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Study on the Effect of Humic Acid Acetamide on the Rheological Properties of Diesel Oil-Based Drilling Fluids

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Abstract. A new kind of humic acid acetamide compoud was synthesized by chemical modification of humic acid with octadecylamine, and the effect of humic acid acetamide on the rheological properties of oil-based drilling fluids was investigated. The results indicated that the humic acid acetamide had excellent dispersing property, and good capacity of depressing fluid loss. Moreover, the humic acid acetamide had better property of depressing fluid loss than oxidated asphalt. As a result, this humic acid acetamide is an excellent fluid loss agent for diesel oil-based drilling fluids, and is an good alternative to oxidated asphalt.

Introduction

Humic acid derivatives have the functions of sealing, anti-sloughing and depressing fluid loss[1]. Meanwhile, humic acid derivatives have good properties of thermostability and salt tolerence, and they are a kind of commonly used additives for drilling fluids[2]. Furthermore, humic acid derivatives are usually water-soluble ionic product, which cannot dissolve or disperse in oil phase, so they could not be used as efficient additive for oil-based drilling fluid system. Hence, humic acid should be treated by a chemical modifying agent, and then they could have certain lipophilic property[3, 4].

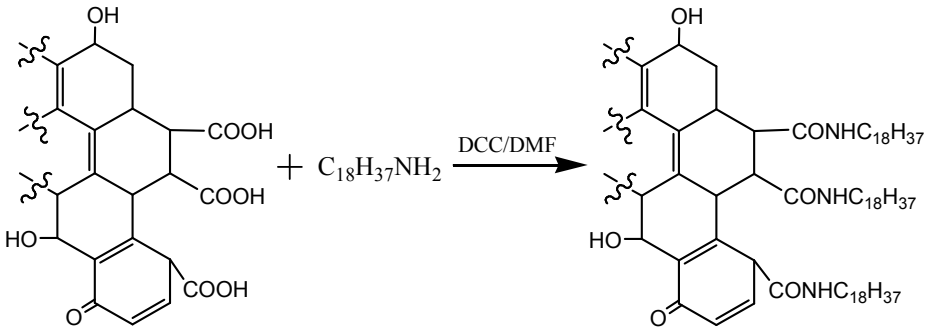
Here, a new kind of humic acid acetamide derivatives is synthesized by chemical modification of humic acid with long chain amine compound. The obtained substituted-ammonium humic acid FLHA has good lipophilic property, so it can dissolve or disperse in oil phase. In addition, FLHA has good temperature resistance property. FLHA has good capacity of fluid loss for oil-based drilling fluids, which is conducive to reducing the leakage of drilling fluids and wellbore stability.

Materials and methods

Materials. Humic acid, industrial grade. octadecylamine, dicyclohexylcarbodiimide (DCC), dimethyl formamide (DMF), are purchased from Beijing Chemical Works. All chemicals, reagents, and solvents are used as received from commercial sources without further purification.

Methods. To a 500 mL, four-necked flask equipped with a nitrogen inlet and a magnetic stirrer, humic acid, octadecylamine, DCC and 200 mL DMF are added. The mixture is stirred at 150 ^{o}C for 16 h in nitrogen. After the solvent is removed at reduced pressure, the product is washed by distilled

water for several times to remove residual DMF, and dried in vacuum at 100 °C for 24 h, pulverized through a 60~80 mesh sieve, and then the humic acid acetamide polymer is obtained as a brown powder.



Scheme 1 Synthetic route to the humic acid acetamide polymer

Results and discussion

The effect of dosage of FLHA on the rheological properties of oil-based drilling fluids. Table 1 illustrates the influence of the dosage of FLHA on the rheological properties of oil-based mud, Fig. 1 shows the relation curve of the dosage of FLHA and HTHP filtration of drilling fluids after rolling 16 h at 150 °C. From Table 1 and Fig. 1, the results indicate that FLHA has very good capacity of depressing fluid loss at 150 °C. The fluid loss effect becomes better and better with the increase of dosage of FLHA.

In addition, FHLA has certain effect of thickening and improving shear strength. All things considered, mainly on account of the cost and depressing effect of fluid loss, the optimum dosage of FLHA at 150 °C is 3%. Moreover, as a oleophylic colloid, FLHA could increase the emulsion-breaking voltage of drilling fluids after rolling 16 h at 150 °C, so the stability of drilling fluids could be improved.

Table 1 Influence of dosage of FLHA on the properties of drilling fluids

Dosage of FLHA/%	Aging condition	AV/mPa·s	PV/mPa·s	YP/Pa	GEL/Pa/Pa	FL_{API}/mL	FL_{HTHP}/mL	ES/V
0	before roll	24	19	5	4.5/5	1.2	-	1200
	after roll	16	16	0	0.25/0.5	-	37	420
1%	before roll	31	20	11	5/6	0.5	-	1136
	after roll	21	20	1	1.5/2	-	20	526
2%	before roll	33	22	11	5.5/6	0.4	-	960
	after roll	25.5	21.5	4	2/2.5	-	10.4	560
3%	before roll	35	23	12	5/5.5	0.3	-	900
	after roll	28	22	6	2.5/3	-	6.6	680
4%	before roll	36	24.5	11.5	4.5/5	0.2	-	810
	after roll	38	28.5	9.5	3/3.5	-	5.6	580

Note: (1) Formula for preparing oil-based mud: 240 mL diesel oil + 60 mL calcium chloride aqueous solution with concentration of 20% + 2% organic clay + 2% primary emulsifier + 2% auxiliary emulsifier + 1% wetting agent + 0-4%FLHA + 1% calcium oxide + barite (The density of mud is 1.20 g/cm^3)。

(2) The rolling condition is 150 °C × 16 h, HTHP filtration condition is 150 °C × 3.5 MPa.

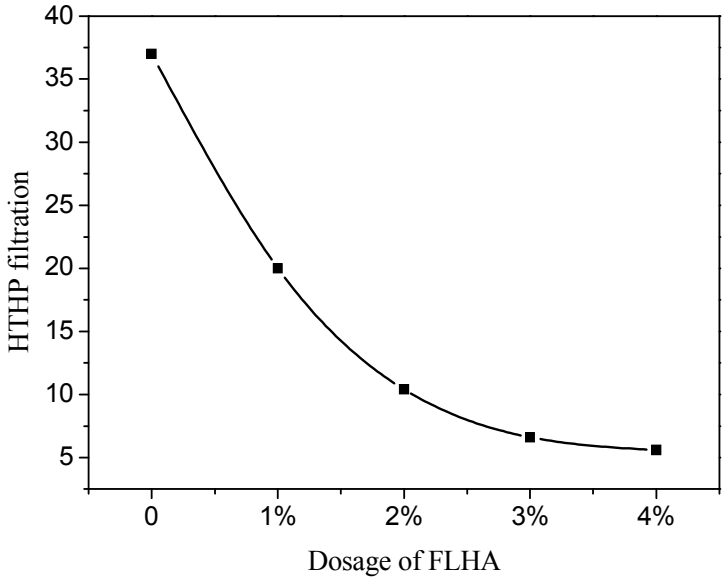


Fig. 1 The relation curve of dosage of FLHA and HTHP filtration (after rolling at 150 °C)

The temperature resistance properties of FLHA. The rheological properties of oil-based drilling fluids at various temperatures is showed in Table 2. When the temperature is less than 150 °C, the experimental results indicate that HTHP filtration of drilling fluids increases gradually as the rolling temperature rises. When the rolling temperature is 180 °C, the HTHP filtration of drilling fluids is large, mainly due to humic acid acetamide being easily degraded at 180 °C. As a result, the temperature resistance capacity of FLHA can be up to 150 °C, and FLHA has very good capacity of fluid loss when the temperature is less than 150 °C.

Table 2 The rheological properties of oil-based drilling fluids before and after rolling 16 h at various temperatures

Rolling temperature/°C	Aging condition	AV/mPa·s	PV/mPa·s	YP/Pa	GEL/Pa/Pa	FL_{API}/mL	FL_{HTHP}/mL	ES/V
90	before roll	34	21	13	5/5.5	0.3	-	900
	after roll	33.5	25	8.5	3.5/4	-	1.4	686
120	before roll	34	21	13	5/5.5	0.3	-	900
	after roll	29	22.5	6.5	3/3.5	-	2.3	626
150	before roll	34	21	13	5/5.5	0.3	-	900
	after roll	28	22	6	2.5/3	-	6.6	680
180	before roll	34	21	13	5/5.5	0.3	-	900
	after roll	41	36.5	4.5	3/3.5	-	36	313

Note: Formula for preparing oil-based mud: 240 mL diesel oil + 60 mL calcium chloride aqueous solution with concentration of 20% + 2% organic clay + 2% primary emulsifier + 2% auxiliary emulsifier + 1% wetting agent + 3%FLHA + 1% calcium oxide + barite (The density of mud is 1.20 g/cm^3)。

The **comparison between FLHA and other fluid loss additives.** FLHA, oxidated asphalt (the softening point is about 170 °C), domestic humic acid fluid loss additive samples CF200 and FB-MOTEX with a concentration of 3% are added to oil-based drilling fluids, respectively, and the rheological properties of oil-based mud formulation at 150 °C are tested as shown in Table 6.

Oxidated asphalt is a kind of additive of good capacity of depressing fluid loss, which is widely applied in China oil fields. Table 3 indicate that the HTHP filtration of drilling

fluids with FLHA at 150 °C is 6.6 mL, which is significant better than oxidated asphalt, CF200 and FB-MOTEX. As a result, substituted-ammonium humic acid FLHA is a better fluid loss agent.

Table 3 The rheological properties of oil-based drilling fluids after addition of different fluid loss agents

Sample	Aging condition	AV/mPa·s	PV/mPa·s	YP/Pa	GEL/Pa/Pa	FL_{API}/mL	FL_{HTHP}/mL	ES/V
FLHA	before roll	37	25	12	5/5.5	0.3	-	900
	after roll	28	22	6	2.5/3	-	6.6	680
Oxidized asphalt	before roll	29	22	7	4/6.5	0.7	-	632
	after roll	28	26	2	2.5/3	-	7.8	534
CF200	before roll	34	25	9	6/6.5	0.5	-	582
	after roll	26.5	24	2.5	2.5/3	-	16	411
MOTEX	before roll	43	32	11	7/7.5	0.4	-	603
	after roll	47.5	41	6.5	4/4.5	-	20	426

Note: (1) Formula for preparing oil-based mud: 240 mL diesel oil + 60 mL calcium chloride aqueous solution with concentration of 20% + 2% organic clay + 2% primary emulsifier + 2% auxiliary emulsifier + 1% wetting agent + 3% Sample + 1% calcium oxide + barite (The density of mud is 1.20 g/cm^3)。

(2) The rolling condition is 150 °C × 16 h, HTHP filtration condition is 150 °C × 3.5 MPa.

Summary

(1) A new type of humic acid acetamide FLHA, suitable for controlling fluid loss in diesel oil-based invert emulsion drilling fluids, is prepared.

(2) FLHA has excellent dispersing property in invert emulsion drilling fluids. Meantime, FLHA has good capacity of depressing fluid loss. FLHA could solve problem that the filtration of oil-based fluids is large under high temperature.

(3) FLHA has good properties of thickening and improving shear strength. FLHA can improve the stability of drilling fluids.

(4) FLHA has better property of depressing fluid loss than oxidated asphalt, CF200 and FB-MOTEX. FLHA is an good alternative to oxidated asphalt, and is conducive to reservoir protection and environment protection.

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