, W2, is [1,3].

For 97% of the powder passing through, the lower limit of the d_{97} domain value is equal to 0 while the upper limit is equal to the maximum particle size of the particle population in the raw material, 130.37, such that W3 is [0,130.37].

(3) Selecting the affiliation function

The traditional method of selecting an affiliation function is to select an affiliation function that can appropriately reflect the parameters according to their properties. According to the experimental kinetic model curve, the ball mill ultrafine grinding process becomes more and more difficult as the depth of the process increases. In this paper, the k times parabolic affiliation function is selected, with k equal to 2 and the values of a (the lower limit) and b (the upper limit) varying with different fuzzy processing objects.

Indicator W1 is a benefit-type indicator (the larger the value, the better), whereas W2 and W3 are both cost-type indicators (the smaller the value, the better). As such, these three indicators require slightly different affiliation functions. The following formulae are used as the W1, W2 and W3 affiliation functions, respectively.

$$f(x; 0, 1.445, 2) = \begin{cases} 0, & x \leq 0 \\ \left(\frac{x}{1.445}\right)^2, & 0 < x < 1.445 \\ 1, & x \geq 1.445 \end{cases} \quad (7)$$

$$f(x; 1, 3, 2) = \begin{cases} 1, & x \leq 1 \\ \left(\frac{3-x}{2}\right)^2, & 1 < x < 3 \\ 0, & x \geq 3 \end{cases} \quad (8)$$

$$f(x; 0, 130.37, 2) = \begin{cases} 1, & x \leq 0 \\ \left(\frac{130.37-x}{130.37}\right)^2, & 0 < x < 130.37 \\ 0, & x \geq 130.37 \end{cases} \quad (9)$$

(4) Fuzzy transformation of indicators

The values of each indicator of the test results were fuzzily transformed into the affiliation degree μ , where μ is normalized to [0,1] by the affiliation degree function. Taking the indicators of the product in the blank tests as examples, the fuzzy transformation of indicators was calculated as follows.

The product of the -10 μm material mass, W1, was 1.31 kg, and since $0 < 1.31 < 1.445$, the degree of membership can be obtained by substitution into the subordinate degree function calculation, given by

$$P1 = f(1.31; 0, 1.445, 2) = \left(\frac{1.31}{1.445}\right)^2 = 0.82.$$

The degree of membership P2 of W2 can be calculated as

$$P2 = f(2.54; 1, 3, 2) = \left(\frac{3-2.54}{2}\right)^2 = 0.05.$$

The degree of membership P3 of W3 can be calculated as

$$P3 = f(14.27; 0,130.37,2) = \left(\frac{130.37-14.27}{130.37}\right)^2 = 0.79.$$

The fuzzy evaluation set of each indicator of this test result is therefore given by P = (0.82,0.05,0.79).

(5) Comprehensive evaluation of multiple indicators

The comprehensive evaluation of multiple indicators is the product of the fuzzy evaluation set of individual indicators and the matrix of the weight distribution set of each indicator, given by

$$Z = P_{1 \times 3} \cdot W_{3 \times 2} = (0.82,0.05,0.79) \cdot (0.69,0.149,0.161)^T = 0.70.$$

3.3.3 Effect of roasting pre-cracking on the fuzzy comprehensive evaluation index of the product

The comprehensive evaluation indexes calculated from the three experimental data of the blank test products are shown as follows.

$$Z = (0.82,0.05,0.79) \cdot (0.69,0.149,0.161)^T = 0.70,$$

$$Z = (0.80,0.06,0.76) \cdot (0.69,0.149,0.161)^T = 0.68,$$

$$Z = (0.89,0.04,0.81) \cdot (0.69,0.149,0.161)^T = 0.75.$$

The comprehensive evaluation indexes calculated from the three experimental data of the ultrafine product by roasting pre-cracking for 1 h are shown as follows.

$$Z = (0.64,0.21,0.71) \cdot (0.69,0.149,0.161)^T = 0.59,$$

$$Z = (0.72,0.10,0.73) \cdot (0.69,0.149,0.161)^T = 0.63,$$

$$Z = (0.74,0.18,0.75) \cdot (0.69,0.149,0.161)^T = 0.66.$$

The comprehensive evaluation indexes calculated from the three experimental data of the ultrafine product by roasting pre-cracking for 2 h are shown as follows.

$$Z = (0.82,0.05,0.79) \cdot (0.69,0.149,0.161)^T = 0.77,$$

$$Z = (0.89,0.16,0.82) \cdot (0.69,0.149,0.161)^T = 0.78,$$

$$Z = (0.93,0.12,0.78) \cdot (0.69,0.149,0.161)^T = 0.78.$$

The mean values and standard deviations were obtained and a comparison of the comprehensive evaluation indicators shows that the ultrafine potash-bearing shale by roasting pre-cracking is not always superior to the direct ultrafine pulverised product in Figure 7.

Figure 7. Fuzzy comprehensive evaluation values of different processing modes.

3.4 Effect of roasting pre-cracking on comminution kinetics

The concept of kinetic breakage has been described by Austin *et al*²². Based on the n order comminution kinetic model that has been accepted by the comminution community²³, d_{max} is given by

$$d_{max}(t) = d_0 \exp(-gt^n) \quad (10)$$

The kinetic curve was fitted using the Levenberg-Marquardt algorithm, where t is time (min), d_0 is the particle size of the largest particle in the feed material (μm), d_{max} is the particle size of the largest particle in the product after t time of comminution (μm), g is a parameter related to the size of the material and n is a parameter related to the nature of the material. The kinetic equations of crushing under different roasting pre-cracking methods were obtained, with correlation coefficients of 0.99 obtained as shown in Figure 8.

Figure 8. Crushing kinetic curves for different roasting pre-cracking methods.

The kinetic equation of comminution with no processing is given by

$$y = 110.06\exp(-0.09x^{0.55}).$$

The kinetic equation of comminution with roasting pre-cracking for 1 h is given by

$$y = 110.06\exp(-0.14x^{0.44}).$$

The kinetic equation of comminution with roasting pre-cracking for 2 h is given by

$$y = 110.06\exp(-0.07x^{0.59}).$$

3.5 Economic feasibility analysis

The previous analysis shows that roasting pre-cracking potash-bearing shale for 2 h can improve the quality of the product, but industrial applications also need to consider the economic applicability. Compared to the blank test, the roasting pre-cracking process mainly consists of increased electricity and equipment depreciation costs. The cost consumption is calculated as 360 days per year at ¥0.9 per kWh of electricity.

For the blank test, the cost consumption is calculated to be $1 \text{ Kw} \times 3 \text{ h} \times 0.9 \text{ RMB/Kwh} + (3 \text{ h} / (15 \text{ a} \times 360 \text{ d} \times 24 \text{ h})) \times 5000 \text{ RMB} = 2.82 \text{ RMB}$. In roasting pre-cracking for 2 h, the cost consumption is calculated to be $4 \text{ Kw} \times 2 \text{ h} \times 0.9 \text{ RMB/Kwh} + (2 \text{ h} / (15 \text{ a} \times 360 \text{ d} \times 24 \text{ h})) \times 4300 \text{ RMB} + 1 \text{ Kw} \times 3 \text{ h} \times 0.9 \text{ RMB/Kwh} + (3 \text{ h} / (15 \text{ a} \times 360 \text{ d} \times 24 \text{ h})) \times 5000 \text{ RMB} = 10.08 \text{ RMB}$.

The fuzzy comprehensive evaluation value increased from 0.71 to 0.78 with a year-on-year growth of 9.86%, while the cost consumption increased from 2.82 RMB to 10.08 RMB with a year-on-year growth of 258.06%. It is clear that roasting pre-cracking potassium-bearing shale is not feasible for industrial applications.

3.6 Microwave Pretreatment

Though it is difficult to put roasting pre-cracking treatment into practice to improve product quality, its potential to improve product quality by microwave pre-cracking treatment remains

unanswered. Microwave pre-cracking treatments in other ore crushing processing tests have proven to have a catalytic effect ²⁴²⁵. Microwave pre-cracking (Microwave oven, MM721AAU-PW, Midea Group, rated input power 1150W, microwave operating frequency 2450MHz) of potassium-bearing shale for 5 min and 15 min and the same method as above was used for the fuzzy comprehensive evaluation of the product. The results are shown in Table 2.

Table 2. The fuzzy comprehensive evaluation of the product by microwave pre-cracking

Processing time	W1 (Kg)	W2 (-)	W3 (μm)	Z (-)
5 min	1.172	2.072	36.08	0.57
15 min	1.253	2.041	32.41	0.64

The fuzzy comprehensive evaluation values of the product after microwave pretreatment of raw materials for 5 min and 15 min shows that microwave pre-cracking is difficult to apply to potassium-bearing shale. The reason may be due to the silicate shelf structure of potassium-bearing shale being relatively stable and that the process of microwave radiation cracking is not enough to grow original cracks or make the particles produce enough cracks. Similar to clay, the microwave roasted potassium-bearing shale in the water quenching process enhances the compressive strength of the grain ¹⁹, the crushing difficulty and product quality.

4. Conclusions

The particle size distributions of both the raw potassium-bearing shale and the ultrafine crushed product by the high energy ball mill conformed to the RRB particle size distribution model with a correlation coefficient of 0.99. Compared to the experimental product without any pre-treatment, the roasting pre-cracking for 1 h did not reduce the particle size of the product while the roasting pre-cracking for 2 h reduced the particle size of the product but with a larger standard deviation. The fractal dimension of the particle size distribution of the three products decreased and then increased slightly. The AHP-fuzzy comprehensive evaluation model, which takes into account both qualitative and quantitative parameters, shows that the roasting pre-cracking for 2 h is effective, with a relative increase of 7.87%. Roasting for 1 h may only increase the hardness, which is not conducive to ultrafine pulverisation. Roasting pre-cracking for 2 h can cause the particles to expand internal cracks, develop new cracks or grow old ones, thus loosening the physical framework of the material, making it more brittle, more efficiently crushed and of better product quality. Roasting pre-cracking for 2 h, however, greatly increases the cost outlay that is not favourable for industrial production. Microwave pretreatment of raw materials for 5 min and 15 min does not improve the product's quality.

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Declaration of competing interest

The authors declare that there are no conflicts of interest in this article.

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Figures

Figure 1

Particle size distribution of raw materials.

Figure 2

XRD of raw materials.

Figure 3

$d_{\max}$ of the ultrafine product by different processing modes.

Figure 4

Particle size distribution of different products.

Figure 5

Calculation of the fractal dimension of the particle size distribution

Figure 6

The fractal dimension of the particle size distribution of different ultrafine products.

Figure 7

Fuzzy comprehensive evaluation values of different processing modes.

Figure 8

Crushing kinetic curves for different roasting pre-cracking methods.