



## Study of modified konjac gum used as green flocculant for waste drilling fluid

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### ABSTRACT

Oilfield chemicals prepared with natural products is an effective way of green oilfield chemistry development. The konjac gum is used as the flocculant for waste drilling fluid, and it is used as a multifunctional oil field additive. By the single factor experiments, the optimal flocculation conditions were screened out. The conditions are as follows: the adding amount is 0.10%, flocculation temperature is at 40°C, pH 7, stirring rate is 200 rpm and stirring is for 5 min. the flocculation efficiency can be significantly improved using composite flocculant with konjac gel and orange peel, and get puff and large flocs. The flocculation efficiency is the best under the mass ratio of 1:5 between konjac gum and orange peel. The flocculation efficiency of the compound flocculant is between polyacrylamide and polyaluminum chloride, but it has obvious an advantage in filtration loss. Experimental results show that the “adsorption bridging” and “charge neutralization” mechanisms in the composite flocculant may coexist and reinforce each other. This research will be beneficial for improving the environmental performance of flocculants in waste drilling fluid treatment.

*Keywords:* Modified konjac gum; Flocculant; Waste drilling fluid; Oil field

### 1. Introduction

For the development of green oilfield chemicals, the preparation of oilfield chemicals from natural products is an effective way [1–3]. Natural vegetable gum and its derivatives have been widely used in oilfield additives, which have the advantages of environmental protection compared with synthetic polymers [4]. However, there are also such as poor efficiency and high-cost performance problems which make it hard to put into use in the practical application field [5,6]. Some of them cannot fulfil the simplification requirements of modern drilling and oil production process [7]. The study of multifunctional and environment-friendly oilfield additives is of great significance for modern oilfield development [8–10]. Konjac gum is an oil field additive extracted from plant gum, which is from

natural vegetable gum after processed and purified [11]. It is mainly used as a drilling fluid treatment agent, thickening for fracturing fluids [12]. Good inhibitory performance in drilling fluid applications and shows great thickening effect on fracturing fluid. So, to a certain extent, that can change the rheological properties of drilling fluid and fracturing fluid. Konjac is mainly composed of non-ionic polyhydroxy grape mannan molecules with long chains, including grape mannan with ortho-trans-hydroxy groups and mannose with ortho-cis-hydroxyl groups (as shown in Fig. 1) [13,14]. The molecular weight of konjac gum is about 1,000,000–1,500,000 [15]. The molecular chain of konjac gum has a large number of active groups including hydroxyl, carboxyl, carbonyl, and its gum has high viscosity [16,17]. So, konjac gum can be used as fracturing fluid thickener [18]. The residue after gel-breaking is used for drilling fluid

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flocculation treatment. Thus, the resources can be fully utilized, and get further improvement in economic benefits. This paper discusses the flocculation efficiency of konjac gum which be applied in waste drilling fluids after being used as thickening agent of fracturing fluid. The effects of dosage, temperature, pH and stirring rate on flocculation are investigated. It provides certain data reference for the development of universal green additives in oil field.

## 2. Experimental set-up

### 2.1. Instruments and materials

Sodium carbonate, sodium hydroxide, hydrochloric acid, cationic polyacrylamide (CPAM), polyaluminum chloride (PAC), these reagents are analytical PURE, produced in Chemical Company of Tianjin No. 3 Chemical Reagent Factory. Calcium-based bentonite (Industrial, Xi'an Yongjiu Chemical Industry Co., Ltd., China) and konjac gum (Ankang, Shaanxi Province) was obtained from Xi'an Changqing Chemical Group Co., Ltd., (China). The following instruments were used in this research: low-speed centrifuge (SC-03, Anhui Zhongke Zhongjia Scientific Instrument Co., Ltd., China), multiple connection Low-Pressure Filter Press (SD6, Qingdao Haitongda Special Instrument Factory), pH meter (pHS-3C, Xianyang Fangzhou Technology Development Corporation), UV-Visible Spectrophotometer (UV-2600, Shimadzu), Micro polariscope (BK-POL, Chongqing Otter Optical Instruments Co., Ltd., China).

### 2.2. Experimental methods

#### 2.2.1. Preparation of flocculants

Weigh a certain amount of konjac gum, add slowly into a mixing cup filled with tap water, stir for 10–20 min until the gel is evenly dispersed, then pour it into a beaker for later use.

#### 2.2.2. Determination of filtrate loss of flocculating drilling fluid

Reference GB/T 16783-1997. The 7.5 min filtrate loss of konjac gum flocculating drilling fluid is measured by the method of filtrate loss of drilling fluid in Field test program of water-based drilling fluid.

#### 2.2.3. Measurement of light transmittance of flocculating solution

Baseline correction with distilled water as reference, the supernatant of waste drilling fluid after flocculated is scanned under full wavelength using UV-2600 UV-Visible Spectrophotometer, and the transmittance is measured.

#### 2.2.4. Centrifugal volume of flocculating drilling fluid test

The flocculant which prepared according to the preceding method in 1.2.2 is added slowly into the prepared drilling fluid in a certain proportion, fully dispersed at a certain stirring rate to make it. After standing for a certain time, the flocculating effect of different flocculants are observed.

Take 10 mL of well-mixed flocculating drilling fluid, measure the volume of supernatant after centrifugation for 10 min at 3,000 r/min.

## 3. Results and discussion

### 3.1. Flocculation efficiency of konjac gum

This research separately investigated how the dosage, temperature, pH and different stirring rates affect flocculation effect of konjac gum. The optimum flocculation conditions were selected.

#### 3.1.1. Effect of the amount of konjac gum on flocculation of waste drilling fluid

Konjac gum which mass ratio is 0.05%, 0.08%, 0.1%, 0.3%, and 0.5% is added to the prepared waste drilling fluid. Filtrate loss of drilling fluid, centrifugal volume of flocculation fluid and light transmittance of supernatant after flocculation are measured until flocculated drilling fluid has been standing for a period of time at room temperature. The experimental results are shown in Table 1, and the flocculation efficiency diagram is shown in Fig. 2.

As shown in Table 1, the filtrate loss, centrifugal volume and light transmittance of waste drilling fluid after flocculated show an initial increase and then a decrease with the increase of konjac gum dosage. This phenomenon indicates that the addition of konjac gum has a certain flocculation efficiency on the waste drilling fluid. The best flocculation effect is at the konjac gum dosage of 0.1%.

#### 3.1.2. Influence of temperature on konjac gum flocculation of waste drilling fluid

0.1% konjac gum is added into prepared waste drilling fluid, then placed them in thermostatic water bath

Table 1  
Influence of the amount of konjac gum on flocculation efficiency of waste drilling fluid

Item	Temperature (°C)		
	25	40	60
Filtration (mL)	47.8	55.3	25.2
Centrifugal volume (mL)	8.1	8.3	5.2
Transmittance (%)	54.0	58.0	45.0

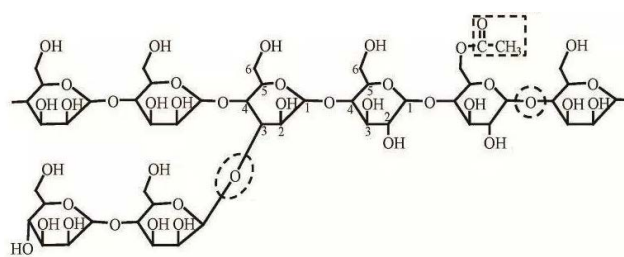


Fig. 1. Molecular structure of konjac gum.

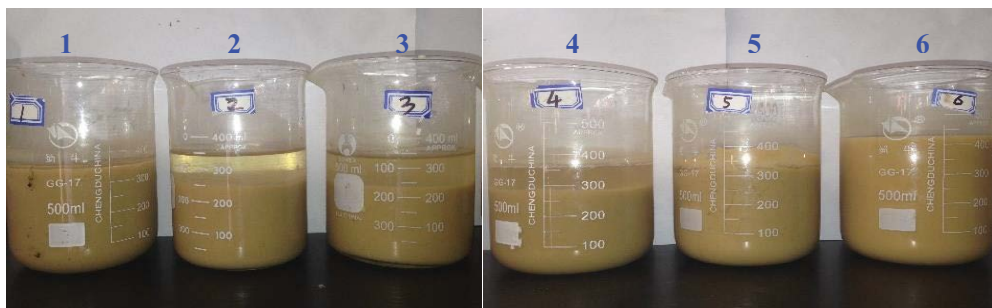


Fig. 2. Flocculation efficiency of konjac gum at room temperature (1–6 addition amount of konjac gum is 0%–0.5%).

separately at 25°C, 40°C, and 60°C for a certain time, and the parameters are measured which include filtration after flocculation, the centrifugal volume of flocculation liquid and the transmittance of supernatant. The experimental results are shown in Table 2, the flocculation efficiency is shown in Fig. 3.

The results are shown in Table 2: The waste drilling fluid system loses its stability at 40°C and shows good flocculation efficiency. This is mainly because Brownian motion intensity of particles decrease at low temperature, so forming flocs takes longer time, and the formed flocs is fine and well dispersed [19]. On the other hand, it's unfavorable to get the best results of flocculant in the conditions of high temperature, smaller flocs or higher water content [20].

### 3.1.3. Influence of pH values on konjac gum flocculation

0.1% konjac gum is added into the prepared waste drilling fluid, adjust the pH of the system to 3, 5, 7, 9, and 11, respectively, stand still in thermostatic water bath for a certain time at 40°C, and the filtration after flocculation, the centrifugal volume of flocculation liquid and the transmittance of supernatant are measured. The experimental results are shown in Table 3. The flocculation efficiency is shown in Fig. 4.

From the results in Table 3, it can be seen that pH has a significant influence on the flocculation process of waste drilling fluid. With increase in pH, the filtration rate, centrifugal volume and transmittance of drilling fluid decrease gradually after being flocculated [21]. Under acidic conditions, the waste drilling fluid system can be destabilized, flocculated and precipitated easily. Considering the pH requirement of filtrate after flocculation for discharge, it is better to make sure the pH of 7. It is because the negative charges on the surface of clay particles react with a large number of positive charges in the system under acidic conditions [22]. This results in the decrease of  $\xi$  potential of clay particles surface [23], double layer compression, the repulsion between clay particles is weakened, so the flocs are easy to form [24].

### 3.1.4. Influence of stirring time and rate on konjac gum flocculation efficiency

During the flocculation process, physical stirring time and stirring rate have great influence on the formation and aggregation process of flocs. Therefore, the optimum dosage

Table 2

Influence of temperature on konjac gum flocculation efficiency in waste drilling fluid

Item	Additive amount of konjac gum (m/v)					
	0.00%	0.05%	0.08%	0.10%	0.30%	0.50%
Filtration (mL)	13.3	15.5	16.2	47.8	40.0	27.2
Centrifugal volume (mL)	4.1	6.0	6.6	8.1	8.0	7.1
Transmittance (%)	40.0	43.0	48.0	54.0	51.0	48.0

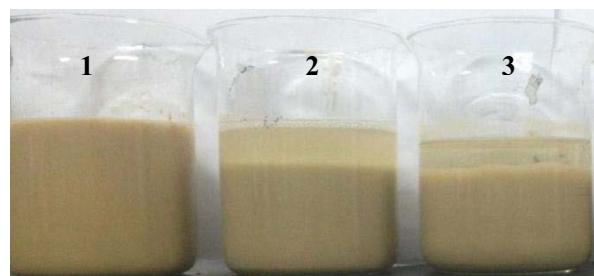


Fig. 3. Effect of konjac gum flocculation at different temperatures (from 1 to 3: 25°C, 40°C, 60°C).

Table 3

Influence of pH on konjac gum flocculation efficiency in waste drilling fluid

Item	pH				
	3	5	7	9	11
Filtration (mL)	105.0	83.2	61.0	30.0	18.2
Centrifugal volume (mL)	6.8	7.5	7.5	6.5	5.5
Transmittance (%)	63.0	59.0	53.0	57.0	55.0

of konjac gum is above 0.1%, the temperature is 40°C, and the pH is 7. Under the above conditions, the effects of different stirring time and stirring rate on the flocculation efficiency of konjac gum are investigated. The experimental results are shown in Table 4. The flocculation efficiency diagram is shown in Fig. 5.

From Table 4, it can be seen that the flocculation efficiency of konjac gum is significantly affected by mixing time and speed. With the increase of stirring rate, filtration loss of waste drilling fluid after flocculation increases first

Table 4  
Effects of stirring time and speed on the flocculation efficiency of konjac gum

Speed (r/min)	Time (min)	Centrifugal volume (mL)	Transmittance (%)	Filtration (mL)
100	5	8.1	53.0	88.2
	10	8.8	57.0	75.0
200	5	9.0	55.0	96.3
	10	8.6	52.0	97.1
300	5	8.2	62.0	83.2
	10	8.6	56.0	81.5
400	5	8.8	60.0	85.9
	10	8.7	52.0	78.4
500	5	8.4	49.0	70.3
	10	8.5	47.0	70.2

followed by decreases. At the same stirring rate, the flocculation efficiency get improvement under longer stirring time. However, at higher stirring rate, too long stirring time will break the floc group has been formed. This is unfavourable for the adsorption between large particles of clay and konjac colloid molecule and the capture process of flocculant on clay. After comprehensive consideration, it can be concluded that the flocculation efficiency of konjac gum is Optimal when the stirring rate is 200 rpm and the stirring time is 5 min.

By investigating how these factors, such as dosage, temperature, pH, stirring time and speed, affect efficiency, the optimal flocculation conditions of konjac gum are as follows: addition of konjac gum is 0.1%, flocculation is 40°C, pH 7, stirring rate is 200 rpm, stirring time is 5 min. But the flocculation efficiency diagram shows that the flocs of waste drilling fluid after flocculation is small, and cannot achieve ideal flocculation efficiency. So, it needs to be compounded with other flocculants.

### 3.2. Flocculation efficiency of konjac gum and orange peel

Orange peel is a concomitant product of citrus production process, which contains a large amount of lignin, pectin,

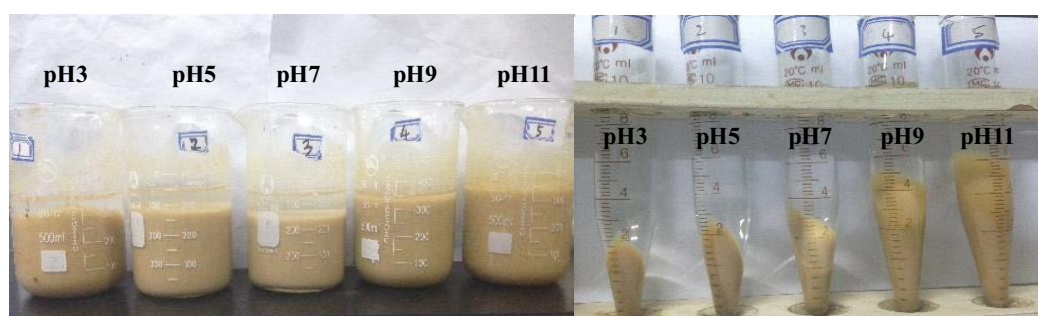


Fig. 4. Flocculation efficiency of konjac gum at different pH values (from left to right: 3, 5, 7, 9, 11).

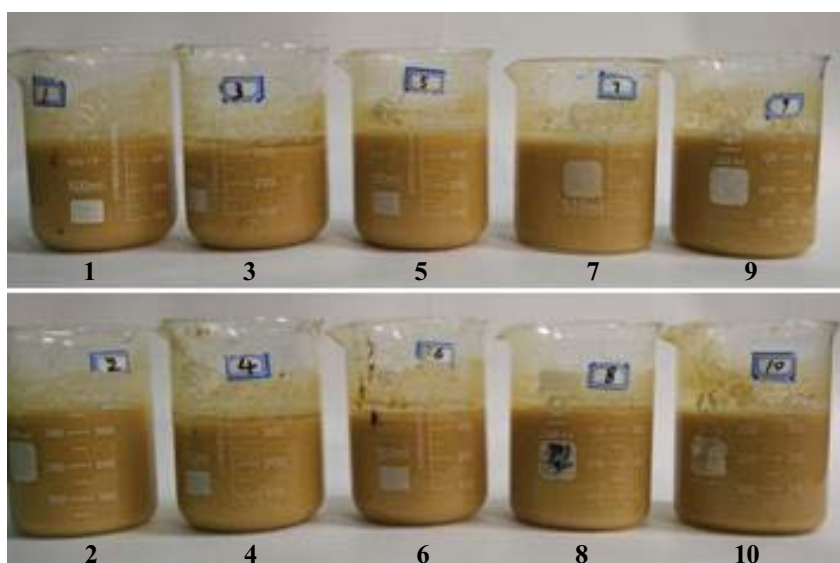


Fig. 5. Flocculation efficiency of stirring time and rate on konjac gum (top layer no. 1, 3, 5, 7, 9: stirring time 5 min, stirring rate 100–500 rpm, bottom layer no. 2, 4, 6, 8, 10: stirring time 10 min, stirring rate 100–500 rpm).

tannin, flavonoids and other plant phenolic compounds [25,26]. Tannin and other plant phenolic compounds can better disperse the drilling fluid system. The orange peel and konjac gum are compounded and to investigate effect of flocculation. The flocculation conditions are as follows: konjac gum addition is 0.1%, temperature is 40°C, pH 7, stirring rate is 200 rpm, stirring time is 5 min. The experimental results are shown in Table 5, and the flocculation efficiency is shown in Fig. 6.

Compared with single konjac gum flocculation efficiency, it can be seen from the flocculation results of konjac gum compounded with orange peel that the flocculation efficiency of waste drilling fluid is obvious with the increasing of the mass ratio of konjac gum and orange peel, resulting in large and loose flocs. When the mass ratio of konjac gum to orange peel is 1:5, the filtration loss of waste drilling fluid reaches the maximum. The filter cake produced after solid–liquid separation is dense; the supernatant is clear. Partly because the longer molecular chain of konjac gum which can absorb clay particles and form a certain bridging structure, so a large number of particles can gather, produce flocs and then make the particles sinking [27]. On the other hand, tannin in orange peel has a certain dilution effect on waste drilling fluid. Sodium tannate which is adsorbed on the  $Al^{3+}$  at the edge of broken bond of clay particles through the coordinate bond [28] can greatly weaken the bonding function between end and end or end and surface between clay particles, thus destroying the spatial network structure formed between clay particles to a certain extent [29]. So, for the system of konjac gum compounded with orange peel, it shows a certain flocculation efficiency macroscopically.

### 3.3. Comparison of flocculation efficiency between composite and common flocculant

CPAM and PAC are widely used in sewage flocculation treatment [30,31]. Through the bridging of macromolecules and the charge neutralization, the flocs can produce rapidly and have a good flocculation efficiency. Flocculant compounded with konjac gum and orange peel, PAC and CPAM are used as flocculant for drilling fluid, and the flocculation efficiency is compared from three aspects: centrifugal volume, transmittance and filtration. The results are shown in Fig. 6.

It can be seen from Fig. 7, CPAM, PAC, compound flocculant of konjac gum and orange peel have different flocculation efficiency on waste drilling fluid. Among them, CPAM has the best flocculation efficiency and less additive

amount. Compared with PAC, the flocculant compounded with konjac gum and orange peel has obvious advantages in reducing filtration after flocculation. However, due to the addition of orange peel, the transmittance of the supernatant after flocculation is not as clear as that of PAC. Analysing from flocculation mechanism, co-existence of adsorbing-bridging and charge neutralization in CPAM and compound flocculant can affect each other. It can accelerate the rapid dispersion of waste drilling fluid colloid system [32]. The flocculation process of PAC is mainly based on the mechanism of “electric neutralization”.

### 3.4. Microanalysis of floc structure and morphology

The flocculation efficiency can be observed through a micrography clearly and directly. Use BK-POL polarizing microscope, dropwise the waste drilling fluid samples

Table 5  
Flocculation efficiency of konjac gum and orange peel

Konjac gum:orange peel ( $w_1:w_2$ )	Centrifugal volume (mL)	Transmittance (%)	Filtration (mL)
1:0	9.0	55.0	96.3
1:1	8.7	52.0	103.5
1:3	8.7	49.0	108.8
1:5	9.0	50.0	117.4
1:10	9.1	43.0	110.1

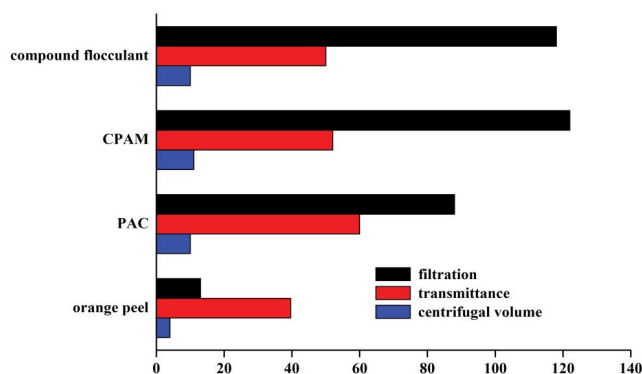


Fig. 7. Comparison of flocculation efficiency of different flocculants (the added concentrations of CPAM and PAC were 10 and 60 mg/L, respectively).



Fig. 6. Flocculation efficiency of mass ratio of konjac gum:orange peel (mass ratio of konjac gum:orange peel from left to right: 1:0, 1:1, 1:3, 1:5, 1:10).

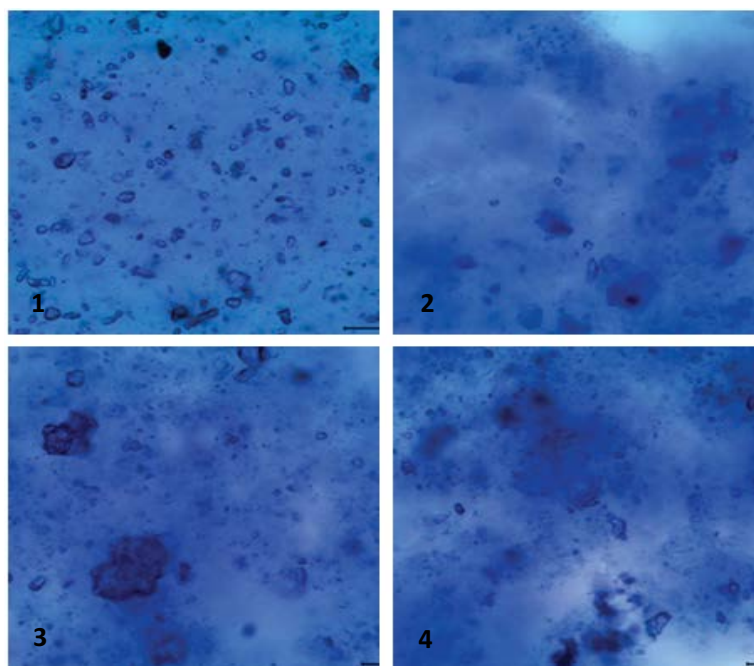


Fig. 8. Flocculation micrograph of different flocculants (1, 2, 3, 4 are waste drilling fluid, konjac gum flocculation drilling fluid, compound flocculant treatment drilling fluid, pam treatment drilling fluid).

treated with different flocculants on the slide, observe the microscopic morphology of flocs at 20 magnifications.

The microscopic results of different flocs morphology are shown in Fig. 8. It shows that the clay particles in the waste drilling fluid are uniformly distributed, the difference in particle size is small, stereoscopy is weak. These show that the waste drilling fluid is relatively stable. The clay particles in the waste drilling fluid are obviously accumulated after flocculation treatment with konjac gum, appear blocky and layered structures, and there is a certain phenomenon of bridge erection. The size of floc increases significantly after flocculation treatment of waste drilling fluid with composite flocculant, distribution is uneven on the whole. Small flocs gather together around large flocs, presenting a three-dimensional network structure. After polyacrylamide flocculate waste drilling fluid, the flocs are interconnected by bridging, floc particles agglomerate together to form larger clusters, display a three-dimensional network structure on the whole. It is conducive to the “net capture” to clay particles.

#### 4. Conclusions

Konjac gum has remarkable flocculation efficiency on waste drilling fluid. The best flocculation effect of drilling fluid is achieved when the dosage of konjac gum is 0.10%, the flocculation temperature is 40°C, pH is 7, the stirring rate is 200 rpm, and the stirring rate is 5 min. The flocculation efficiency of konjac gum and orange peel on waste drilling fluid is more obvious, and the generated flocs are large and loose with the 1:5 mass ratio of konjac gum to orange peel. Compared with common flocculants, the flocculation efficiency of compound flocculant is superior to PAC and inferior to polyacrylamide. The flocculation mechanism of the composite flocculant is mainly through the combined action

of “adsorption bridging” and “electric neutralization” which two can promote each other, the colloid system formed in waste drilling fluid can be dispersed rapidly. Konjac gum derived flocculant will have a good application prospect in the flocculation treatment of waste drilling fluid.

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