Research Article

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Development and performance evaluation of a novel environmentally friendly adsorbent for waste water-based drilling fluids

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Abstract: Adsorbent is an important waste water-based drilling fluid treatment agent, which can adsorb and settle heavy metal ions, high polymer organics, and other soluble harmful substances in the waste drilling fluid. Traditional adsorbents such as polyaluminum chloride and polyacrylamide will produce other metal ions or toxic monomers after hydrolysis, which cannot fully meet the requirements of safety and environmental protection. Therefore, a new environmentally friendly waste water-based drilling fluid adsorbent, named RH-β-CD, was prepared by the Wilson etherification reaction, which was initiated by epichlorohydrin and ceric ammonium nitrate, and successfully grafted rhamnolipid and amine strong adsorption groups onto β-cyclodextrin. The adsorption effect and environmental protection performance of RH-β-CD on the organic matter and chromium ion in waste sulfonated water-based drilling fluid were evaluated and compared with commonly used adsorbents such as activated carbon, PAM, and polyaluminum chloride. The results show that RH-β-CD can effectively adsorb the organic matter in the filtrate of waste water-based drilling fluids, reduce its chemical oxygen consumption, and reduce the concentration of heavy metal ions in the filtrate. The effect is better than PAM, activated carbon, and polyaluminum chloride, with the BOD_5/COD_{cr} >20% and EC_{50} >1,000,000 $mg\cdot L^{-1}$, which is environmentally friendly.

Keywords: waste water-based drilling fluids, cyclodextrin, adsorbent, heavy metal pollution, environmentally friendly

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1 Introduction

With the continuous development of drilling engineering in our country's oil and gas field, the demand for drilling fluid has increased sharply. This makes waste drilling fluid treatment one of the key technologies that need to be solved urgently [1–3]. As an important treatment agent for the waste drilling fluid, the adsorbent can effectively adsorb heavy metal ions, high polymer organic matter, and other harmful substances dissolved in the liquid phase in the waste drilling fluid and settle by agglomeration and adsorption between particles, which is beneficial to the waste drilling liquid that finally achieves harmless treatment [4–6]. In recent years, domestic and foreign scholars have developed a large number of different types of adsorbents for the harmless treatment of the waste drilling fluid [7–9]. Among them, the porous structure and large specific surface area of activated carbon make it an adsorbent in the adsorption process to show the advantages of fast adsorption and good adsorption effect, but its limitation is that it is a nonselective adsorption process and may cause certain environmental problems [10]. Traditional adsorbents such as polyaluminum chloride and polyacrylamide will produce other metal ions or toxic monomers after being hydrolyzed and cannot fully meet the increasingly stringent requirements of safety and environmental friendly on-site [11].

β-Cyclodextrin is an oligosaccharide with a hollow circular truncated cone structure, which is connected by a-1,4-glycosidic bonds by multiple D-glucopyranoses. It has the characteristics of being hydrophobic inside the ring and hydrophilic outside the ring. It is widely used in medicine, food, sewage treatment, and other fields [12–15]. On the one hand, β-cyclodextrin can interact with a variety of guest molecules by noncovalent bonds to form host and guest compounds [16]. Lu et al. [17] used the host–guest interaction between cyclodextrin and azo compounds to prepare responsive supramolecular assemblies. Li [18] used the host–guest interaction between cyclodextrin and adamantane to synthesize targeted compounds. On the other hand, the surface of cyclodextrin has a large number of active hydroxyl groups, and different kinds of functional compounds can be obtained through chemical modification [14,19,20]. Qu et al. [21] used cellulose and cyclodextrin to carry out the cross-linking reaction to synthesize modified cyclodextrin compounds with adsorption of heavy metal pollutants. Zhong et al. [22] used cyclodextrin and epichlorohydrin for the cross-linking reaction and obtained cyclodextrin polymer microspheres with excellent thermal stability. Zhang et al. [23] used epichlorohydrin, corn starch, and γ-cyclodextrin for the cross-linking reaction and obtained a polymer that can absorb dye wastewater. Zheng et al. [12] used starch and cyclodextrin to carry out the cross-linking reaction to prepare a porous absorbent material with excellent adsorption performance. Therefore, modified cyclodextrin compounds have good application value in industrial wastewater treatment, heavy metal adsorption, etc. [24].

Research at domestic and overseas have shown that rhamnose as an adsorbent has the advantages of high mechanical strength, good adsorption effect, short adsorption time, and its inclusion and adsorption effect on some organic and inorganic substances, but it also has the shortcomings of lack of selective adsorption and insufficient chemical stability [25–27]. The molecular structure of polymers containing strong amine adsorption groups is changeable, the degree of protonation in aqueous solution is high, strong multipoint adsorption can occur, and the chemical stability and mechanical strength are high [28,29]. In this article, in response to the above problems, combined with related research results, using Wilson's ether formation reaction, under the initiation of epichlorohydrin and cerium ammonium nitrate, rhamnolipid and amine strong adsorption groups were successfully grafted to β-cyclodextrin, a new environmentally friendly waste water-based drilling fluid adsorbent (RH-β-CD) containing hydrophobic polycyclic rings, carbonyl groups, ether bonds, and other groups was prepared and its performance was evaluated.

2 Materials and methods

2.1 Materials

Polymers containing an imine structure, hydrochloric acid, sodium hydroxide, trimethylamine, ethanol, anhydrous sodium carbonate, glutaraldehyde, and epichlorohydrin were provided by Aladdin Reagent Company as analytical reagents. β-Cyclodextrin was provided by Jinan Lvke Biological Technology Co., Ltd in technical grade. Monorhamnolipid was provided by Zhengzhou Xinghui Biological Technology Co., Ltd in technical grade. Sodium sulfite, perchloric acid, NaCl, cerium ammonium nitrate, and $CaCl_2$ were provided by Sinopharm Reagent Co., Ltd as analytical reagents. Sodium-based bentonite was provided by Weifang Huawei Bentonite Group Co., Ltd. Sulfonated water-based drilling fluid was taken from the drilling site of a well in the southern Sichuan block.

2.2 Synthesis of RH-β-CD

The preparation process of the new environmentally friendly waste water-based drilling fluid adsorbent (RH-β-CD) is shown below, and its molecular structure diagram is shown in Figure 1.

(1) Add a certain amount of rhamnolipid (RHA) and epichlorohydrin into a three-necked flask; then, add 60 mL of toluene and a small amount of perchloric acid, reflux, and allow to react at 80°C for 2 h. Then, filter with suction. Wash the filter cake with acetone three times and dry at 50°C to obtain the product.
(2) Weigh a certain amount of the above product into a three-neck flask; then. add a certain amount of β-cyclodextrin (β-CD), anhydrous sodium carbonate, and distilled water, stir evenly, reflux for 3 h at 60°C, and then filter with suction and wash the filter cake three times with acetone, and finally, dry to obtain the intermediate product CTS-β-CD;
(3) Weigh a certain amount of CTS-β-CD and disperse it in a certain amount of methanol; add 10 mL of 40% sodium hydroxide aqueous solution and then add a certain amount of 3-chloro-2-hydroxypropyl trimethyl chloride at 50°C. Ammonium aqueous solution and polymer containing the imine structure (R-PAI) are reacted for a period of time and then filter with suction. Wash the filter cake three times with ethanol and acetone respectively and dry at 50°C to obtain the target product to obtain as the product RH-β-CD.

3 Results and analysis

3.1 FTIR of RH-β-CD

The FTIR of RH-β-CD is shown in Figure 2. It can be seen that the characteristic absorption peaks of $–CH_3$ are at 2,966 and 2868.22 cm^{-1}, the stretching vibration absorption peaks

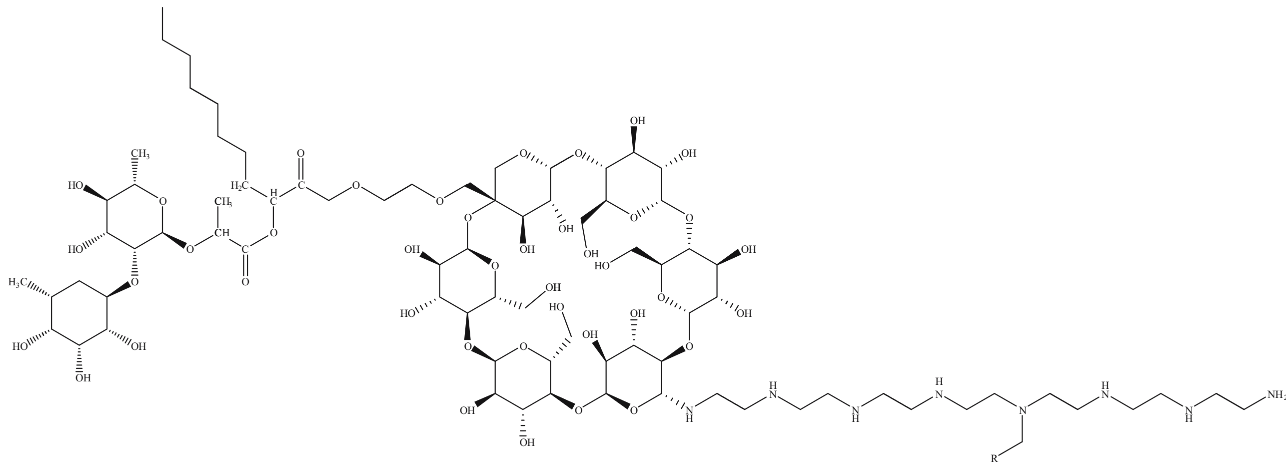

Figure 1: Schematic diagram of the molecular structure of a new environmentally friendly waste water-based drilling fluid adsorbent RH-β-CD.

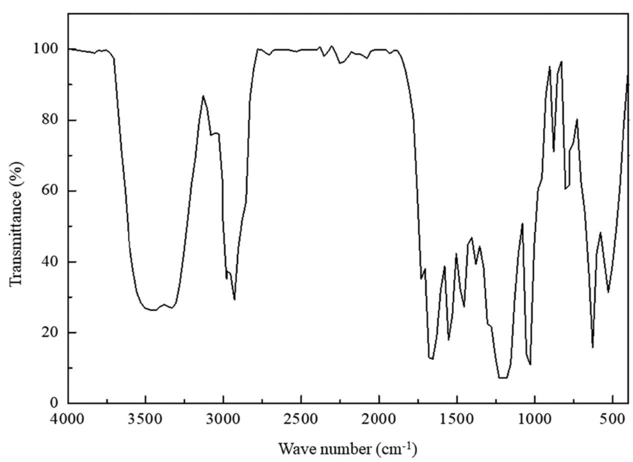

Figure 2: The FTIR image of RH-β-CD.

of C═O and the characteristic absorption peaks of primary/secondary/tertiary amides at 1650.50, and 1658.66 cm^{-1} is the stretching vibration of CO in the polysaccharide structure of the cyclodextrin molecule, 1016.47 cm^{-1} is the stretching vibration absorption peak of CO in –COO–, 1255.60 and 1208.06 cm^{-1} are the vibration absorption peaks of $–C(CH_3)–$ in the rhamnolipid molecule, the vibration absorption peak of C═O connected to the heterocyclic ring at 1350.22–1310.36 cm^{-1}, and the absorption peak of the amine group N–H appears at 1640.25 cm^{-1}. This shows that all the monomers have reacted under the action of the cross-linking agent, and the molecular chain of the synthesized product has the chain-link of the raw material.

3.2 H-NMR analysis

The H-NMR analysis of RH-β-CD is shown in Figure 3. The results show that the chemical shift δ appears at 3.6–4.0 at the proton peak on the carbon chain of cyclodextrin, the chemical shift δ at 1.3 corresponds to the proton peak on the long carbon chain of the rhamnolipid molecule,

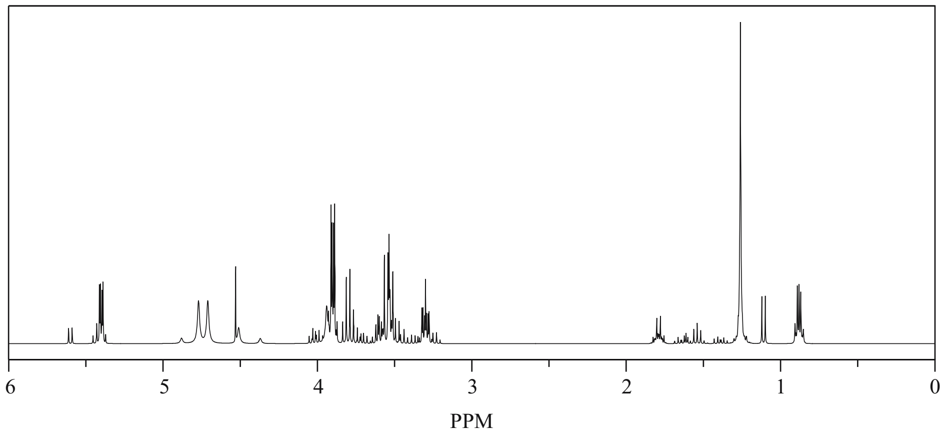

Figure 3: H-NMR spectrum of RH-β-CD.

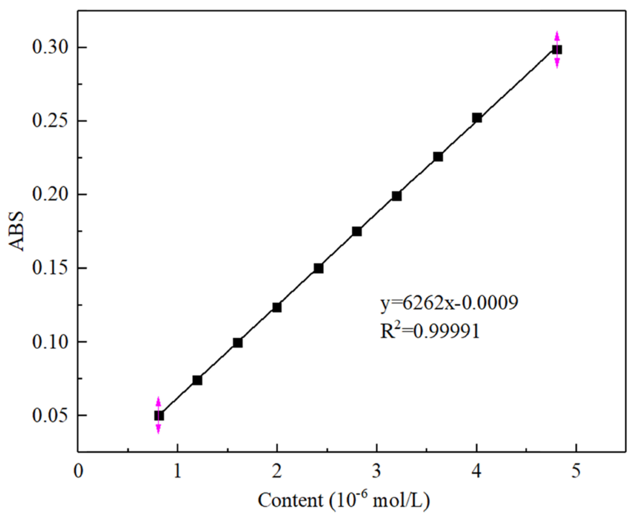

Figure 4: The concentration–absorbance linear adsorption fitting curve of RH-β-CD.

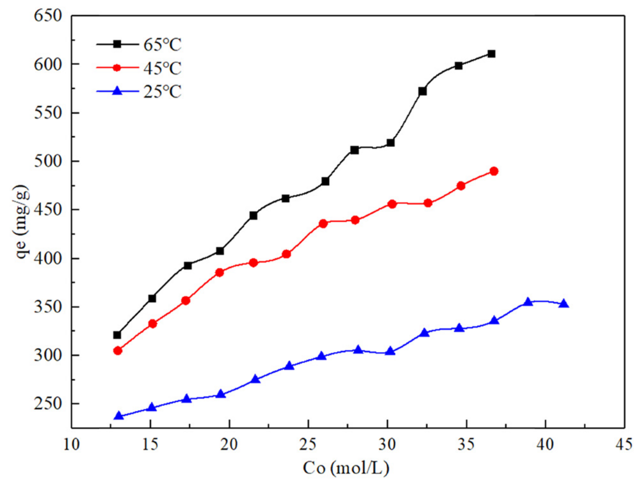

Figure 5: The adsorption isotherm of RH-β-CD.

the chemical shift δ at 4.6 is the methyl proton peak on the cyclodextrin acetyl group, the peak of chemical shift δ at 5.4 is the proton peak of the hydroxyl group on the cyclodextrin backbone, and the chemical shift δ at 3.5–3.9 is the proton peak connected to the nitrogen atom in cyclodextrin. It shows that the rhamnolipid and cyclodextrin have successfully reacted.

3.3 Evaluation of adsorption performance

The concentration–absorbance linear adsorption fitting curve and the adsorption isotherm of RH-β-CD are shown in Figures 4 and 5. It can be seen that the adsorption isotherms of RH-β-CD at different temperatures are roughly linear, indicating that the adsorption process of the treatment agent in the solution is uniform and a single-layer adsorption process. The adsorption process depends on the surface adsorption of a certain number of active sites, which is a chemical adsorption process [30]. The single-layer adsorption can effectively enhance the adsorption strength and adsorption capacity of RH-β-CD, thereby ensuring that it has a better adsorption effect.

3.4 Particle size distribution

The particle size distribution is shown in Figure 6. The results show that after adding RH-β-CD, the particle size

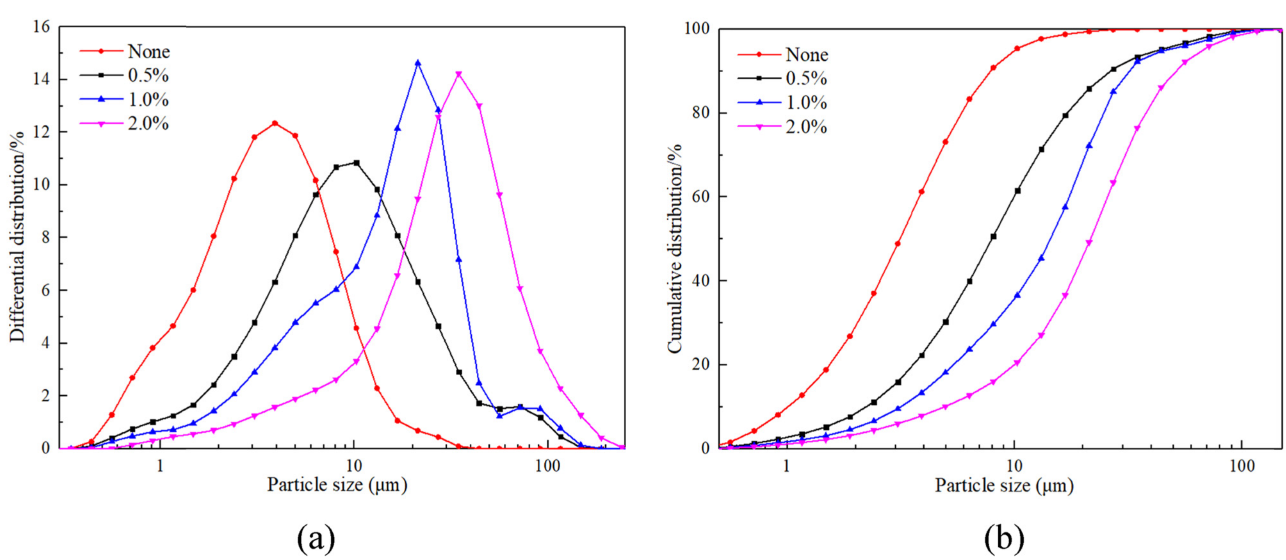

Figure 6: The effect of RH-β-CD on the particle size distribution of bentonite slurry at different dosages: (a) differential distribution and (b) cumulative distribution.

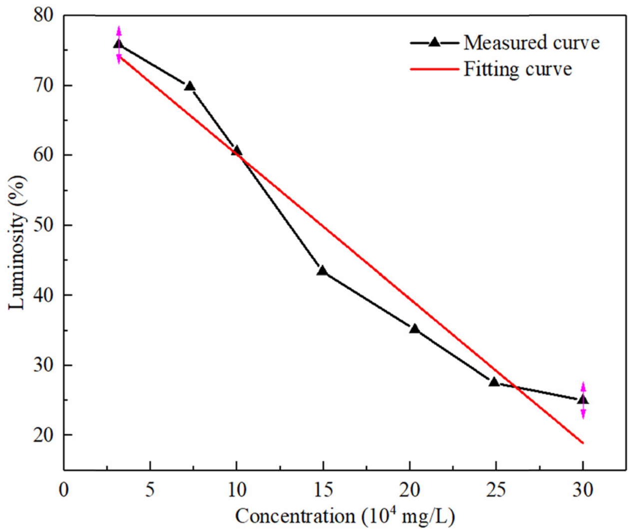

Figure 7: The biological toxicity EC_{50} test linear regression curve of RH-β-CD.

differential distribution curve and cumulative distribution curve shifted to the right, and the right shift gradually increased with the increase in the amount. This shows that as the concentration of RH-β-CD increases, the average particle size of bentonite particles keeps increasing. When the concentration reaches 2.0%, the average particle size of the clay particles added with the new RH-β-CD is 16.315 μm, which is much larger than the blank sample, indicating that RH-β-CD can overlap each other in the solution to form a network structure and its cavity can trap free clay particles in the solution. At the same time, it can adsorb clay particles through electrostatic attraction and aggregate clay particles, resulting in an increase in the average particle size and has better adsorption performance and flocculation effect.

3.5 Biological toxicity test

The biological toxicity test is shown in Figure 7 and Tables 1 and 2, referring to the National Standards of the People's Republic of China GB/T 15441-1995 and GB/T 11914. When the luminosity of the luminescent bacteria is 50%, according to the linear regression equation (y = 80.82 − 2.06x), the EC_{50} value of RH-β-CD is about 14.96 × 10^4 mg·L^{-1}. The test results show that EC_{50} >100,000 mg·L^{-1} and BOD_5/COD_{Cr} >20%. This shows that the new waste water-based drilling fluid adsorbent RH-β-CD is nontoxic and relatively easy to biodegrade.

3.6 Evaluation of the treatment effect of RH-β-CD on the waste drilling fluid

(1) In the process of drilling, the waste water-based drilling fluid comes from a wide range of sources, is difficult to deal with, and does great harm to the environment. Its treatment has always been one of the difficult problems for oilfield enterprises, and its treatment cost accounts for a high proportion of the total drilling cost, which is the main technical problem restricting oilfield production and development. The typical waste water-based drilling fluid from a well in Shengli Oilfield was selected, and the

Table 1: Biological toxicity test results of RH-β-CD

Sample	Result			Standard	
	EC_{50}	EC_{50} fitted value	Toxicity classification	EC_{50}	Toxicity classification
RH-β-CD	>100,000	14,960,000	Nontoxic	<1	Highly toxic
				1–100	Highly toxic
				100–1,000	Moderate toxicity
				1,000–10,000	Slightly poisonous
				>10,000	Practically nontoxic
				>30,000	Emission Standards

Table 2: Biodegradability test results of RH-β-CD

Sample	BOD_5 (mg·L^{-1})	COD_{Cr} (mg·L^{-1})	BOD_5/COD_{Cr} (%)	Biodegradability
RH-β-CD	10.4	48.2	21.57	Relatively easy to degrade

Table 3: Analysis of main properties and pollutants of the waste water-based drilling fluid

Components	Content
pH	10.04
ρ (g·cm^{-3})	1.12
Hydrocarbon (mg·L^{-1})	25.45
COD (mg·L^{-1})	446.8
Fe (mg·L^{-1})	2.2342
Cr (mg·L^{-1})	0.6163
Pb (mg·L^{-1})	1.2029
Cu (mg·L^{-1})	6.2345
Color	Brown black

filtrate was used as the test liquid to evaluate the treatment effect of pollutants after preliminary solid–liquid separation. Its main components can be seen in Table 3. Refer to GB 8978-1996, GB/T7574-1987, and other standards. The adsorption effect of different dosages of RH-β-CD on seven pollution items of waste drilling fluid (total chromium, total mercury, total arsenic, total lead, COD, hydrocarbon, pH value) was determined, and the results are shown in Table 4. It can be seen from the test results that RH-β-CD can effectively adsorb the harmful substances in the waste water-based drilling fluid at a 2.0% increase, and the relevant testing indicators of the waste drilling fluid filtrate after the treatment have reached the national standard [3,31]. It shows that it has excellent adsorption and wastewater treatment effects.

(2) Effect of different types of treatment agents on COD of the waste water-based drilling fluid. Refer to GB 11914-89. We comparatively evaluated the effects of four commonly used treatment agents (activated carbon, cyclodextrin, polyacrylamide, polyaluminum chloride) and the new RH-β-CD treatment agent on reducing the chemical oxygen consumption of waste water-based drilling fluids. The test result is shown in Figure 8. Five different treatment agents have a certain effect on reducing the COD

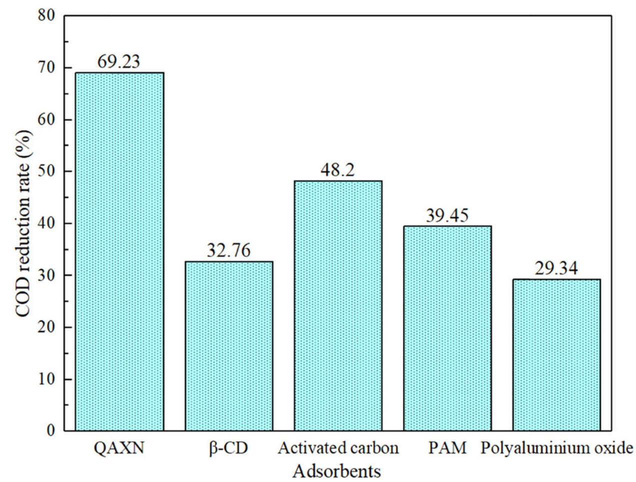

Figure 8: The influence of different treatment agents on the COD value of the waste water-based drilling fluid filtrate.

value of the waste water-based drilling fluid filtrate. Among them, RH-β-CD has the best effect, with a COD reduction rate of 69.23%, followed by activated carbon with a COD reduction rate of 48.2%. This shows that RH-β-CD and activated carbon can effectively adsorb the organic matter in the waste water-based drilling fluid filtrate and reduce its chemical oxygen consumption. This is because the rhamnolipid in RH-β-CD has good surface activity and can reduce the surface tension of the filtrate. Cyclodextrin has a hydrophilic hydroxyl group on the outside and a hydrophobic cavity on the inside. The imine structure can be fully protonated in water and electrostatically attracted to free fine particles. The synergistic effect of the three can firmly adsorb the organic substances free in the water in the hydrophobic cavity of the RH-β-CD molecule, so it has a better effect.

(3) Adsorption of different types of treatment agents to heavy metal ions. The adsorption of four types of treatment agents (activated carbon, cyclodextrin, polyacrylamide, polyaluminum chloride) and the new RH-β-CD

Table 4: Treatment effect of RH-β-CD waste water-based drilling fluid filtrate pollutants

Concentration (%)	pH	COD		Hydrocarbon		Cr		Pb	
		Test value (mg·L^{-1})	Removal efficiency (%)	Test value (mg·L^{-1})	Removal efficiency (%)	Test value (mg·L^{-1})	Removal efficiency (%)	Test value (mg·L^{-1})	Removal efficiency (%)
0.5	7–8	184.54	58.70	18.34	27.94	0.5284	14.26	0.6584	45.27
1.0	6–7	137.45	69.24	5.38	78.86	0.2845	53.84	0.2846	76.34
1.5	6–7	63.56	85.77	4.28	83.18	0.0925	84.99	0.0944	92.15
2.0	6	27.45	93.86	1.63	93.60	0.0174	97.18	0.01654	98.62
Standard	6–9	100	—	5.0	—	0.50	—	1.0	—

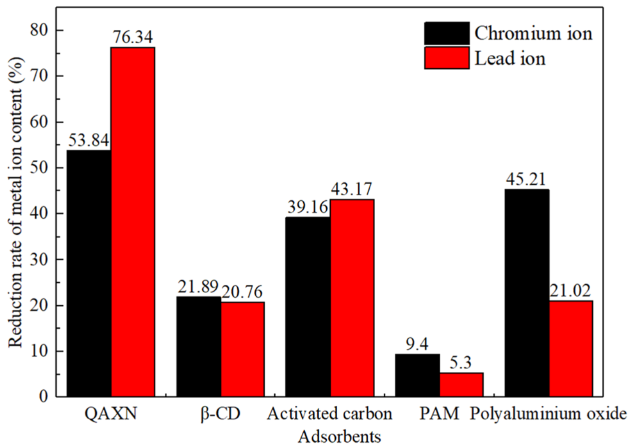

Figure 9: The influence of different treatment agents on the content of metal ions in the waste water-based drilling fluid filtrate.

treatment agent to chromium ions and lead ions in waste water-based drilling fluids were comparatively evaluated. The test result is shown in Figure 9; five different treatment agents have a certain degree of adsorption of chromium ions in the waste water-based drilling fluid loss. Among them, RH-β-CD has the best effect. The chromium ion content is reduced by more than 50%, followed by polyaluminum chloride, and the chromium ion is reduced by 45%. This shows that RH-β-CD and polyaluminum chloride can effectively adsorb heavy metal ions in waste water-based drilling fluid loss.

4 Conclusions

(1) Using Wilson's ether formation reaction, under the initiation of epichlorohydrin and cerium ammonium nitrate, the rhamnolipid and amine strong adsorption groups were successfully grafted onto β-cyclodextrin, and a new environmentally friendly waste water-based drilling fluid adsorbent RH-β-CD was prepared.
(2) The adsorption effect and environmental protection performance of RH-β-CD on organics and chromium ions in waste sulfonated water-based drilling fluids were evaluated, and the performance was compared with commonly used adsorbents such as activated carbon, PAM, and polyaluminum chloride. The results show that RH-β-CD can effectively adsorb the organic matter in the waste water-based drilling fluid filtrate, reduce its chemical oxygen consumption, and reduce the concentration of heavy metal ions in the filtrate. Its effect is better than the current commonly used PAM, activated carbon, and polyaluminum chloride.
(3) The results of the biotoxicity test and biodegradability test show that the BOD_5/COD_{Cr} >20% and EC_{50} >100,000 $mg{\cdot}L^{-1}$ for the new waste water-based drilling fluid adsorbent RH-β-CD, which is nontoxic, relatively easy to biodegrade, and is environmentally friendly.

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