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Study of oily sludge treatment by centrifugation

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ABSTRACT

Centrifugation has been widely applied as a pretreatment method for oily sludge. The effect of centrifugation on pure oily sludge was stable and unsatisfactory under different speeds, times, or temperatures. A new parameter, the mean volume diameter of solid surface layer particle (SPD) was proposed to evaluate centrifugal effect. The results showed that water content of about 90% together with a TPH (Total Petroleum Hydrocarbons) content around 4% and SPD of 31–32 μm was the limit for treatment of oily sludge in the low speed range. Higher speeds may be more effective in increasing the number or quality of critical particles. The analysis of water distribution by differential scanning calorimeter showed the dewatering rate of free water was much higher than bound water, and the bound energy of oily sludge centrifuged at 4000 rpm was about 2392 kJ/kg, which was much higher than that of municipal sludge. The solid particle migration was calculated using the grade efficiency model and the deviation between the simulated and experimental results suggested this model should be further studied to improve simulation accuracy and practical application.

Keywords Oily sludge; Centrifugation; Water distribution; Simulation

1. Introduction

A large amount of oily sludge is generated from the petroleum industry during crude oil production, transportation, storage, and refining [1]. In China, the annual output of oily sludge is about 3,000,000 tons [2]. The sludge is mainly composed of emulsified oil, water, and solid matter, which typically account for 30–50%, 30–50%, and 10–12% by mass [3]. Oily sludge is recognized as a good potential energy source due to its high concentrations of hydrocarbons, but it contains various components that are hard to degrade, including heavy metals, toxic species including benzene, phenol, anthracene, and pyrene [4]. Thus, this sludge is classified as hazardous waste in many countries. The improper treatment or disposal of oily sludge can pose serious threats to the environment [5], so it has become more important to find effective treatment methods.

During the past years, a variety of treatment methods have been developed, such as solvent extraction [6], pyrolysis [7], photocatalysis [8], ultrasonic [9], microwave irradiation [10], and freeze/thaw [11]. The advantage of these methods were successfully demonstrated in previous studies, and in the actual industrial production process, other physical treatment technologies such as centrifugation treatment and froth flotation have been used due to simplicity and operability [12]. In particular, centrifugation has been widely applied to field-scale oily sludge treatment [1]. A centrifugal force is used to separate different density components in a short time. As a quick treatment technology, it is often used before final disposal [13]. However, this process requires high energy to generate enough force to separate water/oil from the sludge [14], and centrifugation effect in a low speed range is not ideal. So, additional research is required to improve centrifugal performance and reduce energy consumption.

The objective of this study is to investigate the limits of high centrifugal performance in the low speed range. The solids were separated from the water/oil phase by

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centrifugal force, so the barrier affected the water distribution and the bound energy associated with solid particles. The solid particle migration was also important because it can influence the rate of ultimate particle removal and the quality of recovered water/oil mixture. In this study, we developed a new parameter, the mean volume diameter of solid surface layer particle (SPD) to analyze the centrifugation barrier, and investigated the meaning and application of this new index. Additionally, the water distribution and bound energy were investigated in order to better illustrate and interpret this barrier. Finally, a solid particle migration model was used to further clarify the centrifugal function and the solid surface particle distribution.

2. Materials and methods

Oily sludge was obtained from the water treatment plant of the Sinopec Yangzi petrochemical company Ltd in Nanjing, China. The sludge was stored at 4˚C in a capped stainless-steel bucket before treatment. The oily sludge samples were centrifuged at room temperature (20 ± 2°C) in centrifugation containers 10 mm in height using a Xiang Yi L-550, Xiang Yi Centrifuge Co. Ltd, China). The samples were centrifuged using different conditions with a rotation speed of 0–4000 rpm, centrifugation time of 0–10 min, and temperature of 20–60°C.

Characterization of the oily sludge was performed including pH, zeta potential, water content, the total hydrocarbon content, and solid content. The pH of the oily sludge samples were measured using a PD 320 portable pH meter. Dried sludge was used to measure Zeta potential after water removal. The oily sludge was heated to 105°C for 24 h in an oven, and the mass lost from the sludge was the water content. The total hydrocarbon content (TPH) was evaluated by Soxhlet extraction for 6 h using petroleum ether with a boiling point of 60–90°C as the solvent. The solid content was calculated based on the contents of water and total hydrocarbons in the sludge. Additionally, the viscosity was measured by NDJ-8S digital viscometer using 4 rotor at 3 rpm speed for 3 min. Analytical-grade chemical reagents were purchased from Sinopharm Chemical Reagent Shanghai CO., Ltd.

The diameter of the solid particles of the surface layer was determined using a Microtrac s3500 Laser diffraction particle size analyzer. The solid surface layer was added to 2 mL pure water after achieving solid-liquid separation during centrifugal process. The surface particles were rinsed by pure water and the particle size distribution was measured immediately. The mean volume diameter (MV) of the particle distribution of surface layer was defined as the SPD. The MV value represents the center of gravity in the particle size distribution, usually the "average diameter," and was strongly influenced by the particle weight itself. Obviously, there is a close relationship between SPD and solid particle migration during centrifugation. The fine SPD shows better migration of the oil/water upper layer particles, which can result in good centrifugal effect. The particle size distribution of all oily sludge samples was determined by suspending in distilled water and maintained in an ultrasonic bath for 5 min to obtain a uniform distribution of the particles before analysis.

The water distribution was determined by differential scanning calorimeter NETZSCH DSC 204F1, the temperature of the specimen was decreased to –60°C b a rate of 10°C/min and then increased to ambient temperature at the same rate.

3. Results and discussion

3.1. Properties of the oily sludge

The composition of the oily sludge is shown in Table 1.

Oily sludge has a higher moisture and lower oil content compared to typical oily sludge and contains 30–50% water or oil content in mass. The gross appearance of the sample was still sticky with black color but with a higher water content. The appearance is shown in Fig. 1.

The relation between the zeta potential and pH is shown in Fig. 2. Oily sludge particles have greater electro-negativity and this can prevent small particles from clustering into larger particles. This is a counter force to centrifugal action which aims at the maximum separation of fine particles, and therefore is a resistance to be overcome during centrifugation.

The article size distribution of dried sludge and purified sludge without water and oil is shown in Fig. 3. The MV values of the purified and dried sludge were 21.85 μm and 34.99 μm, respectively, indicating that the purified sludge is much smaller than the dried sludge. The changes of particle size distribution might result from the electrostatic repulsion of sludge particles and the dispersion of fine water/oil droplets. These two centrifugal forces are significant obstacles to be overcome in centrifugation process.

Table 1
The property of the sludge

Component	Value
pH	7.42 ± 0.9
Bulk density	923.1kg/m^3
Water	95.6%
Hydrocarbons	0.4–2.1%
Solids	3.1–3.5%
Zeta potential (nature pH)	–12mV

Fig. 1. The appearance of the oily sludge

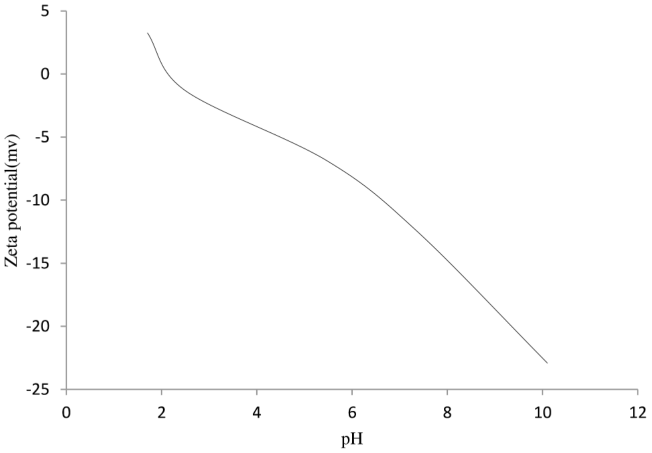

Fig. 2. The relationship between zeta potential and pH.

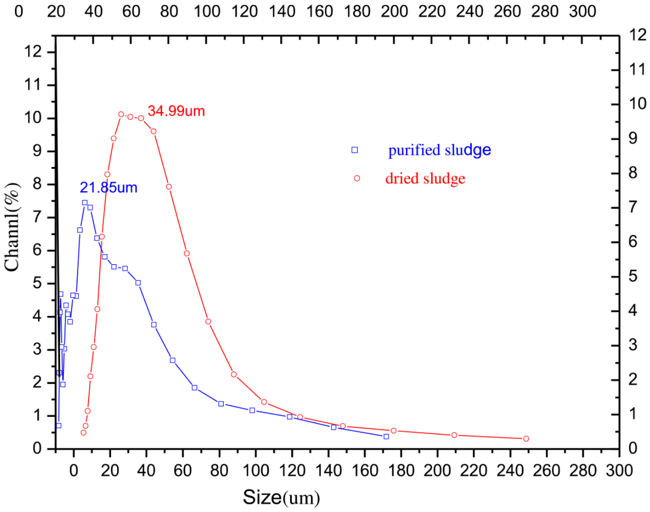

Fig. 3. Particle size distribution of the dried sludge and the purified sludge

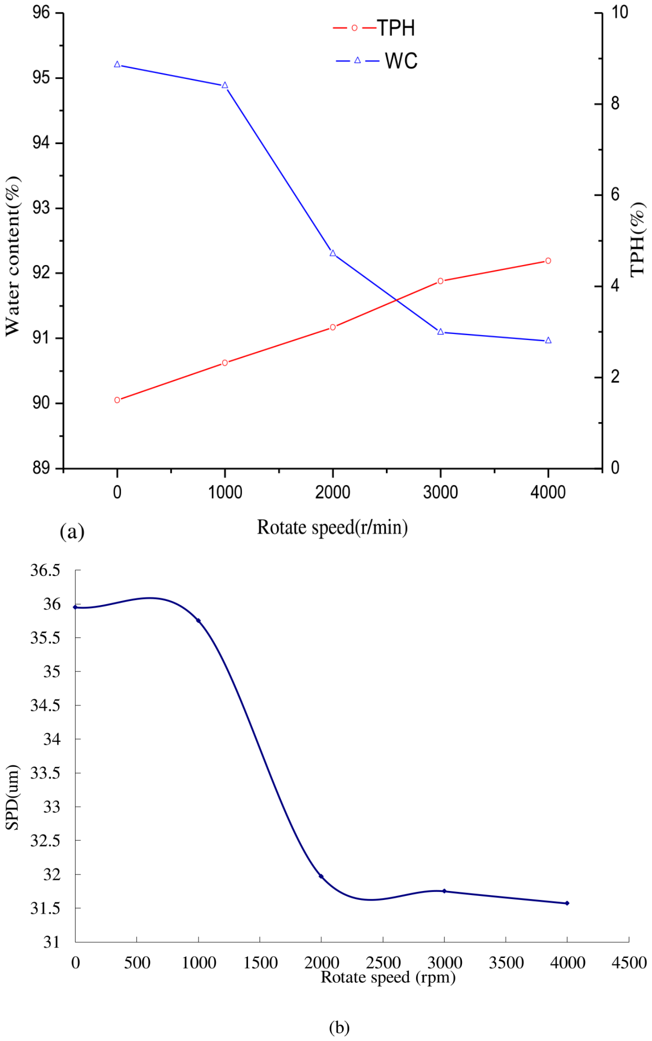

Fig. 4. The influence of rotation speed.

3.2. Influence of rotation speed

The influence of rotation speed on the water and TPH content is shown in Fig. 4a. The water content of the oily sludge decreased with increased rotation speed, and the TPH content increased with increased speed. Although centrifugation can remove partial TPH, TPH content still increased because the relatively lower water content improved the proportion of TPH. The centrifugal process can remove moisture, but this dewatering effect was incomplete, and the water content still remained 90.96% at a centrifugal speed of 4000 rpm. The dewater rate was only 4.24% compared to raw sludge's 95.2% water content, possibly due to the electro-negativity of the particles. The point of zero charge of oily sludge particles as determined by zeta potential measurements was close to a pH of 2.2; the zeta potential in neutral environment was about –12v. Particles with a stable electric double-layer structure show little coalescing of small particles with each other. The reduction in water content was constant at 0–3000 rpm, and then decreased at 3000–4000 rpm. In the centrifugal process, energy consumption is one of the most important indicators and is positively related to the square of the speed. Therefore, maintenance of appropriate speed is important to effectively reduce energy consumption. The relationship between SPD and rotation speed is shown in Fig. 4b. In this study, SPD was chosen as the key factor to assess settlement according to Stoke's law followed by the centrifugal process [15]. Obviously, the smaller the particle diameter, the better the effect of settlement. As can be seen in Fig. 4b, the SPD was stable after 2000 rpm, and ranged from 31–32 µm. This means that particles larger than this diameter range settled completely but smaller particles exhibit partial settlement. Although the sludge dewatering effect increased at higher speeds, there was no increase in particle settlement, which suggested that speeds of at least 2000 rpm might be more effective to increase the number or quality of critical particles. Therefore, 2000 rpm was selected as the appropriate economic rate because this speed can effectively separate critical particles.

3.3. Influence of centrifugation time

The influence of centrifugation time on water and TPH content was studied at 2000 rpm. The water and TPH content decreased and increased with increased time, respec-

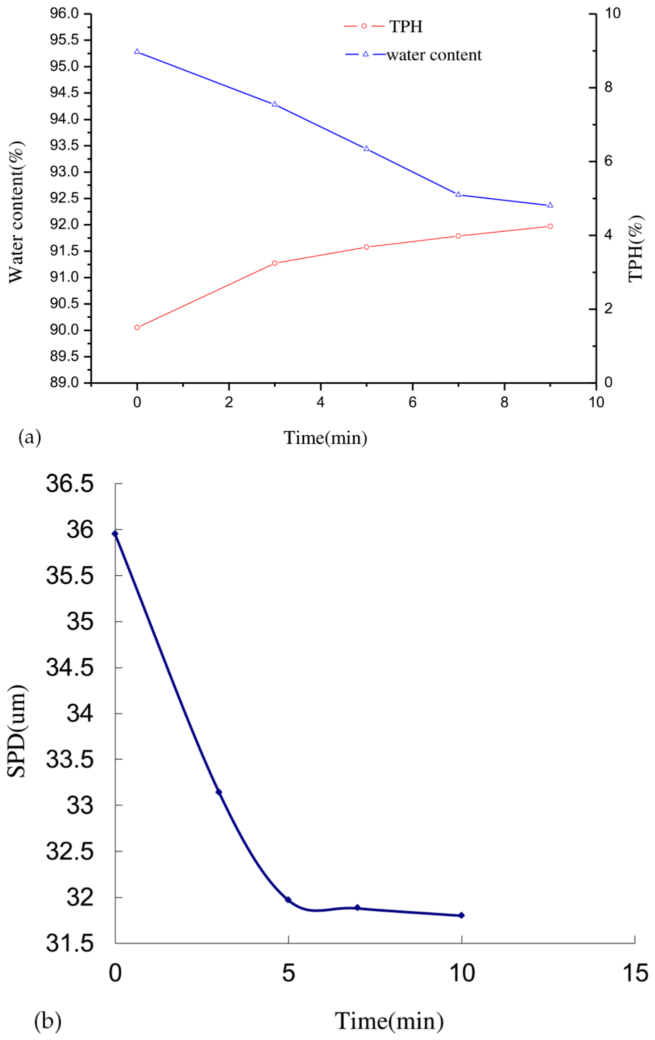

Fig. 5. The influence of centrifugation time.

tively. The relationship of SPD with centrifugation time is shown in Fig. 5. Similar to Fig. 4, although the water content decreased with increased centrifugation time, the overall dewatering efficiency was less than 5%. The water content reduction rate slowed after 7 min, suggesting that centrifugation was a quick process for treatment of oily sludge. The SPD was stable and the MV ranged from 31.5–32 µm after 5 min, which coincided well with the findings of the effects of rotation speed. Additional time will improve the dewatering efficiency but has little effect on settlement diameter. Thus, the increase in dewater efficiency was mainly due to the increase in the number or quality of sludge particles once the SPD value stabilized after 5 min.

3.4. Influence of centrifugation temperature

Decreasing the viscosity of the oily sludge may increase dewatering efficiency, so the influence of temperature was investigated. The relationship between the dynamic viscosity and centrifugation temperature is shown in Fig. 6. As seen in Fig. 6, the dynamic viscosity decreased as temperature increased. At higher temperatures, the viscosity was lower, and the flow resistance then decreased accordingly. This is equivalent to increasing centrifugal force or speed at a constant viscosity condition, so sludge dewatering is easier at higher temperatures. However, the trend of viscosity was qualitative, because the oily sludge was not a Newtonian fluid and its value varies with treatment speed and time.

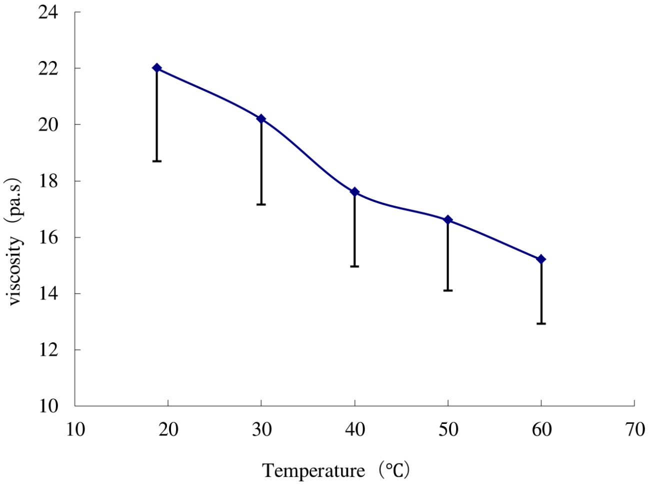

Fig. 6. The influence of temperature on viscosity

Fig. 7 shows the relationship between water and TPH content and centrifugation temperature. As shown in Fig. 7, the moisture content decreased with increased temperature. That is likely because reduced viscosity improved sludge dewatering performance and the lower water content corresponded to higher TPH content. The influence of centrifugation temperature had little effect on the water content. When the temperature increased from 20°C to 60°C, the water content reduced only slightly from 92.4 to 90.8%. This indicated that heat was not a good method to reduce the water content, at least in this temperature range. The SPD of the surface layer was also measured, but there was no significant difference in particle size at different temperatures. The diameter distribution showed no regularity at different temperatures, and particle sizes ranged from 31 to 32.5 µm.

Overall, the oily sludge water content remained around about 90% and TPH content remained around about 4%, and

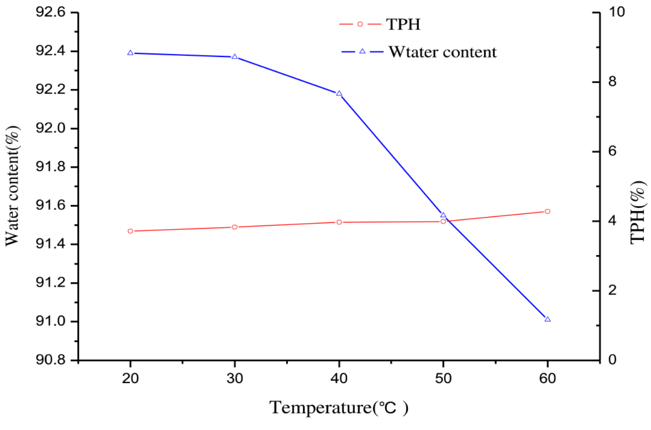

Fig. 7. The influence of temperature on water content and TPH.

the particle size ranged from 31 to 32.5 μm despite increased speed, time, or temperature. This suggests that there was a centrifugal barrier that prevented further increase in the dewater efficiency, so this barrier was investigated from the aspects of water distribution, bound energy, and solid particle migration.

3.5. Water distribution and bound energy

Differential scanning calorimeter (DSC) is an effective tool to measure the water content in oily sludge. Q. Huang first characterized the emulsified water in petroleum sludge using DSC [16], and the overall water content was deduced by the amount of heat absorbed and the specific melting enthalpy. In our study, the water content was much higher, the oil content was relatively lower, and the water was less influenced by solid particles or oil emulsification. So the water content can be divided into free water and bound water. Of these, the free water is not influenced by solid particles but bound water is [17,18]. This application has been widely adopted in municipal sludge, and solid-liquid bound energy can be measured based on thermodynamics [17].

Measuring the water in sludge is based on the principle that at the limiting temperature –60°C, the bound water does not freeze and the heat released during measurement corresponds to the free water content. The total water quantity measured by drying at 105°C minus the free water quantity is the quantity of bound water.

The bound water is calculated by the formula (1)

$$W_B = W_T - W_F = W_T - \frac{\Delta H}{\Delta H_0} \quad (1)$$

where W_B is the bound water content, W_T is the total water content, W_F is the free water content, ΔH is the heat absorbed by the sample during ice melting, and ΔH_0 is the standard melting enthalpy of frozen water, 334.7 kJ/kg

The DSC curve of sludge samples centrifuged at different speeds is shown in Fig. 8. The data shows that different endothermic and temperature values corresponded with different rotation speeds. The higher the speed, the smaller the calculated endothermic value. The moisture content of the oily sludge was lower when the sludge was treated with higher speed. The higher speed results in a lower water content and lower quality of free water. The endothermic value is only associated with the free water because of the assumption that bound water does not freeze at the limiting temperature of –60°C. The approximate conclusion is that the free water became less and less as speed increased, as indicated by the five endothermic values. Smaller adsorbed heat is required to melt the smaller amount of frozen free water, and the lower quality of the free water required less heat and the melting temperature was also reduced.

Effect of rotation speed on free water content is shown in Fig. 9. As can be seen from Fig. 9, the free water content decreased as speed increased, and the free water content change was no longer evident after 2000 rpm. This supported the choice of 2000 rpm as an appropriate speed for the removal of free water. Combined with the influence of the rotation speed on water content, this allows the conclusion that bound water can be removed at the speed of more than 2000 rpm and its removal rate is lower than free water. The smaller the particles, the association and effect of the bound water was greater. When the SPD ranged from 31 to 32 μm, the binding strength between the solid and bound water was bigger, and the removal rate of bound water decreased.

The water and solid distribution of sludge is shown in Fig. 10. As can be seen from the data in Fig. 10, the solid content increased with increased rotation speed, and this means that the water content of oily sludge decreased correspondingly. The free water content decreased as speed increased, and the proportion of free water in whole sludge also decreased. The bound water proportion improved conversely, and experiments showed that centrifugation could remove part of the bound water. However, the dewatering rate of the free water was much higher than that of the bound water, leading to an increase in the proportion of bound water. This indicates that free water removal of the oily sludge was primary in the low-speed centrifugal

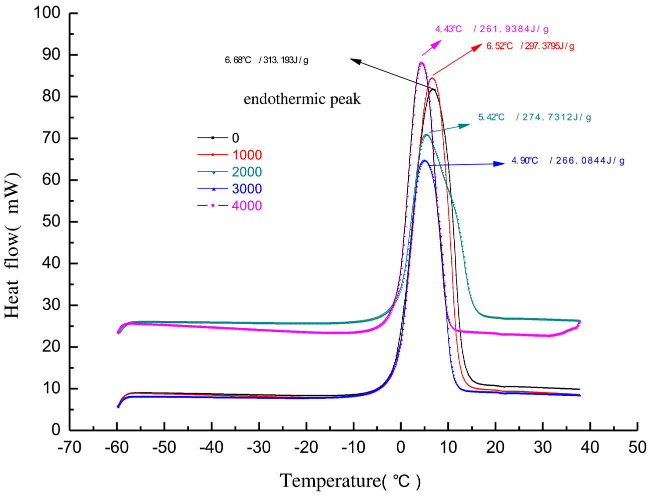

Fig. 8. The DSC curve of sludge samples centrifuged at different rotation speeds.

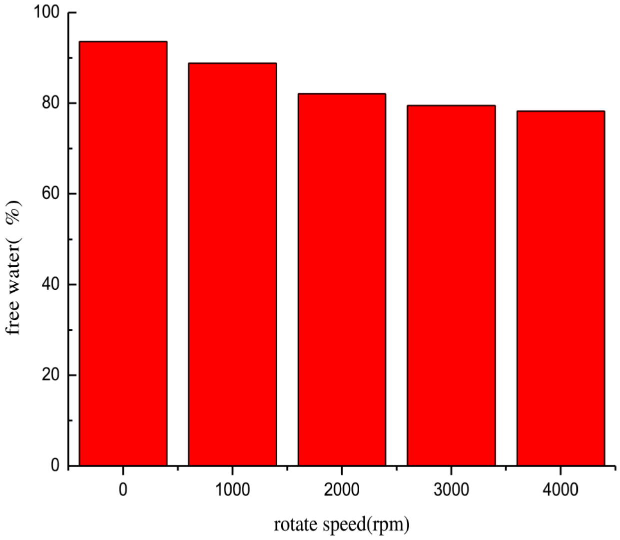

Fig. 9. The free water mass ratio.

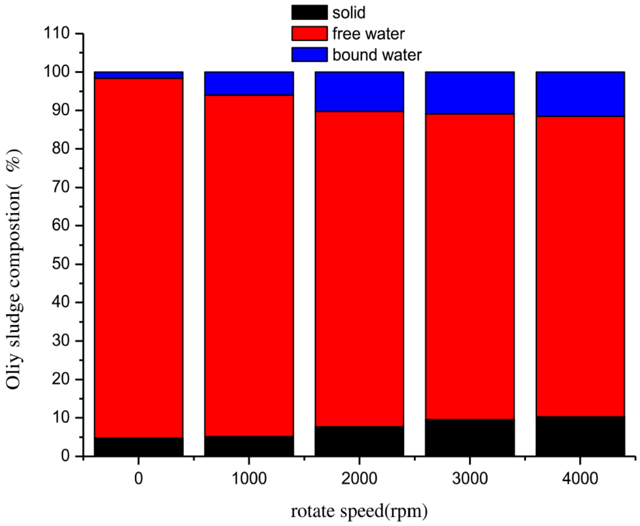

Fig. 10. Water and solid distribution.

process and the bound water was the key factor affecting dewatering. This provides a good explanation for why the moisture content of oily sludge did not improve as the speed increased, and illustrates a disadvantage of the centrifugal process. Other measures should be taken to further reduce the bound water content as required.

Because the centrifugal process is less able to remove bound water and the bound water is the key factor affecting dewatering, it is necessary to analyze the binding energy associated with solid particles, or the bound energy. D.J. Lee supposed a thermodynamic interpretation of bound energy. The ability of bound water to remain unfrozen at low temperatures may be a consequence of the reduction of free energy due to the binding energy acting on the water. The temperature and the bound energy can be calculated by the following formula [17]

$$T_{\mathrm{sh}} = \frac{E_B T_{\mathrm{f}_0}}{\Delta H_{\mathrm{f}}} + T_{\mathrm{f}_0} \tag{2}$$

where T_{sh} is the limiting temperature, –60°C, E_B is the bound energy, T_{f0} is the threshold temperature of freezing, and ΔH_f is the exothermic heat of freezing from ambient temperature to limiting temperature.

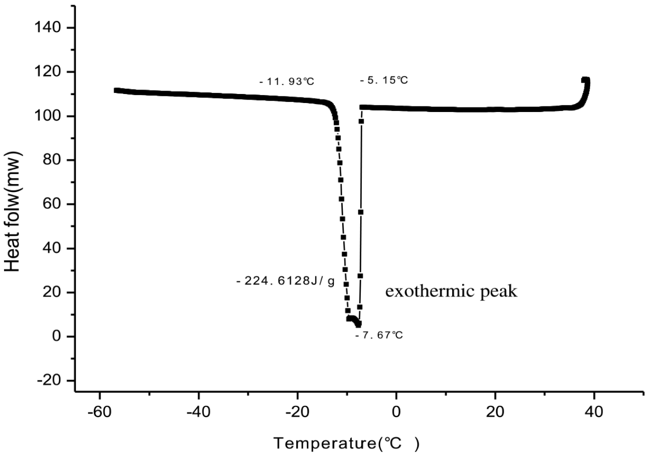

Fig. 11. The exothermic curve of oily sludge

The bound energy of the oily sludge treated by centrifugation at 4000 rotation speed was 2392 kJ/kg. This value is much higher than the sewage sludge value of 65 kJ/kg which was calculated using the same formula and conditions [19], suggesting the binding energy of emulsified water and solid particles in oily sludge was much larger than that of solids and water in sewage sludge. Therefore, for oily sludge, the centrifugation process must overcome greater energy requirements than sewage sludge.

3.6 Simulation of solid particle migration

The sedimentation velocity of particles in the water/oil emulsion during centrifugation can be described by Stoke's law

$$v = \frac{D^2(\rho - \rho_0)\omega^2 r}{18\mu} \tag{3}$$

where v is the sedimentation velocity, μ is the viscosity of water/oil phase, D is the solid particle diameter, and ρ and ρ_o are the densities of the solids and the water/oil phase respectively. ω is the centrifugal angular speed andis the radius of the centrifuge, which indicates the distance between the sample and the center of the centrifuge rotor.

During sedimentation, the settling distance Δl can be expressed with the sedimentation velocity v and centrifuge time t.

$$\Delta l = vt = \frac{D^2(\rho - \rho_0)\omega^2 r}{18\mu} t \tag{4}$$

According to formula (4), the larger particles settle more quickly and will be at the bottom of the centrifuge tube, and the smaller particles settle at lower speeds and will be in the upper location of the tube [20]. The solid particles of the critical size will be just recovered and located in the most surface of the sediment layer in the centrifuge. In a centrifugal experiment, the critical diameter of solid particles can be calculated as follows

$$D_c = \sqrt{\frac{18u\Delta l_{\max}}{\omega^2 r(\rho - \rho_0)t}} \tag{5}$$

where D_c is the limit of the size of the particles that will be recovered in the centrifuge. $\Delta l_{\max}$ is the distance between the surface of the oily sludge to the bottom of the centrifuge tube.

Particle sedimentation in a centrifuge can be predicted using the grade efficiency concept. For any particle, the grade efficiency can be used to determine whether it can be separated or not [21]. Mannweiler and Hoare introduced the Rosin–Rammler–Sperling–Bennett (RRSB) distribution function, $F(D)$, to describe the grade efficiency curve for particles in a disc-stack centrifuge using D/D_c as an independent variable while $D/D_c \leq 2.5$, which can be expressed as Eq. (6)

$$F(D) = 100 - 100\exp\left[-\left(\frac{kD}{D_c}\right)^n\right] \tag{6}$$

Table 2
The detailed parameters of the model

Parameter	Value
Viscosity (mPa·S)	3.01
Density of solids(kg/m^3)	1103
Density of water/oil phase(kg/m^3)	985
Centrifugal angular speed(rad/s)	$\frac{2\pi}{60}\times rs(rpm)$
Radius of the centrifuge(m)	0.012
Total height of oily sludge(m)	0.05
Centrifuge time (s)	300

where k and n are empirical parameters determined experimentally.

According to the RRSB distribution and its significance, *F(D)* is the total sedimentation rate of all the particles of diameter smaller than *D*, so for any particle of diameter *D*, the distribution function *f(D)* can be calculated from formula (6). The volume of particles within a finite size range is usually measured and plotted in form of a distribution histogram during particle size analysis. The difference of two values of the cumulative distribution *f(D)* between two particle diameters D_1 and $D_2 = D_1+\Delta d$ is given by

$$f(D_{1-2}) = F(D_1) - F(D_2) \tag{7}$$

In many model simulations, it was assumed the solid particles had different settleability compared to the emulsified water droplets during centrifugation because of different density, and the interaction and coalescence of water

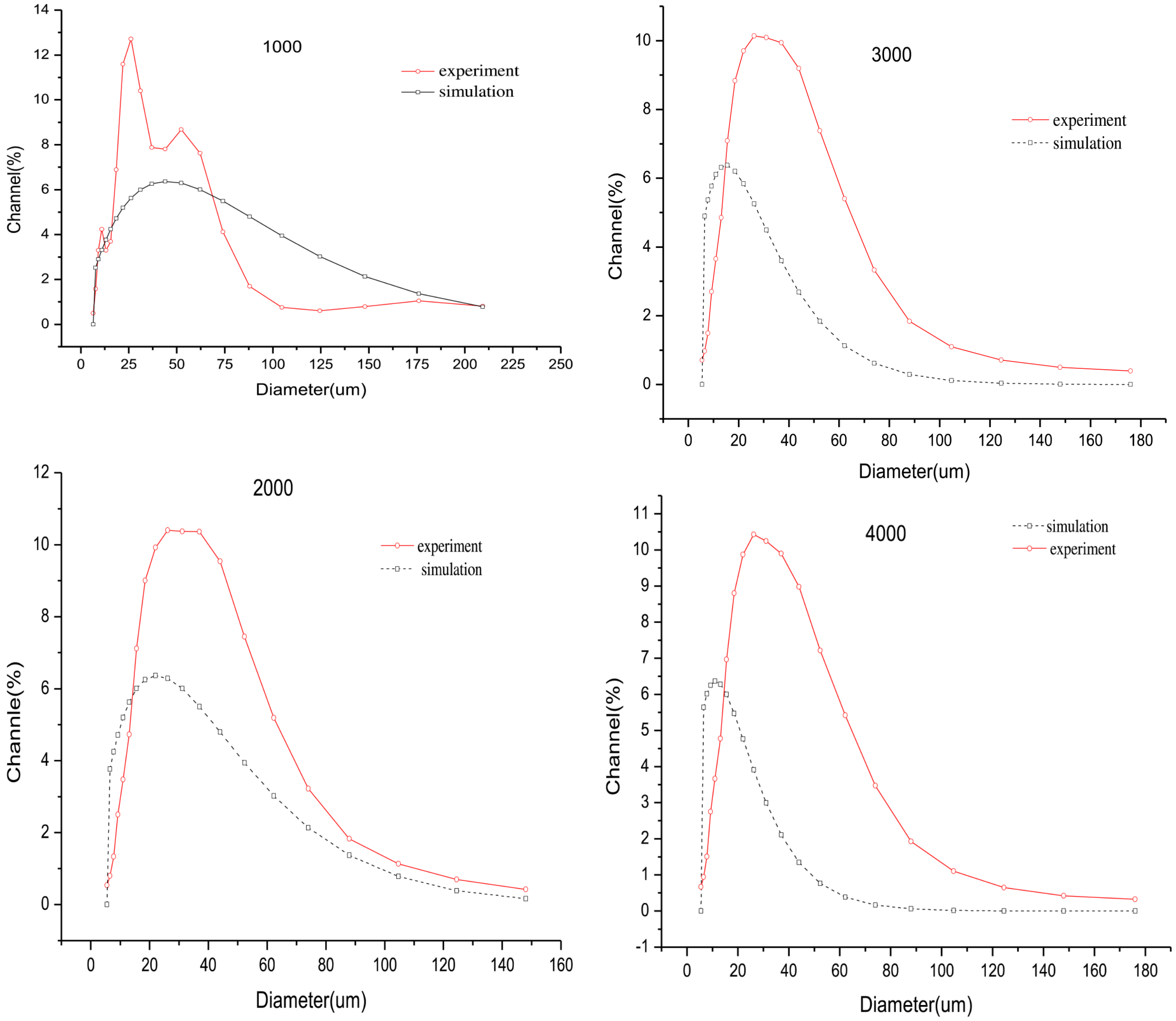

Fig. 12. The particle size distribution of the surface layer

droplets could be negligible [22]. Therefore, there may be differences between the assumptions and reality. In our study, it was assumed that the settling velocity in each interface was uniformly based on zone settlement theory in turbulent flow, which may be more agreement with actual conditions.

Using the definitions and the assumption above, the particle size distribution of solid surface layer $f(D_n)$ can be calculated.

The detailed parameters of the model are shown in Table 2.

The critical diameter was calculated by formula (5), and the grade efficiency curve for particles centrifuged at different speeds can be calculated according to formula (6) based on the calculated D_c value, and $f(D_n)$ can also be obtained based on formula (7). The calculated and experimental values of the particle size distribution in the surface layer are shown in Fig. 12. This comparison shows there is definite deviation between the simulated and experimental results. The simulation results gave smaller particle diameters and lower channel. This may be because smaller particles are more easily affected by water flow and some small particles may return to the aqueous phase. Another reason for the differences may be the error between the model hypothesis and the actual experiment, such as the D_c calculation formula was induced in $Re < 0.5$. It will be necessary to improve this model further to improve the simulation accuracy for practical application.

4. Conclusions

Centrifugation is a quick method to dewater oily sludge, however, its efficiency is not high at low-speed rotation speeds. The SPD is a new efficient parameter proposed to evaluate the centrifugal results. A 90% water content, 4% TPH content, and 31–32 μm SPD were the limit of centrifugation. The analysis of water distribution showed the dewatering rate of the free water was much higher than the bound water. The bound energy of the oily sludge centrifuged at 4000 rotation speed was 2392 kJ/kg; this energy may explain the difficulty in centrifugal treatment. The model should be further perfected to optimize the process and improve the efficiency of centrifugation treatment.

Acknowledgments

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