

Study on two-step process of petroleum substances recovery from oily sludge

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Abstract. Oily sludge has received increasing attention for its hazardousness as well as its high potential as an energy resource. In this paper, a study, in order to obtain the essential parameters in the relevant engineering design or practices, the two-step process of thermochemical demulsification separation and air floatation separation was used for oil material recovery from oily sludge (light oily sludge and heavy oily sludge) that was obtained from an oilfield. The light oil sludge and heavy oily sludge were diluted with of 3 times and 4 times mass water respectively and heated to 75 °C and 80 °C, respectively, and both adjusted pH to 6. The self-made emulsion breaking agent with the mixture mass fraction of 0.3% and 0.4% were added separately, and the thermochemical demulsification separation was carried out for 20 min and 25 min respectively under stirring. The mixture of mud and water after the thermochemical demulsification separation was further air floatation at normal temperature. The oil content of the mud cake formed by the centrifugal separation of the mixture after air floatation separation was under 2%. And the result that centrifugal separation mixture liquid for the next oily sludge treatment was great. Furthermore, the test result of planting clover on the soil sample composed of the dry mud cake and some seedbed soil showed that the seed germination and growth were normal.

1. Introduction

Oily sludge is a stable oil-in-water and water-in-oil systems and mainly produced in the processes of crude oil exploitation, transportation, storage and oily wastewater treatment; its composition is complex and contains aged crude oil, wax, asphaltene, salt, colloid, bacteria, and chemical agents, such as corrosion inhibitor, coagulant, fungicides and scale inhibitors [1]. According to the characteristics of oily sludge, it can be divided into light oily sludge and heavy oily sludge. Compared with light oily sludge, the heavy oily sludge has the characteristics of high viscosity, serious emulsification, high wax and asphalt content, and the longer it is placed, the greater the proportion of heavy oil components [2].

The oil content of oily sludge is generally higher than 20%, which can reach up to 50%, with the high potential as an energy resource. However, it has complex components, large viscosity, fine particles, high degree of emulsification and difficulty in dehydration [3]. The petroleum substances in oily sludge should be recovered first because of its high oil content from the point of view of hazardous waste treatment and disposal [4]. There are pyrolysis and separation methods for petroleum material recovery. Pyrolysis is the simultaneous distillation and thermal decomposition of the sludge under the condition



of isolation of air and high temperature. The main purpose of the pyrolysis is to obtain fuel oil. The pyrolysis method is suitable for sludge treatment with low water content and high hydrocarbon content; Thorough, fast, high fuel recovery rate, low secondary pollution, but large equipment investment, complex operation and high energy consumption [5]. The separation methods generally contain extraction separation and gelation phase separation. The extraction separation method uses an organic solvent as an extractant to extract petroleum substances in the oily sludge. The advantage of this method is that the petroleum material has good recovery effect, but the treatment cost is high, and the safety protection measures are strict [6].

The gelation separation method is to separate the petroleum substances from the mud water and recover it by using ultrasonic or heating to destroy the sludge stabilization system under the condition of dilution. At present, many oily sludge treatment systems are generally slow in processing speed at home and abroad, which cannot reach the designed capacity, and some even only have 1/5 of the designed capacity, which is difficult to meet the increasing requirements of actual sludge production [7]. The reason is that the selection of the emulsion breaking agent is improper and the oily sludge treatment process is problematic. Based on the research and application practice of coagulation technology for many years, the research team based on the analyzing and summarizing the technology of oily sludge breaking phase separation at home and abroad, we put the self-made emulsion breaking agent applied to the separation and recovery of oily sludge. The simple, fast separation speed, good effect, low operating cost, and zero-discharge sludge disposal process provide technical support for improving oil sludge processing speed.

2. Material and methods

2.1. Material

Samples of light oily sludge (LOS) and heavy oily sludge (HOS) were taken from the first and seventh oil production factory in northeast China. The oily sludge was dark brown and viscous, and the content of water, oil and mud were 5% - 10%, 35% - 42%, 48 - 56%, respectively. The planting test soil was taken from the flowerbed soil with the moisture content was about 10%, after grinding, pass 2mm sieve, standby application (A sample). Self-made emulsion breaking agent (white powder, Al_2O_3 mass fraction 23 - 25%, 10% aqueous solution is colorless and transparent, pH 3 - 5).

2.2. Methods

2.2.1. Optimization of separation conditions of petroleum materials in oily sludge. Weigh 25 g of oily sludge that has been removed from large pieces of debris in a 100 ml beaker, dilute with water in proportion, adjust the pH, and slowly stir with a glass rod until the heavy oily sludge sample is evenly dispersed. The emulsion breaking agent was added after the water bath was heated to the specified temperature, and the thermochemical demulsification separation was carried out under stirring conditions. After the separation was completed, the mixture was allowed to cool to room temperature, and the upper layer of oil was scraped off, the step of thermochemical demulsification separation was completed. The remaining mixture of mud and water was stirred uniformly, vacuum filtered, and the dewatering performance was examined to determine the oil content of the mud cake after dehydration. The muddy-water mixture after the thermochemical demulsification separation was further air floatation separation. After the air-floating, the upper layer of oil is scraped off, Stirring the remaining mud-water mixture evenly, and the dewatering performance was examined by vacuum filtration to determine the oil content of the mud cake. Under optimized conditions, the oily sludge amplification experiment was carried out, and the scale-up experiments were carried out according to 50 g, 100 g, 200 g, 500 g, and 1000 g. Three parallel experiments were performed at each stage to measure the oil content of the mud cake.

2.2.2. Filtrate recovery test. Under optimized conditions, the mixture of mud and water after crude oil recovered was vacuum filtered, and the separation fluid was replenished with water, adjusted pH and appropriate amount of emulsion breaking agent (measured by Al_2O_3 content) was added to carry out a new round of oily sludge separation test to investigate the separation effect.

2.2.3. Planting test. The mud cake was air-dried at room temperature after the two-step process of petroleum substances recovered, ground through a 2 mm screen and ready for use (B sample). Mix A sample and B sample in a mass ratio (4:0, 3:1, 2:2, 1:3, 0:4), and put them into experimental flower pots, 500 g per pot, and soak 30 clover seeds, Three parallels per process. Watering once a day and keep the soil moisture more than 85%, observe the germination of the seeds and the growth of the branches and leaves. The seed germination rate was counted at 20 days, and the plant height and fresh weight were measured at 40 days.

2.2.4. Sample analyses. Determination of water content of oily sludge by petroleum product moisture determination [8]. The oil content of the mud cake was determined by the method of infrared spectrophotometry [9]. The mud content in the sample was calculated after removing oil and water content. The content of Al_2O_3 was determined by titration with zinc oxide standard solution [10].

2.2.5. Statistical analyses. A software of the Statistical Package for the Social Science (SPSS) was used for the analysis of variance (ANOVA), which was performed for data treatment using Duncan's multiple range test (DMRT) at the 5 % and 1% level of probability.

3. Results

3.1. Optimization test of the thermochemical demulsification separation of oily sludge

The results of preliminary orthogonal experimental shows that the important factors affecting the thermochemical demulsification separation of oily sludge were acidity and alkalinity of the mixture, temperature, sludge dilution liquid-solid ratio and amount of emulsion breaking agent. The optimization conditions test of the thermochemical demulsification was carried out on these basis, and the vacuum filtration performance of the mixture of mud and water after the sludge phase separation ($-0.09 \sim -0.1$ Mpa, the time required for 100 mL of sludge to be filtered until the cracks appeared) and the oil content of the mud cake were investigated. The basic conditions with the mixture pH, demulsification temperature, amount of emulsion breaking agent, liquid-solid ratio, stirring speed and stirring speed for the treatment of the light oily sludge and heavy oily sludge were 6, 75 °C, 0.3%, 3, 200 r·min⁻¹, 20 min and 6, 80°C, 0.4%, 4, 200 r·min⁻¹, 25 min, respectively.

3.1.1. Effect of different pH for oily sludge thermochemical demulsification separation. The mixture of light oily sludge and heavy oily sludge were vacuum filtered for a long time under alkaline conditions, and the oil content of the mud cakes were high, that is, the separation effect was poor (Figure 1). In addition, the time required was often more than 1 h, and the separation liquid after standing were turbid, and the color of mud cake were dark under alkaline conditions. On the contrary, the thermochemical demulsification separation of the oily sludge can achieve good results in 20~30 min under acid conditions. When the pH of the mixture was 6, the separation liquid of the light oily sludge and the heavy oily sludge were clear. The time required for vacuum filtration of the mixture were 55 s and 75 s, respectively, and the oil content of the mud cake was less than 5%, indicating that the separation effect of petroleum substances in the sludge was good.

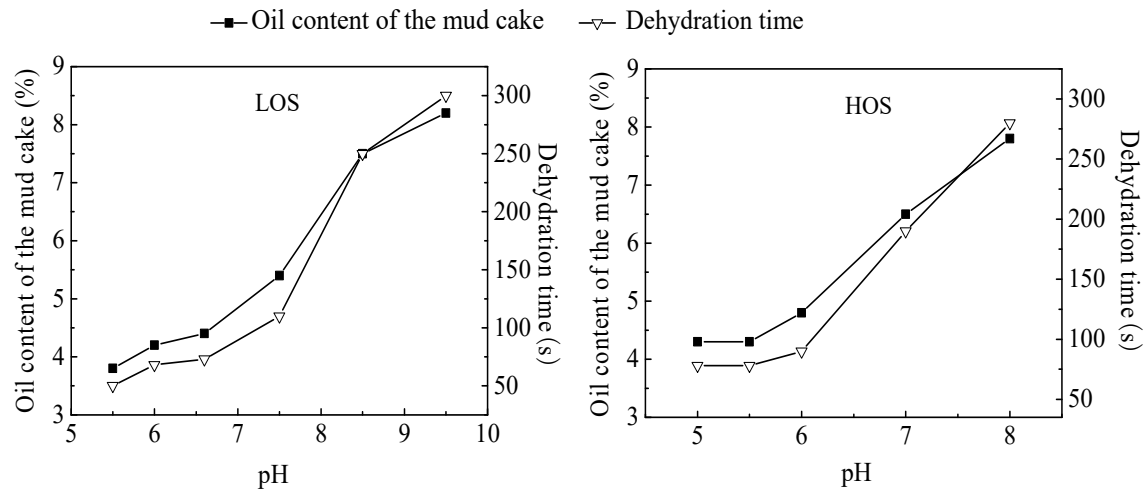


Figure 1. Effect of different pH for oily sludge thermochemical demulsification separation. LOS, light oily sludge; HOS, heavy oily sludge.

3.1.2. Effect of different temperature for oily sludge thermochemical demulsification separation. The mixture of mud and water can be quickly broken and layered when the light oily sludge and the heavy oily sludge were heated to 75 °C and 80 °C, respectively (Figure 2). The sludge-water interface was clear after statically separated. The mixture of mud and water after deoiled was vacuum filtered, and the mass fraction of oil in the mud cake was about 4%.

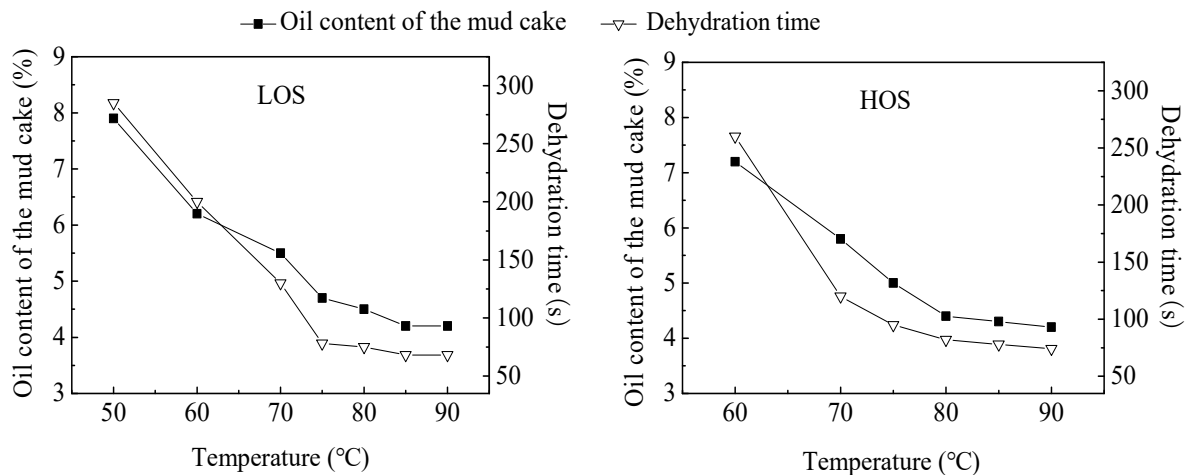


Figure 2. Effect of different temperature for oily sludge thermochemical demulsification separation. LOS, light oily sludge; HOS, heavy oily sludge.

3.1.3. Effect of different amount of emulsion breaking agent for oily sludge thermochemical demulsification separation. The oil content of the mud cakes and the dehydration time of the mixture gradually decrease with the dosage of emulsion breaking agent increased (Figure 3). When the dosage of light oily sludge and heavy oily sludge emulsion breaking agent was 0.3% and 0.4%, respectively, the demulsification separation effect of the sludge was the best, and the oil content of the mud cake was less than 5%. However, the oil content of the mud cakes and the dehydration time of the mixture from the light oily sludge and heavy oily sludge were increased when the emulsion breaking agent was exceed 0.4% and 0.5%, respectively.

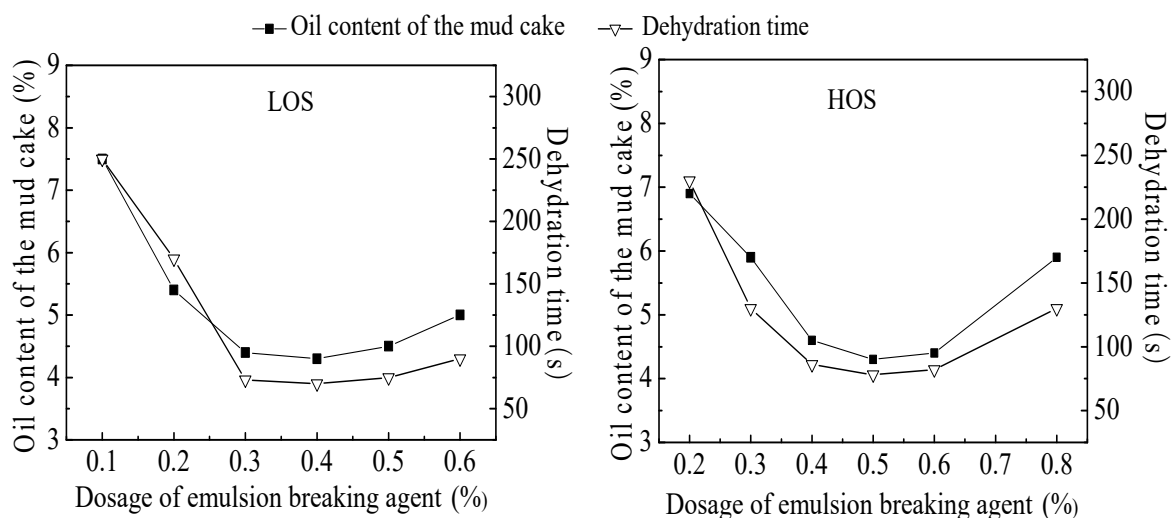


Fig. 3 Effect of different amount of emulsion breaking agent for oily sludge thermochemical demulsification separation. LOS, light oily sludge; HOS, heavy oily sludge.

3.1.4. Effect of different liquid-solid ratio for oily sludge thermochemical demulsification separation.

The separation effect of the light oily sludge and the heavy oily sludge demulsification were very good after the liquid-solid dilution ratio was 3 and 4, respectively (Figure 4), and the sludge dewatering was rapid and the layered interface was clear. The mixture of mud and water after deoiled was vacuum-filtered and dehydrated, and the separated mud cake has low oil content.

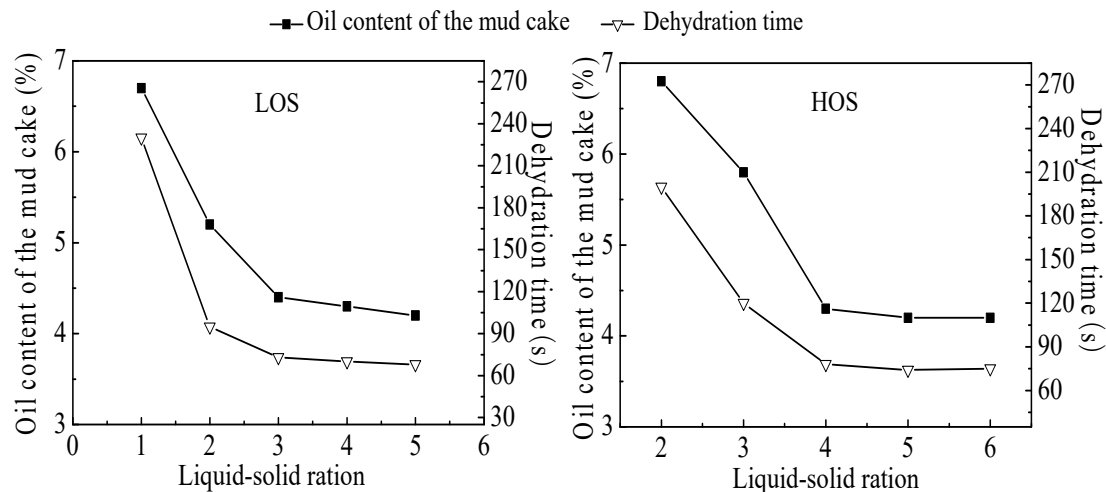


Figure 4. Effect of different liquid-solid ratio for oily sludge thermochemical demulsification separation. LOS, light oily sludge; HOS, heavy oily sludge.

3.1.5. Effect of different stirring speed for oily sludge thermochemical demulsification separation.

The oil content of the light oily sludge cake decreases first and then increases with the increase of the stirring speed, and reaches the lowest at 200 r min⁻¹. When the oil content exceeds 200 r min⁻¹, the oil content of the mud cake gradually rising (Figure 5). The oil content of the mud cake with heavy oily sludge reached the lowest when the stirring speed was 200-250 r min⁻¹, and the oil content of the mud cake gradually increased when it exceeded 250 r min⁻¹ (Figure 5).

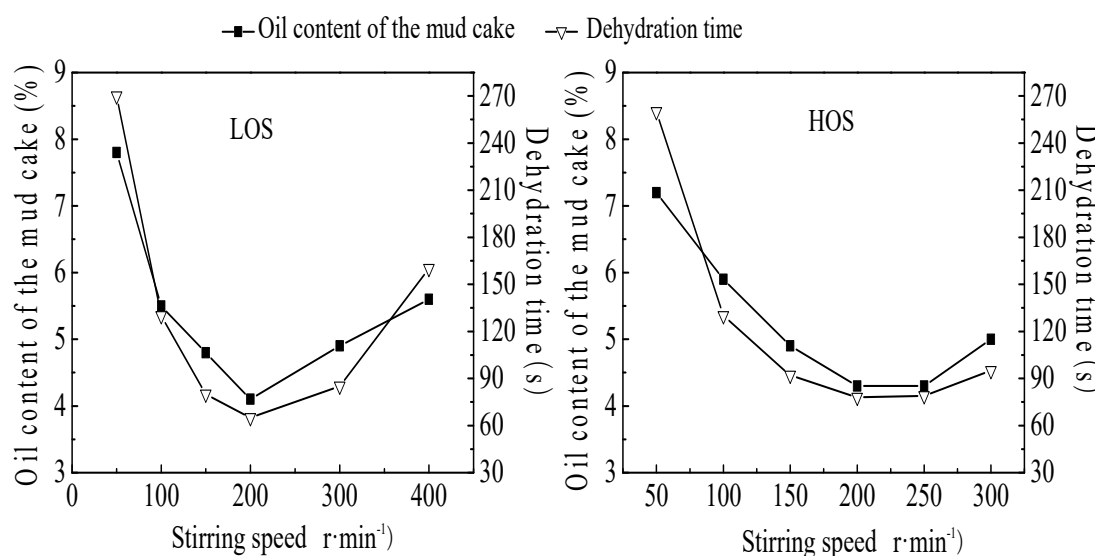


Figure 5. Effect of different stirring speed for oily sludge thermochemical demulsification separation. LOS, light oily sludge; HOS, heavy oily sludge.

3.1.6. Effect of different stirring time for oily sludge thermochemical demulsification separation. The oil content of the mud cake with the light oily sludge and the heavy oily sludge were both decreased with the increasing of the stirring time, and then stabilized and finally rose (Figure 6). Because when the stirring time was less than 20 min, the action time of the agent and the sludge were not enough, and the effect of the agent can not be fully exerted to achieve the demulsification; when the stirring time was 20 - 40 min, the oil content of the mud cake after dehydration was basically the same; However, after 40 minutes, the oil content of the mud cake increased (Figure 6).

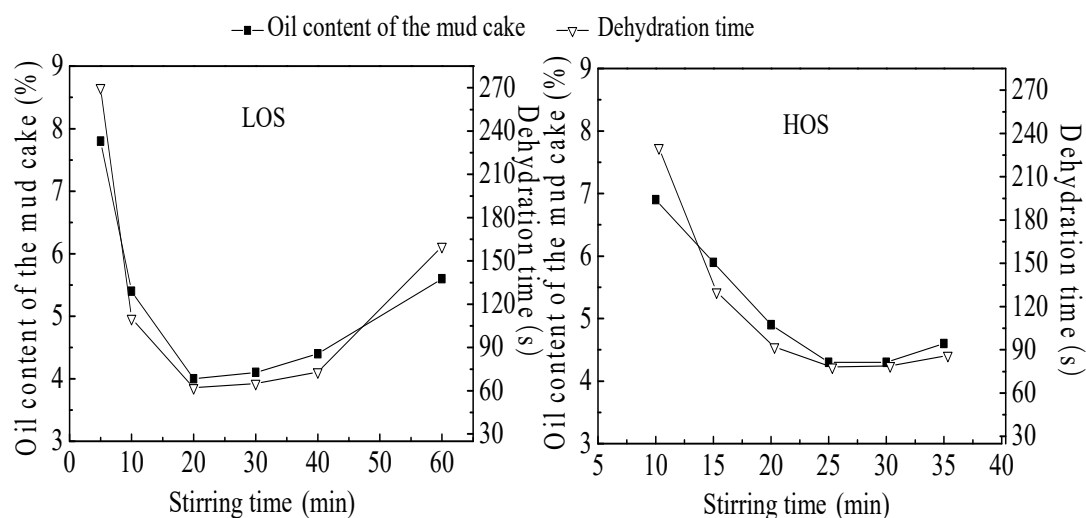


Figure 6. Effect of different stirring time for oily sludge thermochemical demulsification separation. LOS, light oily sludge; HOS, heavy oily sludge.

3.1.7. Comprehensive Experiment. Under the conditions with the mixture pH, demulsification temperature, amount of emulsion breaking agent, liquid-solid ratio, stirring speed and stirring time for the treatment of the light oily sludge and heavy oily sludge were 6, 75 °C, 0.3%, 3, 200 r·min⁻¹, 20 min and 6, 80 °C, 0.4%, 4, 200 r·min⁻¹, 25 min, respectively. Five parallel trials show that the thermochemical demulsification separation can achieve good results. The static layering interface were clear. The vacuum dehydration time of slurry mixture of mud and water after deoiled was 60~90 s, and the oil content of the mud cake was about 4.0% (Table 1).

Table 1. The thermochemical demulsification separation under the under optimized conditions. LOS, light oily sludge; HOS, heavy oily sludge.

	Number	Oil content in muddy cake before treatment (%)	Oil content in muddy cake after treatment (%)	Drying time of mud cake (s)
LOS	1	37.17	4.02	70
	2	39.41	3.97	65
	3	37.63	4.07	75
	4	39.48	3.90	63
	5	39.56	3.94	62
HOS	1	38.17	4.23	74
	2	39.42	4.28	78
	3	38.63	4.21	73
	4	39.87	4.36	81
	5	38.94	4.26	76

3.2. Secondary air floatation separation

Currently, there was no unified international standard for oil content in oily sludge. In Canada and the United States, the soil for landfill required that oil content was less than 2%, while the standard for road paving was less than 5%. The Netherlands requires soil oil content was less than 10 mg l⁻¹, while France requires soil oil content was less than 0.5% and dry land was less than 2% for areas with high precipitation and belonging to wetlands. Singapore and Arabia required oil content in soil which for landfill were less than 1% and 5%, respectively [11]. While mineral oil content in agricultural sludge in China required no more than 0.3%. Combined with the treatment standards of oily sludge at home and abroad, and taking the difference between the actual production and experimental research process conditions into account, the mixture of mud and water after the separation of hot demulsified petroleum substances was subjected to secondary air-floating deoiling test. The results show that the petroleum substances that have not been separated in the thermochemical demulsification can be further removed under the secondary air-floating deoiling at room temperature. With the increase of aeration and aeration time, the oil content of the mud cake was gradually decreased. However, the oil content was basically unchanged when the aeration rates of thin oily sludge and heavy oily sludge exceed 3 m³ (m³ h)⁻¹ and 3.5 m³ (m³ h)⁻¹, respectively; and aeration time exceeds 20 min and 30 min, respectively. It was indicating that the oil has been basically removed; the mixture of mud and water after the scum of the petroleum substance was vacuum dehydrated about 30 s can be dried and crack. The oil content dropped to about 1% (Figure 7).

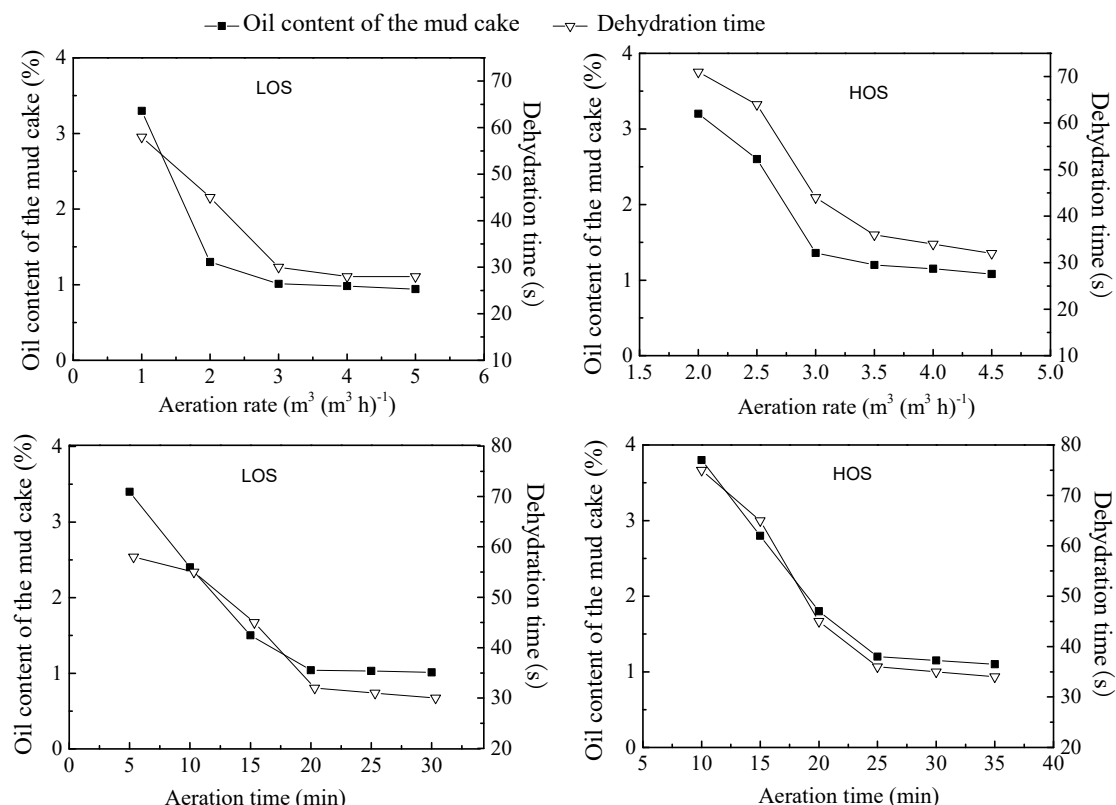


Figure 7. Effect of different floatation amount and floatation time on residual oil type material removal. LOS, light oily sludge; HOS, heavy oily sludge.

3.3. Filtrate recovery test

The separation liquid after dehydration of the mixed liquid of the two-step process of oil material recovered to the dilution for the new oily sludge sample, which can make this process technology realize zero discharge of the wastewater in actual production. Considering the actual production, the sludge dewatering equipment was generally a centrifuge, and the mixture of mud and water after two-step process of oil material recovered was carried out centrifugal dewatering, and the obtained separation liquid was 60-80% of the diluted water consumption of the original oil-containing sludge. The Al_2O_3 mass fraction showed little change and was slightly turbid. After adding water to the Centrifugal separation fluid and for dilute the new sludge. According to the above optimization conditions, adjust the pH value, add demulsifier, carry out a new round of sludge separation test, and repeat the cycle. The results show that the separation of oil and petroleum substances was still fast. The content of oil in the mud cake after centrifugal separation was less than 2%, and the dewatering time of the mixture of mud-water was less than 50 s.

3.4. Stepwise amplification test

Comprehensive experiments show that the oil content of mud cake treated with heavy oil sludge was less than 2% under the optimized condition. The mud cake can be dried and cracked after being vacuum dehydrated for 50 s, the dewatering performance is good, and the separation liquid can be effectively reused. There was no wastewater discharge during the treatment process. Therefore, the stepwise amplification test was carried out in the laboratory under the optimized condition. When the treatment capacity was 1000 g, the oil content of the mud cake was less than 2% (Table 2), and the effect of stepwise amplification was good.

Table 2. Stepwise amplification test. Within columns, means followed by the same capital letter and lowercase are not significantly different at $P = 0.01$ and $P = 0.05$, respectively.

Number	Amount of sample (g)	The oil content of mud cake with LOS (%)	The oil content of mud cake with HOS (%)
1	25	1.12±0.02Bb	1.21±0.02Bc
2	50	1.13±0.03Bb	1.23±0.02Bbc
3	100	1.15±0.03Bb	1.22±0.03Bc
4	250	1.15±0.02ABb	1.25±0.03Bbc
5	500	1.17±0.03ABab	1.27±0.02Bb
6	1000	1.21±0.03Aa	1.38±0.03Aa

3.5. Planting test

The level designed by the experiment had no significant effect on the germination rate, plant height and fresh weight of clover seeds (Table 3, $p > 0.05$). It was indicated that the residual oil content of the treated mud cake was within the tolerance range of the clover seed and does not have a significant impact on the growth of the plant, which provides a pathway for subsequent phytoremediation.

Table 3. The result of planting test. Within columns, means followed by the same capital letter and lowercase are not significantly different at $P = 0.01$ and $P = 0.05$, respectively.

A:B	Seed germination rate (%)	Plant high (cm)	Fresh weight (g)
4:0	77.3±1.1Aa	9.5±1.3 Aa	0.26±0.01 Aa
3:1	76.8±1.2Aa	9.2±1.5 Aa	0.25±0.02 Aa
2:2	76.5±1.5Aa	9.0±1.4 Aa	0.24±0.01 Aa
1:3	76.0±1.0Aa	8.9±1.3 Aa	0.23±0.02 Aa
0:4	75.5±1.6Aa	8.8±1.2 Aa	0.22±0.01 Aa

4. Discussion

Production practice shows that the content of petroleum substances is the decisive factor for the dehydration performance of the mixture of mud and water after sludge separation. The lower the content of petroleum substances, the better the dehydration performance of mixture of mud and water; And vice versa. The mixture of mud and water was vacuum filtered, and the soft oily solid particles (such as asphaltene, colloid, paraffin, etc.) that have not been separated were changed during the dehydration process, resulting in an increased of the filtration specific resistance; if the soft particles adhere to the filter material, plug the filter holes, resulting in a decrease in the efficiency of vacuum filtration. During centrifugal dehydration, the soft viscous particles adhere to the sand soil, making the density of the sand soil become smaller and the centrifugal separation effect become worse. The operation of the whole system will be obstruct when the viscous particles block the outlet of the centrifuge under the extreme cases. The key to improving the speed of oil recovery was the efficient separation of petroleum substances from oily sludge, and the key to the efficient separation of petroleum substances was the choice of demulsifier and the process conditions used.

4.1. Effect of different factors on the separation effect of oily sludge in thermochemical demulsification separation

The oxidation will change the molecular structure of hydrocarbons and produce active groups mainly based on carboxyl groups during the process of oily sludge piling. Under alkaline condition, the mixture of oil and mud will produce self-emulsification. At the same time, the colloids and asphaltenes in the oily sludge contain active groups such as carboxyl groups; It is difficult to break the emulsion of oily sludge (Figure 1). Under acidic conditions, H^+ ions are combined with carboxyl groups, the self-

emulsification of the sludge mixture was lost, and the demulsification was easy to destabilize; In addition, oil beads, expanded clay minerals and flocs in oily sludge were all electronegative. The colloid was a stable state under the repulsive force of each other. After the addition of acid, the electrical properties of oil beads were changed, and the double electric layers of clay minerals were compressed, which was helpful for emulsion breaking, clay dehydration and destruction of oil, water and solid three-phase stability system [12]. The oil content of the mud cake gradually decreases with the increase of temperature (Figure 2). Heating can destabilize the emulsified system, and the molecular motion of the oil particles in the mixed solution will gradually increase with the increase of temperature, and the frequency of collision and aggregation of each particle will increase; the viscosity of the sludge will decrease with the increase of temperature, which plays a very important role in the subsequent uniform regulation of the dosing mixture, and finally achieves the purpose of oil-water separation [13]. The addition of emulsion breaking agents can play a dual role of positive charge neutralization and adsorption bridging with the mixed liquid system, further compress the double electric layer of the emulsion of oily sludge and destroy the stability of the emulsion system [14]. The demulsification effect or the emulsion demulsification of the oily sludge were not enough when the amount of emulsion breaking agent was insufficient, and the separation of petroleum substances was incomplete. On the contrary, the oily sludge system will reverse and become stable again if addition of emulsion breaking agents was excessive (Figure 3). For complex oily sludge systems, water dilution can transform the emulsifying system and facilitate the thermochemical demulsification separation of petroleum materials (Figure 4). The washed oil will form an oil-in-water emulsion in the water phase with stirring and emulsification, and the system will be stable again when the stirring speed was too high (Figure 5). Stirring can fully mix the sludge with water and the shearing force will generated during the process, which can accelerate the removal of crude oil from the muddy sand. This is because as the stirring time increases, the separated oil emulsifies to form an oil-in-water emulsion in the liquid phase. The longer the stirring time, the higher the degree of emulsification, resulting in decreased oil recovery rate and increased oil content in the mud cake (Figure 6).

4.2. The influence of various factors on the separation effect of oil sludge steam floatation stage

Aeration of normal air or ozone into the oily slurry emulsion can make oil particles whose diameter was 0.25-25 μm fall off from the mud sand, mainly dispersing oil and emulsifying oil as bubbles reach the surface of the emulsion. The effect of air floatation and oil removal were proportional to the number of bubbles produced and inversely proportional to the diameter of the bubbles. The oil content of the mud cake was basically unchanged when the aeration rates of thin oily sludge and heavy oily sludge exceed $3 \text{ m}^3 (\text{m}^3 \text{ h})^{-1}$ and $3.5 \text{ m}^3 (\text{m}^3 \text{ h})^{-1}$, respectively; and aeration time exceeds 20 min and 30 min, respectively (Figure 7). The mixture of mud and water which skimming the petroleum material scum was dehydrated by vacuum 30s and could dry and crack. The oil content of mud cake dropped to about 1%.

5. Results

The combination of thermochemical demulsification and air flotation was used to treat oily sludge, recover oil and purify the soil. Under optimized conditions, the dehydration performance of the mixed oil of the light oily sludge and the heavy oily sludge were both good, and the oil content of mud cake was less than 2%. The separation liquid is recycled to the sludge for dilution, and the subsequent separation effect was still good. After the deoiled mixture was dehydrated, the clover was planted with clover, and the seed germination and leaf growth were normal.

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