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PHYSICAL PURIFICATION OF DIATOMITE BASED ON LAMINAR-FLOW CENTRIFUGAL SEPARATION

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Abstract. **N**atural diatomaceous earth or diatomite of good quality becomes rare and a demand for it increases every year. In this paper, we develop a novel method based on laminar-flow centrifugal separation to purify mid- and low-grade diatomite for industrial production purposes. Effects of the drum cone angle, drum speed, feeding concentration, feeding flow rate and feeding time on separation were investigated experimentally. The interdependency of these variables was studied using a response surface experiment. Operating conditions of a laminar-flow centrifugal separator were further optimized. Results showed that the feeding flow rate had a great influence on a silicon dioxide content of diatomaceous in concentrate and tailing. The optimal separation results were achieved as 87.5 wt.%, of SiO_2 content of diatomaceous in concentrate and 6.98 wt.% in tailing. The optimal operating conditions included the drum cone angle of 0.0087 rad, the drum speed of 89.62 rad/s, the feeding concentration of 24.66 wt.%, the feeding flow rate of 2.33×10^{-4} m^3/s, and the feeding time of 90 s. A scanning electron microscopy (SEM) clearly indicated that the pore blockage on the surface of diatoms was cleared out. The variation about the breakage rate of diatom shells was lower than 5% through the pilot purification production line. The characterization of original diatomite and derived products after purification were determined by X-ray diffraction (XRD). The results indicated that the impurity content of purified diatomite was improved significantly.

Keywords: *diatomite, physical purification, laminar-flow centrifugal separation, response surface experiment*

Introduction

Diatomaceous earth or diatomite is a powder and a non-metallic mineral. The main components are diatoms, which are very small (generally < 100 μm) algae. Many diatoms form colonies, which are deposited in the bottom sediments, along with clay, quartz and other minerals. Diatomite is formed over hundreds of millions of years of accumulation and the geological transition of the mixed minerals. Skeletal diatomite has special physical and chemical characteristics, namely a large specific surface area

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and high adsorption capacity. A microscopy analysis reveals the presence of numerous fine microscopic pores, cavities and channels (Martinovic et al., 2006; Ibrahim and Selim, 2011, 2012). The unique cell-wall structure of diatom means that diatomite is widely used in the fields of chemical engineering, environmental engineering, constructional material, agriculture and industrial filtration (Zheng, 2009; Koyuncu, 2012). The demand for this nonrenewable resource increases annually with a rapid industrial development. Though diatomite resources in China are rich, natural and high grade diatomite mines are becoming exhausted. Applications for mid- and low-grade diatomite are limited because of the impurities in the mineral. Beneficiation of diatomite is necessary to enrich the diatomaceous shell and eliminate the clay, quartz, metal oxide minerals and organic matter from a run-of-mine. However, the special microscopic structure of diatom makes it difficult to purify diatomite.

There are many methods to purify diatomite, which include dry classification, wet gravity preparation, acid leaching, magnetic separation, hot flotation and calcinations. The complexity of the flow sheet and effects of purification depend on the planned application of diatomite. Dry classification involves drying a crude ore, crushing it and then putting it through several stages of cyclone classification (Zheng, 1994). An overflow of the cyclone is tailings and an underflow is diatom and a coarse sand. Further classification produces a diatomite concentrate with different quality. Wet gravity preparation involves completely dispersing diatoms and the impurities after scrubbing the mixture of diatomite and water, and subsequent stratifying the diatomaceous slurry by natural gravity settling in a slot-type pool (Zheng, 1994; Wang and Wang, 1995; Zheng et al., 1997; Meng, 1999; Shi et al., 2001; Martinovic et al., 2006; Mohamed, 2009). The coarser material that settles quickly is the diatomite concentrate and the suspended particles in the slurry are clay minerals. Acid leaching involves mixing and agitating a hydrofluoric and sulfuric acid solution with the diatomaceous raw earth (or concentrate processed by wet gravity preparation) (Wang, 1980; Yang and Zhang, 1991; Wang et al., 1995; Li et al., 1997; Qiu et al., 2001; Franca et al., 2003; Gu, 2003; Wang et al., 2006; Zheng et al., 2006; Osman et al., 2009). The impurities such as aluminum oxide (Al_2O_3), ferric oxide (Fe_2O_3), calcium oxide (CaO) and magnesium oxide (MgO) became soluble sulfate. The remaining insoluble component is siliceous diatomite. A high-grade diatomite concentrate is obtained through filtration, washing and drying the insoluble component. The disadvantages of acid leaching are high cost and the production of large quantities of waste acid. Diatomite with high iron content can be purified by magnetic separation (Videnov et al., 1993; Ma et al., 2012). A hot flotation method introduces steam into the scrubbed diatomite slurry with a concentration of 17–18 wt.%, while maintaining the slurry temperature between ~333–343 K (Shi and Zhang, 1993). The diatomite remains and impurities are stratified under these conditions. Diatomite is calcined at ~873–1073 K to vaporize the organics from the raw earth (Wang et al., 2006). Integral flow sheets, which are a combination of the methods mentioned above, are applied to further purification of diatomite (Shi and Zhang, 1993; Ding et al., 1995; Cui et al., 1996; Yuan, 2001; Gu et al., 2003; Wang et al., 2006; Zheng et al., 2006).

Among methods mentioned above, wet gravity preparation is the simplest, most environmentally friendly and most effective. However, at present the industrial application of this method relies on natural gravity settling in slot-type pools to separate diatoms and impurities, which has associated problems of low efficiency and productivity, high labor intensity and unstable product quality. To overcome these problems, we submit the method of laminar-flow centrifugal separation to process mid- and low-grade diatomite. Based upon the differences in particle size and hardness between diatoms and impurities, the run-of-mine is scrubbed by adding water and a dispersant agent at set proportions to increase the dispersion difference between diatoms and the minerals (e.g., montmorillonite) in the water, and to stratify the constituents of diatomite. A screening process is implemented to separate the coarse quartz, feldspar and metal oxide minerals. The undersize slurry enters the laminar-flow centrifugal separator to separate the diatom shells from impurities. The sedimentary deposit is the diatomite concentrate, whereas the overflow (i.e. fine-grade component) is the clay minerals (Zheng, 1994; Zheng et al., 1997). Laminar-flow centrifugal separation is promising as an improved method to purify diatomite since a centrifugal acceleration is hundreds of times greater than a gravity acceleration. In this paper, we study the influence of process and structure parameters of the laminar-flow centrifugal separator on the characteristics of the purified diatomite concentrate. Our goal is to determine the optimal operating condition and operational mode of the separator.

Materials and methods

Materials

The run-of-mine was obtained from the disused diatomite mine in Liudaogou, Linjiang City, China. Table 1 lists the main chemical composition of diatomite. The silicon dioxide (SiO_2) content in the run-of-mine is 80.17 wt.%. It is the typical clayey second-grade diatomite. A diatom morphology was examined with a SEM S-3500N scanning electronic microscope (SEM), as shown in Fig. 1. The diatom types present according to the surface shape as shown in Fig. 1 are mainly disc diatoms, as well as a small number of cymbiform diatoms, straight chain diatoms, and debris from striated diatoms. The shape of diatom is not perfect, with small and broken diatom shells attached on the surface. Complete diatom frustules (cell walls) measure ~20–50 μm in diameter.

Experiment diagram

Figure 2 shows a pilot-scale experimental technical flow sheet of the laminar-flow centrifugal separation. In Figure 2, number 1 indicates a separator specially designed for purifying the diatomaceous earth. The drum end with a short diameter is connected with the base plate, which is fixed on the main axis. The feeding pipe extends into the drum. The separated products discharge from the large end of the drum, with a diameter of 0.8 m. The inclined length of drum's inner wall is 0.6 m. The maximum speed of the drum is 157 rad/s. The actual speed can be changed on-line. The maximum separation factor is 1000, which is computed by dividing the centrifugal acceleration by the gravity acceleration. The panel in the slurry current direction changing box (6 or 7) is driven by an electromagnet operated by the controller, to fulfill the conversion of the slurry flowing direction.

Table 1. Chemical composition of raw diatomite from Liudaogou, Linjiang City, China

Components	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	K_2O	Na_2O	Loss-on-ignition
Content (wt.%)	80.17	5.72	2.48	0.31	0.35	0.64	0.96	0.39	8.31

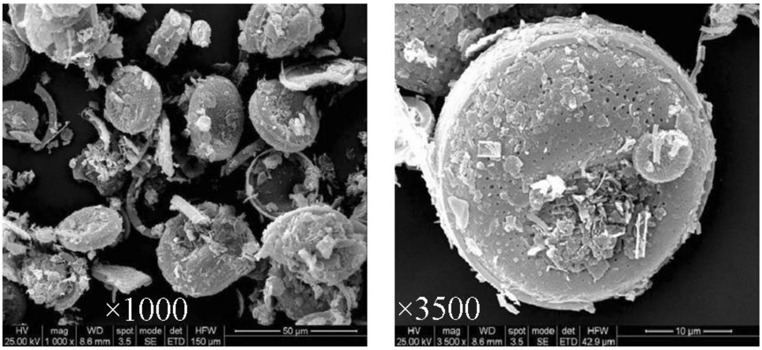

Fig. 1. SEM of the raw diatomite

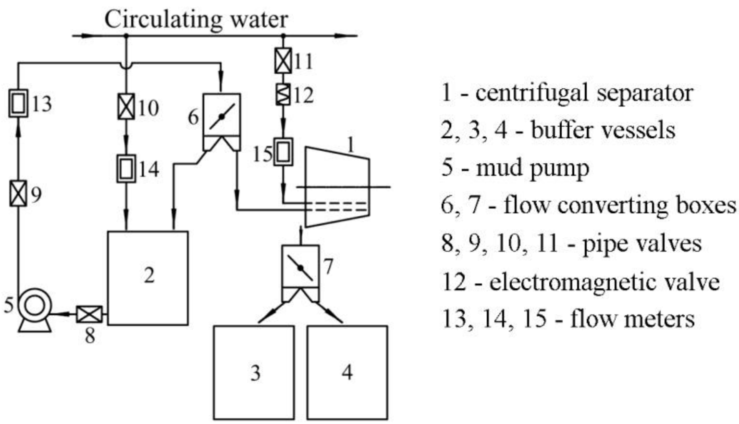

Fig. 2. Pilot-scale experiment flow sheet

The working mode of the laminar-flow centrifugal separator includes four stages: feeding, interval, washing and resting. The raw slurry in the buffer vessel 2 in Fig. 2 is carried by the mud pump 5 to the slurry current direction changing box 6. The panel is set at the zero position, so the slurry flows back to the buffer vessel 2. The feeding stage starts after the drum speed reaches the target speed, at which time the panel changes to the opposite position. Now, the slurry flows into the laminar-flow centrifugal separator. The diatomaceous concentrate deposits on the inner wall of the drum because of the centrifugal force. The tailings flow out of the separator and enter the tailings buffer vessel 4 through the slurry current direction changing box 7. After time t_1 the process reaches the interval stage and the panel in the slurry current direction changing box 6 is reset to the zero position. This stage lasts time t_2 to ensure the slurry in the feeding pipe flows into the separator and the tailings in the separator flow into tailings buffer vessel 4. Now, the washing stage occurs. The panel in the slurry current changing box 7 moves to the opposite position. The electromagnet 12 turns on to open the circulating water. The diatomaceous concentrate on the inner wall of the drum is washed off, and enters into concentrate buffer vessel 3. The electromagnet 12 for washing water is turned off after time t_3. After waiting for time t_4, the panel in the slurry current changing box 7 is reset to the zero position. Then, the next separation cycle commences.

Content analysis

A multi-element express analyzer DHF82 was used to analyze the chemical composition, mainly SiO_2, Fe_2O_3 and Al_2O_3 contents of raw diatomite and purified concentrates. The crystalline phase identification of samples was undertaken by a PANalytical X'Pert PRO X-ray diffractometer employing CuKα radiation at a goniometer rate of 2θ= 4°/min. It is common to choose SiO_2 content as the criterion to evaluate the quality grade of diatomite. The content of SiO_2 for the first class diatomite is over 85 wt.%. High-grade diatomite contains a minimum of SiO_2 content about 95 wt.%. The change in the surface morphology for the separated diatomite in contrast with the raw ore was observed by SEM. The breakage rate of the diatom shell through physical purification was quantified as the total diatom numbers 500 divided by the broken diatom numbers. Counts were performed with the JX-2000 image analyzer in the best observation field. The more complete the diatom shell construction, the more valuable diatomite for high-end and specialized field applications.

Results and discussion

Effects of drum speed and cone angel on purification

Figure 3 shows the changes in product concentration of the laminar-flow centrifugal separator with feeding time (75 s, 90 s) and cone angle (0.07 rad, 0.0087 rad) at a fixed feeding concentration of 24 wt.%, feeding flow rate of $2 \cdot 10^{-4}$ m^3/s, washing water pressure of $4 \cdot 10^5$ Pa, washing water flow rate of $5 \cdot 10^{-4}$ m^3/s and washing time of 20 s. From Figure 3 it can be seen that when the cone angle is 0.07 rad, the diatomite concentration in tailing is > 8.5 wt.% (Fig. 3 a) and for the concentrate it is less than 4.0 wt.% (Fig. 3 b), which does not increase even with prolonged feeding time. However, when the cone angle is changed to 0.0087 rad, the concentrate concentration clearly increases at the same drum speed. It reaches 7.0 wt.% even at a lower drum speed of 83.73 rad/s (Fig. 3 b). The cone angle of drum determines the inner surface area, inclination length and particle movement route. This cone angle affects the axial force and acceleration of particles, as well as the moving speed of diatomite slurry in the radial and axial direction, and motion track. The larger the cone angle, the faster the slurry passes through. Therefore, at this cone angle some part of the slurry directly flows out of the drum without separation and moves into the tailings. This condition leads to a thinner deposit on the inner wall in the laminar-flow centrifugal separator. When the cone angle is 0.07 rad, the drum speed and the feeding time have less influence on the deposit thickness. This phenomenon was verified by examining the inner wall of the drum after the separator was stopped. Under the same working conditions, the concentrate recovery should be low and the separation effect should be worse if the concentration of diatomite concentrate is low. The most diatomaceous concentrate flowed with the overflow into the tailings. Therefore, the subsequent experiment was carried out under a cone angle of 0.0087 rad.

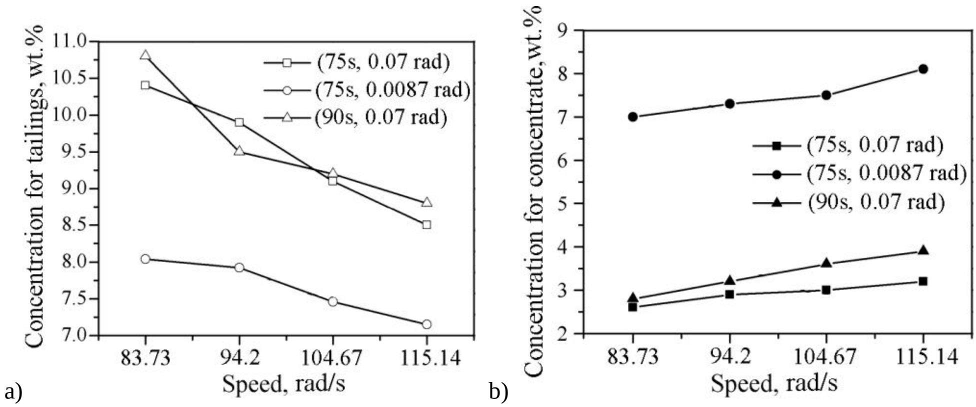

Fig. 3. Influence of cone angle and drum speed on laminar-flow centrifugal separation

Single factor experimentation

The experiments compare the separation effects of the laminar-flow centrifugal separator at different drum speeds. The tests were carried out with the cone angle at 0.0087 rad, raw slurry concentration at 24 wt.%, feeding flow rate at $2 \cdot 10^{-4}$ m^3/s, feeding time at t_1 = 75 s, interval time at t_2 = 6 s, washing time at t_3 = 22 s, resting time at t_4 = 6 s, washing water pressure at $4 \cdot 10^5$ Pa, and washing water flow rate at

$5 \cdot 10^{-4}$ m^3/s. The results are shown in Figs. 4(a) and 4(b). With the increase in drum speed, the SiO_2 content in the diatomaceous concentrate also increases, but the concentration in tailings decreases. It shows that the centrifugal force of the particle depends on the drum speed. The separation factor increases as the drum speed increases, which speeds up the settlement of the particles. However, the separating effects of the diatomaceous slurry are also influenced by particle size and particle density. The drum speed of 31.42–52.36 rad/s is enough to separate the fine metallic minerals. However, since the apparent density for diatomite is 0.58–0.65 × 10^3 kg/m^3, far less than that of fine metallic minerals (Zheng, 2009), the separation of diatomite needs a higher drum speed in laminar-flow centrifugal separation.

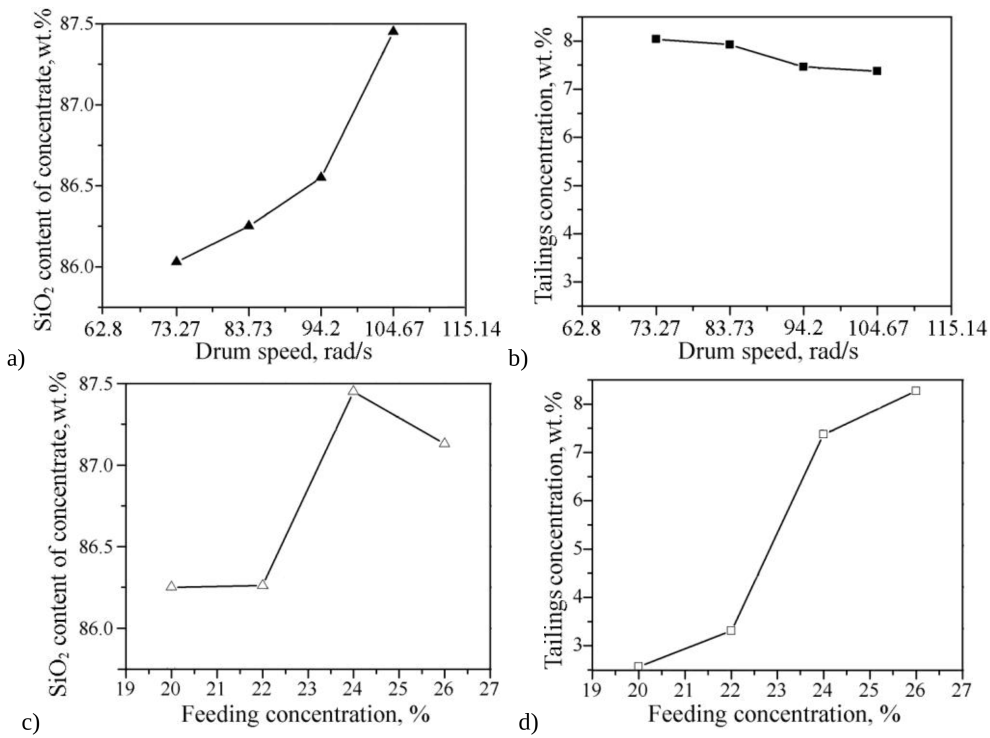

Fig. 4. Influence of drum speed and feeding concentration on laminar-flow centrifugal separation

In this paper we study the influence of different feeding concentrations on the separating effect based on the experimental conditions discussed above (Figs. 4 (c) and (d)). The results indicate that with the increase in the feeding concentration, the SiO_2 content of concentrate first increases and then decreases when the feeding concentration surpasses 24 wt.%. However, the tailings concentration increases with the feeding concentration. If the slurry concentration increases, the fluidity degrades and the viscosity increases. Further, stratification becomes slow. Therefore, the processing capacity increased, but part of deposited concentrate flowed out, which led to a decrease in the SiO_2 content.

Figure 5 shows the relationship between SiO_2 content in concentrate and tailings concentration with different feeding times and feeding flow rates. If the feeding time is prolonged from 45 s to 60 s, the SiO_2 content of concentrate increases by 2.17 wt.% (Fig. 5 a). However, the increase is very small with further prolonging the feeding time. The tailings concentration also increases with the feeding time and then remains stable (Fig. 5 b). This result shows that it is helpful to purify diatomite by increasing the feeding time to a certain degree. Once the deposit thickness on the inner wall reaches a certain extent, the protracted feeding time only increases the processing capacity and does not improve the SiO_2 content in concentrate. In addition, the experiments show that the tailings concentration increases with the increase in feeding flow rate (Fig. 5 c). However the SiO_2 content in concentrate increases with the increase of the feeding flow rate, and then decreases after the feeding flow rate reaches $2\cdot10^{-4}$ m^3/s (Fig. 5 d). It shows that too high a feeding flow rate will lead to diatom loss under this separation condition.

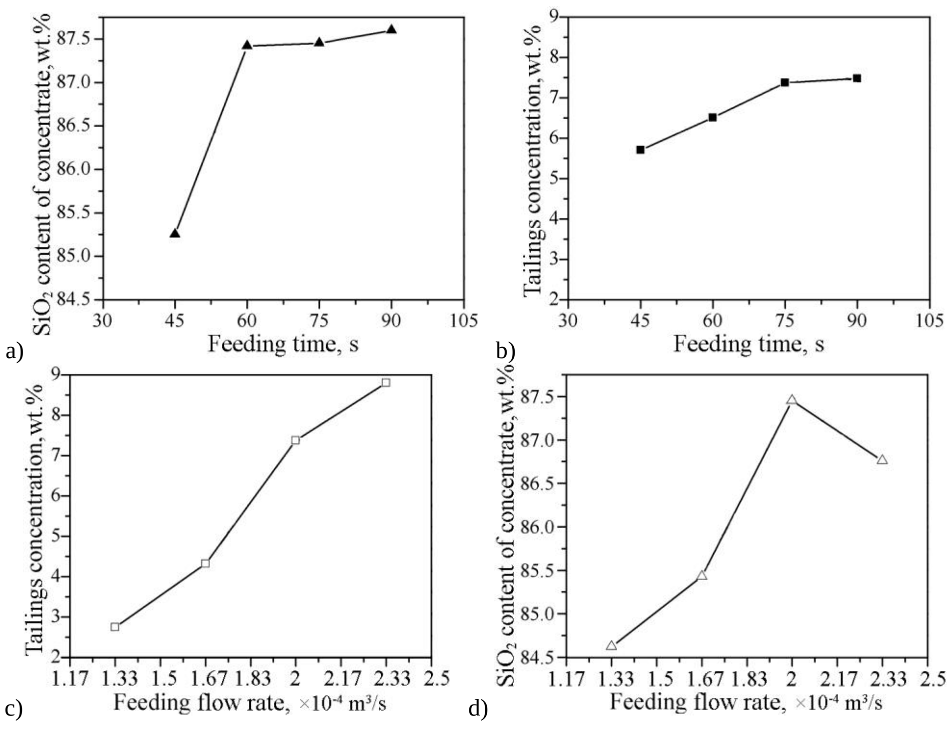

Fig. 5. Influence of feeding time and feeding flow rate on laminar-flow centrifugal separation

Response surface experiment

In order to search for the optimal separation condition of the laminar-flow centrifugal separator, a 4-factor × 3-level response surface experiment, including three central points, was designed to study the influence of drum speed, feeding concentration, feeding flow rate and feeding time on the separating effects based on the single factor experimentation. Table 2 lists the factor and level parameters while Table 3 presents the response surface design and results. The linear model analysis of the standard least squares fitting showed that the influential sequence of the technical parameters on the silicon dioxide content of concentrate from high to low was: feeding flow rate, feeding concentration, drum speed, and feeding time. The feeding flow rate had the most significant influence. The influential sequence for the tailings concentration from high to low was: feeding concentration, feeding flow rate, feeding time, and drum speed. From all these parameters, the feeding concentration and the feeding flow rate had the most significant influence. Therefore, the feeding flow rate had the strong influence on both the silicon dioxide content of concentrate and the tailings concentration.

Table 2. Factors and their levels

Levels	Feeding concentration (wt.%)	Drum speed (rad/s)	Flow rate ($\times 10^{-4}$ m^3/s)	Feeding time (s)
1	22	83.73	1.67	60
2	24	94.20	2.0	75
3	26	104.67	2.33	90

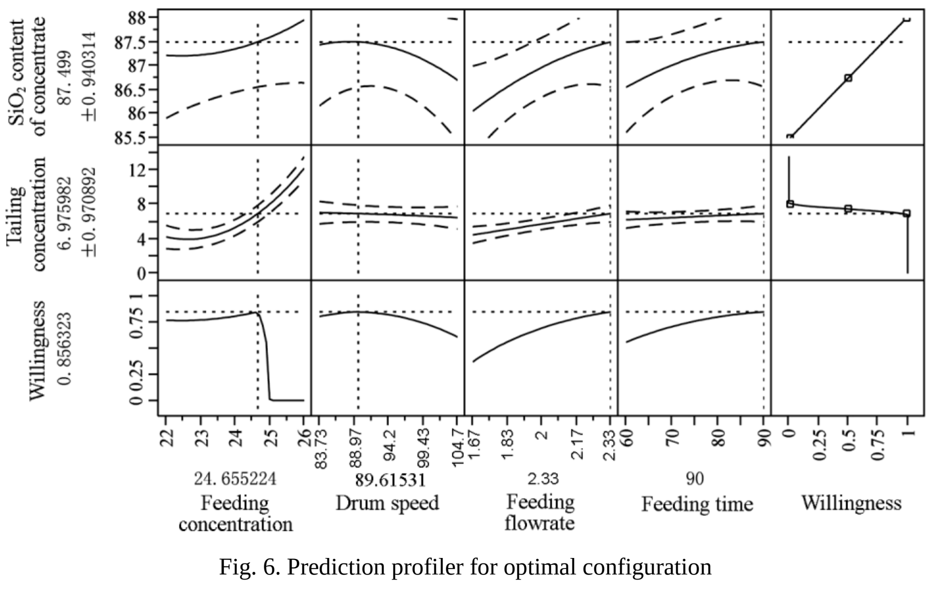

Fig. 6. Prediction profiler for optimal configuration

Table 3. Response surface design and results

No.	Feeding concentration (wt.%)	Drum speed (rad/s)	Flow rate ($\times10^{-4}$ m^3/s)	Feeding time (s)	SiO_2 content of concentrate (wt.%)	Tailings concentration (wt.%)
1	22	83.73	2.0	75	86.21	3.22
2	22	94.20	1.67	75	86.18	2.0
3	22	94.20	2.0	60	86.11	2.75
4	22	94.20	2.0	90	86.55	3.23
5	22	94.20	2.33	75	87.58	4.42
6	22	104.67	2.0	75	87.29	3.11
7	24	83.73	1.67	75	85.98	2.62
8	24	83.73	2.0	60	85.67	3.79
9	24	83.73	2.0	90	85.92	4.61
10	24	83.73	2.33	75	86.93	4.63
11	24	94.20	1.67	60	86.64	2.11
12	24	94.20	1.67	90	86.17	2.81
13	24	94.20	2.0	75	87.49	3.71
14	24	94.20	2.0	75	86.84	4.08
15	24	94.20	2.0	75	86.55	3.48
16	24	94.20	2.33	60	86.64	4.15
17	24	94.20	2.33	90	87.29	4.93
18	24	104.67	1.67	75	86.42	2.59
19	24	104.67	2.0	60	87.51	2.96
20	24	104.67	2.0	90	86.71	4.01
21	24	104.67	2.33	75	86.17	5.17
22	26	83.73	2.0	75	87.13	10.78
23	26	94.20	1.67	75	86.63	8.68
24	26	94.20	2.0	60	87.28	10.17
25	26	94.20	2.0	90	87.93	10.7
26	26	94.20	2.33	75	87.57	12.03
27	26	104.67	2.0	75	87.13	8.27

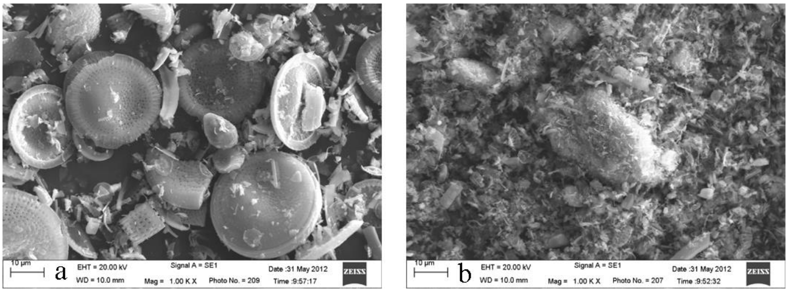

Fig. 7. SEM comparison of the diatomite purification (a: concentrate, b: tailings)

The experimental goal was to find the optimal separation condition to purify diatomite with the laminar-flow centrifugal separator. The requirement is that the SiO_2 content in concentrate is greater than 85 wt.% (the first-grade earth standard) and the tailings concentration is not too low. A low tailings concentration indicates that some clay minerals were deposited on the inner wall of the drum due to centrifugal force, and then entered into the diatomaceous concentrate resulting in decreasing the concentrate grade. Therefore, these two factors should be considered at the same time during the evaluation. The optimal parameter combination was obtained by the model prediction profiler according to the maximum willingness (Fig. 6), that is the feeding concentration 24.66 wt.%, drum speed 89.62 rad/s, feeding flow rate $2.33\cdot10^{-4}$ m^3/s, and feeding time 90 s. The predicted results were: SiO_2 content of 87.5 ± 0.94 wt.% and tailings concentration of 6.98±0.97 wt.%.

A continuous running experiment was carried out based on the optimal combination above. The results were: SiO_2 content = 87.41 wt.%, Al_2O_3 content = 3.81 wt.%, Fe_2O_3 content = 1.64 wt.%, and tailings concentration = 6.14 wt.%. The SEM results from the diatomaceous concentrate and the tailings through beneficiating are shown in Fig. 7. The pore blockages in the diatom frustules are obviously improved by laminar-flow centrifugal separation.

The analysis with a graphic granulometer showed that the breakage rate for the diatomaceous raw mineral was 67.4%, for the feeding raw diatomite 69.4%, and for the separated diatomaceous concentrate 63.5%. The breakage rate of diatoms after being scrubbed increased by 1.9%, but it decreased by 5.8% after separation with the laminar-flow centrifugal separator. This probably was due to broken diatoms entering the overflow and becoming the tailings, because their diameter was close to that of clayey minerals. Therefore, it can be seen that the diatomaceous raw minerals themselves were fragmentary. The breakage rates had no prominent change after physical separation, but the breakage rate of the diatomaceous concentrate decreased when compared with raw diatomite. The destruction to the diatom frustule was very small by using the physical preparation method reported in this paper.

Mineralogical analysis

X-ray diffraction (XRD) is one of the most useful techniques to study the structural geometry and texture of impurities in diatomite. The XRD patterns of raw diatomite before purification and different products (oversized product, centrifugal overflow and centrifugal sediment) obtained after purification are given in Fig. 8. It shows an essentially amorphous phase, but a significant amount of crystalline phases is also found in raw diatomite. The XRD pattern shows the high content of amorphous SiO_2. The main impurities in the raw sample can be divided into two types. One type is clay minerals such as montmorillite, kaolin and muscovite, and the second type is crystalline silicious sands, such as quartz and feldspar. The main mineral composition of the oversized product is coarse feldspar and quartz, which agrees with our expectations. The typical XRD patterns of clay minerals such as montmorillite, kaolin and muscovite appear in the centrifugal overflow, determined as the tailings. The purified powder sample exhibits less crystalline structure after purification. From Figure 8, it can be seen that a high-grade amphorous silica material with less crystalline structure is obtained after purification.

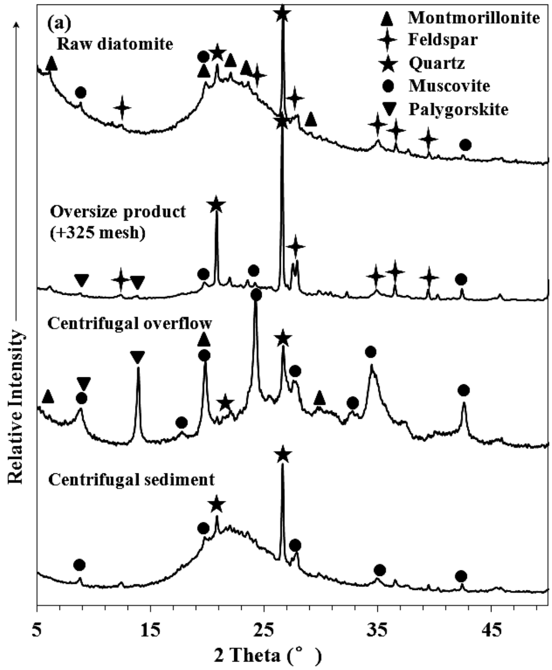

Fig. 8. XRD patterns of raw diatomite and different products obtained after purification

Conclusions

High grade diatomite can be successfully prepared from mid- and low-grade raw diatomite through physical purification by the laminar-flow centrifugal separator. The following salient conclusions related to centrifugal settling treatment and preparation of diatomite powders can be advanced.

A single factor experimentation shows that the SiO_2 content increases with increasing the drum speed, feeding concentration and feeding flow rate. However, SiO_2 content decreases when the feeding concentration exceeds 24 wt.% or the feeding flow rate is faster than $2 \cdot 10^{-4}$ m^3/s.

A response surface experiment indicates that the influential sequence of technical parameters on the SiO_2 content in concentrate is feeding flow rate > feeding concentration > drum speed > feeding time. The feeding flow rate has the most significant influence. The influential sequence for the tailings concentration is feeding concentration > feeding flow rate > feeding time > drum speed. From all these parameters, the feeding concentration and the feeding flow rate have the most significant influence.

The optimal parameter combination for diatomite separation was obtained by the model prediction profiler according to maximum willingness. The optimal combination was the feeding concentration 24.66 wt.%, drum speed 89.62 rad/s, feeding flow rate $2.33\cdot10^{-4}$ m^3/s and feeding time 90 s. The predicted results were: SiO_2 content = 87.5 ± 0.94 wt.% and tailings concentration = 6.98 ± 0.97 wt.%.

The feasibility of this method was demonstrated by the pilot-scale continuous production, based on the optimal parameter combination above. The SiO_2 content in concentrate was 87.41 wt.%. The SEM analysis of the diatomaceous concentrate indicated that the pore blockages in the diatom shell were clearly improved by laminar-flow centrifugal separation. Graphic granulometry indicated that the breakage rate was not markedly changed using this method.

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