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The Study of Optimization of Flocculation and Destabilization Technology of Waste PEM Drilling Fluid in Bohai Oilfield

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In light of the difficulty of solid-liquid separation of waste PEM drilling fluid in the Bohai oilfield, constructing an inorganic-organic flocculation system is proposed and the processing method of destabilization technology is optimized. The biggest influence factor on the flocculation process of PEM drilling fluid was determined by designing an orthogonal test. The flocculation mechanism was researched through single factor optimization, combined with zeta potential and particle size distribution test. The results showed that the most significant factors affecting the flocculation of waste PEM drilling fluid were the dosage of inorganic flocculant $CaCl_2$ and flocculation pH value. When the dosage of inorganic flocculant $CaCl_2$ was 1.2% (w/v), the dosage of organic flocculant SDYJ-2 was 0.05%, the flocculation pH value was 3, and the flocculation time was 5 min, the flocculation technology reached the optimization and then the liquid yield can reach 70.96%. The mechanism of flocculation and destabilization was as follows: the inorganic flocculant of $CaCl_2$ mainly reduced the zeta potential of clay particles through electric neutralization. 1% $CaCl_2$ could reduce the potential mean value of drilling fluid system from -38.1 mV to -32.5 mV, and then decrease the repulsion among suspensions. Through bridging curling and electric neutralization, the organic flocculant of SDYJ-2 can absorb and wrap the clay particles after flocculation destabilization to form a network spatial structure, which made clay particles aggregate into large flocs and particles. D50 can increase by 21.5 times, when the concentration of SDYJ-2 was 0.15%.

Keywords: PEM drilling fluid, inorganic-organic flocculation system, liquid yield, electric neutralization, bridging-curling

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1 INTRODUCTION

Bohai Sea is the only semi-enclosed inland sea body in China, and the ecological environment has a serious impact on the daily life of coastal residents. With the increasingly strict requirements for environmental protection, more attention has been paid to the effective disposal of waste drilling fluid by offshore drilling (Malachosky et al., 1993; Hou-ming et al., 2012). Solid-liquid separation is one of the best methods for environmental protection due to the limitation of spatial position on offshore drilling (Wang et al., 2021). Currently, PEM drilling fluid is used in most areas of Bohai oilfield, including the advantages of low filtration loss, rheology, thermal stability, and good inhibition (Xun et al., 2011; Paul et al., 2020). However, PEM drilling fluid has high polymer content and high solid content, which make the drilling fluid system remarkably stable (Shaocheng

TABLE 1 | Basic properties of PEM drilling fluid.

MW/(g·cm^{-3})	1.46
PV/(mPa·s)	58
FL (ml)	2.8
Φ3/Φ6	15/20
AV (mPa·s)	95
YP (Pa)	37
pH	75

TABLE 2 | Factor level table.

Factor	Level		
	1	1	3
A/(%) ($CaCl_2$ concentration)	1.3	1.5	1.7
B/(%) (815 emulsion concentration)	0.03	0.05	0.07
C/(s) (Flocculation mixing time)	3	5	7
D (Flocculation pH value)	4	7	Original state

TABLE 3 | Test scheme table.

Factor / Level / No	A	B	C	D
1	1.3	0.03	3	4
2	1.3	0.05	5	7
3	1.3	0.07	7	Original state
4	1.5	0.03	5	Original state
5	1.5	0.05	7	4
6	1.5	0.07	3	7
7	1.7	0.03	7	7
8	1.7	0.05	3	Original state
9	1.7	0.07	5	4

et al., 2001; Fan et al., 2011). Wai Li et al. (Li et al., 2017) studied the solid-liquid separation technology based on chemical flocculation and centrifugation, and comprehensively investigated the effects of flocculant type, concentration, and treatment conditions on the separation degree of drilling fluid. Zhou JL et al. (2012) carried out solid-liquid separation of waste drilling fluid by centrifugation and adding surfactant, and studied the ability of surfactant enhanced washing to treat the waste drilling fluid and remove the hazardous hydrocarbons. Wang Youhua et al. (Cui et al., 2011) applied low pressure distillation technology to solid-liquid separation of drilling fluid. This process mainly contains such three steps as chemical destabilization, fold and spiral dehydration, and low pressure distillation. Field analysis results of disposed water have shown that the content of fluoride, heavy metals, oil, COD, BOD, and chromaticity can reach the class II water quality requirements of China national standards. In order to improve the flocculation destabilization efficiency and simplify the field construction for the characteristics of waste PEM drilling fluid in Bohai oilfield, this paper proposes to build an inorganic organic flocculation system, optimize the optimal treatment methods of different flocculation destabilization processes, and analyze its flocculation destabilization mechanism for waste PEM drilling fluid in Bohai oilfield.

2 EXPERIMENTAL MATERIALS, INSTRUMENTS, AND METHODS

2.1 Experimental Materials and Instruments

Polyferric chloride (PFC), polyaluminum chloride (PAC-1, liquid), polyferric chloride sulfate (SPFS), polyferric chloride (PFC), polyferric sulfate (PFS), and polyaluminum chloride (PAC-2, solid) were obtained from Zibo Heying Water Purification Materials Co., Ltd. (China). $CaCl_2$ (analytical grades) was provided by Sinopharm Chemical Reagent Co., Ltd. (China). There were SDYJ-1 emulsion, SDYJ-2 emulsion, SDYJ-3 emulsion, and SDYJ-4 emulsion, which were configured in the laboratory. Citric acid (analytical grades) and NaOH (analytical grades) were purchased from Aladdin Chemistry Co., Ltd. (China). The waste PEM drilling fluid used in the experiment was taken from the offshore drilling platform of Bohai oilfield, and the basic parameters were shown in **Table 1**.

Medium pressure water loss instrument and high-speed frequency conversion stepless speed mixer were purchased from Qingdao Tongchun Petroleum Instrument Co., Ltd. (China). ZNN-D68 electric six-speed viscometer was supplied by Qingdao Haitongda Special Instrument Co., Ltd. (China). TA6-3 program-controlled coagulation test mixer was provided by Wuhan Hengling Technology Co., Ltd (China). GT10-1 high-speed Bench Centrifuge was obtained from Beijing Shidai Beili Centrifuge Co., Ltd. (China). PHS-3 C T precision desktop pH meter was produced by Shanghai Dapu Instrument Co., Ltd. (China). There were beakers (150 ml), measuring cylinders (10 ml, 50, and 100 ml), and glass rods.

2.2 Experimental Materials and Instruments

In the first, the method of flocculation sedimentation was applied to the waste PEM drilling fluid. A total of 100 ml of waste PEM drilling fluid taken in the beaker, and placed it in the TA6-3 program-controlled coagulation test mixer. Then the flocculant was added under high-speed stirring at 450 rpm, which was mixed evenly for 3 min after slowly stirring at 50 rpm for 3 min, and finally stood for 30 s. This was followed by the centrifugal experiment. The drilling fluid after flocculation standed was placed in the GT10-1 high-speed bench top centrifuge at the rate of 3000 rpm for 3 min. Thereafter, the volume of the supernatant was measured and the liquid yield for the waste PEM drilling fluid can be obtained by **Equation (1)**.

$$f = \frac{V_1}{V_2} \tag{1}$$

where f is the liquid yield, V_1 is the volume of liquid phase removed from drilling fluid, mL, and V_2 is the initial drilling fluid volume, mL.

TABLE 4 | Analysis table of test results.

Factor	A	B	C	D	Liquid yield/(%)
Level					
No	1	2	3	4	
1	1.3	0.03	3	4	59.19
2	1.3	0.05	5	7	54.26
3	1.3	0.07	7	Original state	55.00
4	1.5	0.03	5	Original state	48.00
5	1.5	0.05	7	4	49.47
6	1.5	0.07	3	7	45.54
7	1.7	0.03	7	7	46.51
8	1.7	0.05	3	Original state	50.00
9	1.7	0.07	5	4	53.00
	k1/(%)	56.15	51.23	51.58	53.89
Level	k2/(%)	47.67	51.24	51.76	48.77
	k3/(%)	49.84	51.18	50.33	51.00
Range/(%)	8.48	0.06	1.43	5.11	
Better level	A1	B2	C2	D1	
Factor primary and secondary	1	4	3	2	

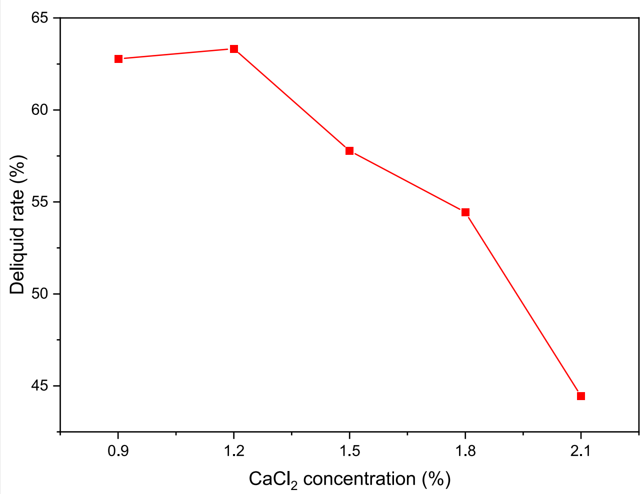

FIGURE 1 | Effect of $CaCl_2$ addition on flocculation.

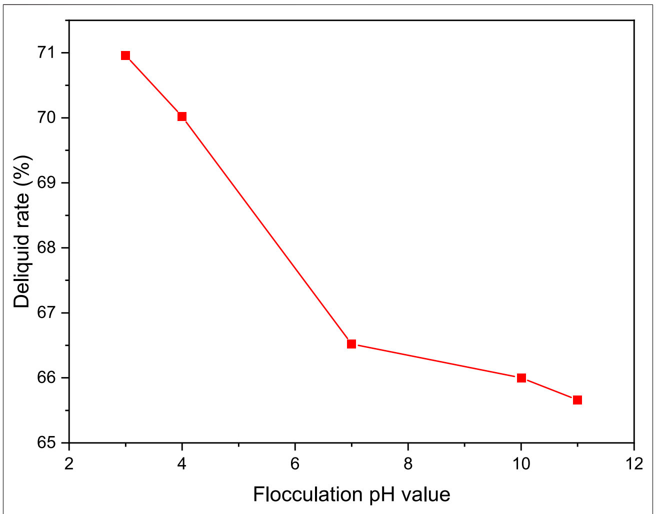

FIGURE 2 | Influence of different pH value on flocculation effect.

3 FLOCCULANT OPTIMIZATION AND STUDY ON EXPERIMENTAL CONDITIONS

The waste PEM drilling fluid contained many gel protecting agents, which increased the solid content of surfactant in the drilling fluid and made them more difficult to flocculate and destabilize. The colloidal stability of the drilling fluid system was destroyed through adding flocculants which changed the physical and chemical properties of the drilling fluid. Promote the suspended particles in the drilling fluid to agglomerate into large flocs, separated by solid and liquid through centrifuge. Therefore, different flocculation and destabilization processes are carried out to study and analyze the flocculation effect of waste PEM drilling fluid.

3.1 Orthogonal Experimental Design

$CaCl_2$ (inorganic flocculant) and 30% cationic SDYJ-2 emulsion (organic flocculant) were designed as flocculating agent. There were four factors including $CaCl_2$ dosage (A/(%)), SDYJ-2 emulsion dosage (B/(%)), flocculation agitation time (C/(s)), and flocculation pH value (D). In addition, each factor contained 3 levels, the factor level table is shown in **Table 2**.

The factors were arranged on the L9 3^4 orthogonal table, and then translated into the test scheme table, as shown in **Table 3**.

The liquid yield was taken as the reference index (**Table 4**) by means of the orthogonal test.

It can be obtained from **Table 4** that the order of influence on the treatment of waste PEM drilling fluid was: inorganic flocculant dosage > pH > flocculation mixing time > organic flocculant dosage.

TABLE 5 | Effect of inorganic flocculant on Zeta potential of PEM drilling fluid.

Flocculant/(%)	Zeta potential/(mV)			
				Average value and standard deviation
–	–34.0	–38.0	–42.3	–38.1 ± 4.151
SPFS	–33.1	–35.0	–35.6	–34.57 ± 1.305
PFS	–35.2	–37.3	–37.8	–36.77 ± 1.380
PFC	–34.3	–35.3	–34.9	–34.83 ± 0.503
PAC-1	–34.6	–34.5	–37.9	–35.67 ± 1.935
PAC-2	–32.6	–35.1	-35.5	–34.4 ± 1.572
$CaCl_2$	–31.9	–33.2	–32.4	–32.5 ± 0.656

TABLE 6 | Effect of $CaCl_2$ with different concentrations on Zeta potential of PEM drilling fluid.

Flocculant concentration	Zeta potential/(mV)			
				Average value and standard deviation
–	-34.0	-38.0	-42.3	-38.1 ± 4.151
0.5%	-31.7	-33.4	-34.8	-33.3 ± 1.55
1%	-31.9	-33.2	-32.4	-32.5 ± 0.656
1.5%	-30.1	-30.5	-29.8	-30.13 ± 0.35
2%	-28.0	-29.5	-29.6	-29.03 ± 0.90

TABLE 7 | Effect of inorganic flocculant on particle size distribution of PEM drilling fluid.

Flocculant/(1%)	D10/(μm)	D50/(μm)	D90/(μm)
–	1.543	29.84	293.5
SPFS	5.966	40.34	813.2
PFS	1.954	32.73	190.4
PFC	4.576	29.05	394.0
PAC-1	2.793	32.37	265.1
PAC-2	3.146	87.54	242.9
$CaCl_2$	2.187	29.84	198.1

Therefore, the experimental study on the optimal dosage of inorganic flocculant and flocculation pH value was carried out.

3.2 $CaCl_2$ Dosage Optimization

Figure 1 shows $CaCl_2$ has an obvious effect on flocculation destabilization. When the $CaCl_2$ dosage is less than 1.2%, with the increase of $CaCl_2$ dosage, the liquid yield of waste PEM drilling fluid increases slowly after solid-liquid separation. For $CaCl_2$ addition the amount was less than 1.2%, with the increase of $CaCl_2$ dosage, the liquid yield of waste PEM drilling fluid increases slowly after solid-liquid separation. The liquid yield reaches the highest value with 63.33% for the $CaCl_2$ addition reached 1.2%. The liquid yield showed a downward trend with the $CaCl_2$ addition above 1.2%. Therefore, the optimum dosage of $CaCl_2$ was 1.2%.

3.3 Optimization of Flocculation pH Value

The experimental results of different flocculation pH for flocculation destabilization were shown in the **Figure 2**.

TABLE 8 | Effect of $CaCl_2$ concentration on particle size distribution of PEM drilling fluid.

Flocculant concentration	D10/(μm)	D50/(μm)	D90/(μm)
–	1.543	29.84	293.5
0.5%	1.635	29.01	258.2
1%	2.187	29.84	198.1
1.5%	1.666	30.80	238.4
2%	2.134	25.54	229.2

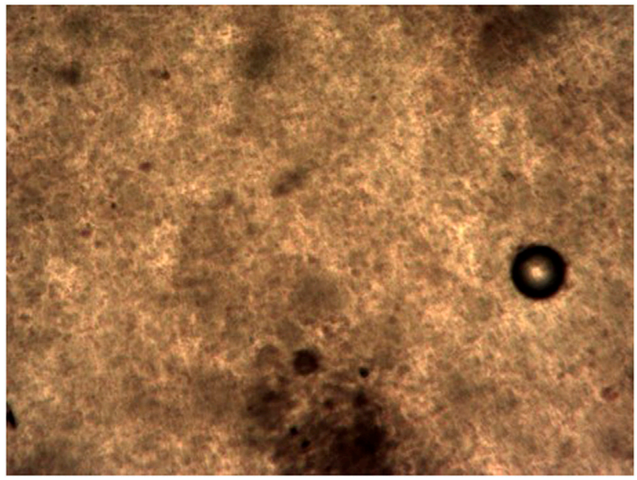

FIGURE 3 | Microscopic morphology of PEM drilling fluid.

The flocculation destabilization efficiency of waste PEM drilling fluid was significantly better in acidic conditions than in alkaline conditions. Under acidic conditions, with the decrease of flocculation pH value, the liquid yield increased linearly and increases greatly. In addition, when the flocculation pH value was 3, the liquid yield can reach 70.96%. The liquid yield decreases slowly with the increase of flocculation pH value under alkaline conditions.

4 RESULTS AND DISCUSSION

4.1 $CaCl_2$ Dosage Optimization

4.1.1 Optimization of $CaCl_2$ Flocculation Destabilization Study Quantity

The addition of Ca^{2+} can neutralize the negative charge in the waste PEM drilling fluid, so as to reduce the negative charge on the surface of clay particles and Zeta potential in the drilling fluid, resulting in the reduction of repulsion between clay particles. When the dosage of Ca^{2+} was too large, Ca^{2+} entered the adsorption layer on the particle surface, making the clay particles positively charged, and the potential changed from negative to positive, resulting in "re-charged" clay particles. The drilling fluid was again stabilized by reverse gelling and

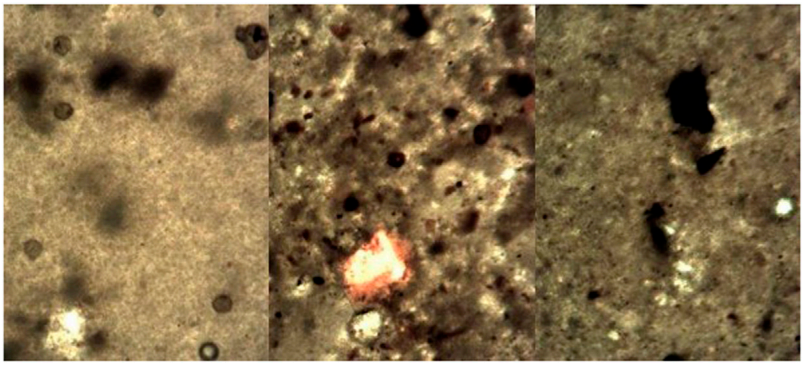

FIGURE 4 | Microscopic morphology of PEM drilling fluid treated with inorganic flocculant.

TABLE 9 | Effect of organic flocculant on Zeta potential of PEM drilling fluid.

Flocculant/(0.05%)	Zeta potential/(mV)			
				Average value and standard deviation
–	–34.0	–38.0	–42.3	–38.1 ± 4.151
SDYJ-1	–35.1	–37.0	–37.6	–36.57 ± 1.31
SDYJ-2	–35.0	–36.9	–38.1	–36.67 ± 1.56
SDYJ-3	–34.2	–35.5	–36.0	–35.23 ± 0.93
SDYJ-4	–33.3	–34.6	–34.5	–34.13 ± 0.72

TABLE 10 | Effect of organic flocculant on particle size distribution of PEM drilling fluid.

Flocculant/(0.05%)	D10/(μm)	D50/(μm)	D90/(μm)
–	1.543	29.84	293.5
SDYJ-1	19.79	117.8	411.8
SDYJ-2	26.21	122.6	859.4
SDYJ-3	27.94	306.1	1,170
SDYJ-4	37.80	132.8	1,028

TABLE 11 | Effect of SDYJ-2 emulsion with different concentrations on the Zeta potential of PEM drilling fluid.

Flocculant concentration	Zeta potential/(mV)			
				Average value and standard deviation
无	–34.0	–38.0	–42.3	–38.1 ± 4.151
0.03%	–35.5	–36.7	–37.3	–36.5 ± 0.92
0.05%	–35.0	–36.9	–38.1	–36.67 ± 1.56
0.1%	–30.5	–31.7	-34.2	–32.13 ± 1.89
0.15%	–29.1	–29.8	–31.3	–30.07 ± 1.12

TABLE 12 | Effect of SDYJ-2 emulsion with different concentration on particle size distribution of PEM drilling fluid.

Flocculant concentration	D10/(μm)	D50/(μm)	D90/(μm)
–	1.543	29.84	293.5
0.03%	14.68	31.73	748.2
0.05%	26.21	122.6	859.4
0.1%	35.78	278.4	888.2
0.15%	40.68	641.9	1,203

the liquid yield will show a downward trend (Zhang et al., 2005).

4.1.2 Study on Flocculation and Destabilization of pH Value

Because the waste PEM drilling fluid contained some organic emulsifying functional groups, the change of flocculation pH value caused the phenomenon of "inactivation" of the organic emulsifying functional groups, and the drilling fluid lost its stability under the original conditions (Hu et al., 2017). When H^+ was added to PEM drilling fluid, the negative charge on clay surface can be neutralized, and the dispersive emulsification ability of organic functional groups was weakened, resulting in instability of colloidal system, local shrinkage, cementation, and dehydration of the drilling fluid. When the flocculation pH value was greater than 7, the increase of flocculation pH value created an alkaline condition for the PEM drilling fluid, which increased the Zeta potential between clay particles and the repulsive force between clay particles, making it difficult for clay particles to condense (Xie et al., 2011).

4.2 Study on Action Mechanism

4.2.1 Effect of Inorganic Flocculant on Flocculation of PEM Drilling Fluid

The Zeta potential of the waste PEM drilling fluid system was high, and the system was stable (**Table 5**), indicating that natural sedimentation and stratification will not occur. The Zeta potential of the clay particles of the system was decreased due to the addition of inorganic flocculants, reducing notably system stability. Among them, the inorganic flocculant $CaCl_2$ had the greatest influence on the Zeta potential of the system with the average value to -32.5 mV and the standard deviation to ±0.656 mV. With the increase of the concentration of $CaCl_2$, the Zeta potential of the drilling fluid system gradually decreased. In **Table 6**, the addition of concentration of $CaCl_2$ reached 2%,

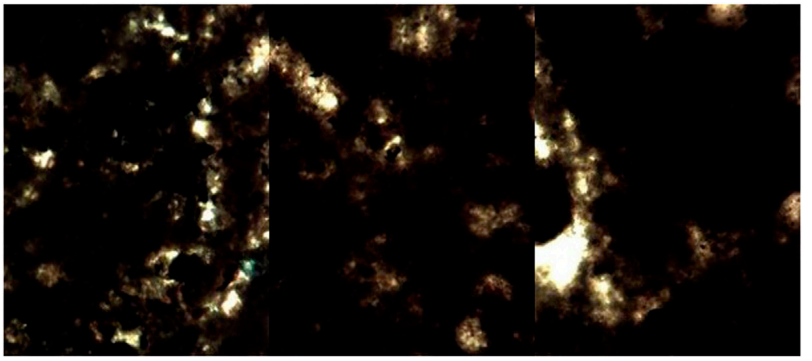

FIGURE 5 | Microscopic morphology of PEM drilling fluid treated with organic flocculant.

the Zeta potential of the system with the average value was -29.03 mV, and the standard deviation was ±0.90 mV.

The small increase in particle size in PEM drilling fluid was accompanied by the addition of inorganic flocculants, which makes the system easier to flocculate to a certain extent (shown in **Tables 7** and **8**). The solid phase in the lower part of the flocculated drilling fluid is made and the floc morphology is observed under the microscope, as shown in **Figures 3** and **4**. The floc particle distribution of PEM drilling fluid treated with inorganic flocculant is relatively loose and has no trend of connection. Combined with the Zeta potential test results, it indicated that the inorganic flocculant mainly reduces the Zeta potential of the system through electric neutralization, reducing the stability of the PEM drilling fluid system.

4.2.2 Influence Law of Organic Flocculant on Flocculation of PEM Drilling Fluid

Polyacrylamide emulsions with different cationic degrees have no obvious effect on the zeta potential of the PEM drilling fluid system because of the small amount of organic flocculants, and the Zeta potential decreased slightly and slightly fluctuated as shown in **Tables 9** and **10**. The organic flocculant remarkably increases the particle size distributed in the PEM drilling fluid, which was more conducive to the flocculation and sedimentation of the system. In addition, as the cationic degree of the organic flocculant increases, the particle size also increases. **Tables 11** and **12** showed that with the increase of SDYJ-2 emulsion concentration, the overall trend of the Zeta potential gradually decreased and the particle size increased gradually. After the lower solid phase is made, it is observed under the microscope. As shown in **Figure 5**, the drilling fluid particles are connected with each other to form a network structure, which is more prone to flocculation and sedimentation. When the concentration of SDYJ-2 is 0.15%, the D50 increases by 21.5 times. Jointing the Zeta potential test results, it shows that the organic flocculant mainly helps the system destabilization through bridging connection, and also partially reduces the Zeta potential of the system through electric neutralization, so as to reduce the stability of the PEM drilling fluid (Xue et al., 2008).

5 CONCLUSION

1) The $CaCl_2$ dosage and flocculation pH value had the remarkable influence on the flocculation and destabilization of PEM mud through the orthogonal test, and the optimal flocculation conditions are optimized. The deliquid rate of flocculation destabilization can reach 70.96%.
2) The Zeta potential of the waste PEM drilling fluid had reduced by electric neutralization for the inorganic flocculant to reduce the repulsion between suspended solids and to lose stability; organic flocculants contain cations and long molecular chains, which wrap clay particles by bridging-bending and electric neutralization, so that suspended particles gather into large flocs and large particles.

DATA AVAILABILITY STATEMENT

The original contributions presented in the study are included in the article/Supplementary Material. Further inquiries can be directed to the corresponding authors.

AUTHOR CONTRIBUTIONS

YL: Conceptualization, methodology, investigation, writing—original draft; YZ: Supervision, validation, writing—review and editing, funding acquisition; TX: Supervision, funding acquisition; MY: Formal analysis, resources; DW: Resources; BL: Resources.

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Conflict of Interest: The authors YL, YZ, TX, MY and DW were employed by China National Offshore Oil Corporation (China) Co. Ltd. Author BL was employed by China Oilfield Services Co. Ltd.

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