A novel method for purification of low grade diatomite powders in centrifugal fields

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Abstract.
This study presented a novel method for purification of three different grades diatomite from China by scrubbing technique using sodium hexametaphosphate (SHMP) as dispersant combined with centrifugation. Effects of pH value and dispersant amount on the grade of purified diatomite were studied and the optimum experimental conditions were obtained. The characterizations of original diatomite and derived products after purification were determined by scanning electron microscopy (SEM), X-ray diffraction (XRD), infrared spectroscopy (IR) and specific surface area analyzer (BET). The results indicated that the pore size distribution, impurity content and bulk density of purified diatomite were improved significantly. The dispersive effect of pH and SHMP on the separation of diatomite from clay minerals was discussed systematically through zeta potential test. Additionally, a possible purification mechanism was proposed in the light of the obtained experimental results.

Keywords: Diatomite; Centrifugal separation; Purification; Sodium hexametaphosphate

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1. Introduction

Diatomite or diatomaceous earth is a non-metallic material composed mainly of a wide variety of shape and sized skeletal remains of diatoms [1, 2]. Because of highly porous structure, low density, high absorption capacity, low thermal conductivity, chemical inertness, relatively low price and high abundance, diatomite can be used extensively as filtration media, adsorbents, conventional catalyst supports [3-8]. It can also be applied in manufacturing of certain membranes and in the separation of biocatalytic proteins and cells [9]. China has substantial diatomite resources, most of which are of moderate quality, having a SiO$_2$ content of about 60 %-80 %. There are a lot of impurities such as alkaline and alkaline earth metals and some organic substance, which decrease the microporosity and limit the use of diatomite. Hence, raw diatomite needs to be industrially purified before its commercial utilization. Several approaches have been reported, such as acid leaching, classification, crushing, gravitational sedimentation, calcinations and ultrasonic treatment [9-13]. Chemical treatments, such as acid treatment, can significantly improve the quality of raw diatomite. However, waste water generated after acid leaching is very difficult to deal with, which causes severe environmental problems and great operative expenses. Other beneficiation processes have some other weaknesses, such as inefficiency, high processing cost, inadequate effect, fusion problem and destruction of diatom structure. The advantages and disadvantages of different diatomite purification techniques are concluded in Table 1. Among these methods, scrubbing technique has been preferred due to its cheapness and the high-efficiency of the low grade diatomite purification especially for pretreatment. However, there are few industrial achievements on diatomite purification now.

High speed scrubbing technique is applied to make the impurities coated on the surface of raw diatomite separated from the diatom units initially. Agitator scrubbing machine is a kind of conventional dressing equipment, which has an extensive application in mineral processing. Centrifugal separation is one of the technically and economically acceptable solutions for the treatment of waste water, flotation slurries and chemical industry [14-16]. The advantage of centrifugal separators lines in the fact that the settling velocity is much higher than that of gravitational separators, which results in smaller separator dimensions, higher separation efficiency and precision. Because of small particle size of impurities and diatomite, the centrifugal separation can shorten the particles settling time, which can ensure the separation process efficiently and continuously.

Some kinds of dispersants, such as sodium hexametaphosphate and sodium pyrophosphate, are
employed during the scrubbing process. Uniform and relatively stable dispersion of diatomite and its impurities in the scrubbing suspension is the premise of effective centrifugal separation. The sodium hexametaphosphate (SHMP) is widely used in mineral processing as floatation regulator and dispersant [17]. The influence and mechanism of SHMP on the floatation and dispersion of different minerals has been studied by many researchers [18-19]. However, the relationship between the settling property and the dispersion of diatomite and its impurities has not been systematically studied before.

In the light of these, three natural diatomite powders, selected from different regions of China, were purified by scrubbing technique combined with centrifugal separation using sodium hexametaphosphate as the dispersant. It is the aim of the investigation to show whether different grades of raw diatomite can be effectively purified through this method. Effects of pH value and dispersant amount on the grade of purified diatomite were discussed. The influence of pH and sodium hexametaphosphate on the dispersion of the diatomite and clay minerals was systematically studied based on the zeta potential analysis of pure materials, and the possible purification mechanism was further proposed.

2. Materials and methods

2.1. Materials

Three typical natural original diatomite powders were obtained from Linjiang of Jilin province (LJ-DE), Huade of Inner Mongolia (HD-DE), and Shengxian of Zhejiang province (SX-DE) in China respectively, respectively. Sodium hexametaphosphate and sodium hydroxide were purchased from Beijing Chemical Reagent Company (Beijing, China). The chemical compositions of the raw diatomite are listed in Table 2. As shown in Table 2, the main component of raw diatomite is SiO₂. Ferric oxide and alumina are the main impurities in raw diatomite. Based on the SiO₂ content, the grade of three diatomites decreases in the following order: LJ-DE > HD-DE > SX-DE. The pure minerals for zeta potential analysis were purchased from NCS Testing Technology Co., Ltd. (Beijing, China) without any further purification. The chemical compositions of the pure minerals were summarized in Table 2. The microstructure of the pure diatomite is displayed in Fig. 1. It is clear that the purity of the diatomite sample was very high from the chemical compositions analysis and SEM pictures.

2.2. Purifying methods
The flowsheet for the processing of raw diatomite is given in Fig. 2. As shown in Fig. 2, the purification was undertaken by the following procedure: 100 g of raw diatomite samples was initially dispersed in 400 mL of water with a laboratory-scale model XFD12 agitator scrubbing machine for about 25 min with a stirring rate of 1500 rpm. A certain amount of sodium hydroxide was pre-dissolved in 20 mL of deionised water and then it was slowly added to the scrubbing suspension at room temperature in order to adjust the pH value of the suspension. The suspension was stirred for further 10 min, and then the selected amount of sodium hexametaphosphate was added to the suspension. After stirring for a further 5 min, the suspension was collected and sieved using a 325 mesh standard screen (45 μm). After sieving, the oversized products were collected and dried in an oven at 105 °C for 12h. The obtained sample was labeled as oversized product. The undersized suspension was then transferred to a 100 mL centrifuged tube. After shaking, the mixture was centrifuged at 1500 rpm (RCF (relative centrifugal force) = 300) for 15 min. After the centrifugation, the centrifugal sediment was obtained and then dried at 105 °C for 12 h. The centrifugal overflow was filtrated by vacuum filter and washed with distilled water 2-3 times and then dried at 105 °C for 12h.

2.3. Characterization

The chemical compositions of raw diatomite and purified diatomite are determined by X-ray fluorescence (SPECTRO X-LAB2000). 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. Morphology of samples was examined by a scanning electron microscopy (S-3500N, FEI Company). Infrared emission spectroscopy was carried out on a Nicolet Nexus 870 FTIR spectrometer, which was modified by replacing the IR source with an emission cell. Spectral manipulation such as baseline adjustment, smoothing and normalization was performed using the Spectra calc software package (Galactic Industries Corporation, NH, USA). Surface area analyses based upon N₂ adsorption/desorption were carried out with a Micromeritics Tristar 3000 automated gas adsorption. From the measurement, pore structure parameters were also characterized at liquid nitrogen temperature. The samples were pre-heated at 105 °C under the flow of N₂ on a Micrometrics Flowprep 060 degasser before the measurement.

2.4. Zeta potential measurement
A certain amount of pure diatomite, kaolin and montmorillite was added into distilled water firstly. The suspension was stirred for 15 min using a magnetic stirring apparatus before the test. At different pH values, zeta potential of different samples was measured in a Nano ZS90 type ζ-potential analyzer (Malvern, Britain). The pH value of the solution was automatically adjusted by adding HCl or NaOH in range of 2–12.

3. Results and discussion

3.1 Characterizations of purified diatomite

3.1.1 Chemical analysis

The effect of pH values of the scrubbing suspension on SiO₂ content of purified diatomite was examined through a series of centrifugal separation experiments while other variable kept constant. The dosage of SHMP keeps 3 wt. ‰. Changes of the SiO₂ content of the purified diatomite at different pH are given in Fig. 3. It is clear that the SiO₂ content of purified diatomite was enhanced with the increase in pH. The SiO₂ content of purified diatomite reached the highest values when the pH was up to 10, so the optimum pH value is determined as 10. It can be confirmed that a high-grade silica material, more than 87 wt. % SiO₂ was obtained when pH was 10 for the purification of LJ-DE and HD-DE. On the other hand, the removal of the impurities for SX-DE was more difficult compared with the other two diatomites, and the SiO₂ content of the purified diatomite was just increased to 74.01 wt. % when pH was 10.

The dosage of SHMP is another key factor on the grade of purified diatomite and its influence is shown in Fig. 4. The pH values of the scrubbing suspension were kept 10. From the Fig. 4, the SiO₂ content of purified diatomite was increased with the increase in the dosage of SHMP. As for LJ-DE, when the dosage of SHMP was up to 2 wt. ‰, the grade of purified diatomite reached the highest value. As for HD-DE and SX-DE, the optimum SHMP dosages are determined as 3.5 wt. ‰ and 3 wt. ‰, respectively. The difference of optimum SHMP dosage might be caused by the different contents and forms of impurities in three raw diatomites. The yield of the purified diatomite for LJ-DE, HD-DE and SX-DE are 81.37%, 64.21% and 77.28% respectively under the optimum experimental conditions.

3.1.2 Mineralogical analysis

X-ray diffraction (XRD) is one of the most useful techniques to study the structural geometry and
texture of impurities in diatomites. The XRD patterns of three raw diatomites before purification and different products (oversized product, centrifugal overflow and centrifugal sediment) obtained after purification are presented in Fig. 5. **An essentially amorphous phase was observed in three raw diatomites**, but a significant amount of crystalline phases were also found. Among three raw diatomites, the XRD pattern of LJ-DE showed the highest content of amorphous SiO₂, followed by HD-DE and then SX-DE. The main impurities in raw LJ-DE samples can be divided into two types. One is clay minerals such as montmorillite, kaolin and muscovite, and the other one is crystalline siliceous sands, such as quartz and feldspar. The main mineral compositions of the oversized product are 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. All the purified powder samples exhibit less crystalline structure after purification. From Fig. 5 (a) and (b), it can be seen that a high-grade amphorous silica material with less crystalline structure is obtained after purification. As shown in Fig. 5(c), the reduction of impurities is rather insignificant for SX-DE, indicating that the purification effect of SX-DE is the worst. In addition, the content of clay minerals is the highest for SX-DE among three diatomites. The results indicate the effect of purification might be influenced to a large extent by the content of clay minerals in raw diatomites and the relative higher content of clay minerals in SX-DE results in the undesirable purification effect.

### 3.1.3 Microstructural analysis

Microstructural observations have been applied to identify the success of producing highly-porous diatomite particles and maintaining porous structural integrity of diatom through the applied scrubbing and centrifugation treatment. A selection of SEM images of raw diatomite and purified diatomite from Linjiang are present in Fig. 6. The characteristic disc-shaped structure of diatom particles is clearly shown in raw LJ-DE with the diameter of about 20 μm. Compared with purified diatomite, the raw diatomite exhibits diatomite frustules with closed-pores dispersed in clay and some other fine particles. In addition, integrated morphology of diatom in raw diatomite and purified diatomite is also displayed (Fig. 6 LJ-a and LJ-b). It was found that the pores on the surface of diatoms could be seen more clearly after purification, which might be due to the fact that the impurities were removed from the clogged pores. Moreover, the purified diatomite still kept integrality after purification.
Naturally it is also observed that some impurities have been deposited on the diatomite particles in HD-DE (see Fig. 7 HD-a1 and HD-a2). The diatoms in raw HD-DE are mostly cracked inherently. The diameter of integrated diatom in HD-DE sample is approximately 10 μm, which is smaller than that of LJ-DE sample. There are fewer impurities in purified diatomite (Fig. 7 HD-b1). Fig. 7 HD-b2 indicates undamaged forms of diatom in purified diatomite with extremely clean pores, which demonstrates the approach has a significant improvement on the pore structure of diatomite.

Fig. 8 displays the microstructure of raw SX-DE sample and purified diatomite samples. The presence of cylindrical diatom together with small particles of impurities could be noticed in micrographs of raw SX-DE sample and purified diatomite. Considerable content of small impurities such as quartz, feldspar and clay minerals was detected on the surface of diatomaceous earth and such high content of impurities makes SX-DE greyer than the other two raw diatomite samples. Compared with the undamaged forms of diatomaceous earth in raw SX-DE sample, the outside pore structure on the surface of cylindrical diatom looks cleaner after purification. However, there are still a mass of impurities embedded in the pore of cylindrical diatom. The shearing force under the current scrubbing conditions can not remove such embedded impurities quickly and effectively. The embedded distribution of impurities might be another key factor why the SX-DE sample is harder to be purified than the other two samples.

3.1.4 IR analysis

A comparison of the IR spectra of three raw diatomite samples and purified diatomite are presented in Fig. 9. As for all samples, the IR spectra show two major absorption bands which were observed at about 3430 and 1630 cm\(^{-1}\) [21]. The band at 3430 cm\(^{-1}\) is due to the stretching vibrations of adsorbed water, and the band at 1630 cm\(^{-1}\) represents H-O-H bending vibration of water. The bands at about 1100 cm\(^{-1}\) reflects the asymmetric stretching modes of Si–O–Si bonds and the Si-O-Si symmetric stretching vibration peaks appear at about 800 cm\(^{-1}\) [22, 23]. Compared with the raw LJ-DE and HD-DE sample, the bending modes of Al-O-H at 912 and 914 cm\(^{-1}\) appear in the IR spectrum of centrifugal overflow sample, which indicates that the presents of some clay minerals [24]. The bending vibrations of Al-O-H are observed in all three samples of SX-DE, which means the content of clay minerals are high and the removal effect after purification is not very well, which is in line with the result of the previous SEM analysis.
3.1.5 Surface area measurement

As shown in Fig. 10, the nitrogen adsorption/desorption isotherms of raw diatomite, centrifugal overflow and sediment exhibit a type II sorption behavior in the classification of Brunauer, Deming, Deming and Teller (BDDT) [25]. The specific surface area, pore volume and average pore size of raw diatomites and products after purification are summarized in Table 3. It is clear that the raw diatomite, tailing and purified diatomite have the same form of isotherms and hysteresis loop. The hysteresis loop is associated with the filling and emptying of the mesopores by capillary condensation [26]. The adsorption of $N_2$ of centrifugal overflow is much higher than that of purified diatomite, indicating that the overflow possesses a higher specific surface area. The result is in line with the XRD result since the overflow is mainly composed of clay minerals. Compared with the raw diatomite, purification did not evidently change the shape of the isotherms. However, purification resulted in a dramatic increase in nitrogen adsorption and an enhancement of the hysteresis, indicating the improvement of the microporous structure of diatom. As shown in Table 3, the specific surface areas of purified diatomite powder become smaller than that of raw diatomites. It is easy to understand that the decrease of specific surface areas is caused by the removal of impurities with relative higher specific surface areas. Similar specific surface areas changes were also obtained in previous studies [9, 10, 27, 28].

3.2 Mechanism of purification

The relationship between zeta potential and pH value of pure diatomite, kaolin and montmorillite is presented in Fig. 11. It can be seen that the pH value at the isoelectric point of diatomite, kaolin and montmorillite is about 2.0. The surface of diatomite, kaolin and montmorillite particles shows negative electricity in the pH range of 2-12. In the acidic environment, the absolute value of zeta potential of diatomite, kaolin and montmorillite particles is remarkably lower and aggregation occurs easily among the particles due to the relative lower electrostatic repulsive force [18, 19]. Moreover, with the increase in the concentration of SHMP, it is obvious that sodium hexametaphosphate could lower the zeta potential of diatomite, kaolin and montmorillite. As for diatomite and kaolin, the absolute value of surface charge of diatomite and kaolin kept increasing with increase of pH in the range of 2-12. On the other hand, the surface charge of montmorillite decreased firstly and then increased when pH was up to 9. A similar variation trend for the three
minerals was observed in different concentrations of SHMP. The above results illustrate that the optimum dispersion conditions for diatomite and clay minerals are in the weak alkaline condition at a relative high concentration of SHMP, which is in agreement with the previous obtained optimum purification conditions.

Based on the above results, the mechanism of purification can be comprehensively concluded as follows: Under the high speed shear force, the impurities were taken out from the surface and the porous structure of raw diatomite. Due to the high electrostatic repulsive force, uniform and relatively stable dispersion of diatomite and impurities in the scrubbing suspension can be obtained in the weak alkaline condition at a relative high concentration of SHMP. Then, the coarse fractions (+45 μm) containing mainly quartz and feldspar were firstly removed to prevent these large and heavy impurities from entering the centrifugal process. After suitable centrifugation, the diatom and impurities were separated from each other. The grade of diatomite was further improved due to the remarkable difference of particle density and size in centrifugal field.

4. Conclusions

Purification effects of scrubbing technique combined with centrifugal separation using sodium hexametaphosphate as dispersant on three typical diatomite powders were studied. The simple technique could effectively remove most of the impurities containing clay minerals and organic matters from LJ-DE and HD-DE samples. From the SEM analysis, the porous structure of all three raw diatomites can be improved after purification. It is deduced that the strong centrifugal force in scrubbing process can effectively separate the diatomite and impurities. Uniform and relatively stable dispersion of diatomite and clay minerals in the scrubbing suspension can be obtained in the weak alkaline condition at a relative high concentration of SHMP. Based on the difference of partial density and size between diatom and impurities, the diatomite was effectively purified in centrifugal field. The embedded distribution and relatively high content of clay minerals result in the inadequate separation before centrifugation, which might be the main reason for the undesirable purification effect of SX-DE. It is concluded that the technique will be potentially used as good purification method of low grade diatomite in the industry.

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References


classification-calcination process. J. Ore Dressing 12, 24-32.

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Fig. 6. SEM pictures of raw diatomite sample (LJ-a$_1$ ×500 and LJ-a$_2$×1000) and purified diatomite sample (LJ-b$_1$ ×800 and LJ-b$_2$×1500) from Linjiang.

Fig. 7. SEM pictures of raw diatomite sample (HD-a$_1$ ×500 and HD-a$_2$×1000) and purified diatomite sample (HD-b$_1$ ×1000 and HD-b$_2$×1500) from Huade.

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Fig. 10. Nitrogen adsorption-desorption isotherms of three raw diatomites and different products obtained after purification, (a) LJ-DE, (b) HD-DE, (c) SX-DE.

Fig. 11. Relationship between zeta potential and pH value at different concentrations of SHMP, (a) diatomite, (b) kaolin, (c) montmorillite
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Table 2. Chemical compositions of three raw diatomite samples (wt. %).

Table 3. BET specific surface area ($S_{\text{BET}}$), pore volume ($V_p$) and pore diameter for raw diatomites and products after purification.
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<th>Techniques</th>
<th>Main advantages</th>
<th>Main disadvantage</th>
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<td>Acid leaching treatment</td>
<td>Good removal of impurities</td>
<td>High cost; Release of a large amount of acid wastewater causing environmental problems</td>
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<td>Scrubbing method</td>
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<td>Not effective for purification of all raw diatom</td>
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<tr>
<td>Classification</td>
<td>Simplicity of application</td>
<td>Low efficiency; Difficult for large scale industrial application</td>
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<tr>
<td>Calcination</td>
<td>Simplicity of application; good removal of organic</td>
<td>High energy consumption; Incapable of removal of some impurities, such as clay minerals</td>
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<tr>
<td>Gravitational sedimentation</td>
<td>Simplicity of application</td>
<td>Low efficiency; Large water consumption; Rigorous weather conditions</td>
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<td>Ultrasound treatment</td>
<td>Effective purification at lab scale; Good removal of</td>
<td>Difficult for large scale industrial application</td>
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<td></td>
<td>impurities embedded in the porous structure of diatom</td>
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<td>Magnetic separation</td>
<td>Good removal of magnetic impurities</td>
<td>Incapable of removal of nonmagnetic substance</td>
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<td>Flotation</td>
<td>Good removal of impurities</td>
<td>High cost; Release of a large amount of wastewater causing environmental problems because of the addition of the flotation reagents</td>
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<td>Undesirable damage of original porous structure of diatom</td>
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<td>Pure montomolite</td>
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$^a$ Loss on ignition
Table 3. BET specific surface area ($S_{BET}$), pore volume ($V_p$) and pore diameter for raw diatomites and products after purification.

<table>
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<tr>
<th>Sample ID</th>
<th>$S_{BET}$ (m$^2$ g$^{-1}$)</th>
<th>$V_p$ (cm$^3$ g$^{-1}$)</th>
<th>BET (nm)</th>
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* BJH desorption cumulative pore volume of pores between 1.7 and 300 nm in diameter.

* adsorption average pore diameter (4V/A by BET)