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Synthesis of nano-β-CD@Fe_3O_4 magnetic material and its application in ultrasonic treatment of oily sludge

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ABSTRACT

The extraction process of Tarim oil field in Xinjiang is accompanied by a large amount of oily sludge generation, which seriously restricts the progress of oil and gas development and causes serious pollution to the environment due to its large production, complex composition, and difficult treatment. Nanomaterials combined with ultrasound have been demonstrated to be a promising method for the disposal of hazardous oily sludge. In this paper, a magnetic material Nano-β-CD@Fe_3O_4 was prepared by hydrothermal method and surface modification method. Nano-β-CD@Fe_3O_4 can be intelligently enriched at the oil–water interface and oil-solid interface, and it can be stably dispersed to form nanofluid under the action of ultrasound. Nano-β-CD@Fe_3O_4 can cause changes in oil composition when it is exposed to ultrasound, resulting in the decrease of viscosity and increase of fluidity. The experimental results of treating oily sludge in Xinjiang Tarim showed that the best treatment effect was achieved when the concentration of Nano-β-CD@Fe_3O_4 was 0.5 %, the ultrasonic frequency was 60 Hz and the temperature was 60℃. This solution can reach 90.17 % oil removal efficiency within 45 min, and the secondary oil removal efficiency of Nano-β-CD@Fe_3O_4 recovered by magnetic separation could still reach 85.65 %. This efficient oily sludge treatment method proposed in our study provides valuable information for the development of oily sludge treatment technology.

1. Introduction

Oily sludge is a semi-solid waste generated during oil extraction, storage, and transportation. Typically composed of 15–50 % petroleum hydrocarbons, 30–85 % water and 5–46 % solid particles [11,14]. Oily sludge has the characteristics of large production and difficult disposal, it can cause serious damage to the atmosphere, water, and soil, and if not treated properly, it will result in secondary pollution to the environment [7]. It is worth mentioning that although oily sludge is a waste, it contains a large amount of recyclable hydrocarbon and non-degradable petroleum hydrocarbon resources [26], which is a renewable energy source with high potential value. Therefore, if a proper treatment method is used to separate the oil components in the oily sludge, its utilization rate can be greatly improved. For this reason, scholars have been actively exploring how to efficiently treat oily waste to achieve the recovery and utilization of oil and gas resources. At this stage, the commonly used methods at home and abroad mainly include solvent extraction, mechanical centrifugation, surfactant cleaning, pyrolysis, ultrasonic emulsion breaking, supercritical state separation, and processing, etc. [3,18,20,30]. However, these methods are inefficient and reveal drawbacks such as high energy consumption and long processing cycles.

Therefore, we need to explore a new method to treat oily sludge in simple, low energy consumption and high effectiveness. Ultrasound is an elastic mechanical wave that propagates in a medium. Due to its unique mechanical vibration, cavitation, and thermal effects [26], in recent years, scholars have applied ultrasound to the treatment of oily sludge to improve oil fraction recovery [12]. Compared to traditional cleaning technologies, ultrasound can release high energy density to break down sludge agglomerates and further remove absorbed contaminants on solid particles. Xu et al. observed that flocculated oily sludge decomposed into small particles under ultrasonic irradiation [32]; Gao et al. applied ultrasound to treat oily sludge on a pilot scale and found that sufficient acoustic pressure amplitude was the key to overcomeing

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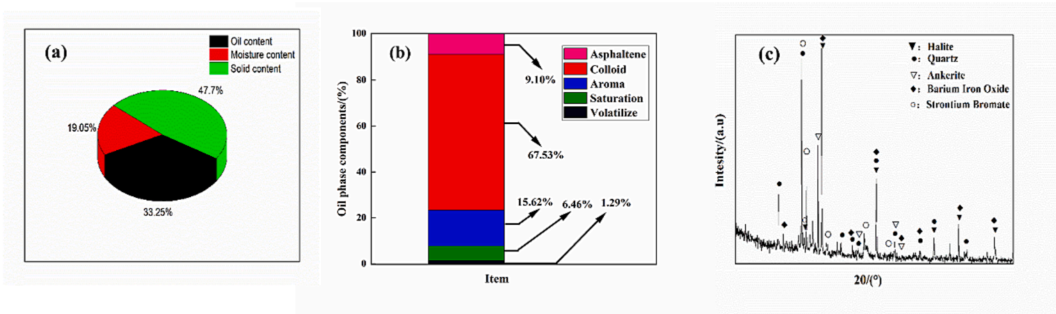

Fig. 1. (a) Three-phase; (b) Oil phase components; (c) XRD of Solid.

adhesion in the oily sludge [9]; Luo et al. investigated the effects of acoustic parameters (frequency, acoustic intensity and treatment time) and operating conditions (water sludge ratio, pH and surfactant) on the recovery of oily sludge [19]). The complexity of oil-bearing sludge components makes it impossible to achieve satisfactory oil removal by ultrasound alone. Kim et al. investigated the combined ultrasound-alkali pretreatment of oily sludge to achieve solubilization of oil fraction [13]. Gao et al. found that the removal rate of oily sludge after ultrasonic cleaning was 82–90 % with the assistance of surfactant washing [6]. In most studies, scholars have introduced chemical cleaning agents, catalysts, and nanomaterials to improve the treatment efficiency based on ultrasound.

Recently, magnetic nanoparticles (MNP) have been widely implemented in various sectors like medicine, food, electronics, oil, and gas owing to their distinctive properties, including nano-scale size, quantum effects, and massive surface area [2,24,25,28]. Taheri et al. exploited the synergistic interaction between magnetic nanoparticles and ultrasound for efficient catalysis [27]; Abdullah achieved good results in the separation of methylene blue in water using ultrasound in combination with Fe_3O_4 [1]. The synergistic application of ultrasound and magnetic nanoparticles in the treatment of oily sludge is also an important problem that needs to be studied urgently.

In this paper, an intelligent magnetic material Nano-β-CD@Fe_3O_4 was synthesized using a hydrothermal method and surface modification, which has strong magnetic properties and can be adsorbed at the oil–-water interface. In this study, Nano-β-CD@Fe_3O_4 was combined with ultrasonic waves for oily sludge treatment in Xinjiang Tarim block to improve the treatment efficiency.

2. Experimental studies

2.1. Experimental sample

2.1.1. Xinjiang Tarim block oil sludge

The oily sludge was used in Xinjiang Tarim block in this experiment. The sample sludge has good fluidity. The physical and chemical properties of oily sludge were analyzed, as shown in Fig. 1.

Fig. 1(a) is a three-phase mass ratio diagram. It can be seen from Fig. 1(a) that the oil content of the oily sludge can reach 33.25 %, and the recoverable components are high; the moisture content is small, which is 19.05 %; the solid phase accounted for 47.7 %. Fig. 1(b) shows the content of oil phase components. From Fig. 1(b), it can be seen that the relative proportion of asphaltene in the oil component is small, and the cross-linking between the three phases is weak. Fig. 1(c) shows the XRD pattern of the solid phase in the oily sludge. It can be seen that the solid phase of the oily sludge is mainly basic mineral component. Through analysis and investigation of oily sludge, mainly uses oil as the continuous phase and water and solid matter as the dispersed phase. Therefore, in order to recover the oil components and discharge the solid content, it is necessary to realize the stripping recovery of the oil components.

2.1.2. Materials and experimental apparatus

Materials: $FeCl_3 \cdot 6H_2O$(number average molecular weight: 50,000, AR ≥99.0 %), H_2NCONH_2 (number average molecular weight: 50,000, AR ≥99.0 %), Beta-Cyclodextrin (β-CD, number average molecular weight: 25,000, AR ≥99.0 %), Ethylene Glycol($(CH_2OH)_2$, number average molecular weight: 50,000, AR ≥99.5 %), Polyethylene glycol ($HO(CH_2CH_2O)n$, number average molecular weight: 50,000, AR ≥99.0 %), H_2O_2 (35 wt% solution in water, stabilized).

Experimental Apparatus: Ultrasonic Cleaner (KQ3200DE), Vacuum Drying Oven, PTFE Reactor. Stability Analyzer (Formulaction-MLS), Fourier transform infrared spectroscopy (FTIR)(BRUKER, TENSORII) and XRD patterns (BRUKER, AXios) were used for the structural characterization. The field emission environmental scanning electron microscope (SEM, Gemini SEM 300) was used for the morphological observations test.

2.2. Synthesis of Nano-β-CD@Fe_3O_4 treatment agent

2.2.1. Preparation stage

First, 1.15 g of ferric chloride hexahydrate ($FeCl_3 \cdot 6H_2O$) was dissolved in 30 mL of ethylene glycol (EG) and magnetically stirred to obtain a homogeneous orange liquid. Then 2.0 g of urea (H_2NCONH_2) and 1.0 g of polyethylene glycol 2000 were added to the orange liquid and magnetically stirred to make it fully dissolved and form a uniform solution. Third, the uniform solution obtained in the previous step was added to the reaction vessel and kept at 200 °C for 18 h, 20 h, 22 h, and 24 h after sealing. Finally, after cooling the reaction vessel to 25 °C, the product was transferred to a beaker, washed repeatedly with deionized water and ethanol, and dried to obtain a black powder, which is the Fe_3O_4 precursor. Fe_3O_4 with different synthesis times were tested and analyzed by XRD diffraction analyzer and SEM microscopic analyzer.

2.2.2. Experiment procedure

Firstly, 0.5 g of the prepared nano Fe_3O_4 precursor was taken out and evenly dispersed in ultrasonication, then 1.5 g of β-CD was dissolved in 100 mL of distilled water to obtain a uniform solution. Then, the pH value of the nano Fe_3O_4 solution was adjusted to 12 using a 5 % NaOH aqueous solution. Then, the β-CD solution was added to the nano Fe_3O_4 solution. Followed by 9 mL of H_2O_2 which was added to the solution three times at an interval of 1 h. The product Nano-β-CD@Fe_3O_4 was obtained.

Finally, the solution was placed in an ultrasonic field at 60 °C for 8 h. After the reaction, the product was magnetically separated and washed several times with deionized water and ethanol. The modified Nano-β-CD@Fe_3O_4 was analyzed by FTIR spectroscopy.

2.3. The performance test of Nano-β-CD@Fe_3O_4 intelligent, magnetic, and dispersion

The intelligent and magnetic properties of β-CD modified Fe_3O_4 were tested and studied, and the stability of Nano-β-CD@Fe_3O_4 dispersion

Table 1
L_9 (3^4) orthogonal experimental formula.

Level	Factor		
	A (Density/%)	B (Ultrasonic frequency /Hz)	C (T/℃)
0	0.3	60	30
1	0.5	80	45
2	1	100	60

under the action of ultrasound was tested by Stability Analyzer (Formulaction-MLS).

2.4. Testing the effect of ultrasonic synergistic Nano-β-CD@Fe_3O_4 on the rheological properties of oil components in oily sludge

The oil fraction separated from the oily sludge was taken and reacted with the oil fraction by ultrasound, which was combined with Nano-β-CD@Fe_3O_4 at 90 °C for 8 h. The viscosity change of the oil fraction before and after the reaction was tested by Thermo Scientific HAAKE Rheostress.

2.5. Degreasing experiment

By designing orthogonal experiments to explore the influence of treatment agent concentration, ultrasonic frequency, and temperature on oil removal efficiency, the best conditions can be optimized. The horizontal distribution of orthogonal experiment factors is shown in Table 1.

The concentration was set in the table to 0.3 %, 0.5 %, and 1 % respectively. Including ultrasonic frequency which has been set to 60 Hz, 80 Hz, and 100 Hz. Considering that environmental protection processing requires low energy consumption processing, the temperature was selected at 30 °C, 45 °C, and 60 °C. 20 mL of treatment agent concentration was taken to carry out a degreasing experiment on 4.5 g of oily sludge. Under optimal conditions, the relationship between ultrasonic treatment and combined ultrasonic treatment over time was compared. The oil removal efficiency in 45 min was explored and the curve to analyze the molecular dynamics of the oil removal process was fitted.

3. Results and discussion

3.1. Characterization of Nano-β-CD@Fe_3O_4 treatment agent

The XRD component analysis and microscopic analysis of Fe_3O_4 prepared at different holding times were performed and the results are shown in Fig. 2.

Fig. 2(a) shows the XRD pattern of nano-Fe_3O_4 synthesized at different holding times at 200 ℃. In the figure, the synthesis time from bottom to top is 18 h, 20 h, 22 h, and 24 h respectively. Through the analysis of the XRD peak spectrum, it is found that the Fe_3O_4 synthesized under 20 h has good crystallinity, the crystal peak diffraction angle is sharp, and the diffraction peak gradually becomes flat with the increase of temperature. Combined with Fig. 2(b) (SEM images of nano-Fe_3O_4 with different holding times), it is also found that as the synthesis time increases, the spherical structure gradually breaks down and becomes dispersed particles. The nano-Fe_3O_4 microspheres synthesized under 20 h are uniform in size (all between 200 nm and 300 nm), and the structure is good. Therefore, nano-Fe_3O_4 microspheres synthesized under a hydrothermal holding time of 20 h are selected as the modified precursor.

Cyclodextrin, an important class of polysaccharides with a cyclic oligosaccharide, is formed by the action of cyclodextrin glucosyltransferase on starch and belongs to the cage-type molecule family. The most common β-cyclodextrin consists of 7 α-d-glucopyranose units connected through α-(1,4) glucosidic linkers [5,21]. The structure of β-cyclodextrin is characterized by a dimensionally stable hydrophobic cavity and hydrophilic outer surface with primary hydroxyl groups lying on the above and the secondary hydroxyl groups below. When cyclodextrins are grafted on spherical nano-Fe_3O_4, the new material Nano-β-CD@Fe_3O_4 can have both magnetic and amphiphilic properties, comparing the FTIR spectra of ferric tetroxide before and after the modification, as shown in Fig. 3.

Fig. 3(a) shows the FTIR spectroscopy of Fe_3O_4 and Fig. 3(b) shows the FTIR spectroscopy of Fe_3O_4 after modification. By analyzing the

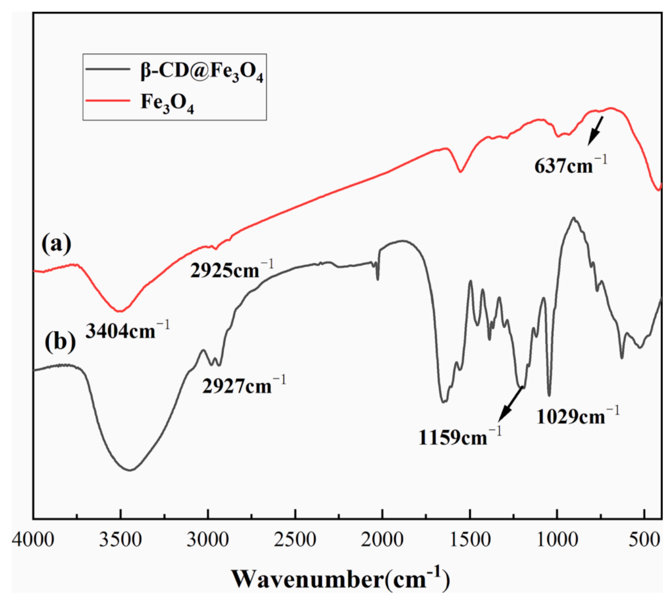

Fig. 3. (a)FTIR spectroscopy of Fe_3O_4; (b)FTIR spectroscopy of Nano-β-CD@Fe_3O_4.

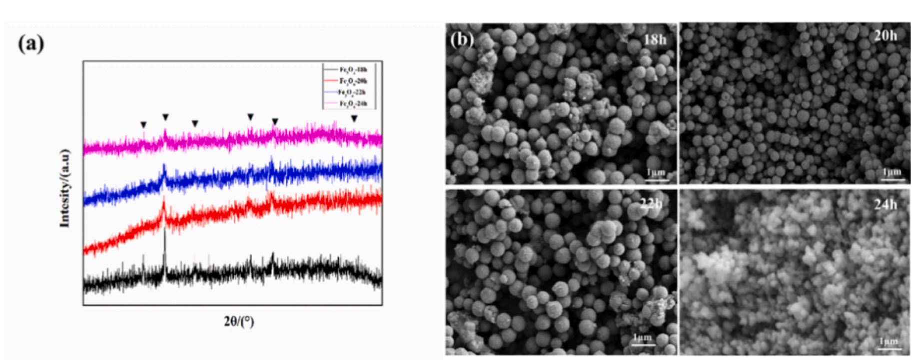

Fig. 2. (a) XRD of nano-Fe_3O_4; (b) SEM of nano-Fe_3O_4.

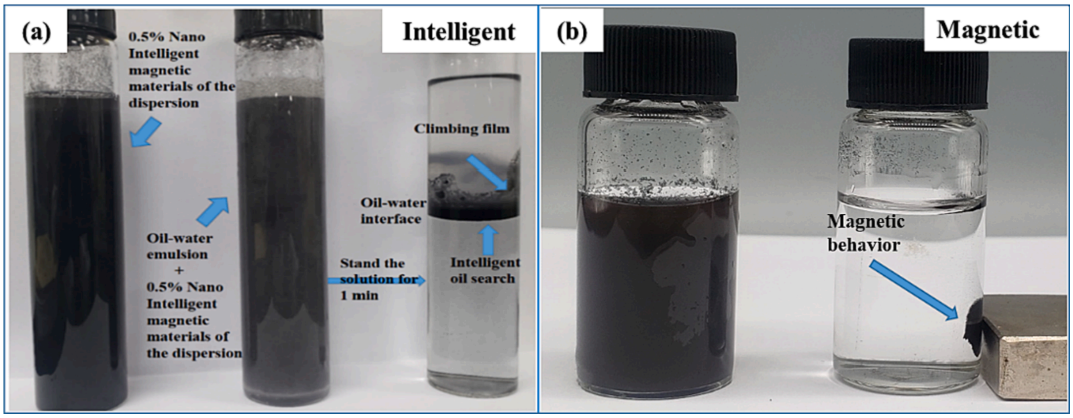

Fig. 4. (a) intelligent oil–water interface seeking of Nano-β-CD@Fe_3O_4; (b) Magnetic adsorption of Nano-β-CD@Fe_3O_4.

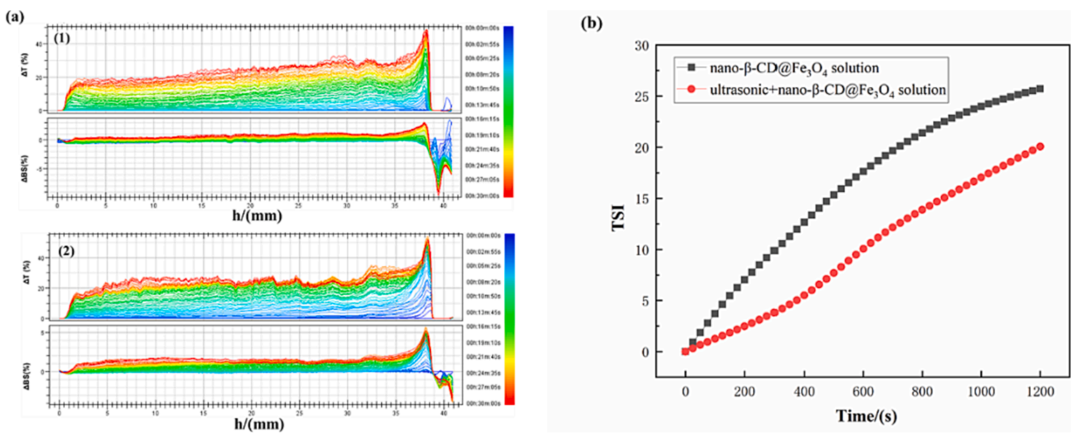

Fig. 5. Effect of ultrasonic synergistic Nano-β-CD@Fe_3O_4 on the rheological properties of oil components in oily sludge.

graph, 2927 cm^{-1}, 1159 cm^{-1},and 1029 cm^{-1} are the characteristic absorption peaks of β-CD. It can be concluded that β-CD is successfully combined with nano-Fe_3O_4 microspheres to form a cyclodextrin coating shell, and the modified product is Nano-β-CD@Fe_3O_4.

3.2. *Performance analysis of Nano-β-CD@Fe_3O_4*

The performance of the prepared Nano-β-CD@Fe_3O_4 was investigated to ensure its application in the treatment of oily sludge.

Fig. 4(a) is an intuitive diagram of intelligent oil search. Through the analysis of this figure, the synthesized nano smart material can be adsorbed at the oil–water interface and can form a climbing film on the oil-solid interface. In the process of combining with oily sludge, Nano-β-CD@Fe_3O_4 will be enriched at the oil–water and oil-solid interface, destroying the original structure of oily sludge and achieving three-phase separation of oily sludge in the combined treatment with ultrasonic. Fig. 4(b) shows the magnetic adsorption phenomenon of Nano-β-CD@Fe_3O_4 fluid under the applied magnetic field, and the strong magnetic properties can realize the recycling of Nano-β-CD@Fe_3O_4 in the application process.

Fig. 5 shows the dispersion stability test of Nano-β-CD@Fe_3O_4 under ultrasonication. The results show that Nano-β-CD@Fe_3O_4 TSI values are smaller and have better stability to form stable nanofluid in an ultrasonic environment, which confirms that when Nano-β-CD@Fe_3O_4 and ultrasonication are used in conjunction, it can be dispersed and adsorbed on various surfaces of oily sludge for the deconstruction of oily sludge under the synergistic effect of ultrasonication.

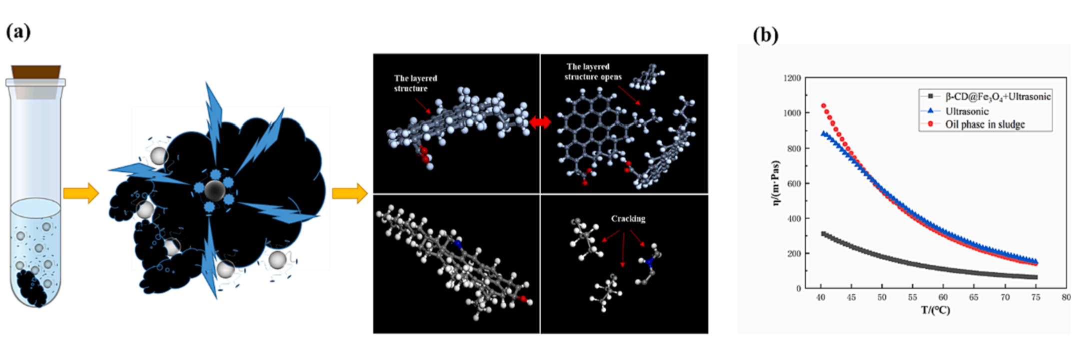

Fig. 6. (a) destructive effect of ultrasound combined with Nano-β-CD@Fe_3O_4 on the structure of oily sludge; (b) rheological properties of the oil phase in oily sludge before and after the reaction.

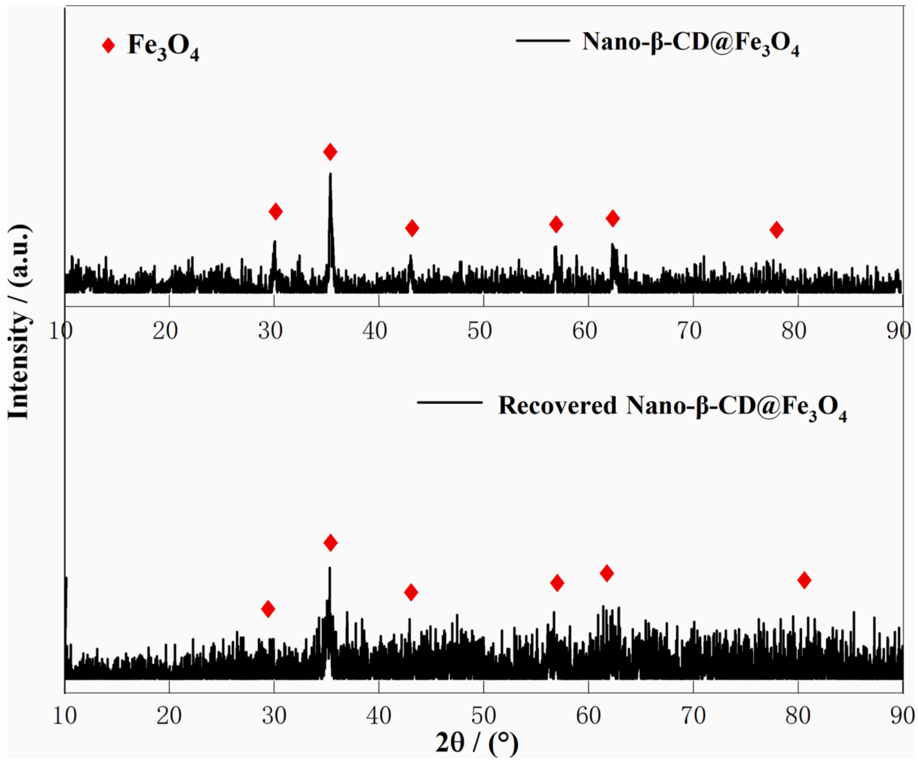

Fig. 7. XRD of Nano-β-CD@Fe_3O_4 before and after the cleaning reaction.

3.3. Effect of nano-β-CD@Fe_3O_4 synergistic sonication on the rheological properties of oil components

Oily sludge is mainly a mixture composed of oil phase wrapped with tiny solid phase particles and a small amount of water, where gum and asphaltene are the main reasons for the solid cementation of the mixture [4,23]. The key to achieving the deconstruction and destruction of oily sludge is to achieve the stripping of the oil phase and water, oil phase and solid phase [10]. Nano-β-CD@Fe_3O_4 can be adsorbed at the oil–-water interface and oil-solid interface and realize the enrichment at the interface, which provides powerful conditions for the cavitation of ultrasound [17,22].

Ultrasound and the usual acoustic wave propagation in the medium is a linear motion. The speed of motion is related to the medium, and all kinds of sound transmission mediums have a fixed acoustic impedance rate [8,34]. As the resistance of iron and oil, water is very different, therefore when the ultrasonic wave travels to the interface of the two media with a large difference in acoustic impedance, the main reflection occurs, the reflection of the ultrasonic wave back and forward in the synthesis of the ultrasonic wave, when each point of the phase difference remains stable, resonance occurs, and in some fixed positions superimposed on each other to strengthen the medium in these positions easy to produce cavities. [16,31]. The energy emitted during the destruction of the cavity can be transferred to the dirt and cause their dissociation and dispersion through the interaction of the media masses with the dirt particles [6].

As shown in Fig. 6(a), ultrasound combined with Nano-β-CD@Fe_3O_4 can achieve the dissociation and dispersion of oily sludge and can lead to the dispersion of asphaltene molecules in the oil fraction, as well as the breakage of long chains. From the viscosity-temperature curve in Fig. 6 (b), it can be seen that the viscosity of the oil fraction decreases, and the mobility is enhanced after the reaction of ultrasonic combined with Nano-β-CD@Fe_3O_4.

Fig. 7 shows the XRD patterns of Nano-β-CD@Fe_3O_4 before and after the sludge cleaning reaction. From the figure, it is observed that the diffraction peak signal of Fe_3O_4 diminishes after the reaction, indicating the presence of partially dissolved Fe_3O_4. However, the basic crystal shape can be maintained, which confirms that Nano-β-CD@Fe_3O_4 has a recovery potential and is less affected by oily sludge and ultrasonication.

3.4. Analysis of the results of ultrasonic synergistic Nano-β-CD@Fe_3O_4 degreasing

It can be seen from the trend chart in Tab.2 that when the solution concentration is 0.5 %, the oil removal rate is the highest. This is because Nano-β-CD@Fe_3O_4 is dispersed and enriched at the oil–water and oil-solid interface. Ultrasonic waves produce a large number of cavitation cores on the surface of Nano-β-CD@Fe_3O_4 and cause Nano-β-CD@Fe_3O_4 to generate mechanical vibrations, making crude oil easier to be stripped. When the concentration is too low, Nano-β-CD@Fe_3O_4 cannot be completely enriched at the oil-solid and oil–water interface, resulting in low efficiency [29]. When the concentration is too high, the Nano-β-CD@Fe_3O_4 accumulates to form micelles, which consume ultrasonic energy to disperse and produce resistance to the separation of oil components. The ultrasonic frequency directly affects the cavitation threshold of the cleaning liquid: the higher the ultrasonic frequency, the higher the cavitation threshold. Therefore, the higher the energy required for the occurrence of the cavitation phenomenon, which makes it difficult for the cavitation effect to occur. Therefore, it is concluded that the oil removal efficiency is the highest at 60 Hz. As the oily sludge contains a lot of colloids and asphaltenes, when the cleaning temperature increases from 35℃ to 60℃, the viscosity of the oily sludge decreases. Nano-β-CD@Fe_3O_4 can absorb heat energy to reduce the adhesion of crude oil. At the same time, the movement resistance of each particle in the system is reduced, the movement speed is accelerated [15], and the crude oil and the sediment particles are separated under the Nano-β-CD@Fe_3O_4 slip. In addition, when ultrasonic waves propagate in water, the sound energy density, sound velocity, and temperature have the following relationships [33]:

C = 331.4 + 0.16 t3-1.**

Table 2
$L_9(3^4)$ orthogonal experimental formula.

Level	Factor		
	A(Density)	B(Ultrasonic frequency /Hz)	C(T/℃)
0	0.3 %	60	30
1	0.5 %	80	45
2	1 %	100	60

Samples number	Factor			
	A	B	C	Oil removal rate
1	0	0	0	54.93 %
2	0	1	1	49.53 %
3	0	2	2	67.43 %
4	1	0	1	59.54 %
5	1	1	2	69.19 %
6	1	2	0	44.96 %
7	2	0	2	61.33 %
8	2	1	0	41.73 %
9	2	2	1	49.12 %

$E = 1/2\rho\omega^2 cA^2$ 3-2.

Formulas 3–1 and 3–2, show that the sound energy density and wave speed of ultrasonic waves is proportional to temperature, which means that the higher the temperature, the faster the ultrasonic wave speed and the higher the sound energy density, The more ultrasonic energy Nano-β-CD@Fe_3O_4 accepts, the better it is beneficial to the treatment of oily sludge (see Table 2).

Fig. 8 is a visual diagram of the orthogonal experiment after degreasing. In this figure, the upper layer is the oil phase, the middle layer is low-oily sewage, and the bottom layer is the solid phase. Experiments have verified that β-CD@ Fe_3O_4 combined with ultrasonic can be used to treat oily sludge, it enables the separation of oil sludge components and effective oil component recovery. Through elemental analysis of the separated solid phase, as shown in Fig. 9, the results show that the solid phase component has a low heavy metal content and meets the emission standard.

The best experimental conditions for treating oily sludge were screened out through orthogonal experiments. Under the best experimental conditions, the degreasing efficiency of Nano-β-CD@Fe_3O_4 with 0.5 % concentration of water and ultrasound was compared with that of Nano-β-CD@ Fe_3O_4 with ultrasound to confirm the interface effect of Nano-β-CD@Fe_3O_4. At the same time, the influence of Nano-β-CD@Fe_3O_4 on ultrasonic cavitation and mechanical vibration is also verified.

As shown in Fig. 10, within 45 min, Nano-β-CD@Fe_3O_4 combined with ultrasonic treatment can reach 90.17 %, which is 40.24 % higher than the efficiency of water plus ultrasonic. The recovery, cleaning, and dispersion of Nano-β-CD@Fe_3O_4 are carried out by magnetic separation, and the efficiency of the combined ultrasonic degreasing of Nano-β-CD@Fe_3O_4 after recovery can reach 85.65 %. It is proved that Nano-β-CD@Fe_3O_4 can be recycled.

4. Conclusion

In this study, a microsphere material Nano-β-CD@Fe_3O_4 with a particle size of about 200 nm was synthesized by hydrothermal and surface modification methods using $FeCl_3$-$6H_2O$ and β-CD as raw materials for the treatment of oily sludge. This material has strong magnetic properties and can be intelligently enriched at the oil–water interface and oil-solid interface. In the process of oily sludge cleaning in Xinjiang Tarim block, a treatment method using ultrasound combined with Nano-

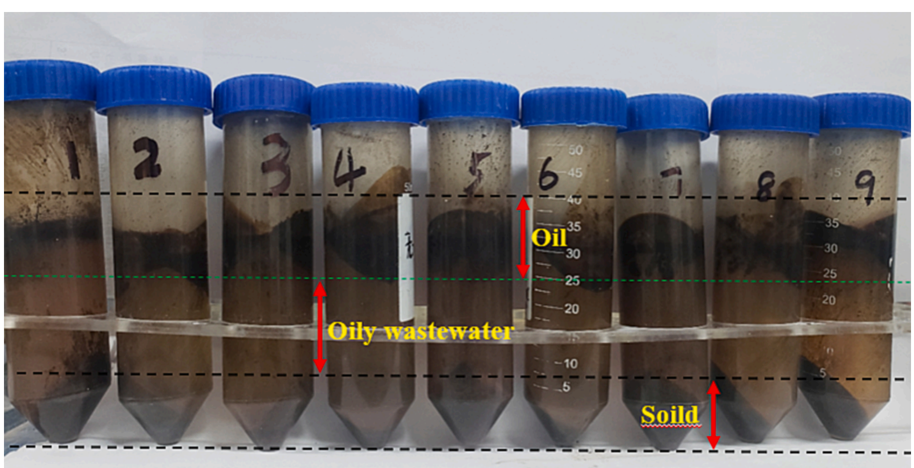

Fig. 8. Component separation.

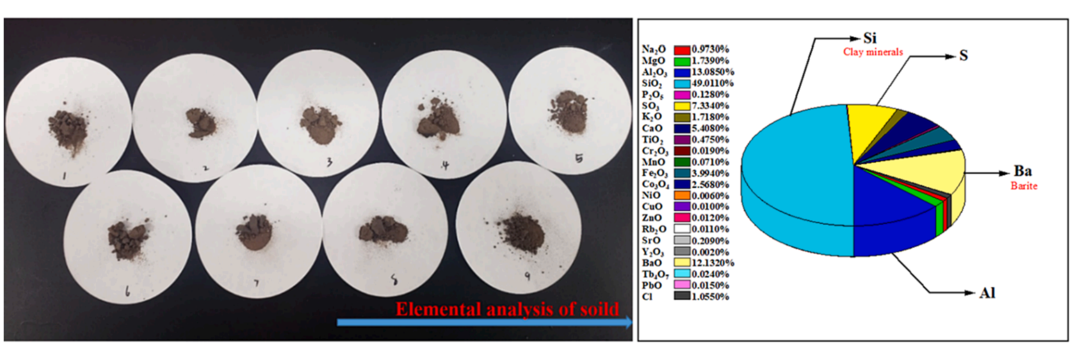

Fig. 9. Elemental analysis of solid phase.

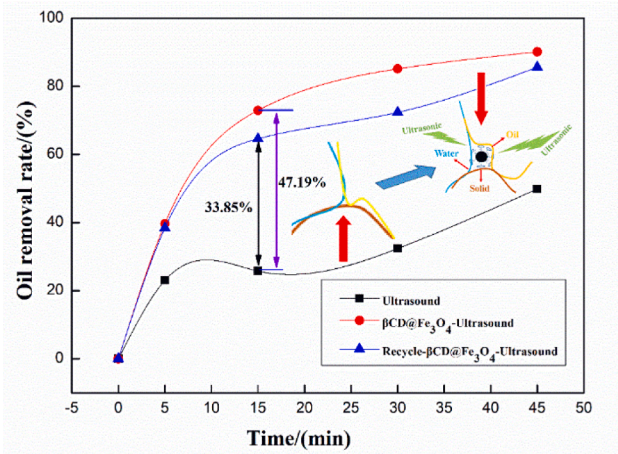

Fig. 10. Oil removal efficiency curve.

β-CD@Fe_3O_4 is proposed. Nano-β-CD@Fe_3O_4 can be stably dispersed under the action of ultrasound, and its adsorption at the oily sludge phase interface provides a strong condition for the cavitation of ultrasound and improves the energy transfer of ultrasound. The treatment efficiency of oily sludge can reach 90.17 % at 0.5 % β-CD@Fe_3O_4 concentration, 60 Hz, 60 °C optimum conditions, and Nano-β-CD@Fe_3O_4 can be recovered and reused by magnetic separation, and the oil removal efficiency can reach 85.65 % after reuse.

CRediT authorship contribution statement

Song Hanxuan: Conceptualization, Methodology, Writing – original draft. **Ye Yan:** Funding acquisition, Project administration. **Zheng Weiru:** Investigation, Formal analysis. **Essouma Essouma Ariane Felicite Bibiche:** Writing – review & editing. **Zhang Qingwen:** Writing – review & editing. **Guo Jixiang:** Writing – review & editing.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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