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Original Paper

A new environmentally friendly water-based drilling fluids with laponite nanoparticles and polysaccharide/polypeptide derivatives

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ABSTRACT

Considering the increasing environmental pressure, environmentally friendly and high-performance water-based drilling fluids (WBDFs) have been widely studied in recent years to replace the commonly used oil-based drilling fluids (OBDFs). However, few of these drilling fluids are entirely composed of natural materials, which makes it difficult to achieve real environmental protection. Using laponite nanoparticles and various derivatives of natural materials, including crosslinked starch, cellulose composite, gelatin ammonium salt, poly-l-arginine, and polyanionic cellulose, a kind of environmentally friendly water-based drilling fluid (EF-WBDF) was built for drilling in environment-sensitive areas. The properties of this EF-WBDF were evaluated by thermal stability tests on rheology, filtration, inhibition, and salt contamination. Besides, biological toxicity, biodegradability, heavy mental content and wheat cultivation tests were conducted to investigate the environmental factor of EF-WBDF. Results showed that EF-WBDF displayed satisfactory thermal resistance up to 150 °C, and the rheological properties did not suffer significant fluctuation, showing potential application in high-temperature wells. The optimal rheological model of EF-WBDF was Herschel–Bulkley model. This EF-WBDF performed an eligible filtration of 14.2 mL at 150 °C and a differential pressure of 3.5 MPa. This fluid could still maintain colloidal stability after being contaminated by 7.5% NaCl or 0.5% $CaCl_2$. Meanwhile, rather low clay swelling degree of 2.44 mm and high shale recovery of more than 95% ensured the inhibitive capability of EF-WBDF. Furthermore, EF-WBDF presented a half maximal effective concentration (EC_{50}) of 51200 mg/L and a BOD/COD ratio of 47.55%, suggesting that EF-WBDF was non-toxic and easily biodegradable. The wheat cultivated in EF-WBDF could grow healthily, beneficial for reducing the adverse impact on ecological environment. The formed EF-WBDF has a promising future for drilling in environment-sensitive and high-temperature areas.

1. Introduction

Drilling fluids perform vital roles in oil and gas drilling operations, such as 1) cooling and cleaning the drilling tools, 2) carrying cuttings from the bottom of borehole to the surface, 3) reducing the friction between drilling strings and sides of the hole, 4) stabilizing the borehole, 5) preventing the inflow of formation fluids (well-control), 6) forming a filter cake to seal pores, 7) assisting in the collection and interpretation of information, and 8) enhancing the rate of penetration (Al-Ansari et al., 2005; Apaleke et al., 2012; Caenn and Chillingar, 1996; Johannes, 2012; Li M. et al., 2015a). With the increasingly stricter technical and environmental requirements in complex formations (Dilmore et al., 2015; Hossain and Apaleke, 2015), the selection of drilling fluids must take account of the thermal stability, salt tolerance, cost, safety, and especially environmental factor (Gao et al., 2021; Zhong et al., 2021). In the past, oil-based drilling fluids (OBDFs) have been commonly applied due to their excellent thermal stability, inhibition, and lubrication performance, especially in some easily hydrated areas (Zhao et al., 2008; Zhuang et al., 2019). However, OBDFs are expensive, disadvantageous to well logging, and easy to

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pollute environment (Liu et al., 2014; Ogeleka and Tudararo-Aherobo, 2011). Later, synthetic-based drilling fluids (SBDFs), which could exhibit excellent properties similar to those of OBDFs under acceptable environmental factor, were proposed and applied in some offshore areas (Celino et al., 2022; Friedheim, 1997; Li W. et al., 2016a, 2016b). But shortcomings on high cost and rheological control limited their application to some extent. These challenges have driven the search for a high-performance WBDFs that can provide performance similar to OBDFs and are environmentally acceptable in sensitive areas (Al-Hameedi et al., 2021; Attia et al., 2010; Lv et al., 2012).

Previously, some WBDFs have been studied to achieve remarkable performance. A novel and environmentally compliant water-based fluid system (HPWBF) has been developed and performed extremely inhibitive and lubricious properties at 160 °C (Attia et al., 2010). Yang et al. have presented a kind of salt water mud with high-temperature resistance of 130 °C for shale gas horizontal drilling by combination of silica nanoparticles and environmentally friendly materials (Yang et al., 2017). Aramendiz and Imqam suggested a WBDF formulation using silica and graphene nanoparticles for unconventional shale applications, which showed a low high-temperature/high-pressure (120 °C/3.5 MPa) filtration of 9.9 mL (Aramendiz and Imqam, 2019). Lyu et al. have also prepared a biodegradable polymer drilling fluid, and this fluid has been applied in Qinshui Basin below 70 °C (Lyu et al., 2019). Zhang et al. studied the environmental friendliness of fuzzy-ball drilling fluids, indicating that environmental indicators of the new fluids were within the ceiling limits of related national standards (Zhang et al., 2019). Besides, glycerin based drilling fluid (Duarte et al., 2021), methylglucoside based drilling fluid, natural plant based drilling fluid (Wajheeuddin and Hossain, 2017), and some others (He and Zheng, 2009; Li F. et al., 2013; Zhu et al., 2018) have been also prepared and evaluated as environmentally friendly WBDFs. However, these fluids seldom performed excellent technical and environmental properties simultaneously. Especially, the contradiction between high-temperature resistance and biodegradability was difficult to balance. Furthermore, the environmental influence of these drilling fluids has been not evaluated systematically and completely. Therefore, developing high-performance WBDFs with environmental acceptability and high-temperature resistance is still a continuous effort (Jiang et al., 2021).

Laponite is a kind of synthetic nanoparticles (Tomas et al., 2018) and exhibits excellent properties in WBDFs (Xiong et al., 2019). It is a synthetic hectorite clay with trioctahedral 2:1 layered structure and has the following chemical formula, $Si_8(Mg_{5.45}Li_{0.4})H_{4.0}O_{24}Na_{0.75}$ (Morariu and Bercea, 2013). Similar to bentonite, the surfaces of disc-shaped laponite nanoparticles are permanently negative-charged and the edges are positively charged (depending on pH), easily leading to the formation of a “house of cards” structure in water. However, the dimension of the laponite particle is only 25 nm for the diameter and 1 nm for the height, which is much smaller than that of bentonite particles (Huang et al., 2021). Due to the nanoscale-sized layers and crystal structure, laponite easily hydrates and forms a stronger gel structure in water. The rheological parameters of laponite gel such as storage modulus, yield stress or viscosity can sharply increase with ageing time (Au et al., 2015). Then, these nanoparticles can exhibit high viscosity and excellent shear-thinning behavior, even at high temperatures, displaying great potential as effective rheological modifiers in bentonite suspensions (Liu et al., 2017; Ruzicka and Zaccarelli, 2011). Moreover, laponite has been also proved to be effective in reducing the static and dynamic sag tendency of weighted water-based drilling fluids (Mohamed et al., 2021). As inorganic nanoparticles, laponite can also enhance the thermal stability of polymers. The water-based drilling fluids consisting of bentonite, terpolymer and laponite have performed substantially higher viscosity than that without laponite (Huang et al., 2021). The composite of polymer and laponite could exhibit excellent filtration property in bentonite WBDFs under high-temperature conditions (Shen et al., 2020). The poly(ethylene glycol)(PEG)/laponite and poly(propylene glycol)(PPG)/laponite aqueous dispersions were beneficial for optimizing the formulation of WBDFs (Morariu et al., 2022). However, seldom research have presented the application of laponite in complete drilling fluid systems, especially at high temperatures. In addition, a low fluid loss and strong inhibition capability are also greatly important for WBDFs. Some natural materials are commonly considered abundant, easily biodegradable, and easily chemically modified. They have been usually used to develop effective drilling fluid additives with environmental performance (Li M. et al., 2015b; Moslemizadeh et al., 2017; Zhong et al., 2021). For example, environmentally friendly lightweight biopolymer drilling fluid from xanthan gum and starch has shown typical Herschel-Bulkley rheological characteristics (Khalil and Mohamed, 2012). A degradable polymer drilling fluid system containing xanthan gum and polyanionic cellulose (PAC) has been developed for coalbed methane well (Lyu et al., 2019). A natural vegetable gum drilling fluid with temperature resistance of 140 °C was developed for reducing environmental damage (Li F. et al., 2014). In recent years, some other natural materials, such as lignin, guar gum (Adewole and Muritala, 2019), soy protein isolates (Li W. et al., 2015), grasses (Wajheeuddin and Hossain, 2017), mandarin peel powder (Al-Hameedi et al., 2019), peanut shells (Al-Hameedi et al., 2021), and aloe vera (Bagum et al., 2022), have been also employed as environmentally friendly additives in bentonite WBDFs. Although some environmentally friendly WBDFs containing natural materials have been developed, there are still two issues that need further research: (1) some drilling fluids are claimed to be environmentally friendly. But some hard-biodegraded materials such as sulfonated resins have been still used in the formulas, making it difficult to achieve real environmental protection; (2) some drilling fluids are entirely composed of natural or biodegradable materials. But the high-temperature resistance of these fluids is always less than 150 °C. The preparation of high-temperature resistant, and environmentally friendly WBDFs still need an effort.

Using laponite nanoparticles and environmentally friendly additives based on natural materials, this paper presented a kind of high-temperature resistant WBDF, in which all of additives were natural materials or modified natural materials. Tests on thermal stability of rheology, filtration, salt contamination, inhibition, and rheology model analyses were conducted to evaluate various properties of the environmentally friendly WBDF (EF-WBDF).

2. Materials and methods

2.1. Materials

Clay and shale. Bentonite used in this study was sodium salt form and consisted of 76.4% clay (Table 1). It was a commercial product purchased from Huaian Tengfei Development Co., Ltd. (China). The content of montmorillonite in this bentonite was 66.5%. Bentonite particles were commonly applied as the skeletal materials of filter cakes, which also provided viscosity for WBDFs. The outcrop shale (Table 2) employed for hot-rolling recovery tests was supplied by the Chuanqing Drilling Engineering Company Limited (China) from Sichuan shale gas field.

Inhibitor. There were two inhibitors used in this study. One was the gelatin quaternary ammonium salt (denoted as GT), which was self-prepared by etherification reaction between gelatin and 2,3-epoxypropyl trimethyl ammonium chloride. GT mainly exerted

Table 1
Mineral compositions of the bentonite and shale cuttings.

Sample	Mineral composition, %							
	Quartz	K-feldspar	Plagioclase	Calcite	Dolomite	Magnesite	Anhydrite	Clay mineral
Bentonite	8.1	5.6	7.1	—*	1.6	1.2	–	76.4
Shale	37.3	1.7	10.5	18.7	0.9	–	2.6	28.3

Note: * indicates that there is no this composition.

Table 2
Clay mineral compositions of the bentonite and shale cuttings.

Sample	Clay mineral composition, %			
	Smectite	Illite	Kaolinite	Chlorite
Bentonite	87	5	8	–
Shale	36	62	1	1

inhibition through encapsulation effect, and performed good high-temperature resistance of 150 °C (Li X. et al., 2019). Another one was the poly-l-arginine (denoted as PArg), which could be synthesized using l-arginine hydrochloride by high-temperature melt polymerization (Li X. et al., 2021b; Xuan et al., 2015). In comparison with GT, PArg could display better high-temperature resistance of 180 °C.

Fluid loss additive. A kind of starch composites (denoted as SPL), obtained by crosslinking among starch, plant polyphenol, and sodium lignin sulfonate (Li X. et al., 2021a), was employed as fluid loss additive in this paper. These composites were colloidal substances in water and had good filtration property when the temperature was below 160 °C. The scanning electron microscopy (SEM) micrograph of SPL was shown in Fig. 1. SPL polymer showed an irregular cystic structure with a ten-micron particle size. Some SPL particles displayed special hollow structure. Besides, white asphalt (NFA-25), an environmentally friendly composite of cellulose, walnut shell flour, resin, and surfactants, was also employed as a plugging additive.

Rheological modifier. Various nanoparticles have been employed as efficient agents for drilling fluids. As mentioned above, laponite was a kind of synthetic nanoparticles with similar structure as natural bentonite (Fig. 2). Laponite easily hydrated in water and formed a gel structure, showing excellent shear-thinning properties in WBDFs. In this study, laponite nanoparticles were utilized as rheological modifiers.

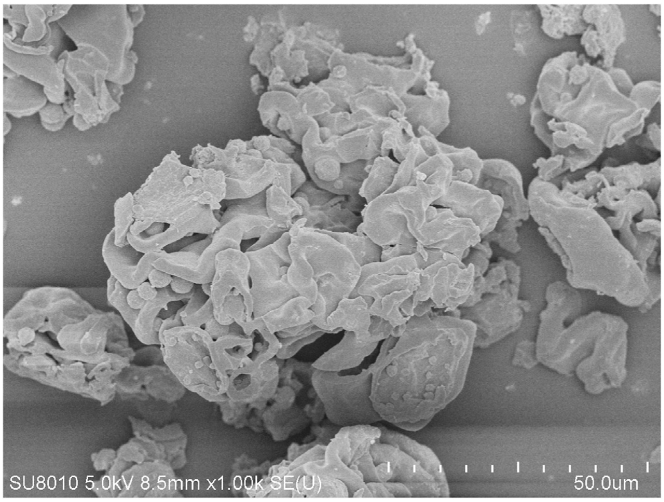

Fig. 1. SEM image of SPL.

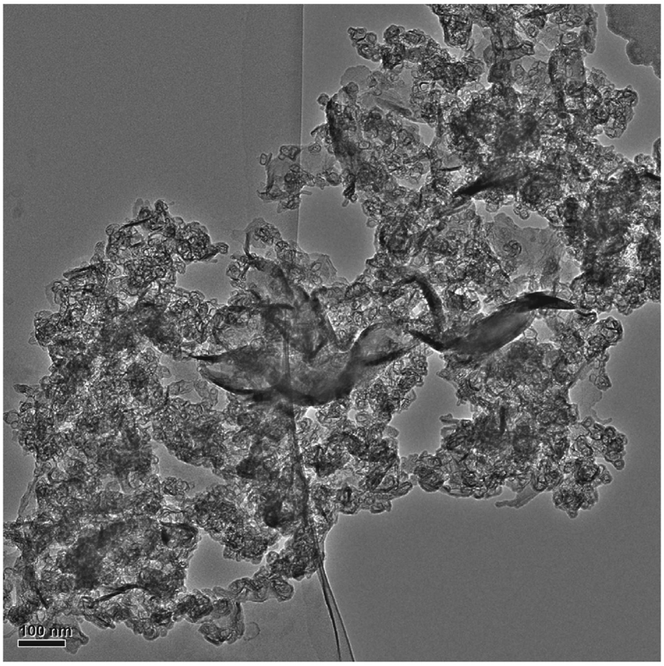

Fig. 2. Transmission electron microscopy (TEM) image of laponite nanoparticles.

All of the additives used in EF-WBDF have been clearly summarized in Table 3.

2.2. Methods

Preparation of the EF-WBDF. Firstly, bentonite dispersion with a fixed dosage of 2 w/v% was prepared. Secondly, while continuing to stir at 8000 r/min, a certain quality of additives was cooperated slowly with 300 mL bentonite dispersion according to the formula (the mass/volume concentration). After each additive was added, the stirring process was continued for 20 min to ensure the full solution and dispersion of the materials. Finally, the EF-WBDF was stirred for another 30 min to get it mixed evenly.

Rheological property. The rheological properties of a serious of EF-WBDFs composed of different additives or dosages were tested. Before each measurement, the EF-WBDF was stirred at high speed of 8000 r/min for 10 min. Then, various rheological parameters including the apparent viscosity (AV), plastic viscosity (PV), yield point (YP), and stress (τ) were surveyed by a ZNN-D6L viscometer. AV, PV, and YP were calculated.

Filtration property. Filtration volumes were measured on the basis of the American Petroleum Institute (API) standards (API Recommended Practice 13B-1 Field Testing Water-based Drilling Fluids). The filtration tests at room temperature were conducted under a pressure of 0.69 MPa by using N_2 gas charge. Filtration volume (denoted as FL_{API}) was recorded after 7.5 min. For filtration testing at high-temperature high-pressure condition (FL_{HTHP}), EF-WBDFs were added to a GGS-71 HTHP loss meter. The fluid loss

Table 3
Main additives in EF-WBDF.

Material	Short name	Type	Main component	Function
Bentonite	Bentonite	Natural material	Montmorillonite	Filtration, tackifying
Gelatin ammonium salt	GT	Modified natural material	Gelatin modified by glycidyl trimethyl ammonium chloride	Inhibition
Poly-l-arginine	PArg	Synthesized material	Synthesized by l-arginine	Inhibition
Starch composite	SPL	Modified natural material	Crosslinked composite of starch, lignosulfonate, polyphenol	Filtration
White asphalt	NFA-25	Modified natural material	Composite of cellulose, resin, walnut shell flour, surfactant	Filtration, plugging
Polyanionic cellulose	LV-PAC	Modified natural material	Cellulose	Tackifying, filtration
Laponite	Laponite	Synthesized material	Laponite	Rheological modification
Fine calcium carbonate	$CaCO_3$	Inorganic material	$CaCO_3$	Plugging

was determined at a set high temperature and 3.5 MPa for 30 min, and the FL_{HTHP} was twice of the recorded result.

Inhibition property. The swelling height of compacted bentonite immersed in different filtrates obtained from filtration tests was determined using a CPZ-2 dual-channel linear swell meter. For each measurement, bentonite (5 g) was compressed into a cylindrical device under 10 MPa pressure for 5 min. Then, 15 mL of sample solution was added to immerse the bentonite pellet. The height of the pellet was recorded every 30 s through the transducer for 24 h.

In shale recovery test, a total of 20 g of shale cuttings with 6–10 mesh size as well as 350 mL EF-WBDFs were added to a jar together. The mixture was hot-rolled at 150 °C for 16 h. After cooling, the remaining cuttings were screened using a 40-mesh sieve and the cuttings retained on the sieve were dried at 75 °C until a constant weight. The recovery percentage was calculated by the following equation:

$$R = \frac{m_2}{m_1} \times 100\% \tag{1}$$

where R is the recovery percentage, %; m_1 is the mass of shale cuttings before hot rolling, g; and m_2 is the mass of shale cuttings after hot rolling, g.

Environmental factor. Three tests were conducted to access the environmental influence of EF-WBDF. Firstly, the lethal concentration of 50% (EC_{50}) was measured through luminescent bacterial method. Secondly, the biodegradability of EF-WBDF was tested by BOD/COD method. Thirdly, wheat was planted in different WBDFs to compare their effect on the growth of the wheat. The growth situation of different samples was photographed and recorded in a constant temperature incubator. In this test, two fluids were used as the base solutions for planting, including nutrient soil, and soil contaminated by hot-rolled EF-WBDF. The concentration of WBDF in the contaminated soil was 50%. Furthermore, to verify the environmental characteristics of EF-WBDF, a sulfonated polymer-based WBDF (donated as SP-WBDF) was used to compare with EF-WBDF. The formula of SP-WBDF was similar with EF-WBDF, which included 2% bentonite, 0.4% potassium polyacrylate (KPAM), 0.5% polyamine (PA), 2.15% amphoteric polymer, 3% sulfonated lignite resin (SPNH), 4% NFA-25, 3% $CaCO_3$ and barite.

3. Results and discussion

3.1. Optimization of the additives

To establish an outstanding EF-WBDF, the selection of additives and the optimization of dosage were conducted based on a basic formula. The basic formula was as follows: 2% bentonite dispersion + 1.5% laponite + 0.5% LV-PAC + 0.5% PArg + SPL + 4% white asphalt + 3% $CaCO_3$ + barite. The density of the basic WBDF was 1.35 g/cm^3. The above listed order was also the order of adding additives during the preparation process.

First of all, SPL with different concentrations was added as the main fluid loss additive. Generally, the viscosity of a drilling fluid is located in 10–100 mPa s while the stress is between 0 and 20 Pa, which can be adjusted according to the drilling requirements (Wu et al., 2002). As shown in Table 4, when SPL was not cooperated, the EF-WBDF performed an AV value of 29 mPa s, low YP value of 2 Pa, and high FL_{HTHP} of 27.6 mL. Correspondingly, AV, PV, and YP raised after adding SPL as a fluid loss agent. In detail, AV, PV, YP and stress significantly increased as increasing the dosage of SPL at room temperature, but increasing the dosage of SPL had little effect on the variations of AV, PV, and YP at high temperatures. High temperatures were beneficial for the dissolution of SPL and white asphalt, thereby exerting their stable functions. The AV of EF-WBDFs maintained in the range of 37–40 mPa s while the YP kept 4–6 Pa. Besides, FL_{HTHP} gradually decreased as increasing the dosage of SPL. When the dosage of SPL exceeded 3.0%, FL_{HTHP} varied slightly and was below 13.6 mL, ensuring the safe and effective drilling. The results demonstrated that the addition of 3.0% SPL was enough for the filtration control.

Secondly, laponite was used as a rheology modifier and its dosage was determined based on the basic formula containing 3.0% SPL. Table 5 gave the variations of the properties of EF-WBDF with different dosages of laponite. It could be obviously seen that the YP was improved gradually with increasing the dosage of laponite. When the amounts of laponite were 1.5% and 1.75%, the values of YP after aging at 150 °C were the same with a value of 4.5 Pa. However, the static stress differential between τ_{10s} and τ_{10min} was only 1.0 Pa at a laponite dosage of 1.5%, while that achieved 3.0 Pa at a laponite dosage of 1.75%. As exhibited in Table 6, based on the force balance (Hong and Li, 1995) and "Stokes settlement formula", the required static stress for EF-WBDF was calculated and achieved approximately 1.0 Pa. Meanwhile, the settling rate of barite particles was about 2.07×10^{-4} cm/s. In practice, experience showed that the stress that could effectively suspend barite was generally at least 1.4 Pa (Yan, 2014). Thus, the stress of EF-WBDF containing laponite was enough. What's more, shear thinning behavior and thixotropic behavior were also required for quality drilling fluids, which were closely related to barite suspension performance and cutting transport performance. The static stress differential between τ_{10s} and τ_{10min} enhanced with increasing the dosage of laponite, demonstrating the improved thixotropic behavior. Moreover, with increasing the dosage of laponite, the value of YP/PV increased, showing an enhanced shear thinning behavior. The low value of flow index (n) in Table 10 also proved the shear thinning behavior. Thus, EF-WBDF performed satisfactory rheological performance. Considering the thixotropy and static stress, a laponite dosage of 1.75% was appropriate for EF-WBDFs. Meanwhile, the other parameters including AV, PV, YP, and FL_{HTHP} were also satisfactory.

Thirdly, different inhibitors were also tested in EF-WBDF. In the basic formula, the dosages of SPL and laponite were set at 3.0% and 1.75%, respectively. GT, PArg, and their mixture were used as

Table 4
The properties of EF-WBDF with different concentrations of SPL.

SPL, %	Temperature, °C	AV, mPa s	PV, mPa s	YP, Pa	τ_{10s}/τ_{10min}, Pa/Pa	FL_{HTHP}, mL
0	150	29	27	2	3.0/4.0	27.6
1.5		38	34	4	2.5/4.0	16.8
3.0		38	32	6	4.5/6.5	13.6
4.0		39.5	35	4.5	5.0/7.0	12.4
5.0		37	32	5	5.0/8.0	12.0

Table 5
The properties of the environmentally friendly WBDF with different dosages of laponite.

Laponite dosage, %	Temperature, °C	AV, mPa s	PV, mPa s	YP, Pa	YP/PV Pa/(mPa s)	τ_{10s}/τ_{10min}, Pa/Pa	FL_{HTHP}, mL
0	150	32.0	29.5	2.5	0.08	3.0/4.0	15.1
1.5		37.5	33.0	4.5	0.14	5.0/6.0	14.8
1.75		37.5	33.0	4.5	0.14	6.0/9.0	13.5
2.0		37.0	28.0	9.0	0.32	7.0/12.0	12.4

Table 6
The required stress for EF-WBDF.

Parameter	Formula	Result	Note
Static stress τ_s, Pa	$\tau_s = \frac{d(\rho_s - \rho)g}{6}$	0.95	The barite particle was taken as the example: ρ_s is the barite density, $\rho_s = 4.2$ g/cm^3; ρ is the drilling fluid density, $\rho = 1.35$ g/cm^3; d is the particle median diameter, $d_{0.5} = 7.0$ μm; η is the viscosity, $\eta = 37.5$ mPa s.
Corrected static stress τ_s, Pa	$\tau_s = \frac{4}{\pi}\cdot\frac{d(\rho_s - \rho)}{6}$	1.21	
Settling rate v, cm/s	$v = \frac{d^2}{18\eta}(\rho_s - \rho)g$	2.07×10^{-4}	

inhibitors. Table 7 demonstrated that EF-WBDF added with 0.5% PArg had a shale recovery of 81.55% after aging at 150 °C. Generally, the shale recovery in high-performance WBDFs could reach more than 90%. Then, the dosage of PArg was increased to 1.0%, unfortunately resulting in a similar shale recovery value and poor fluid loss. The deterioration of filtration property might be due to excessive inhibitors inhibiting the clay hydration and destroying the colloidal stability, which could be proved by the significantly reduced viscosity. To overcome the inadequacy of inhibition, PArg and GT were employed together in EF-WBDF. Adding 0.5% PArg and 0.4% GT increased the shale recovery to 95.55%, suggesting efficient inhibition. The rheological and filtration properties also kept stable. It was worth noting that stress increased after adding PArg and GT, compared with that when PArg was used alone. It was inferred that GT and clay formed network structure, which had a resistance to flow under shear force. Consequently, the combination of PArg and GT exerted prominent inhibition and excellent compatibility.

According to the optimization results, the formula of EF-WBDF was determined as follows: clay (2% bentonite, and 1.75% laponite), tackifiers (0.5% PAC-LV), shale inhibitors (0.5% PArg, and 0.4% GT), fluid loss additives (3% SPL), plugging agents (4% NFA-25, and 3% $CaCO_3$), and weighting agent. Furthermore, the densities of the fluids were adjusted and properties at 150 °C were shown in Table 8. The viscosity and stress increased with increasing the density. On the whole, various properties of EF-WBDFs with different densities maintained stable. However, when the density reached 2.0 g/cm^3, the viscosity of EF-WBDF before hot rolling was too high and the fluidity of drilling fluid was not good. The upper limit of the density was 1.7 g/cm^3, and the solid tolerance of this drilling fluid system was relatively low. According to actual requirements and downhole conditions, the EF-WBDF formula could be adjusted flexibly.

Furthermore, according to previous literatures (Hu et. 2011; Li J. et al., 2014; Sun et al., 2018), the properties of established EF-WBDF with a fixed density of 1.35 g/cm^3, including high-temperature resistance, salt resistance, inhibition, and especially environmental factor, were tested systematically below.

3.2. Thermal test on filtration and rheology of EF-WBDF

As exhibited in Table 9, the filtration and rheological properties of EF-WBDF at different temperatures were evaluated. After aging at 120–160 °C, the AV of EF-WBDFs lied in the range of 41–56 mPa s, while the YP fluctuated in the range of 9–17 Pa, displaying stable viscosity and high stress. However, after aging at 180 °C a sharp decrease in viscosity and an increase in fluid loss volume were observed, demonstrating the complete invalidation of the properties. When hot rolling at 150 °C, the FL_{HTHP} of EF-WBDF was 14.2 mL, which could also meet the drilling requirements in easily collapsed strata. After the temperature raised to 160 °C, the

Table 7
The properties of EF-WBDF with different kinds and dosages of inhibitors.

Inhibitor	Temperature, °C	AV, mPa s	PV, mPa s	YP, Pa	τ_{10s}/τ_{10min}, Pa/Pa	FL_{HTHP}, mL	Recovery, %
0.5% PArg	150	37.5	33	4.5	6.0/9.0	13.5	81.55
1.0% PArg		19	19	0	0.5/0.5	30.6	85.25
0.5% PArg + 0.4% GT		41	32	9	5.5/11	14.2	95.55

Table 8

The properties of EF-WBDFs with different densities.

Density, g/cm^3	AV, mPa s	PV, mPa s	YP, Pa	τ_{10s}/τ_{10min}, Pa/Pa	FL$_{HTHP}$, mL
1.07 (without weighting)	34	27	7	5.0/9.0	14.6
1.35	41	32	9	5.5/11.0	14.2
1.55	48	34	14	7.0/13.0	13.8
1.70	59.5	48	11.5	6.0/10.0	14.0

Table 9

The properties of the environmentally friendly WBDF before and after hot rolling.

Temperature, °C	AV, mPa s	PV, mPa s	YP, Pa	τ_{10s}/τ_{10min}, Pa/Pa	FL$_{HTHP}$, mL
25	60.5	23	37.5	16/23	4.0
120	56.5	39	17.5	9/15.5	8.4
150	41.0	32	9.0	5.5/11	14.2
160	43.5	32	11.5	6/10	17.5
170	15.5	14	1.5	1.5/2	64.0

Table 10

Rheological equations fitting at different temperatures for EF-WBDFs.

Rheological model	Room temperature	120 °C	150 °C	160 °C
Bingham ($\tau = \tau_0 + \mu_{pV}\gamma$)	$\tau = 25.9746 + 0.0390\gamma$ $R^2 = 0.9202$	$\tau = 12.3769 + 0.0463\gamma$ $R^2 = 0.9849$	$\tau = 6.8296 + 0.0352\gamma$ $R^2 = 0.9938$	$\tau = 6.8239 + 0.0384\gamma$ $R^2 = 0.9862$
Power law ($\tau = K\gamma^n$)	$\tau = 13.2687\gamma^{0.2137}$ $R^2 = 0.9675$	$\tau = 2.5240\gamma^{0.4429}$ $R^2 = 0.9549$	$\tau = 0.7990\gamma^{0.5652}$ $R^2 = 0.9618$	$\tau = 0.8087\gamma^{0.5742}$ $R^2 = 0.9790$
Casson ($\tau^{1/2} = \tau_c^{1/2} + \eta_\infty^{1/2}\gamma^{1/2}$)	/	$\tau^{1/2} = 7.8286^{1/2} + 0.0223^{1/2}\gamma^{1/2}$ $R^2 = 0.9994$	$\tau^{1/2} = 3.7858^{1/2} + 0.0196^{1/2}\gamma^{1/2}$ $R^2 = 0.9974$	$\tau^{1/2} = 3.7185^{1/2} + 0.0220^{1/2}\gamma^{1/2}$ $R^2 = 0.9995$
Herschel–Bulkley ($\tau = \tau_y + K\gamma^n$)	$\tau = 18.2740 + 1.4857\gamma^{0.4878}$ $R^2 = 0.9989$	$\tau = 9.5637 + 0.2532\gamma^{0.7573}$ $R^2 = 0.9999$	$\tau = 5.5801 + 0.1053\gamma^{0.8430}$ $R^2 = 0.9996$	$\tau = 4.6535 + 0.1912\gamma^{0.7706}$ $R^2 = 0.9997$

FL$_{HTHP}$ increased to 17.5 mL, which was relatively higher. According to relative industry standard (SY/T 7377-2017, Petroleum and Natural Gas Industry Standards of the People's Republic of China - specification of drilling fluid programs), the FL$_{HTHP}$ of WBDFs should be below 20 mL for non-reservoir well sections with good wellbore stability, while that should be less than 15 mL when drilling in reservoir well sections or formations with wellbore instability. Thus, EF-WBDF could resist 150 °C. To ensure safe and effective drilling, this fluid was not suggested to be used in reservoir well sections or formations with wellbore instability, especially when the temperature was higher than 150 °C. In addition, compared with that at room temperature, aging at high temperatures obviously decreased the YP. The initial gel strength (τ_{10s}) and final gel strength (τ_{10min}) also showed a similar variation tendency with YP. Generally, a space truss structure existed in the thixotropic system. Due to the small size of laponite nanoparticles, the gel strength of the laponite suspension was strong, which could offer large stress for drilling fluids. However, in the drilling fluid system, the effect of enhancing stress decreased with increasing temperature. This might be because that the adsorption of laponite onto other additives at high temperatures increased and the nano-size effect was reduced. From the stress value, it was refereed that the gel structure between bentonite, polymer and laponite in EF-WBDF was affected to some extent by high temperatures. However, the YP and gel strength, which were far larger than 1.4 Pa could still kept good level to transport and suspend the cuttings in downhole.

To further verify the rheological properties of EF-WBDFs, the plots of shear stress versus shear rate for EF-WBDFs at different temperatures were shown in Fig. 3. Moreover, the Bingham plastic, power law, Herschel–Bulkey, and Casson models were applied to fit their shear stress–shear rate curves (Nasiri and Ashrafizadeh, 2010), and the corresponding fit parameters were summarized in Table 10. In comparison with the Bingham plastic, power law, and Casson models, the Herschel–Bulkey model provided a better fit for the shear stress–shear rate curves, which was evidenced by the higher values of R^2.

3.3. Salt contamination test

In some areas, the drilling fluids usually met complex formations containing salt layer. High salt concentration would deteriorate the hydration of bentonite and cause serious flocculation adversely, leading to destabilized dispersion. Consequently, the permeability of the formed filter cake increased and induced large fluid loss. Therefore, the salt-resistance performance should be conducted for WBDFs. As displayed in Table 11, the addition of 7.5% NaCl or 0.5% $CaCl_2$ hadn't undermine the colloidal stability of EF-WBDFs. In comparison, salt contaminate increased the viscosity and stress to some extent, which might due to the weak flocculation structure caused by electrolytes. The result proved that EF-WBDF could exhibit stable performance under the contamination of salts.

3.4. Inhibitive evaluation

Shale inhibition was quite vital for drilling fluids, especially when WBDFs were used in shale formations. To meet the requirements of drilling engineering, high-performance WBDFs must contain effective inhibitors. As displayed in Fig. 4, at 150 °C, the shale cuttings used in this study performed a low recovery of 32.95% in pure water, indicating the easy hydration and dispersion. In contrast, EF-WBDF and SP-WBDF increased the recovery to 95.55% and 93.15%, respectively. These two fluids showed alike outstanding inhibition on the dispersion of shale cuttings. However, EF-WBDF exhibited a slightly higher recovery than SP-WBDF. The used inhibitor for SP-WBDF was polyamine, which was

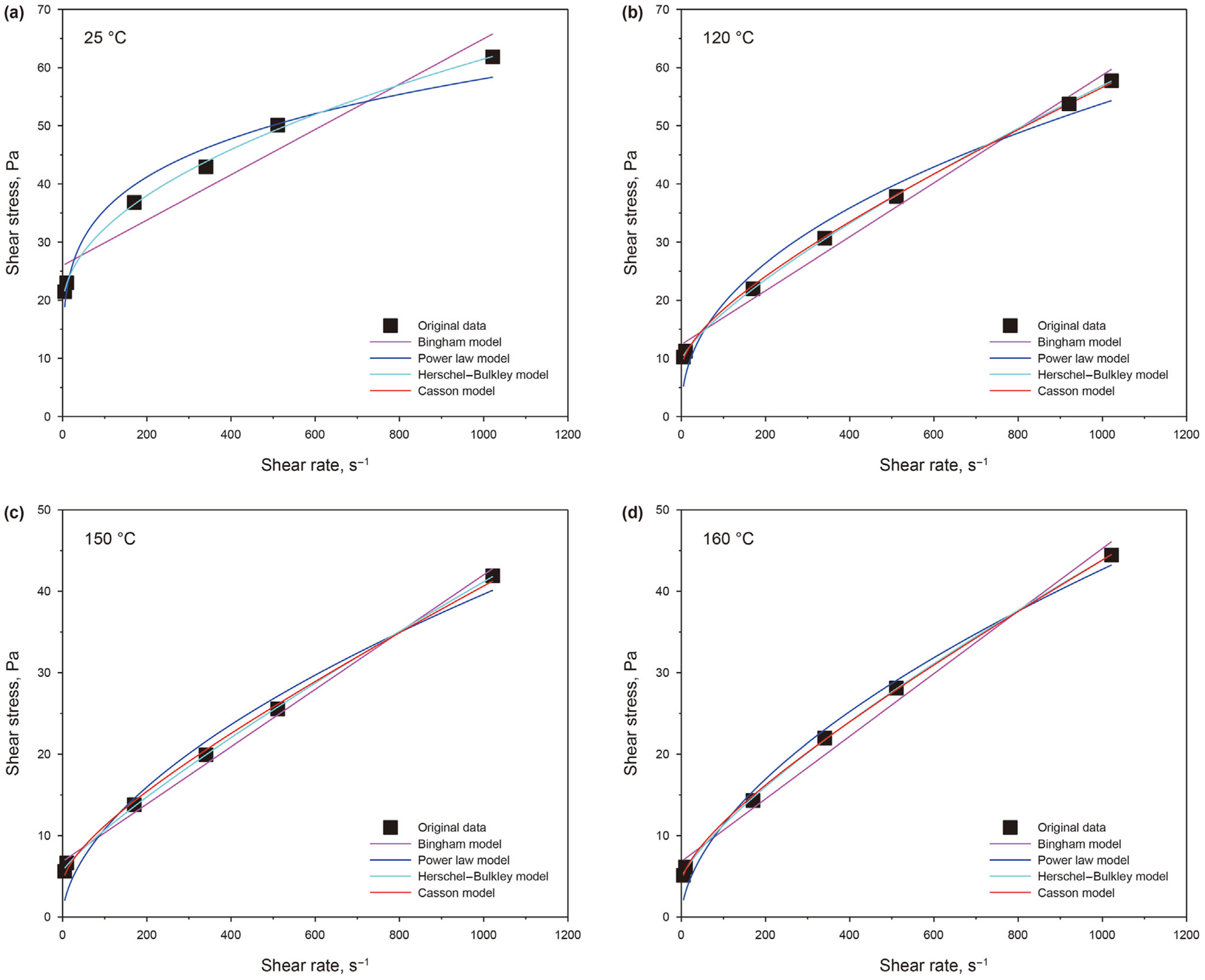

Fig. 3. Rheological curves fitting at different temperatures for EF-WBDFs.

Table 11
The anti-salt performance of EF-WBDF.

Salt content	Temperature, °C	AV, mPa s	PV, mPa s	YP, Pa	FI_{HTHP}, mL
0	25	60.5	23	37.5	4.0
	150	41.0	32	9.0	14.2
7.5% NaCl	25	62.0	30	32.0	4.8
	150	52.0	28	24.0	16.2
0.5% $CaCl_2$	25	66.0	34	32.0	5.2
	150	43.0	21	22.0	17.4

commonly used and considered as an efficient shale inhibitor in WBDFs (Patel, 2009). The result indicated that the inhibition of the combination of PArg and GT was better than polyamine. Meanwhile, the linear swelling height of bentonite pellet in filtrate obtained from HTHP filtration tests was also determined (as shown in Fig. 5). Compared with the swelling height of 6.95 mm in pure water, the swelling height in filtrate obtained from EF-WBDF and SP-WBDF at 150 °C was reduced to 2.44 and 3.41 mm, respectively, displaying efficient inhibition on swelling of bentonite. These results from linear swelling tests and shale recovery tests were consistent and proved the excellent inhibition of EF-WBDF.

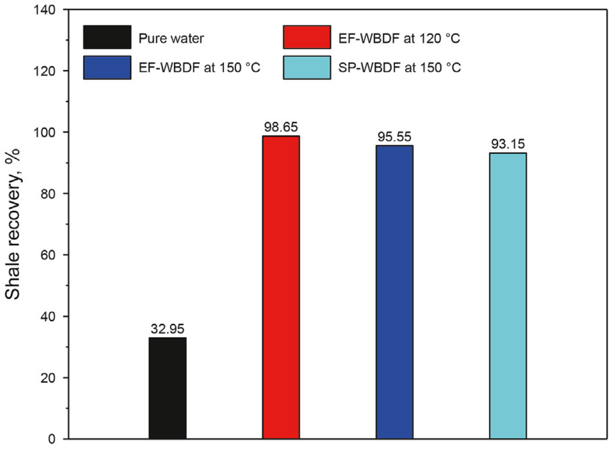

Fig. 4. The shale recovery of different WBDFs at different temperatures.

3.5. Environmentally friendly test

Finally, the environmental performance of EF-WBDF was comprehensively evaluated through four methods, including

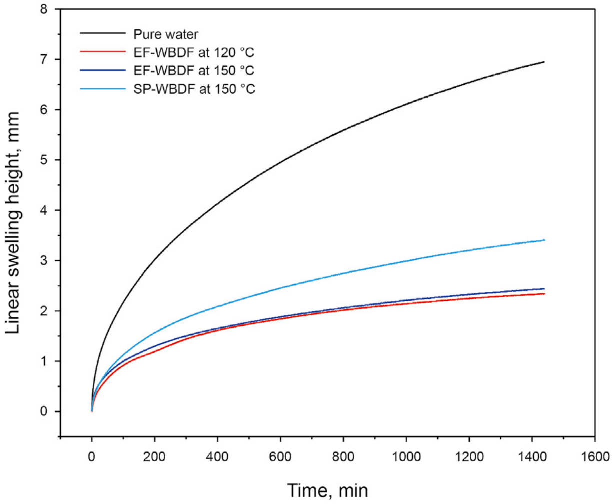

Fig. 5. The linear swelling height of bentonite in filtrates from different WBDFs.

Table 12
The biotoxicity of EF-WBDF.

Sample	EC_{50}, mg L^{-1}	Biotoxicity
EF-WBDFs	51200	Non-toxic

Table 13
The biodegradability of EF-WBDF.

Sample	BOD, mg L^{-1}	COD, mg L^{-1}	BOD/COD	Biodegradability
EF-WBDF	13600	28600	47.55%	Easy-biodegradable

biological toxicity, biodegradability, plant cultivation test, and heavy metal content measurement.

3.5.1. Biological toxicity analysis

The EC_{50} of EF-WBDF was tested by the luminescent bacteria method according to relative industry standard (Q/SY 111–2007, Garding and determination of the biotoxicity of chemicals and drilling fluids—Luminescent bacteria test) (Ma et al., 2014), and the results are listed in Table 12. The EC_{50} of this system was as high as 51200 mg/L. According to the biological toxicity grade classification standard, when the EC_{50} was larger than 25000 mg/L, WBDF was nontoxic. If the EC_{50} exceeded 30000 mg/L, the emission standard was achieved (Yang et al., 2017). Therefore, EF-WBDF was totally considered non-toxic.

Table 14
Heavy metal content in EF-WBDF.

Test item	Standard requirement, mg/kg	Test result
Hg	15	N.D.
Cd	20	N.D.
As	75	N.D.
Cr	1000	N.D.
Pb	1000	N.D.
Ni	/	N.D.

Note: N.D. indicates that the result was below the detection limit.

3.5.2. Biodegradability analysis

The biodegradability of EF-WBDF was measured by the BOD/COD ratio method. Generally, when the value of BOD/COD was greater than 25%, it was easy to degrade (Qu et al., 2020). As shown in Table 13, EF-WBDF performed a high BOD/COD ratio of 47.55%, displaying an easily biodegradable property. This was mainly because that the main additives of EF-WBDF were based on natural materials or their derivatives. These natural components were easily biodegradable in natural environment.

3.5.3. Plant cultivation analysis

Wheats were cultivated in natural soil dispersion, EF-WBDF and SP-WBDF under the same conditions, respectively. As exhibited in Fig. 6, the wheats grew healthily and were luxuriant in soil after 15 days. In comparison, the wheat planted in soil contaminated by EF-WBDF grew slightly weaker, while that in soil contaminated by SP-WBDF did not grow healthily and only a small part of the wheat survived. Moreover, the solid in SP-WBDF occurred crusting phenomenon, which might due to the added synthetic polymers of high molecular weight. These results further demonstrated that this EF-WBDF had little effect on the growth of wheat, and was quite environmentally friendly.

3.5.4. Heavy metal content analysis

The contents of different heavy metals including Hg, Cd, As, Cr, Pb, and Ni in EF-WBDF were tested through inductively coupled plasma atomic emission spectrometer (ICP-AES). As shown in Table 14, the EF-WBDF did not contain these heavy metals. According to industry standard (SY/T 6787-2010: Technical requirements of environmental protection for water-soluble oilfield chemicals), the highest contents of Hg, Cd, As, Cr, and Pb were 15, 20, 75, 1000, and 1000 mg/kg, respectively. Therefore, the content of metals in EF-WBDF met the criterion.

3.6. Mechanism analysis

Compared with other environmentally friendly drilling fluids, all of the additives used in this EF-WBDF were natural materials or

Fig. 6. The growth of wheat in soil (**a**), soil contaminated by EF-WBDF (**b**), and SP-WBDF (**c**) after 15 days.

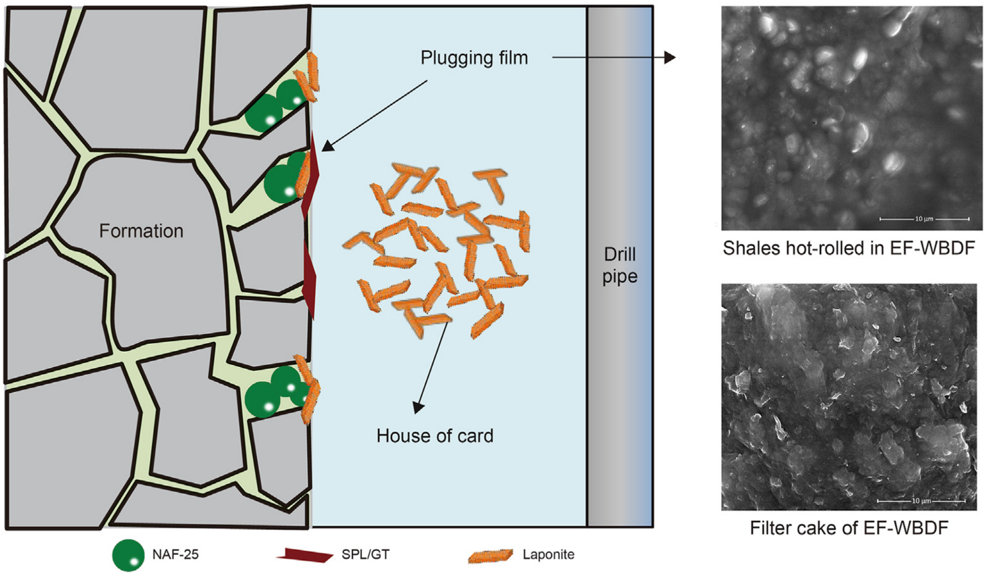

Fig. 7. The interaction behavior of different additives in EF-WBDF.

modified natural materials. Although laponite nanoparticles were evaluated as efficient agents in other studies, their applications in drilling fluid system at high temperatures were seldom. This research proved that laponite nanoparticles and natural materials performed synergistic effect and satisfactory properties in EF-WBDF.

As illustrated in Fig. 7, laponite nanoparticles easily hydrated and formed a "house of cards" structure. Moreover, due to the small size of laponite nanoparticles, the gel strength of the laponite suspension was strong, which could offer large stress for drilling fluids. NAF-25, as a polymer composite, could bridge on the borehole wall and plug the pores and fractures. SPL exhibited a colloidal substance in high temperature environment and could adsorb onto the wall and plug the pores, while GT could also encapsulate the shales. Under the synergistic effect of these additives, a dense filter cake and smooth cutting surface were formed. A "polymer film" was observed, which could efficiently stop the invasion of water and stabilize the borehole.

4. Conclusions

Using laponite nanoparticles and environmentally friendly additives based on natural materials as the main additives, an EF-WBDF was optimized and built. Various properties of EF-WBDF were evaluated. Several findings could be drawn as follows:

(1) All of the additives used in this EF-WBDF were natural materials or modified natural materials.
(2) The EF-WBDF embodied effective thermal stability up to 150 °C, showing satisfactory filtration, rheological, and inhibitive properties. The optimal rheological model for EF-WBDF was the Herschel–Bulkley model.
(3) This EF-WBDF had an EC_{50} value of 51200 mg/L and a BOD/COD ratio of 47.55%, performing non-toxic and easily biodegradable characteristics. There were no heavy metals in the EF-WBDF. The wheat cultivated in the EF-WBDF could grow healthily.
(4) Laponite nanoparticles and natural materials used in this EF-WBDF performed synergistic effect. A polymer film could be formed on the wall for plugging and stabilizing the borehole.

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