

## Article

# Study on Plugging Microfracture by Using High-Temperature Emulsified Bitumen

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**Abstract:** A new kind of modified emulsified bitumen used to plug a microcrack was studied. The sizes of high-temperature emulsified bitumen were fit for the sizes of the microcrack, which were approved by the scanning electron microscope and laser particle size analyzer. Some tests have been designed to demonstrate that the polymer could be used to promote the softening point of modified asphalt, and the high-temperature emulsified bitumen has also shown an excellent performance in terms of static filtration, the viscous coefficient, and extreme pressure lubrication, as well as to inhibit shale expansion. The permeability recovery could reach 88.26%, which meets the specification requirements. The mud cake, which was formed by high-temperature emulsified bitumen as an additive, was thin, tough, and dense, which was proved by the scanning electron microscope. The process used to obtain this additive was simple, and the performance of the plugging microfracture was excellent, so this kind of plugging agent could have a better application future.

**Keywords:** emulsified bitumen; borehole stability; water base drilling fluid



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## 1. Introduction

Drilling fluid leakage refers to a phenomenon in which drilling fluid or other media (cementing cement mud, etc.) leak into pores, microfractures, and other spaces. As a common situation that occurs in the drilling process, this leakage makes economic loss fair. What is more, serious leakage leads to the decline of wellbore stability, affects the drilling speed, and even threatens the personal safety of operators, which brings great trouble to oil and gas exploration and protection [1]. Therefore, a series of measures need to be taken to control the occurrence of leakage [2]. Adding lost circulation additives to drilling fluid can plug microcracks, avoid drilling mud or cementing cement mud leakage during drilling operations [3], reduce economic losses during drilling, and improve the service life of oil wells.

Preparing lost circulation additives is a valid way to minimize filter loss. Some inorganic materials, such as CaCl<sub>2</sub>, which use ion exchange to reduce clay hydration [4], and organic materials, such as polymeric quaternary amines, have been prepared [5,6]. At the same time, some natural polymers and synthetic polymers [6,7] have been applied as lost circulation additives by forming layers to stop the leakage of drilling fluid or cementing cement mud [8,9]. But in super deep boreholes, with well temperatures higher than 120 °C, the performance of preventing drilling fluid or cementing cement mud leakage is reduced [10,11]. In high-temperature boreholes, the degradation of the polymeric lost circulation additives results in remarkable enhanced filtration loss [12]. T. Marriott et al. [13] investigated foamed conventional lightweight cement slurries with an ultra-low density and low equivalent cyclic density (ECD) to address the issue of cyclic loss. Long et al. [14] proposed that nanomaterials can effectively fill microfractures and reduce drilling fluid

loss. Bore-plate [15] is a native asphalt mixture containing high and low softening point asphalt, alkaline lignite, and non-ionic surfactants used to address cyclic loss issues. To reduce the lost circulation, a mixture of bitumen and surfactant with a wide particle size distribution [16] was studied. Thus, the traditional lost circulation additives with excellent plugging performance used in harsh conditions could not solve the wellbore stability and filtration loss led by the microfracture [17]. However, common cationic emulsified asphalts have been studied to reduce losses at low temperatures, leading to microfractures with good results. In contrast, there are no reports on the use of high-temperature emulsified asphalt for reducing well leakage in ultra-deep boreholes where the well temperature exceeds 130 °C.

## 2. Experimental

Water-borne epoxy resin (WEP) was bought from Aladdin. The emulsifier and base asphalt were obtained from Sobute New Materials Co., LTD. (Nanjing, Jiangsu, China). The sulfonated bitumen (SAS) and a kind of oil-soluble resin polymer plugging agent (FT-3000) were obtained from Shandong Deshunyuan Petroleum Sci. & Tech. Co., Ltd. (Dongying, Shandong, China). Sulfonated phenolic resin (SMP-II), hydrolyzed polypropylene ammonium salt (ammonium salt), and a polymer inhibitor were purchased from Hebei Yan Xing Chemical Co., Ltd. (Cangzhou, Hebei, China). Sodium carbonate was bought from Arkema (Suzhou) Polyamides Co., Ltd. (Suzhou, Jiangsu, China). The test equipment contains a scanning electron microscope (Quanta 250, Phenom, Waltham, MA, USA) and laser particle size analyzer (HELOS-SUCCELL ranging from 0.1 to 875  $\mu\text{m}$ , SYMPATEC GMBH, Clausthal-Zellerfeld, Germany).

### 2.1. General Procedure for Preparing High-Temperature Emulsified Bitumen (HTEB)

We added 30 g of lignin emulsifier and 40 g of WEP to 320 g of water to obtain 400 g of soap solution, where 37% hydrochloric acid solution was used to regulate the pH value to 2–2.5.

A brown and creamy textured asphalt emulsion was obtained by mixing 600 g of liquid asphalt preheated to 138–142 °C and soap preheated to 50–60 °C in a colloid mill with high-speed shear. The emulsions were stored at room temperature for 8 h and then tested using ASTM D-2444 [18].

### 2.2. General Procedure for Obtaining Drilling Fluid

We added 200 g of bentonite to 5000 L of water and stirred for 24 h at 2000  $\text{r}/\text{min}^{-1}$  to obtain a stable bentonite aqueous solution. Then, 50 g of ammonium salt, 6 g of sodium carbonate, and 25 g of polymer inhibitors were added into the stable bentonite aqueous dispersion, and this was stirred for 30 min at 2000  $\text{r}/\text{min}$  to obtain drilling mud.

### 2.3. General Method for Obtaining Rheological Measurements of Drilling Mud

The ZNN-D6 six-speed rotary viscometer (Meik) was used to investigate the apparent viscosity ( $A_V$ ), plastic viscosity ( $P_V$ ), and yield point ( $Y_p$ ) of drill mud, and the test procedure of the rheological was according to SYT 6864-2020 [19] (calibration method of drilling fluid viscometer). The average value of the three groups of test samples was taken as the test result.

### 2.4. General Method for Obtaining Static Filtration

The GGS-42 High-Temperature and High-Pressure (HTHP) Filter Press (Baroid, Houston, TX, USA) was used to study the value of static filtration, and we used the test method according to (GB/T 16783.1-2014 [20]). Static filtration is the value of the average of the three groups of samples in parallel tests.

### 2.5. General Procedure for Obtaining Lubrication

Extreme pressure lubrication instrument (American) was used to investigate the lubrication of HTEB, and we used the experiment method according to SYT 6094-1994 [21] (evaluation procedure of lubricants for drilling fluids). The lubrication is the value of the average of the three groups of samples in parallel tests.

### 2.6. General Procedure for Obtaining Core Inflation Inhibitory

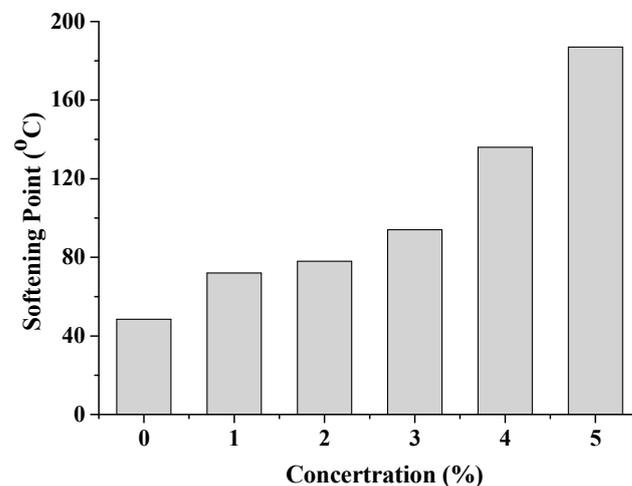
The NP-01 shale expansion tester (Hengtaida) was used to investigate the ore inflation inhibitory property of HTEB, and we used the experiment method according to SY/T5946-1994 [22] (method for the determination of linear core expansion reduction rate). The core inflation inhibitory value was obtained by three groups of samples in parallel tests.

## 3. Results and Discussion

In this paper, the performance of high-temperature emulsified asphalt was verified by static filtration, lubrication, core swelling inhibition, and electron microscope scanning tests, and the results showed that the high-temperature emulsified asphalt obtained from the study can be used as a sealant for water-based drilling fluids, and a new scheme of high-temperature emulsified asphalt as an encapsulant for microfracture repairs was proposed.

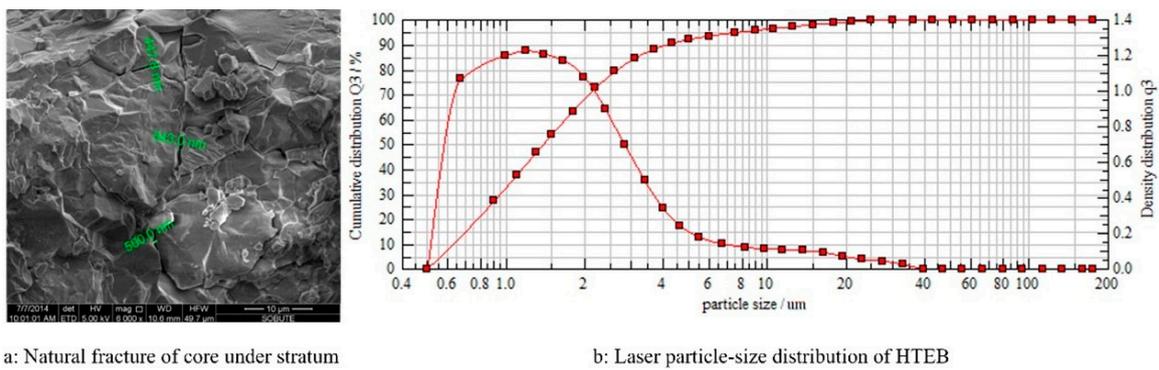
### 3.1. The Microfracture Plugging Size Analysis

This analysis indicates that the softening point of emulsified asphalt is related to the dosage of WEP in HTEB. As shown in Figure 1, a low concentration of WEP results in a low softening point of 120 °C, which is much lower than the specification requirement. As the concentration increases, the temperature at the softening point rises linearly. When the concentration of WEP rises to 4% and 5%, the softening point reaches 136 °C and 180 °C, meeting the specification requirement. For economic purposes, the following study will be continued with the concentration of WEP at 4%.



**Figure 1.** The softening point of emulsified asphalt influenced by the different WEP concentrations.

As shown in Figure 2a, some natural fractures appear randomly in the core, the size of which in the early stage is between nanometer and micrometer. To ensure that these cracks can be effectively filled further, the particle size distribution of HTEB is shown in Figure 2b. The modulus particle size and average particle size of HTEB are 1.05  $\mu\text{m}$  and 1.18  $\mu\text{m}$ , respectively, meaning that they are in line with the sizes of the natural cracks, and that they could be used to seal the natural microcracks.



**Figure 2.** Comparative analysis of the maximum particle size of early cracks and HTEB.

3.2. The Viscosity of Drilling Mud with Different Additives

The thickening effect of mud was used as one method of the evaluation of the influence of filling microcracks, and the SAS and FTI-3000, which are more mature market plugging agent types, were used as control groups to further study the effects of different plugging agents on the rheological properties of mud slurry, and the results are shown in Table 1.

**Table 1.** Effect of different additives on the viscosity of drilling mud.

Additives	Treatment	AV /mPa·s	PV /mPa·s	Yp /Pa	Cutting/Pa	
					Beginning	Final
Blank	a	31	28	1.5	1.5	1.5
	b	20	17	3	1.5	2
SAS	a	55	53	2	2	2
	b	32.5	28	2.5	2.5	2.5
FT-3000	a	43.5	42	1.5	1.5	1.5
	b	24	20	1.5	1.5	1.5
HTEB	a	37	36	1	1	1
	b	24	20	1.5	1.5	1.5

a: Mix additives and drilling mud together and stir for 10 min with 10,000 r/min. b: The drilling mud system was aged in a 130 °C digestion tank for 18 h.

As can be seen in Table 1, three kinds of additives affect the rheology of the mud slurry to varying degrees at room temperature. Among them, SAS has the greatest impact on the fluidity of the slurry, the Av, Pv, and Yp all show a significant increase, where the maximum increase reaches around 53%, while the HTEB parameters changed the least. When the temperature rises to 130 after aging for 18 h, the HTEB and FT-3000 have a relatively excellent index, while SAS still has a more obvious increase. This indicates that the self-developed HTEB can be used as a drilling plugging agent and has little effect on the rheological properties of mud.

3.3. The Static Filtration of Drilling Fluid with Different Additives

The filtration and wall-building performance of drilling fluid is very important in the drilling process, and the static filtration of drilling fluid with the different plugging additives (SAS, FT-3000) and different dosages of self-developed HTEB was studied in this paper, and the results are shown in Table 2.

As shown in Table 2, the static filtration of drilling mud without additives is 25.8 mL. With different types and dosages of additives, the static filtration decreases obviously. When the dosage is 3%, compared with 23.4 mL of SAS and 21.8 mL of FT-3000, the static filtration of HTEB reaches the lowest level at 18.8 mL. This is because, under HTHP conditions, HTEB can still maintain a good three-dimensional network structure. This kind of strongly bonded structure can ensure that the mud cake is thin and dense.

**Table 2.** Static filtration <sup>a</sup> influenced by different additives.

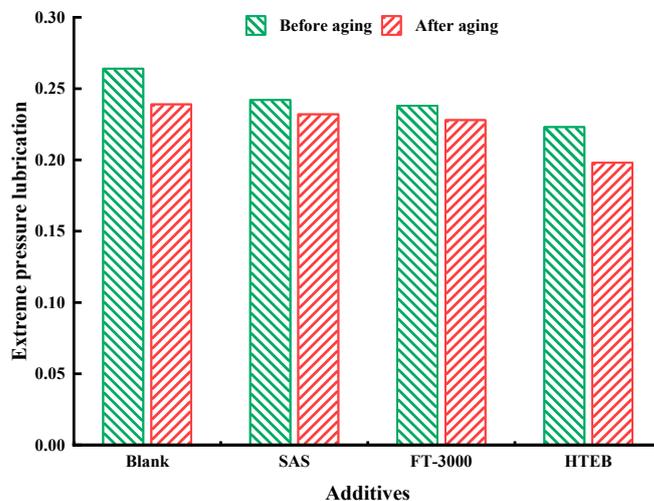
Additives		Static Filtration/L
Blank	-	0.0258
SAS	3%	0.0234
FT-3000	3%	0.0218
	2%	0.0221
HTEB	3%	0.0188
	4%	0.0176
	5%	0.0172

<sup>a</sup>: Mix additives and drilling mud together and stir for 10 min with 10,000 r/min. Simulated temperature: 130 °C; simulated static pressure: 3.5 MPa.

To further prove the change in the static filtration of HTEB with different dosages, a 2%–5% dosage comparison test was carried out, as can be seen from Table 2; with the increase in dosage, the static filtration decreases linearly, and its value decreases from 22.1 to 17.2. When the dosage began at 3%, the frequency of decline gradually changed, considering the economic benefits, so then 4% was deemed the optimal incorporation amount.

### 3.4. The Lubricating Properties of Drilling Fluid with Different Additives

The lubrication performance of drilling fluid plays a vital role in reducing downhole complications such as stuck drilling, ensuring safe and fast drilling. The extreme pressure lubrication characterizes the friction between the well slurry and the drill pipe, and the viscous coefficient is the friction between the drill pipe and the borehole. In this paper, these two coefficients are used to evaluate the lubrication performance of drilling fluids with different additives, and the results are shown in Figures 3 and 4.

**Figure 3.** The effect of different additives on extreme pressure lubrication.

As shown in Figure 3, the extreme pressure lubrication coefficient of the control group after aging shows a similar downward trend; this is because the drilling fluid acts as a lubricant in the drilling process, and the polysulfur drilling fluid substrate forms an oil film on the surface of the drill bit while aging at a high temperature, which can effectively reduce the friction between the drill bit and the rock. Further, with the addition of different additives, HTEB shows the lowest extreme pressure lubrication coefficient no matter whether this occurs before or after aging. This is because powdered SAS and FT-3000 additives do not make it easy for particles to adhere to the drill bit during the lamination process, and HTEB, as an epoxy-modified emulsified asphalt, its asphalt substrate, is lubricating at the same time. The cohesion formed by epoxy curing can make it easier for the drilling fluid to adhere to the oil film on the surface of the drill bit to protect the drill

bit, which further shows that the developed HTEB displays stable and excellent lubrication performance regardless of low or high temperatures.

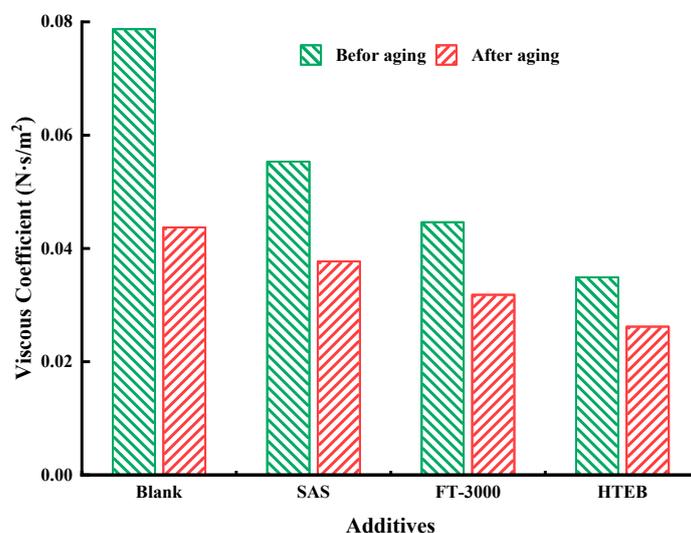


Figure 4. Influence of different additives on viscous coefficient.

As shown in Figure 4, like the change in the law of the extreme pressure lubrication coefficient, the viscous coefficient also shows a downward trend with aging. The reason why drilling fluid without additives decreases significantly is because of the formation of oil film. It can protect the drill pipe to a greater extent and reduce the friction between the borehole wall and the drill pipe. Further, with the addition of additives, HTEB shows the lowest viscous coefficient before and after aging, which is because, on the one hand, the powdered SAS and FT-3000 additives do not make it easy for particles to adhere to the drill pipe during the film lamination process; on the other hand, the epoxy-modified asphalt in HTEB has good bonding properties, which can form a mud cake on the wall. This thin and dense mud cake can greatly reduce the friction between the wall and the drill pipe. At the same time, the emulsified asphalt in HTEB has a lubricating effect, which can reduce the addition of lubricant components in the overall drilling fluid to a certain extent, and effectively reduce costs. This further demonstrates that the developed HTEB has good lubricating properties and excellent economic benefits.

### 3.5. Comparative Analysis of Microscopic Morphology after Plugging

The self-developed HTEB and FT-3000 both have similar excellent leak plugging performance, as revealed by the macro test above. To clarify the difference in the microscopic mechanism of plugging between them, SEM was further used to test the microscopic morphology, and the results are shown in Figure 5.

As shown in Figure 5a, the mud cake which formed after the addition of FT-3000 is very thick and has many microcracks inside, which makes it easy for water to penetrate through the microcracks, leading to the further loosening of the mud cake. On the contrary, it can be found that the mud cake formed after the addition of HTEB is thin and has few microcracks, and the dense mud cake structure makes it difficult for water to penetrate it. Compared with FT-3000, HTEB is an epoxy-modified emulsified bitumen, and the three-dimensional network structure of the epoxy can be better combined with the asphalt molecular chain during the reaction process so that its intermolecular force is enhanced and has stronger cohesion and adhesion. Therefore, the HTEB microcrack repair agent proposed herein for sealing microcracks shows superior performance compared to the market-proven materials (FT-3000).

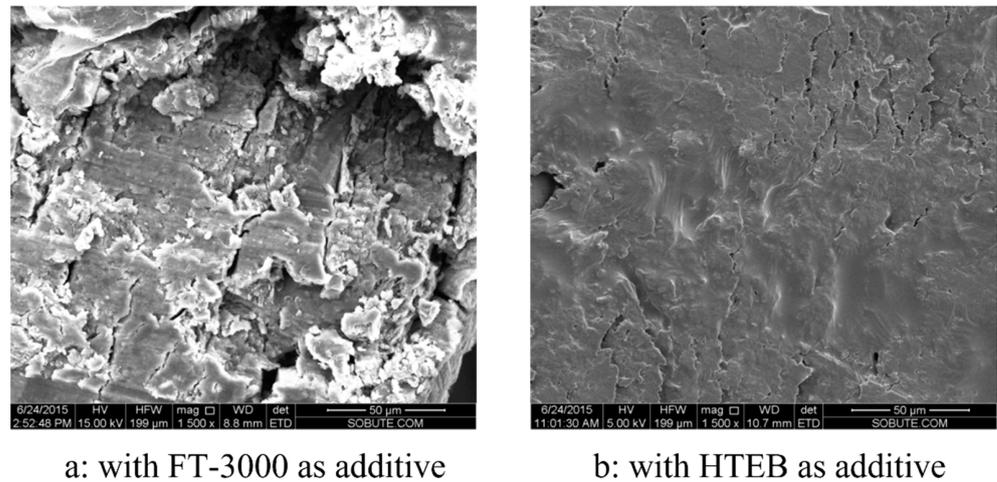


Figure 5. Microscopic morphology of microcrack plugging by different additives.

3.6. The Core Expansion Properties of Drilling Fluid with Different Additives

All kinds of drilling mud with different additives can interact with the core and form a mud cake, which has been studied above with the SEM test. These mud cakes can effectively inhibit the expansion of the core to ensure the stability of the drilling state. In this paper, the drilling fluid with the best additive is selected by comparing the expansion coefficient of different cores before and after aging, and the results are shown in Figures 6 and 7.

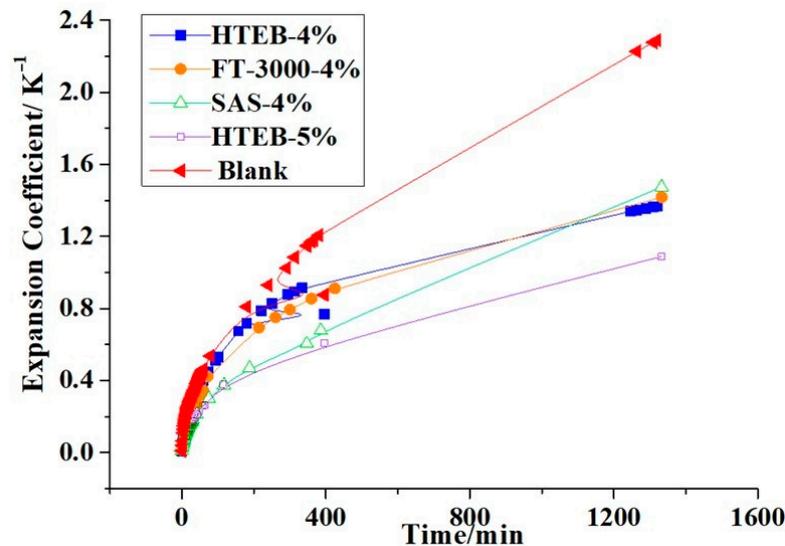


Figure 6. Effect of different additives and dosages on the expansion coefficient before aging.

The expansion coefficient of shale formation in the reservoir affects the stability of the borehole wall. The larger the coefficient of shale expansion, the more easily the borehole wall becomes unstable. As shown in Figure 6, with the extension of time, the curves of the expansion coefficient of drilling fluid along with different additives all show an increasing trend; the drilling fluid without additives increases the most when it reaches 22.5 h, and its expansion coefficient reaches 2.289, which is because, with the drilling process, the drilling fluid cannot form a high-quality mud cake, and the microfracture of the mud cake is not well blocked, resulting in the core expanding, and its expansion rate can reach 27.8%. With the addition of additives, the increasing frequency of its expansion coefficient gradually decreases. Also, when the time reaches 22.5 h, the expansion coefficient of the 4% SAS, 4% FT-3000, 4% HTEB, and 5% HTEB reaches 1.463, 1.418, 1.366, and 1.089, respectively. On the one hand, it is indicated that the addition of additives could effectively plug the cracks

and inhibit core expansion; on the other hand, considering the economic cost, the addition of 4% HTEB met the construction needs, which was the best choice before aging.

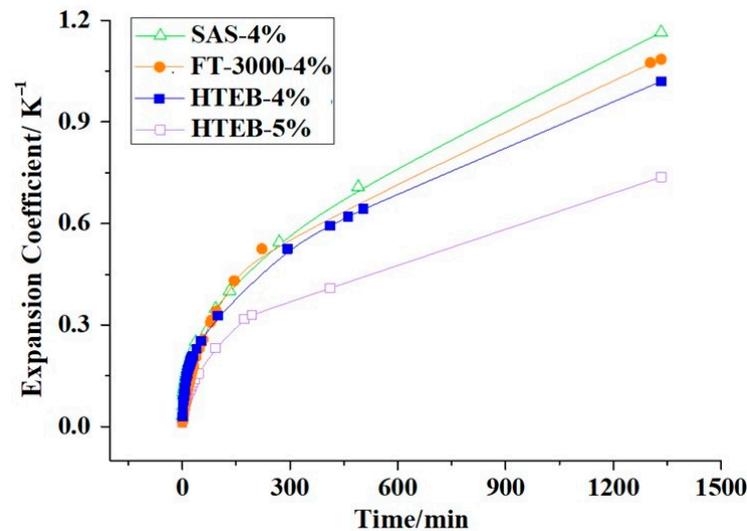


Figure 7. Effect of different additives and dosages on the expansion coefficient after aging.

In terms of further discussion of the expansion coefficient of the control group after aging, it can be seen from Figure 7 that the variation law of the expansion coefficient is consistent with that before aging, all the curves of the expansion coefficients show a slow increasing trend, which is because the drilling fluid with additives can effectively inhibit the expansion of the core and plug the microcrack, and the influence of the drilling fluid by the high-temperature aging environment is also weakened. The expansion coefficients of different dosages of HTEB are both lower than that of FT-3000 and SAS. When the expansion coefficient of 4% HTEB is 1.02, the expansion coefficient of 5% HTEB decreases by 27.7% to 0.737. This is because the epoxy asphalt in HTEB is densely bonded to the microcrack interface during the curing process, forming a network structure, and the emulsified asphalt as a ductile material can also change with the core and become more ductile. To sum up, the 4% self-developed HTEB material not only has good leakage microcrack plugging and core expansion abilities before and after aging but it also meets the need for high performance and saves on costs.

#### 4. Conclusions

In this paper, a new method for plugging microcracks using high-temperature emulsified asphalt (HTEB) is proposed, and the following conclusions are drawn:

- (1) When the content of WEP reaches 4%, it can effectively raise the softening point of the asphalt to 136 °C, which meets the specification requirement (120 °C). Between the nano and micro levels, the size of HTEB can comprehensively fill the microcracks, which indicates that HTEB can be used for drilling fluid modification.
- (2) The addition of different additives will increase the viscosity of the mud, thereby reducing the rheology of the mud, among which the influence of HTEB is the smallest, with almost no difference; at the same time, this degree of thickening can achieve a gradual reduction in the static filtration and ensure the stability of the drilling fluid performance.
- (3) The addition of different additives can effectively reduce the extreme pressure lubrication coefficient and viscous coefficient. Among them, the WEP in HTEB forms a kind of three-dimensional network structure to give a strong adhesion ability, and the emulsified asphalt substrate in HTEB has lubricity, meaning that the HTEB has the best lubricating performance.

- (4) Compared with the drilling fluids of FT-3000 and SAS, HTEB forms a high-quality mud cake, which has the advantages of a dense, thin, and smooth surface; the drilling fluid with HTEB can well fill the microcracks and inhibit the core expansion coefficient rate.
- (5) With the increase in dosage, the drilling fluid with HTEB always shows excellent performance, and the optimal dosage is 4% when in combination with economic cost considerations.

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