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Research on superfine grinding process and kinetics of calcined black talc in planetary mill

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Abstract

This work investigated the size reduction process of calcined black talc using a high energy CJXXM planetary mill. The grinding effects of two different grinding media, aluminia and zirconia, and three kinds of grinding aids, ethanol triethanolamine and sodium hexametaphosphate on the average particle size D50 was compared. The specific surface area, morphology and crystal structure of the ground calcined black talc was characterized by BET, SEM and XRD. The kinetic models of the -1μm particles in the product are also been studied. The result shows that the zirconia is superior to aluminia as grinding media, and ethanol is a better grinding aid for calcined black talc compared with triethanolamine and sodium hexametaphosphate. The D50 of product can be 1.09μm within 15min. No agglomerations between fine particles are formed in the product. The (220) is the most unstable crystal plane**.** Kinetic models of calcined black talc in a planetary mill under dry superfine grinding conditions using zirconia as grinding media with and without ethanol can be expressed as $Q_t = 1 - (1 - Q_0)e^{-0.0315t^{0.7976}}$ and $Q_t = 1 - (1 - Q_0)e^{-0.0295t^{1.0759}}$ respectively.

Keywords: planetary mill; dry grinding; calcined black talc; kinetic equation

1. Introduction

Black talc is a kind of silicate mineral, its main chemical composition is silica and magnesium oxide, it also contains a small amount of potassium, calcium, iron, sodium, etc[1].The main mineral compositions of black talc are

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talc, quartz, calcite, sepiolite, organic carbon, etc. so its color is usually present in black or ash-black. The whiteness of black talc, after calcination process, can be increased by more than 90%. Calcined black talc with good insulation, heat resistance, adsorption, and lubrication performance, can be widely used in ceramic industry, padding industry, paint industry, waterproof materials industry, medicine and cosmetics industry [2-4].

With the development of modern industry and high-tech industry, customers put forward higher and higher request about fineness of the calcined black talc powder. As a result, ultrafine grinding has recently been more and more important than before. Currently, most of ultrafine powder was prepared by using mechanical force in dry condition, the planetary ball mill which can generate a high energy is considered as important superfine grinding equipment. For grinding mills using spherical balls as grinding media, the effectiveness of small grinding balls on the fineness of ground products has been recognized as an important experimental factor[5-9].

Vedaraman [10] studied the effect of the physical properties of liquid additives on dry grinding and found that additives increased the fineness of the ground particles and the powder flow. Fuerstenau[11] and Hasegawa et al. [12] reviewed the use of chemical additives for improving the efficiency of both wet and dry pulverization. The effect of grinding aids, i.e. dispersants or additives, on grinding has been explained mainly by two mechanisms. One is the alteration of the surface and mechanical properties of individual particles, such as a reduction of surface energy, and the other is the change in arrangement of particles and their flow in suspensions[13–16].

In this paper, the effects of different grinding media and grinding aids on dry ultra-fine grinding of calcined black talc in planetary mill were studied, the proper grinding media and grinding aid of calcined black tale were found, and by means of mathematical methods, the grinding models were concluded.

Nomenclature

P_1	product ground by alumina for 30min
P_2	product ground by zirconia for 30min
P_3	product ground with ethanol by zirconia for 15min
I	diffraction intensity
$\triangle P_i$	changes in diffraction intensities between product and feed, calculated by the formula $(I_{feed}-I_{Pi})/I_{feed}$
Q_t	content of coarse particles in product at grinding time t
Q_0	content of coarse particles in feed
Q_{ft}	content of fine particles in product at grinding time t
Q_{f0}	content of fine particles in feed
n	the order of grinding kinetic
k	proportionality coefficient
t	grinding time

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2. Materials and experimental methods

2.1. Materials

Calcined black talc powder was supplied from Zhejiang HongYan Mining Co., Ltd., The size distribution of the calcined black talc is given in Table 1. Alumina ceramic beads and high purity zirconia ceramic beads were chosen as the grinding media for this study. Alumina ceramic beads (3.62g/cm^3, 2mm) were supplied from King's Ceramics & Chemicals Co., Ltd. High purity zirconia ceramic beads (6.00g/cm^3,2mm) were obtained from Zhuzhou Jin Tao Advanced Ceramic co., Ltd. Triethanolamine, ethanol and sodium hexametaphosphate were chosen as grinding aids.

Table 1. The size distribution of the calcined black talc

Cumulative fraction/%	D_3	D_6	D_{10}	D_{16}	D_{25}	D_{50}	D_{75}	D_{84}	D_{90}	D_{97}	D_{100}
Particle size/μm	2.96	3.92	5.11	8.33	11.66	20.17	27.83	31.86	35.72	43.2	50

2.2. Experimental method

Grinding experiment was performed in a CJXXM planetary mill which was produced by China-Russia High-Tech Incubator (Jiaxing) Co., Ltd. This mill consists of a pair of pots installed on a revolving disk as schematically illustrated in Fig. 1. Each pot has lining for the alumina ceramics with an inside diameter of 60 mm and an inner volume of 250ml. The pots are rotated in the counter direction against the disk revolution.
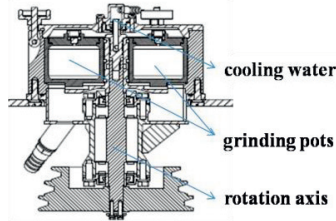

Fig.1 Profile of CJXXM planetary mill

Mass of feed materials, balls and grinding aids were set at 25 g, 150g and 0.125g, respectively. The mill was rotated at 800 rpm. Batch grinding tests were carried out for five spans of 6, 12,18,24 and 30min with zirconia or alumina, absence of grinding aids; According to the former paper[17] , the addition of grinding aids can improve the grinding rate, so the interval is shorted to 3min and the tests were carried out for five spans of 3, 6, 9, 12 and 15min with zirconia and grinding aids. The ground products were characterized by using a centrifugal sedimentation analyser (BT-1500, Dandong Bettersize Instruments Ltd.), nitrogen adsorption surface tester (JW-BK, Beijing JWGB Sci.&Tech. Co.,Ltd.), field emission scanning electron microscope(S-4800, Hitachi, Ltd.) and X-ray diffractometer (D8 ADVANCE, Bruker Co.)

3. Result and discussion

3.1. Effect of media on the particle size D50 and size distribution

Fig.2(a) shows the effect of zirconia and alumina on product particle size D50 within 30min. Clearly, the grinding result of zirconia is significantly better than alumina. The D50 of the product with zircnia as media is smaller throughout the whole 30min grinding period. From the comminution curve of zirconia, it can be seen that particle size decreased obviously before 9min, with the average decrease rate of 2μm/min. After 12min, the particle size decreases slowly, with the average rate of 0.114μm/min. The grinding limit of 1.06μm was reached at 24min. Based on the comminution curve of alumina, it can be seen that particle size decreased obviously before 12min, with the average decrease rate of 1μm/min then decreases slowly after 12min. After grinding 30 min the particle size can just decreased to 4.23μm.
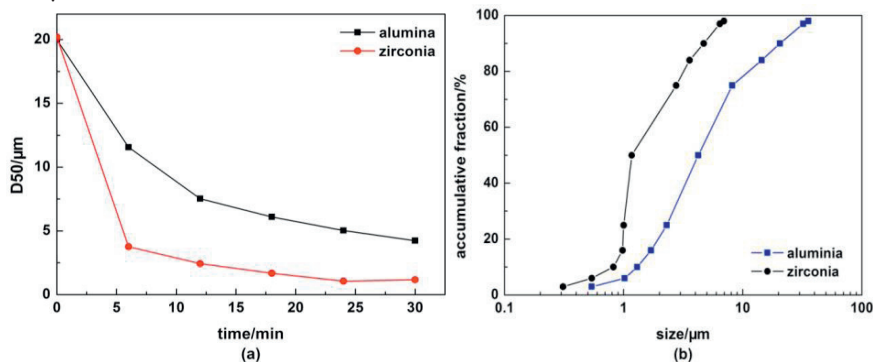

Fig. 2 (a) variation of D50 values with grinding time for different media; (b) the size distribution of ground calcined black talc

Fig.2(b) shows the size distribution of ground calcined black talc at 30min, using zirconia and aluminia as media respectively. From distribution curves we can see that particle sizes corresponding to each cumulative content using the zirconia as media are smaller than those using alumina as grinding media. And the size distribution of product ground by zirconia is more narrow and uniform. The zirconia beads have ability to ground calcined black talc to a range from 0.31μm to 6.95μm, corresponding to the cumulative content of D3 and D98 respectively, while alumina beads can just ground the product to the range from 0.54μm to 35.61μm. As a result, zirconia, for the superior grinding ability, was chose as grinding media at following tests.

3.2. Effect of grinding aids on particle size D50 and size distribution

Fig.3 shows the effect of different grinding aids triethanolamine, ethanol and sodium hexametaphosphate on D50 within 15min. As can be seen from Fig.3, during 15min, the grinding effects of ethanol and triethanolamine on calcined black talc are superior to sodium hexametaphosphate. At 9min, the value of D50 using erhanol as aids is 1.11μm smaller than that of using triethanolamin and hexametaphosphate. After grinding 12 minutes, the median size of product adopted sodium hexametaphosphate, triethanolamine and ethanol is 1.18μm, 1.07μm and 1.1μm respectively. Keep on grinding to 15min, the particle size sustaining decreased to 1.09μm with ethanol as grinding aids, while for triethanolamine and sodium hexametaphosphate, the D50 of product is increased a little to 1.12μm and 1.54μm respectively. So it can be concluded that ethanol has an excellent grinding effect on calcined black talc, compared with sodium hexametaphosphate and triethanolamine.
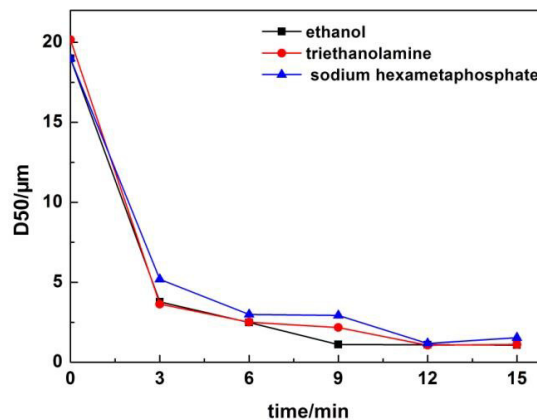

Fig. 3 Variation of D50 values with time for different grinding aids

Fig.4 shows the size distribution of ground calcined black talc at 9min and 15min with different grinding aids. Fig.4(a) indicates that ethanol is superior to trithanolamine and trithanolamine is superior to hexametapho. The ethanol is the best among the three grinding aids, for the finer and narrow distributin span of product. Comparing these two figures, it can be seen that the particle size almost have no further refinement after 9min under condition of adding ethanol as grinding aids, indicating that the limit particle size is reached. Relatively, the distribution curves of triethanolamine and sodium hexametapho still change with grinding time. The curve between D25 and D90 move to left side, indicating that particles in this range continue to be broken into small ones. At 15min, distribution curve of product using trithanolamine reach the same result of ethanol for 9min. This indicating that prolong the grinding time product with triethanolamine and sodium hexametapho can reach the same limit particle size.
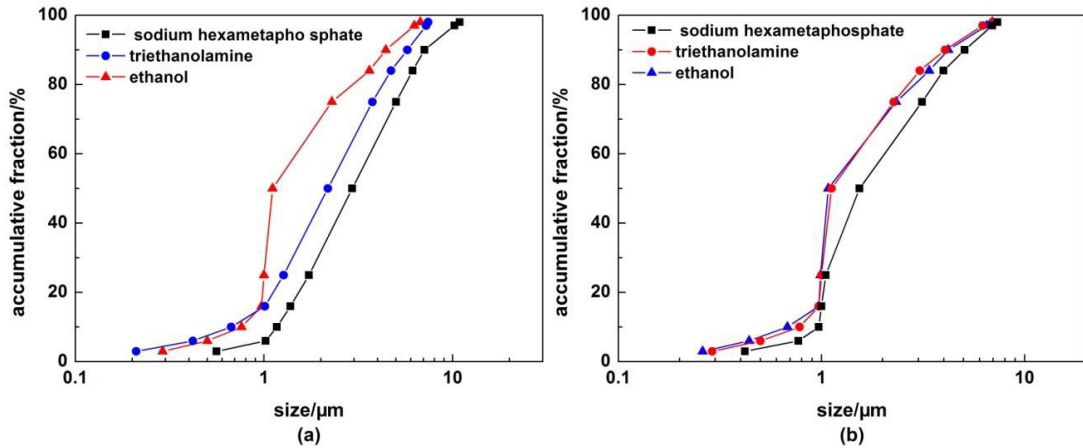
Fig.4 The size distribution of ground calcined black talc with different grinding aids at grinding time of (a) 9min and (b) 15min.

Fig.5 shows the variation of specific surface area and average particle size with time using zirconia beads as media in the case of with and without ethanol. The average particle size and particle size D50 under same grinding condition are nearly the same indicating that the agglomeration is not server, same conclusion can be found in SEM result. From the curve of specific surface area which absence of ethanol, it can be seen that the increase rate of specific surface area is fast before 12min. Between 12min and 18min, the specific surface area stabilizes at a value of about 5.55m^2/g. And then it increases again. At 30min, the value of specific surface area is 6.74 m^2/g corresponding to an average particle size of 0.93μm. From the curve of specific surface area with ethanol, it can be seen that the increase rate of specific surface area is faster before 3min, then became slowly from 3min to 12min. At 30min, the value of specific surface area is 7.39 m^2/g corresponding to an average particle size of 0.85μm. Comparing these curves, we know that adding ethanol in calcined black talc during superfine grinding process can improve grinding efficiency and shorten the grinding time to the desired product size.
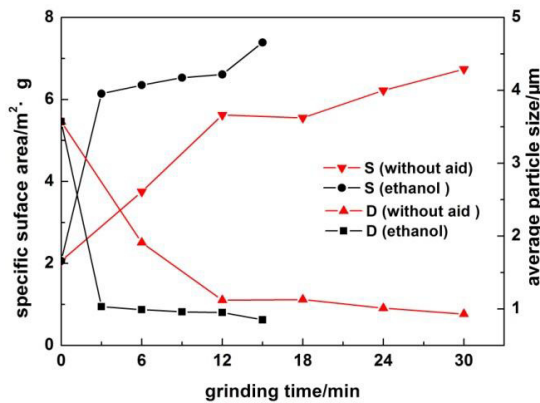

Fig. 5 Variation of specific surface area and average particle size with time

3.3. Morphology of the particle

Fig.6 shows the scanning electron micrographs of unground calcined black talc, ground product using alumina beads and zirconia beads at grinding time of 30min, and ground product added ethanol using zirconia beads at grinding time of 15min. As can be seen from Fig.6A, there are particles of blocky and warped lamellar crystal shape, and aggregates formed by columnar particles. The aggregates can be as large as about 50μm. From the Fig.6B, C

and D, we can see that the aggregates disappear after ground, and no agglomerations between fine particles are found. Comparing Fig.6B and C, we can see that the amount of large particles in product ground by alumina is more than that by zirconia, indicating that zirconia has a superior grinding ability. Comparing Fig.6C and D, we can see that the amount of fine particles in products is almost the same, showing that adding grinding aid ethanol can obtain the same product quality (particle size and distribution) within a short time.
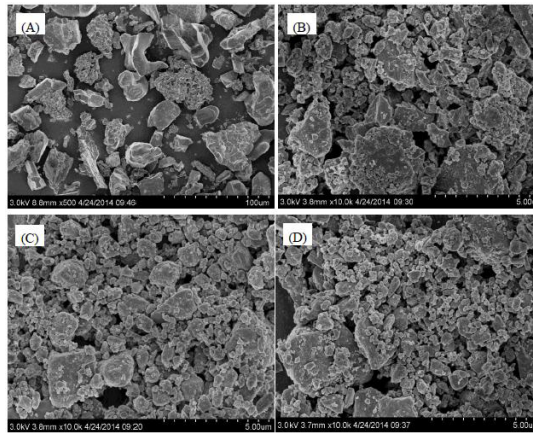

Fig.6 Scanning electron micrographs of: (A) unground calcined black talc; (B) ground product using alumina beads; (C) ground product using zirconia beads and (D) ground product using zirconia beads and ethanol

3.4. Structural changes of ground calcined black talc

Fig.7 shows x-ray diffraction profiles of unground calcined black talc, ground product using alumina beads and zirconia beads at grinding time of 30min, and ground product added ethanol using zirconia beads at grinding time of 15min. The XRD results revealed the calcined black talc are mainly composed of clinoenstatite and silica. After ultrafine grinding, intensity of characteristic peaks for each plane of clinoenstatite and silica is reduced, indicating that the structure of some clinoenstatite and silica particles was distorted and grain size was reduced which resulted in reduction of diffraction intensity.
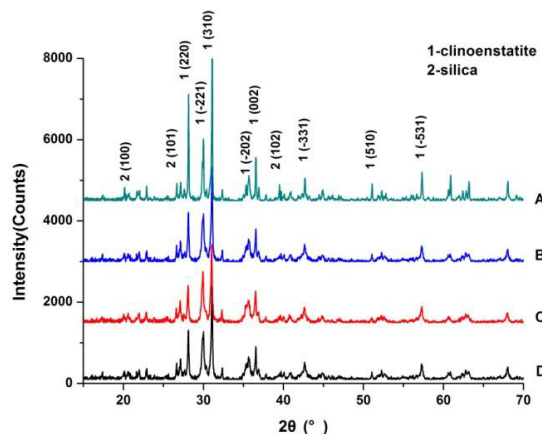

Fig.7 X-ray diffraction profiles of: (A) unground calcined black talc; (B) ground product using alumina beads; (C) ground product using zirconia beads and (D) ground product using zirconia beads and ethanol

Diffraction intensities of characteristic peaks of clinoenstatite for each plane are listed in Table 2. They indicate that different lattice planes are varies in intensity reduction level after fine grinding process. For product P_1, (310) and (220) crystal planes have obvious changes in diffraction intensities, and the intensity changes of (-531) and (-331) are slight, indicating that (-531) and (-331) are more stable than (310) and (220). For product P_2, the result shows that (-221) and (-202) are more stable than (510) and (220). For product P_3, the (-221) and (-331) are more stable than (510) and (220). As a result, (220) is the most unstable crystal plane. For product P_3, the intensities of most crystal plane are stronger than that of P_1, showing that grinding aids can slow down particles become amorphous.

Table 2 Diffraction intensities of the crystal planes of clinoenstatite.

lattice lane	feed	P_1	ΔP_1	P_2	ΔP_2	P_3	ΔP_3
(310)	5379	2539	0.53	2423	0.55	2948	0.45
(220)	3025	1426	0.53	1172	0.61	1547	0.49
(-221)	1865	1163	0.38	1651	0.11	1548	0.17
(002)	1169	815	0.30	877	0.25	859	0.27
(-531)	738	567	0.23	477	0.35	420	0.43
(-202)	723	482	0.33	638	0.12	561	0.22
(-331)	600	425	0.29	476	0.21	517	0.14
(510)	414	215	0.48	140	0.66	184	0.56

3.5. Grinding kinetics of the calcined black talc

In the grinding experiment, the weight of the coarse particles in ground product will be reduced after a period of grinding time. The decreased rate of the weight of coarse particles changed with time (i.e. grinding speed) can be expressed as follows [18]:

$$Q_t = Q_0 e^{-kt^n} \tag{1}$$

Taking the fine fraction content in the material as the dependent variable, Eq. (1) is converted as the following:

$$1 - Q_{ft} = (1 - Q_{f0}) e^{-kt^n} \tag{2}$$

In order to obtain the arguments *K and n*, Eq. (2) is converted as the following:

$$\ln\ln(1 - Q_{f0})/(1 - Q_{ft}) = n\ln t + \ln k \tag{3}$$

The arguments *K and n* can be obtained by regression analysis of the experimental data of particle size distribution.

Fig.8 shows the relationship between $\ln\ln(1-Q_0)/(1-Q_t)$ and $\ln t$ under experimental conditions of adopting zirconia as media with and without ethanol. Fitting a straight line to these data, the slope n and intercept lnk can be obtained by regression analysis. So the equations of these two lines are as following:

$$\ln\ln(1 - Q_0)/(1 - Q_t) = 0.7976\ln t - 3.4590 \tag{4a}$$

$$\ln\ln(1 - Q_0)/(1 - Q_t) = 1.0759\ln t - 3.5230 \tag{4b}$$

Converting Eq. (4) the kinetic equations of calcined black talc are as following:

$$Q_t = 1 - (1 - Q_0)e^{-0.0315t^{0.7976}} \tag{5a}$$

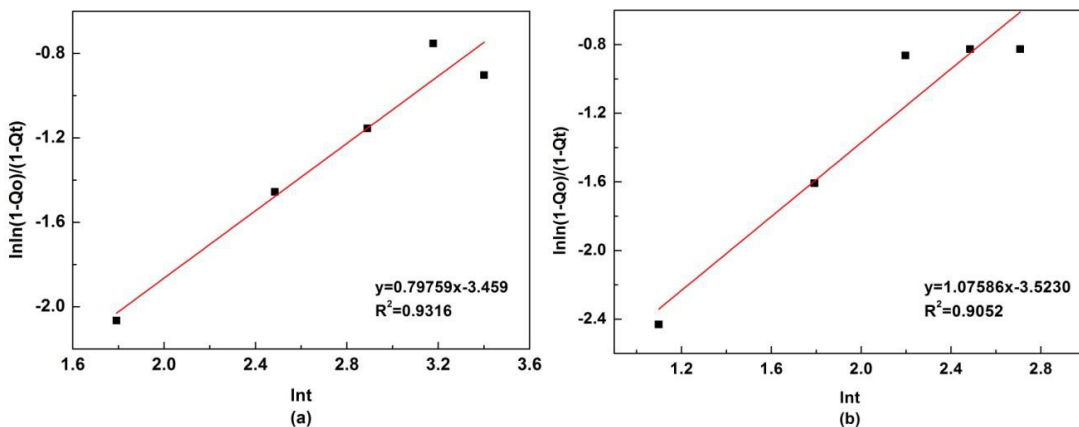$$Q_t = 1 - (1 - Q_0)e^{-0.0295t^{1.0759}} \tag{5b}$$

Fig.8 Kinetic curves of calcined black talc for (a) grinding 30min without ethanol and (b) grinding 15min with ethanol

The content of particles smaller than 1μm calculated by the kinetic models and obtained by the centrifugal sedimentation analyser are shown in Fig.9. It indicates that the models are well agreement with the experiment results.
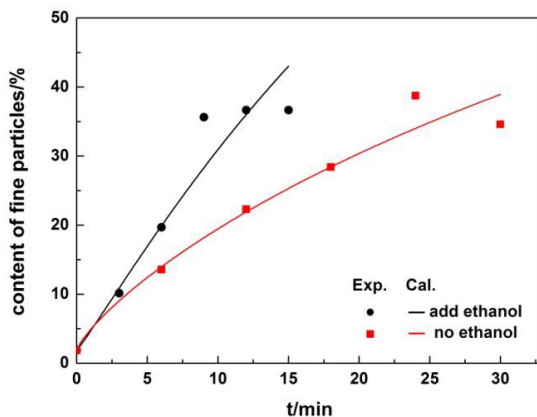

Fig.9 Comparison between calculated and experimental content of particles smaller than 1μm

4. Conclusions

Based on the study on the grinding of calcined black talc with CJXXM planetary ball mill, it can be concluded that using zirconia as grinding media, superior to alumina, the D50 of product can be 1.06μm, after grinding 24min and using ethanol as grinding aid, superior to triethanolamine and sodium hexametaphosphate, the D50 of product can be 1.09μm, after grinding 15min. The SEM and BET results show no agglomerations between fine particles are formed in the product. The XRD results illustrate the (220) is the most unstable crystal plane. Kinetic models of

calcined black talc in a planetary mill under dry superfine grinding conditions using zirconia as grinding media with and without ethanol can be expressed as $Q_t = 1 - (1-Q_0)e^{-0.0315t^{0.7976}}$ and $Q_t = 1 - (1-Q_0)e^{-0.0295t^{1.0759}}$ respectively.

Acknowledgements

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