

Modified Corn Starch as an Environmentally Friendly Rheology Enhancer and Fluid Loss Reducer for Water-Based Drilling Mud

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Summary

Drilling fluid rheology and fluid loss property are fundamental parameters that dictate the effectiveness and easiness of a drilling operation. Maintaining these parameters under high temperatures is technically challenging and has been an exciting research area for the drilling industry. Nonetheless, the use of drilling mud additives, particularly synthetic polymers, threaten ecological environments. Herein, modified corn starch (MCS) was synthesized, characterized, and investigated as an environmentally friendly rheology enhancer and filtration loss controlling agent for water-based mud (WBM) at high temperatures. The experimental results indicated that MCS exhibits better performance in improving rheological properties and fluid loss controlling ability for WBM than the commonly used mud additives. With the addition of an optimal concentration (0.3 wt%), MCS improved the rheology and fluid loss behavior of WBM formulation at harsh aging temperature (220°C) by practically 4 times and 1.7 times, respectively. The MCS was revealed to perform superbly over polyanionic cellulose (PAC) addition at all investigated temperatures. The better performance of the MCS was ascribed to the improved entanglements in the mud system owing to the additional hydroxyl (OH) groups. Besides, the Herschel-Bulkley model was found to be a constitutive model that described the rheological properties of the investigated muds satisfactorily. Moreover, the MCS was found to exhibit acceptable biodegradability properties.

Introduction

Drilling oil and gas wellbores is the most expensive step in recovering oil and natural gas from underground petroleum reservoirs (Sarah and Ebdisseghabofa 2021). Drilling fluid is a key requirement during the drilling operations and is vital in ensuring the safety and productivity of the oil and gas wells (Okon et al. 2015; Kumar et al. 2018). Drilling fluid, which is also called drilling mud, is a particularly designed fluid that is circulated in the borehole to make the drilling activities much easier and free from obstructions (Sedaghatzadeh et al. 2012; Meng et al. 2012; Nasser et al. 2013). It is often used when drilling crude oil and natural gas boreholes, and simpler boreholes for water wells. Drilling mud is normally formulated to encounter certain properties that enable the fluid to execute its basic functions during the drilling operations (Novrianti et al. 2021; Amani et al. 2012; Rana et al. 2019). A properly formulated drilling mud should be able to carry out the following functions: control formation pressures, remove cuttings from the wellbore, seal permeable formations, cool and lubricate the rotating bit and drillstring, transmit hydraulic horsepower to the rotating bit, suspend and release cuttings, maintain wellbore stability, minimize formation damage, control subsurface pressures, facilitate the collection of formation data, control corrosion, and facilitate cementing and completion (Quintero 2002; Samaei and Tahmasbi 2007; Okon et al. 2014; Kafashi et al. 2017; Vryzas and Kelessidis 2017; Saboori et al. 2018; Maiti et al. 2019; Jinasena and Sharma 2020).

The drilling mud is a complex mixture that may consist of solids, liquids, gases, and other chemicals, with the base fluid or continuous phase determining the type of drilling mud (Elkatatny et al. 2012; Dias et al. 2015; Dankwa et al. 2018). Depending on the type of base fluids or continuous phases (water, oil, pseudo-oil, gas, or foam), drilling muds are categorized into WBMs, oil-based muds, synthetic or pseudo oil-based muds, gas-drilling muds, and foam-drilling muds (Agin et al. 2018; Behnamanhar et al. 2014; Dankwa et al. 2018; Onuh et al. 2020). Each type of drilling mud is designed specifically to suit a particular reservoir formation characteristics; there is no one type of drilling mud that fits all reservoir formations. In contrast to other drilling muds, WBM has received special attention because it is cheap and environmentally friendly (Kania et al. 2015; Aughenbaugh et al. 2016; Dankwa et al. 2018; Tiwari et al. 2020; Pinheiro et al. 2021). Besides, the WBMs favorably demonstrate adequate rheological and filtration control properties, good cooling and cutting removal ability, and simplicity to formulate and use it in the oilfields (Warren and Stewart 2003; Li et al. 2015, 2016).

WBMs are mainly composed of water as the base fluid, clay minerals, and a variety of chemical additives similar to other drilling muds (Amanullah and Yu 2005; Tchameni et al. 2019). The continuous phase acts as a carrier for mud additives, which are the final determinants of the mud's properties (Onuh et al. 2017). Drilling mud additives are added to drilling muds during formulation to control the following parameters: viscosity, fluid loss, weight or density, pH, and alkalinity of the drilling muds (Adesina et al. 2012; Akpan et al. 2020). Viscosifying additives, such as clay minerals (bentonite, smectite, sepiolite, and kaolinite; however, bentonite is the most commonly used) and polymers (xanthan gum, guar gum, starch, polyacrylates/polyacrylamides, carboxy-methyl-cellulose, hydroxy-ethyl-cellulose, and PAC), are added to the drilling mud to thicken it and control the fluid loss (Omotoma et al. 2015; Li et al. 2019; Afolabi and Yusuf 2020; Al-Hameedi et al. 2019; Karagüzel et al. 2010; Abdo and Haneef 2013; Ghazali et al. 2015; Vipulanandan and Mohammed 2015; Zarouri

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and Benmounah 2016; Onuh et al. 2017; Sarah and Ebidiseghabofa 2021). Weighting additives (BaSO_4 , CaCO_3 , CaCl_2 , and K_2CO_3) are added to the mud to increase the density of the drilling mud and help control the subsurface pressures (Fink 2015). Deflocculating additives (lignosulfonates, lignites, and phosphates) are added to the mud to disperse particles in the drilling mud and reduce the circulating pressure problems in the borehole (Zhang and Yin 1999; Al-Hameedi et al. 2019; Fink 2012). The pH and alkalinity control additives [NaOH , KOH , Na_2CO_3 , NaHCO_3 , K_2CO_3 , $\text{Ca}(\text{OH})_2$, and $\text{Mg}(\text{OH})_2$] are added to the drilling mud to increase the pH of the drilling mud and reduce the corrosion problems (Amanullah and Yu 2005; Fink 2012). Moreover, KCl and $(\text{NH}_4)_2\text{PO}_4$ are added to the drilling mud as shale inhibitors, but they also stabilize the clay particles and prevent the swelling of the clay formations, whereas NH_4HSO_3 , $(\text{NH}_4)_2\text{SO}_3$, ZnCO_3 , NaNO_3 , and Na_2SO_3 are added as corrosion inhibitors (Al-Hameedi et al. 2019, 2020; Omotioma et al. 2015; Li et al. 2019; Afolabi and Yusuf 2020; Sarah and Ebidiseghabofa 2021; Zhou et al. 2021; Fink 2012; Ghazali et al. 2015; Zarouri and Benmounah 2016). Each one of these additives performs a specific function; thus, the total formulation of the drilling mud is very important, both from a technical and an economic point of view (Fink 2012). Basically, the drilling mud additives improve the mud properties such as rheological properties, wellbore stability under various temperatures and pressures, electrolyte compatibility, and prevent fluid loss (Li et al. 2019).

Generally, mud rheology and fluid loss behavior are the most important parameters to control during the drilling operations (Li et al. 2015; Ivan et al. 2002; Vryzas and Kelessidis 2017). Concerning drilling muds, rheology is the study of the flow of fluids and their deformation under the conditions in which they respond (Alemdar et al. 1999). The rheological properties are very useful in describing the flow behaviors of a drilling mud under different flow conditions (Hajjabadi et al. 2020; Dokhani et al. 2020). The most commonly used rheological parameters to study the flow characteristics of a drilling mud include gel strengths, plastic viscosity (PV), yield point (YP), apparent viscosity (AV), and filter cake (Dankwa et al. 2018). These rheological parameters help to describe the flow behavior of the drilling mud and its capacity to execute the desired functions. Both rheological and fluid loss properties of drilling fluids are usually witnessed to vary extremely owing to variation in operating conditions, such as pressure and temperature (Amanullah and Yu 2005; Wenjun et al. 2014; Vryzas and Kelessidis 2017; Mohammed 2017; Vryzas et al. 2017). This variation has elicited and validated the concept of applying rheology enhancers (Kafashi et al. 2017; Li et al. 2015) and fluid loss reducers (Amanullah and Yu 2005; Saboori et al. 2018) to generate drilling muds with excellent performance.

Polymer-based materials have been widely studied and used as rheology enhancers and fluid loss control agents (Luqman Hasan et al. 2018; Hamad et al. 2020). Synthetic polymers (polyacrylates, polyacrylamides, polyvinyl alcohol, ethylene oxide polymers, and polyalkylene glycols), semisynthetic polymers (hydroxy-ethyl-cellulose, carboxy-methyl-cellulose, and PAC), and natural polymers (xanthan gum, guar gum, starch, cellulose, lignin, chitosan, gelatin, protein, and natural rubber) have been widely applied in the oil fields as rheology enhancers and filtration loss controlling agents in drilling muds (Fink 2015; Oseh et al. 2019, 2020; Coussot et al. 2006; Baba Hamed and Belhadri 2009; Ghazali et al. 2015; Zarouri and Benmounah 2016; Li et al. 2019; Zhou et al. 2021; Al-Hameedi et al. 2020; Sarah and Ebidiseghabofa 2021). Synthetic polymers have been widely used over the decades as drilling mud additives owing to their promising rheological behaviors (Fink 2015; Safi et al. 2015). Unfortunately, some various shortcomings, such as thermal instability, salt intolerance, cost-ineffective, and environmental unfriendliness of synthetic polymers, restricted their applications as drilling mud additives (Al-arfaj et al. 2011; Zhu et al. 2018; Tchameni et al. 2019). Environmentalists have raised concerns regarding their production methods, usages, and discharges (Amanullah and Yu 2005; Saboori et al. 2018; Xu et al. 2019). In recent years, there is an increase in global concern for the protection of the environment from the catastrophic effect of drilling mud additives (Elkatatny 2019). Thus, there is a need for developing environmentally friendly, thermally stable, and cost-effective drilling mud additives that can help to control the rheological properties and improve their performance with the least effects on the environment.

As an attempt to comply with environmental requirements and to moderate costs for drilling operations, naturally occurring polymers are widely studied as environmentally friendly and cost-effective drilling mud additives (Ekeinde et al. 2018; Khalid et al. 2019; Li et al. 2019; Zhou et al. 2021; Al-Hameedi et al. 2020). Both field applications and experimental studies have ascertained the promising performances of naturally occurring polymers from local materials, such as corn/corncoobs, acacia gum, cassava, potatoes, banana peels, mango seeds, rice husks, sugarcane bagasse, mandarin peels, soybeans, coconut shells, and cashew nut shells as drilling mud additives (Taiwo et al. 2011; Olatunde et al. 2012; Omotioma et al. 2015; Talukdar et al. 2018; Li et al. 2019; Afolabi and Yusuf 2020; Abro et al. 2020; Sulaimon et al. 2020; Sarah and Ebidiseghabofa 2021; Al-Hameedi et al. 2019, 2020; Ghazali et al. 2015; Zarouri and Benmounah 2016; Zhou et al. 2021). Unfortunately, naturally occurring polymers decompose at moderate temperatures, limiting their applications as drilling mud additives in high-temperature environments (Thomas 1982; Amanullah and Yu 2005; Nasiri et al. 2018).

Currently, semisynthetic polymers that are obtained through modifications of naturally occurring polymers represent a novel class of environmentally friendly and efficient drilling mud additives (Sarah and Ebidiseghabofa 2021). Researchers and industrial practitioners have currently developed an interest in modified naturally occurring polymers as drilling mud additives with excellent performance (Mahto and Sharma 2004; Amanullah and Yu 2005). Among naturally occurring polymers, cellulose and starch are the most modifiable and widely applied drilling mud additives in the petroleum industry. The modifications are generally achieved by chemical reactions (etherification, esterification, hydrolysis, sulfonation, and amination) with one of the alcohol groups on the glucose ring or glycosidic bonds (Thomas 1982; Kim et al. 2015; Won et al. 2017; Li et al. 2019). Currently, hydroxy-ethyl-cellulose, PAC, and carboxy-methyl-cellulose represent the extensively used chemically modified naturally occurring polymers (Thomas 1982; Amanullah and Yu 2005; Kim et al. 2015; Won et al. 2017; Soto et al. 2020). They are commonly used to enhance the drilling mud rheological and fluid loss properties during drilling operations in the oil and gas industry.

Although the existing modified naturally occurring polymers exhibit an environmental friendliness property and better performance than native ones, their performance under vigorous conditions, such as high temperatures, are challenging. According to previous studies on modified naturally occurring polymers as drilling mud additives (Amanullah and Yu 2005; Oswald et al. 2006; Agin et al. 2018; Thomas 1982), a dramatic decrease in rheological behaviors and excessive fluid losses were witnessed at high-temperature vicinities. Thus, there still is a need for more effective and cheap polymers that can function as drilling mud rheology enhancers and fluid loss reducers at high-temperature vicinities. Chemically modified starches have been reported to have higher rheology and fluid loss controlling ability, and thermal stability (Thomas 1982; Amanullah and Yu 2005; Kim et al. 2015; Won et al. 2017; Sulaimon et al. 2020; Sarah and Ebidiseghabofa 2021). This study, for the first time, presents a modification of naturally occurring polymers in corn starch (CS) as an environmentally friendly, cost-effective, and thermally stable rheology enhancer and fluid loss reducer for WBM formulations. The MCS was semisynthesized, characterized, and investigated experimentally to determine its rheological and filtration loss properties in WBM.

Experimental Part

Materials. The materials used in this study are as follows: fresh water, epichlorohydrin, sodium hydroxide, absolute ethanol, CS, PAC, bentonite, potassium silicate, potassium humate, sodium sulfite, defoamer, and barite. Epichlorohydrin (>98%), sodium hydroxide (99%),

and absolute ethanol (99%) were bought from Aladdin Company (Shanghai, China). CS, PAC, and other mud additives were supplied by Jingzhou Jiahua Technology Ltd., China. All reagents, solvents, and mud additives were used as received. Because bentonite is an important viscosifying additive in WBMs, its mineralogical phases and chemical composition were analyzed by X-ray diffraction and X-ray fluorescence, respectively, before mud formulation as reported in **Table 1**.

Mineralogical Phases (%) by X-ray Diffraction							
Albite	Gypsum	Calcite	Hematite	Quartz	Microcline		
38.01	26.17	24.36	5.59	5.85	0.01		
Chemical Composition (%) by X-ray Fluorescence							
Na ₂ O	CaO	Al ₂ O ₃	SiO ₂	Fe ₂ O ₃	K ₂ O	MgO	TiO ₂
2.86	2.70	14.81	58.60	16.65	0.22	2.67	1.48

Table 1—Mineralogical phases and chemical composition of bentonite.

Synthesis of MCS. MCS was synthesized by modifying CS through etherification and, subsequently, alkali treatment of the resulted intermediates. In summary, 16.20 g of CS (predried at 45°C for 2 days) contained in 160 cm³ of distilled water was reacted with 9.30 g of epichlorohydrin under controlled temperature for sufficient time. Subsequently, 45 cm³ of 10 wt% sodium hydroxide solution was added to the reaction mixture and refluxed at elevated temperature for 8 hours, and then cooled to room temperature. After cooling, enough ethanol was added, mixed, left to settle, and finally filtrated to obtain MCS. The collected product was washed twice with ethanol and then dried to obtain a practical dry MCS. It was theoretically assumed that the reaction between epichlorohydrin and CS components (amylose and amylopectin) happened mostly at 6-OH groups rather than at the 2-OH and 3-OH groups. Thus, the MCS is composed of two components [modified amylose (a*) and modified amylopectin (b*)] as shown in **Fig. 1**. During the synthesis, the reaction conditions were controlled and optimized to ensure maximum reaction conversion.

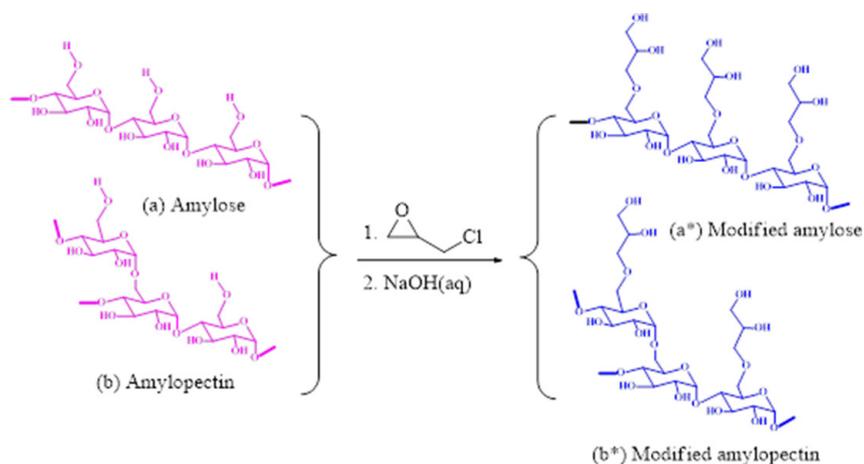


Fig. 1—Modification of CS polymers [amylose (a) and amylopectin (b)].

Characterization of MCS. The chemical structure, morphological structure, and thermostability and dispersibility properties of MCS were characterized by using different techniques. Fourier transform infrared (FTIR) spectroscopy was used for functional groups and chemical structure elucidation. A pH meter, scanning electron microscope (SEM), thermogravimetric analyzer (TGA)-differential thermal analysis (DTA), and zeta potential meter were used to determine the pH, morphological structure, thermostability, and dispersibility behavior, respectively.

- FTIR spectra were recorded by using a Vector-33 FTIR spectrometer in the scanning range of 4000–400 cm⁻¹.
- The pH value of the MCS was measured using a pH meter (pH/ORP meter, HI 2211).
- SEM images of MCS were recorded by using an SEM (SU 8010, HITACHI Japan) under variant resolutions.
- TGA and derivative thermogravimetric (DTG) thermograms were recorded using a simultaneous TGA-DTA analyzer (NETZSCH TG 209F1 Libra), under a gas flow rate of 40 cm³/min and at a heating rate of 10°C/min. The thermoanalytical experiment was carried out in the temperature range of 32–500°C under an inert atmosphere (N₂).
- Zeta potential (ξ) values for 0.01 wt% MCS solutions were measured using a zeta potential meter (JS94, Shanghai Powereach Digital Technology Equipment Co., Ltd.).

Drilling Fluid Formulation. Three WBMs were prepared merely differing in compositions of PAC and MCS as shown in **Table 2**. The WBMs were formulated according to the API standard (*API RP 13B1* 2019); the drilling muds were designed to achieve a mud density of 1.20 g/cm³. The main compositions were fresh water (as a base fluid), viscosity controlling agents, weighting materials, shale stabilizer, defoamer, thermal stabilizer, and fluid loss controlling agents.

Additive	Amount			Function
	WBM	WBM + PAC	WBM + MCS	
Fresh water	350 cm ³	350 cm ³	350 cm ³	Continuous phase
Bentonite	10.5 g	10.5 g	10.5 g	Viscosifying agent
Caustic soda	3.50 g	3.50 g	3.50 g	pH and alkalinity control agent
Potassium silicate	10.5 g	10.5 g	10.5 g	Shale inhibitor
Potassium humate	10.5 g	10.5 g	10.5 g	Fluid loss reducer
Sodium sulfite	10.5 g	10.5 g	10.5 g	Corrosion inhibitor
Barite	55.0 g	55.0 g	55.0 g	Weighting agent
Defoamer	17.5 g	17.5 g	17.5 g	Minimizing foam formation
PAC	0	1.05 g	0	Viscosifying and fluid loss controlling agent
MCS	0	0	1.05 g	Viscosifying and fluid loss controlling agent

Table 2—Drilling fluid formulations for laboratory scale.

Initially, bentonite and fresh water were mixed thoroughly for an hour using a Hamilton Beach mixer (load speed 11,000 rev/min). Other additives were added sequentially to the mixture while mixing. Mud performance enhancers—MCS or PAC (0.3 wt%)—were individually added slowly while concurrently mixing to minimize agglomeration. The WBMs were aged at 180, 200, and 220°C for 16 hours in a hot roller device.

Rheological Behavior. The rheological properties of WBMs were determined using a rotational viscometer (Model No. 1103, Qingdao Chuang Meng Instrument Co., Ltd., China) at 60°C. The data were obtained at fixed rotational speeds (i.e., 600, 300, 200, 100, 6, and 3 rev/min), which give Newtonian shear rates of 1022, 511, 341, 170, 10, and 5 s⁻¹, respectively. The rheological properties investigated in this study include YP, PV, gel strengths, and AV. The YP, AV, and PV were calculated according to the following relations (Eqs. 1–3; Xu et al. 2019):

$$AV = \theta_{600}/2 \text{ (mPa} \cdot \text{s)}, \quad (1)$$

$$PV = \theta_{600} - \theta_{300} \text{ (mPa} \cdot \text{s)}, \quad (2)$$

$$YP = \theta_{300} - PV \text{ (lb/100 ft}^2\text{)}, \quad (3)$$

where θ_{600} and θ_{300} are dial readings at 600 and 300 rev/min, respectively. Moreover, gel strength, which is the shear stress of drilling mud that is measured at a low shear rate when the drilling mud has settled for a while, was also investigated. Herein, G10-s and G10-min, which denote gel strengths at 10 seconds and 10 minutes, respectively, were recorded at 3 rev/min.

Filtration Loss. Filtration loss tests were conducted by using API filter press equipment (Model No. GGS42, Qingdao Chuang Meng Instrument Co., Ltd., China) equipped with a 45- μm filter paper. The filtrate volumes were collected in measuring cylinders after 30 minutes. The fluid losses from 5 to 25 minutes were calculated by using Eq. 4 (Chinwuba et al. 2016):

$$f_1 = f \times \sqrt{T_1}/\sqrt{T}, \quad (4)$$

where f is the known filtrate at a time interval of T (30 minutes), and f_1 is the unknown filtrate at a time interval of T_1 . Besides, the filter (mud) cake thicknesses were recorded in millimeters using vernier callipers. The obtained thicknesses in combination with filtration loss data were used to determine the mudcake permeability.

Filter-Cake Morphological Analysis. The filter cakes obtained after the filtration processes were air-dried at room temperature and then were morphologically characterized using an SEM (Model No. SU 8010, HITACHI, Japan); SEM observations were carried out under different resolutions.

Biodegradability. The biodegradability of MCS and CS was studied by determining the chemical oxygen demand (COD) and the 5-day biochemical oxygen demand (BOD₅). The ratio of BOD₅/COD that gives the information on the biodegradability of the materials was calculated. The COD and BOD₅ were determined by following the protocols described elsewhere (Pereira et al. 2018; Xu et al. 2019).

Results and Discussion

Characterization of MCS. The characterization of MCS was achieved through spectroscopic, thermogravimetric, and electric potential analyses. In general, the analyses inferred that CS was modified successfully, giving a more thermally stable derivative with quite good dispersibility property.

FTIR Analysis. The recorded FTIR spectra of the MCS and CS samples are depicted in **Fig. 2**. Several absorption bands for functional groups incorporated in MCS are displayed. The displayed absorption bands are comparable with those of non-MCS, differing only in the absorption intensities because of the increased O–H and C–O functionalities in the MCS. In general, MCS displayed more pronounced absorption bands 3329, 2920, 1433, 1100, and 1028 cm⁻¹, which are distinctive bands for O–H, CH₂, C–O–C, and C–OH vibrations. The

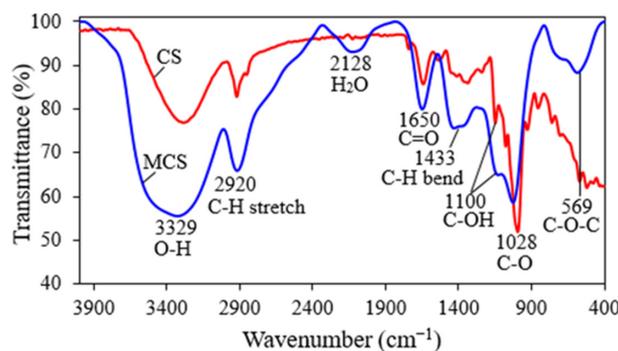


Fig. 2—FTIR spectra of CS and MCS samples.

vibration bands at 2128 and 1650 cm^{-1} presumably arose from trace water molecules in the hydrated MCS sample (Warren et al. 2016). The occurrence of the reaction is signified by the absence of a strong absorption band around 850–550 cm^{-1} in the MCS spectrum.

Determination of the pH Value of MCS. The suspension of MCS with various concentrations was prepared using distilled water, and its pH value was determined. The experimental results in Fig. 3 show that the MCS suspension has a pH around the neutral scale. The pH value of the drilling mud prepared in this study was controlled at approximately 10.27 using caustic soda to reduce the corrosion of the drilling tools and inhibit the dissolution of minerals from the clay formation.

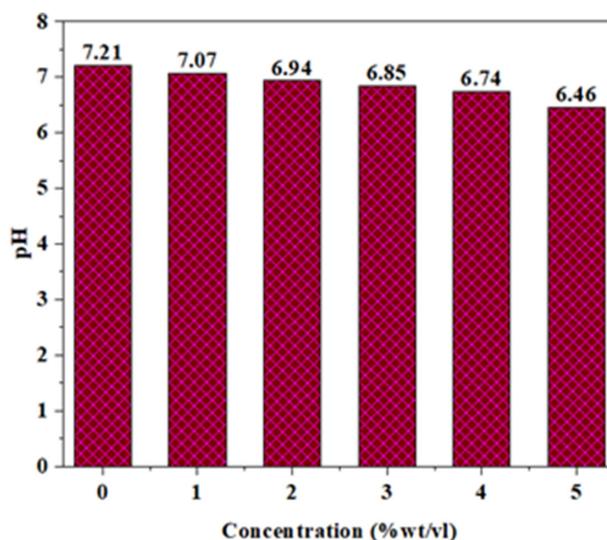


Fig. 3—Results for pH value of different concentrations of MCS suspension.

Thermostability of MCS. TGA is a suitable method to investigate the thermostability and decomposition profile of polymeric compounds (Vega et al. 1996; Liu et al. 2013). The knowledge of thermostability and the mode of decomposition of drilling mud additives under the influence of heat are highly acclaimed in maximizing the performance of drilling fluids. The TGA and the corresponding derivative (DTG) thermograms for the degradation process of MCS and CS at 10°C/min are shown in Fig. 4. As is seen, both TGA and DTG thermograms for MCS show two conspicuous zones/stages in the degradation process of MCS at a temperature range of 32–500°C. The initial mass loss between 36 and 218°C (8.84%) was attributed to physical dehydration that occurred in a single step (Liu et al. 2019). After dehydration, the analyzed sample was stable up to nearly 300°C, and above this temperature, the main degradation stage occurred. This stage is composed of two overlapping steps: 38.49 and 32.29%, which were apparently in the DTG curve. The occurrence of two peaks for the DTG thermogram signified the main thermal degradation in MCS is quite complex and is often reported for starch-based compounds (Janković 2013; Liu et al. 2013). The mass losses of 38.49 and 32.29% were attributed to chemical dehydration and thermal decomposition, respectively. The unmodified CS was found to degrade at a temperature relatively lower compared to MCS and degraded completely when the temperature reached 480°C. In general, the thermostability of MCS was quite better compared to native starches (Vega et al. 1996; Liu et al. 2019) and known modified starches (Liu et al. 2013). An increased thermostability of MCS was possibly a result of increased molecular weight and more stable microstructures owing to additional OH groups, which need more energy to break MCS molecular chains (Liu et al. 2013).

Morphological Analysis. SEM micrographs of the MCS at different magnifications are depicted in Figs. 5a and 5b. The SEM images show the presence of MCS granules, which ranged from small to large sizes. The MCS sample is composed of polygonal, irregular, and fibrous granules, with slight surface smoothness. The SEM analysis indicated that the modification brought noticeable morphological changes in the structure of MCS granules compared with those of natural starches and gelatinized starches reported in previous literature

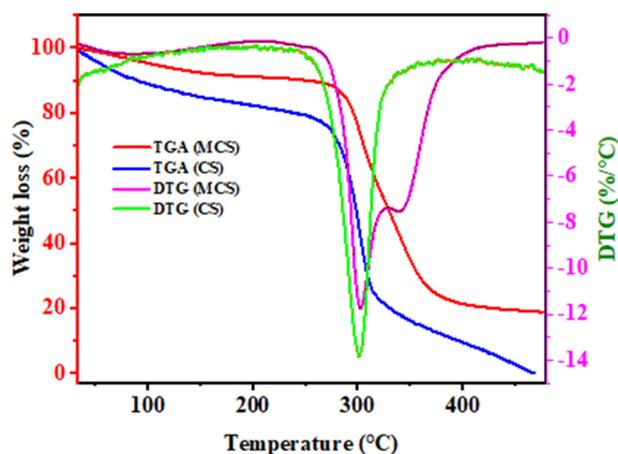


Fig. 4—TGA-DTG thermograms of the CS and MCS.

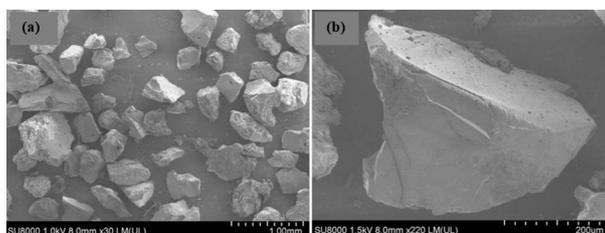


Fig. 5—SEM micrographs of MCS at varying magnifications.

(Kaur et al. 2011; Kim et al. 2015; Babu et al. 2015; Won et al. 2017). The morphological changes confirmed the occurrence of the targeted chemical modification in CS.

Zeta Potential (ζ) Measurement. The average value of ζ measured for three MCS solutions (each 0.01 wt%) was -28.25 ± 0.37 mV. The higher value of ζ (large positive or negative) helps in avoiding flocculation or aggregation of colloidal particles through repulsion (Wongsagonsup et al. 2005; Hamad et al. 2019). Thus, the MCS particles exhibited better dispersibility and may stabilize in colloidal mud systems and, consequently, better mud properties owing to improved dispersion stability of mud components.

Rheological Properties. A successful wellbore drilling in oil and gas fields is achieved through monitoring and controlling the rheological properties of the drilling mud. Herein, the rheological properties of formulated drilling muds were studied systematically at variant high temperatures (180–220°C). The rheological profiles (AV vs. shear rate plots) are depicted in Figs. 6a–6c. As is seen from Figs. 6a–6c, all drilling muds exhibited the shear-thinning (pseudoplastic) behavior. The shear-thinning behavior of drilling muds was defined by the displayed decrease in AV with an increase in shear rate. Drilling muds play a significant role in cleaning the wellbore by removing the cuttings from the drill path. The shear-thinning effect is believed to increase the viscosity in the annular space, thereby improving the drilling mud cleaning efficiency of the well (Salehnezhad et al. 2019). The efficiency of MCS was more pronounced at all temperatures; the mud comprising MCS interestingly exhibited optimal rheological profiles. The MCS was mostly a superior rheology enhancer over PAC at all tested conditions. The superior performance of MCS was attributed to its additional OH groups, which prompted the strong intermolecular and particulate interactions in the aqueous system as discussed and illustrated schematically under the filter-cake permeability determination section. The strong intermolecular and particulate interactions prompted the contact and internal resistance between the molecules and/or particulates, giving better rheology for mud.

Moreover, high temperature presumably degraded the mud components (Besün et al. 1997), dwindling the active components responsible for rheology enhancer in the system. Because of this phenomenon, the rheological qualities of the drilling muds were observed to diminish when the aging temperature increased. Nevertheless, mud comprising MCS exhibited superior rheological properties at all tested temperatures. The addition of MCS not only improved the rheological properties of the drilling mud but also enhanced the thermostability property of the mud. Rheology is a fundamental property that needs to be customized for the development of stable and effective drilling mud. To further understand the effect of MCS on mud rheology, YP (stress), AV, PV, and gel strengths for 10 seconds and 10 minutes were examined systematically.

PV. PV refers to the friction existing between the fluid layers and the solid particles, and, in this work, was studied at different aging temperatures. The experimental results (Fig. 7) revealed the substantial increments in PV owing to the addition of PAC or MCS. The mud, which comprised MCS, demonstrated the optimal PV over those of PAC or without rheology enhancer additive (WBM alone). The inclusion of MCS in mud remarkably heightened PV by 316% and 10% compared with WBM alone and mud with PAC at 220°C, respectively. The remarkable superior performance of MCS was accredited to its stronger intermolecular interactions between fluid layers and/or solid particles, thus increasing PV. For drilling purposes, muds with moderately high PV have potential in enhancing the cutting transport efficiency of the drilling fluid, thereby eliminating the problems related to the cutting settling. Therefore, MCS exhibits promising and potential performance for smart drilling fluids formulation.

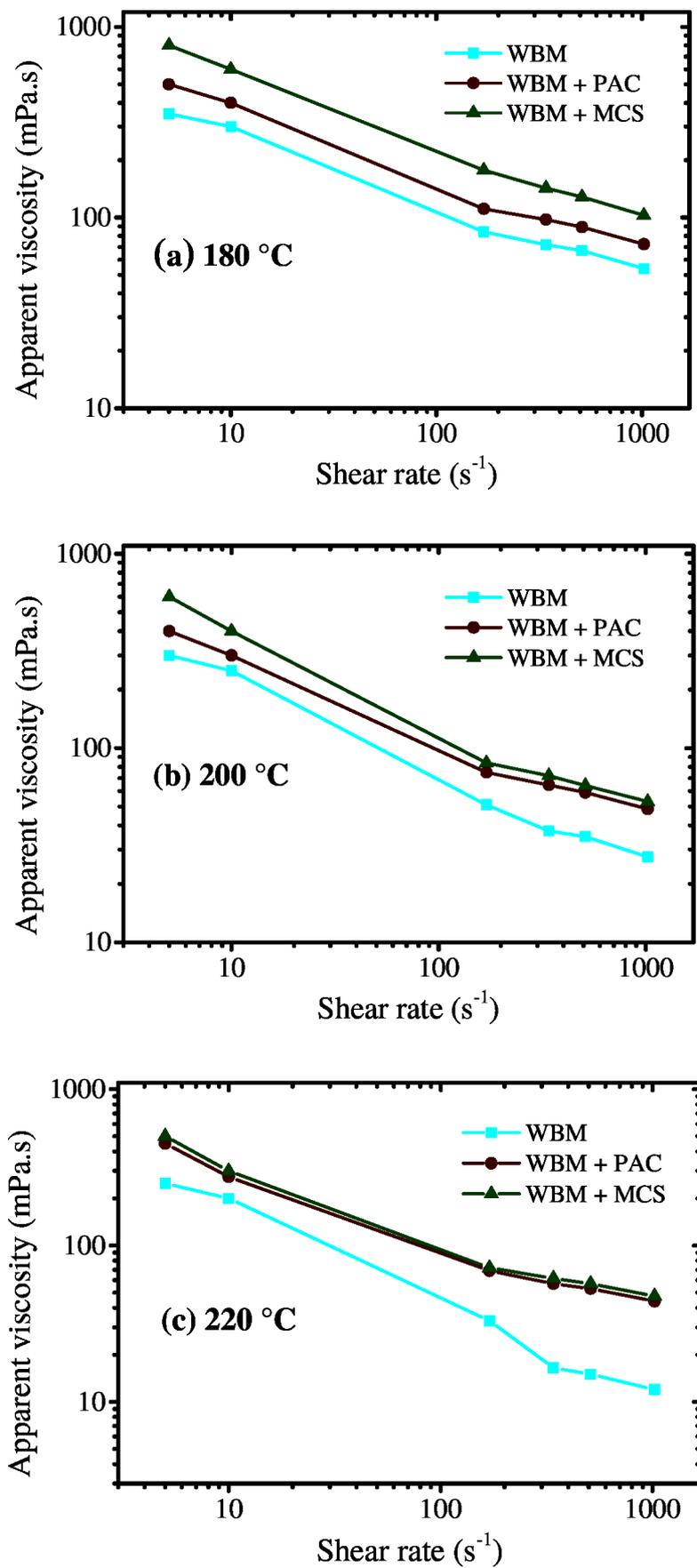


Fig. 6—Apparent viscosities of drilling mud formulation as a function of shear rate at different temperatures.

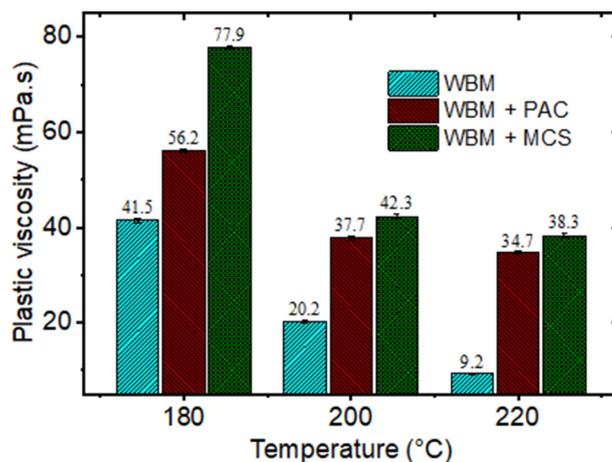


Fig. 7—PVs of drilling muds at varying temperatures.

AV. The AV is one of the main rheological properties of drilling mud, playing a role in preventing various drilling problems and improving well-cleaning efficiency (Al-Khdheawi and Mahdi 2019). The addition of MCS played a substantial role in controlling the AV at various temperatures. As is seen in Fig. 8, at all temperatures, mud formulation comprising MCS exhibited maximal AVs. In contrast to WBM, whose AV decremented severely at 220°C, the incorporated MCS boosted the AV three times more than WBM alone. The better performance of MCS was ascribed to its capability of forming strong hydrogen bonding, which increased the intermolecular interaction. The strong molecular interactions triggered molecular entanglements between the molecular chains of the mud components and MCS, thereby increasing the flow resistance and, subsequently, AV of the mud system increased.

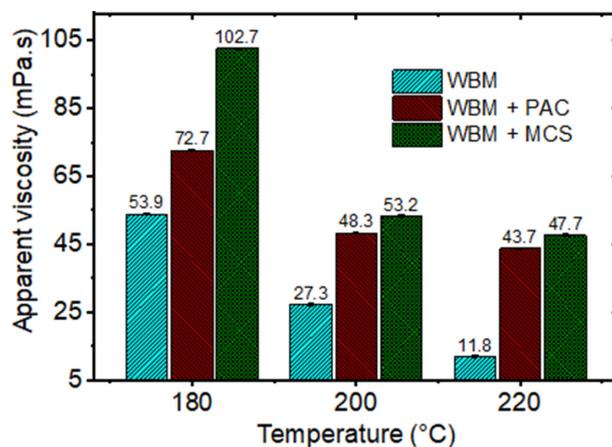


Fig. 8—AVs of drilling muds at varying temperatures.

Nevertheless, temperature elevation weakened the hydrogen bonding, resulting in deteriorated entanglements. Because of weak entanglements, the flow resistance decreased, causing a decrease in AV as the temperature increased. Interestingly, mud comprising MCS showed the maximal AV even at high temperatures (220°C). This is attributed to the formation of crosslinking between the polymeric components, which triggered the temperature-resistance ability. The high AV reflected a high ability of mud to resist deformation under deforming stress (Al-Khdheawi and Mahdi 2019). Mud having high AV has a better ability to suspend the mud solids and, consequently, better suspension of drill cuttings.

Yield Point/Stress. The addition of MCS had a prominent effect on the YP of the formulated drilling mud. As is shown in Fig. 9, the addition of MCS increased the YP by 2 and 1.5 times more than WBM alone and mud with PAC at 180°C, respectively. The YP was observed to decrease with rising temperature, and both PAC and MCS exhibited fairly comparable YPs at 200 and 220°C. The potential application of MCS is more pronounced at 220°C, where the YP of WBM decremented critically. The high YP was linked to an increased internal flow resistance because of the strong internal structure prompted by the addition of MCS. Thus, an extra force was required to break down the strength of the internal structure of the fluid before any movement could occur, hence the high YP.

The strength of the internal structure weakened when temperature increased owing to deterioration of the entanglement and polymer crosslinking. This phenomenon dwindled the amount of force (yield stress) needed to cause fluid movement, and subsequently, decreased YP when temperature increased. Enhancement of YP for mud formulation is of practical importance in drilling operations (Elkatatny 2019). Drilling mud with a moderately high YP has a better ability to transport the cuttings out of the borehole, thus better cleaning operation in the hole.

Gel Strengths (G10-s and G10-min). The gel strengths recorded at 10 seconds and 10 minutes are presented in Table 3. From Table 3, it is clear that the gel strengths of mud formulations increased with time, except for WBM at 180°C where gel strengths did not change. Mud comprising MCS demonstrated both high G10-s and G10-min strengths at all trialed temperatures; the gel strengths were found to

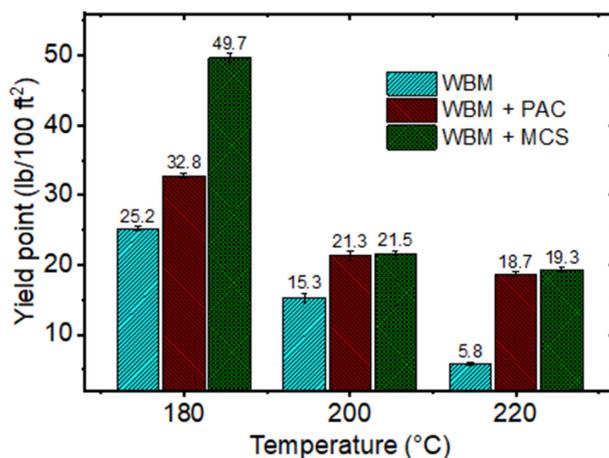


Fig. 9—YPs of drilling muds at varying temperatures.

decrease as temperature increased. The incorporation of MCS in mud formulation prompted strong molecular interactions, causing strong attractive forces between fluid components under static conditions and consequently high gelation. The strength of attractions weakened at high temperatures owing to the deterioration of some mud components. The weakening in attractive forces caused weak gelation under static conditions. Thus, decreased gel strengths were observed when the temperature increased. Optimizing the gel strength of drilling mud aids in avoiding an accumulation of drilled cuttings, which may result in sticking of the drillstring (Alakbari et al. 2018). A drilling mud having optimal gel strength has exceptional ability in suspending drilled solids and weighting materials when circulation stops. Thus, the MCS is the most promising mud additive because it demonstrated the acceptable gel strengths at the experimented temperatures (Machado and Arago 1990; Ekeinde et al. 2018).

Temperature (°C)	Gel Strength (lb/100 ft ²)					
	WBM		WBM + PAC		WBM + MCS	
	G10-s	G10-min	G10-s	G10-min	G10-s	G10-min
180	5	5	6	7	7	9
200	4	5	5	6	6	8
220	2	3	4	5	5	6

Table 3—Gel strengths of drilling muds at different temperatures.

Rheological Models. Rheological models are often used for a quantitative description of the mud rheological properties. For many decades, rheological models such as power law, Carson, Bingham plastic, and Herschel-Bulkley have been used to study the rheological properties of drilling fluids (Livescu 2012). In the present study, Herschel-Bulkley and Bingham plastic models were applied in determining the rheological properties of the formulated drilling muds because they are widely used in describing the rheological properties of drilling muds (Vryzas and Kelessidis 2017). The Bingham plastic model (Eq. 5) constitutes both yield stress and a limiting viscosity at a finite shear rate (Bingham 1916). The Herschel-Bulkley model combines the power law and Bingham plastic models as represented in Eq. 6 (Herschel and Bulkley 1926; Livescu 2012):

$$\tau = \tau_o + \mu_p \dot{\gamma}, \quad (5)$$

$$\tau = \tau_o + k \dot{\gamma}^n, \quad (6)$$

where τ = shear stress (Pa), τ_o = yield stress/point (Pa), μ_p = PV (Pa·s), and $\dot{\gamma}$ = shear rate (s⁻¹). The parameters k and n represent the consistency index (Pa·s ^{n}) and the flow behavior index (unitless), respectively.

The experimental results for the shear rate and shear stress were fitted as per Eq. 5 and Eq. 6; the fitting results are given in **Table 4**. R -squared (R^2) is the linear correlation coefficient, which is the relative measure of fit, and root mean square error (RSME) is the absolute measure of fit. According to the results in **Table 4** and **Fig. 10** (for WBM + MCS mud), the Herschel-Bulkley model demonstrated maximal R^2 (>0.99) and minimal RMSE (<1). Thus, the Herschel-Bulkley model more ideally described the rheological behaviors of the studied muds than did the Bingham plastic model.

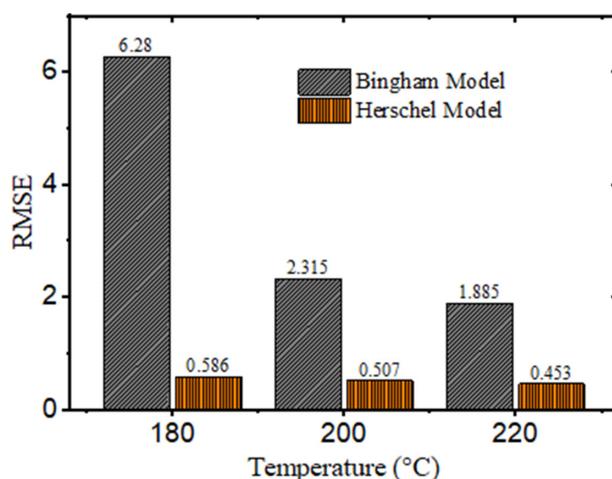


Fig. 10—RMSE for Bingham plastic and Herschel-Bulkley models for WBM+MCS formulation at different temperatures.

Mud Sample	Temp. (°C)	Bingham Model				Herschel-Bulkley Model				
		τ_o (Pa)	μ_p (Pa·s)	R^2	RMSE	τ_o (Pa)	k (Pa·s ^{<i>n</i>})	n	R^2	RMSE
WBM	180	4.442	0.052	0.985	2.746	1.497	0.260	0.770	0.999	0.781
	200	2.986	0.026	0.975	1.783	0.874	0.215	0.699	1	0.196
	220	1.677	0.011	0.988	0.517	1.273	0.034	0.835	0.994	0.404
WBM + PAC	180	5.367	0.071	0.982	4.126	0.761	0.426	0.744	0.999	0.943
	200	3.904	0.047	0.987	2.287	1.421	0.216	0.781	0.999	0.478
	220	3.636	0.042	0.989	1.863	1.648	0.169	0.800	0.999	0.447
WBM + MCS	180	9.335	0.099	0.979	6.280	2.027	0.713	0.718	0.999	0.586
	200	4.936	0.050	0.989	2.315	2.450	0.214	0.793	0.999	0.507
	220	3.887	0.045	0.991	1.885	1.904	0.168	0.813	0.999	0.453

Table 4—Parameters for constitutive rheological models for investigated drilling muds.

Based on the Bingham model, the addition of MCS improved the μ_p excellently compared with PAC addition under the same experimental conditions. The μ_p values were observed to decrease progressively owing to the increase in temperature. The Bingham plastic model predicted consistently the experimented variation of μ_p with temperature despite its inaccuracy in describing the rheological behaviors of the assessed muds.

Flow Behavior Index (n). The flow behavior index is an empirical measure that decides the flow behavior and hence the types of fluids. Following the Herschel-Bulkley model, drilling fluids may be classified as shear-thinning ($n < 1$), Newtonian ($n = 1$), and shear-thickening ($n > 1$) fluids (Vryzas et al. 2017). As is evident in Fig. 11, all mud formulations exhibited the shear-thinning behavior (i.e., all were pseudoplastic fluids as it was stated previously). Despite the decrease in n value observed for WBM at 200°C, the values of n

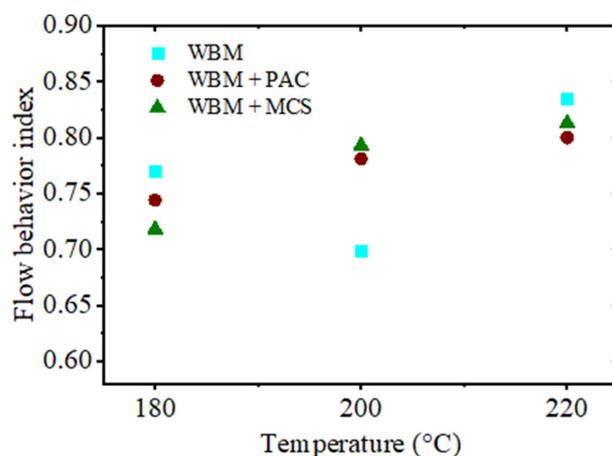


Fig. 11—Flow behavior indices of drilling muds at different temperatures.

generally increased when temperature increased and were fairly comparable at the same temperature. Summarily, the trend observed was comparable to the one described elsewhere (Wang et al. 2016).

Flow Consistency Index (k). Drilling muds display variable consistency indices depending on the structure and stability of the gel (Salehnezhad et al. 2019). Fig. 12 depicts the consistency indices of the studied drilling muds at different aging temperatures. As is seen from Fig. 12, the consistency indices were affected with aging temperature, and the values of k decreased as the temperature increased. The substantial decrements were observed when the temperature was raised from 180 to 200°C for MCS and PAC mud formulations. Meanwhile, an insignificant decrease in k was observed for WBM formulation at the aforementioned conditions. Contrarily, when the aging temperature varied from 200 to 220°C, k decreased substantially for WBM and remained fairly constant for PAC and MCS mud formulations. The consistency index depends on the structure and stability of gel, and it is associated with the compaction and/or entanglement of the structural units (Besün et al. 1997; Salehnezhad et al. 2019). The strong compaction and/or entanglement of the mud components are believed to increase the consistency index. When the aging temperature was 180°C, mud with MCS exhibited the highest consistency index, inferring the strong compaction and entanglement of the mud components owing to the strong interactions between MCS and other mud components. The decrements in consistency indices were explained by losses of molecular weights and breakdown of polymeric chains at high temperatures (Besün et al. 1997). The degradation caused the loss of polymeric properties and the effectiveness of materials, lessening the compactness and entanglement and hence the diminished consistency index.

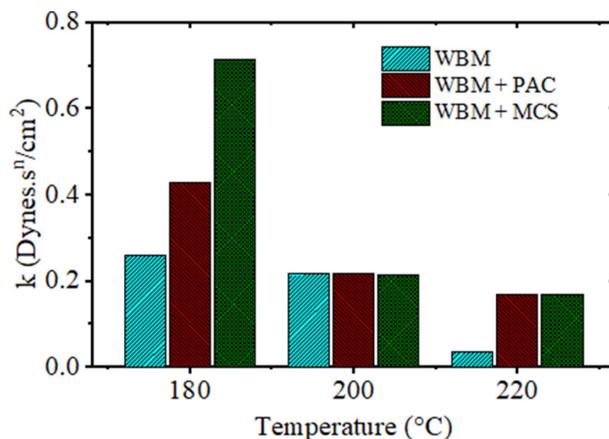


Fig. 12—Flow consistency indices of drilling muds at different temperatures.

Fluid Loss and Filter-Cake Permeability Analysis. Fluid loss refers to mud filtrate loss into a porous permeable formation as a result of high hydrostatic pressure. This behavior results into formation damage, poor cementing, and stuck pipe. The use of fluid loss control agents is well known to alter the permeability of filter cake and subsequently fluid loss behavior (Vryzas and Kelessidis 2017; Li et al. 2015). In this study, the addition of MCS in the mud system substantially reduced the mudcake thickness and permeability, resulting in low fluid loss. The observed phenomenon was ascribed to the strong compactness and entanglement, which resulted after the addition of MCS.

API Filtration Tests. Smart drilling fluid should have a minimal filtration rate under stressful conditions, including high temperatures. To investigate the influence of MCS on fluid loss behavior, API filtration loss tests under low pressure and low temperature were conducted, and the results are presented in Fig. 13. As is clearly shown in Fig. 13, mud comprising MCS exhibited the lowest filtrate volumes at all tested temperatures. Meanwhile, WBM without MCS or PAC demonstrated the largest filtrate volumes at all tested temperatures. The experimental results inferred that MCS is the superior fluid loss control agent compared to PAC. Besides, temperature elevation resulted in a substantial increase in filtrate volumes.

Filter Cake Permeability Determination. The permeability and thickness of the filter cake are important parameters used in prescribing the effectiveness of the filter cake in strengthening the wellbore (Feng et al. 2018). The permeability of the filter cake (k) is an imperative parameter that defines the filtration behavior of the filter cake (Dejtaradon et al. 2019). There are several models to predict the permeability of the filter cake (Elkatatny et al. 2012; Jaffal et al. 2017); herein, we adopted a model that relies on the cumulative filtrate volume and time as shown in Eq. 7 (Li et al. 2018):

$$k = \frac{\mu l \partial V}{\Delta P A \partial t} = \frac{\mu \cdot l \cdot q}{\Delta P A}, \quad (7)$$

where k is the filter-cake permeability, μ is the filtrate viscosity at 25°C (estimated to 1 cp), l is the thickness of filter cake (cm), P is the pressure difference (6.8 atm), A is the effective filter surface (45.8 cm²), and $\partial V/\partial t = q$ is the filtrate rate (cm³/s).

The filtrate rate (q) was deduced as the gradient value for a plot of filtration volume (cm³) vs. time (seconds) as described elsewhere (Li et al. 2018). The deduced filtrate rate (q) and the permeability (k) alongside the recorded filter-cake thickness (l) are summarized in Table 5. Evidently, WBM exhibited the highest q , l , and k values under the same experimental conditions. Meanwhile, mud consisting of MCS demonstrated the lowest q , l , and k values under similar experimental conditions. It is clear that the filtration rates decreased because of the addition of PAC or MCS, and the MCS was superior over PAC. The decrease in filtration rates was ascribed to the reduced permeability of the filter cakes because of the addition of PAC and MCS. These observations are coherent with the API fluid loss results, affirming that the permeability of the deposited filter cakes substantially governed the filtration performance of drilling muds.

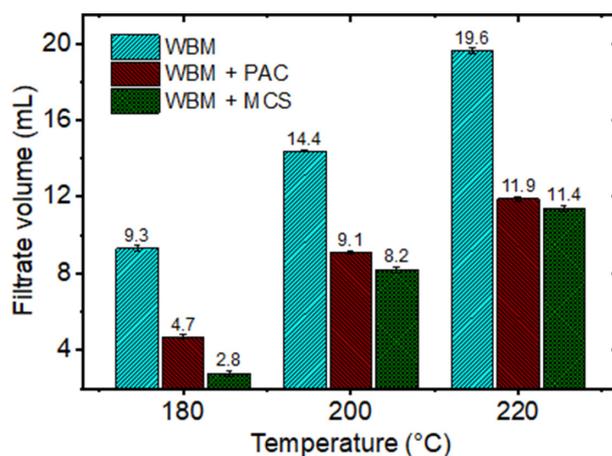


Fig. 13—API filtration losses for studied muds at varying aging temperatures.

Mud Formulation	Temperature (°C)	$q = \times 10^{-3}$ (cm ³ /s)	l (cm)	$k = \times 10^{-3}$ (md)	Percentage Decrease in k (%)*
WBM	180	3.60	0.050	0.580	-
	200	5.60	0.077	1.384	-
	220	7.50	0.150	3.612	-
WBM + PAC	180	1.80	0.027	0.156	73
	200	3.50	0.060	0.674	51
	220	4.50	0.083	1.199	67
WBM + MCS	180	1.20	0.027	0.104	82
	200	3.20	0.050	0.514	63
	220	4.50	0.073	1.055	71

*Decrease in k (%) = $\left[\frac{(k_b - k_a)}{k_b} \right] \times 100$, where k_b and k_a are permeability of WBM and WBM +PAC (or WBM +MCS), respectively, measured under the same conditions.

Table 5—Filtrate rate (q), thickness (l), and permeability (k) of filter cake for the studied drilling muds.

It was proposed that the additional hydrogen bonding owing to the introduced OH groups in MCS accounted for its better capability to control filtration loss. The introduced hydrogen bonding strengthened the molecular interaction, which boosted the packing efficiency. Besides, the branched nature of amylopectin presumably improved the interaction. Because of those hypothetical phenomena, the interaction between the mud components was provoked, causing the formation of more compact networking structures, as illustrated schematically in Fig. 14. The overall effects resulted in the formation of thin filter cakes with compact and well-packed microstructures, thereby reducing permeability and, subsequently, filtration loss. This was contrary to mud formulation comprising PAC, where there is weak hydrogen bonding, and to the WBM system, where the van der Waals interactions were dominant.

The hypotheses formulated were confirmed by the SEM analyses, which revealed the morphological structures on the surfaces of deposited filter cakes (Fig. 15). The molecular entanglement and strong intermolecular interactions arose because of the addition of MCS-enabled mud (WBM +MCS) microstructures to deposit in a compactible way during the filtration process, producing a compact and relatively smoother filter cake (Fig. 15c). This compactness typically condensed and clogged the fluid penetration pathway, producing less permeable filter cake with comparatively superior filtration behavior. Despite the reduction in permeability of the filter cake ascribed to the diminished sizes of pores caused by the addition of PAC (Fig. 15b), the intermolecular interactions were comparatively weaker, causing inefficient packing of microstructures. This resulted in the deposition of comparatively rough and permeable filter cake during filtration measurement. Nonetheless, the filtration performance of the mud comprising PAC was superior over mud without PAC or MCS (Fig. 15a).

Furthermore, temperature elevation was observed to degrade the mud additives. The degradation of materials defined the incremental trends in q , l , and k values. Nevertheless, MCS demonstrated better thermostability than PAC as witnessed from mud formulation comprising MCS, which exhibited better fluid loss control behavior than the one with PAC or without PAC or MCS (i.e., WBM). The addition of MCS into mud formulation provided a desirable filter cake having low permeability and low thickness. The low permeability filter cake is required to prevent drilling fluid invasion into the formation. Also, a thin filter cake is necessary for avoiding drilling fluid problems including stuck pipe, excessive torque, and drag (Feng et al. 2018).

Biodegradability. The biodegradation of the MCS and CS was studied by measuring COD and BOD₅. BOD₅ was used to measure biodegradability, whereas the extent of environmental pollution was assessed by considering the magnitude of COD. The BOD₅/COD ratio was used to measure the degree of biodegradation of the materials. As is seen in Table 6, MCS exhibits lower COD, BOD₅, and BOD₅/COD than unmodified CS because it possesses many oxygen atoms and OH groups in its structure than unmodified CS. In

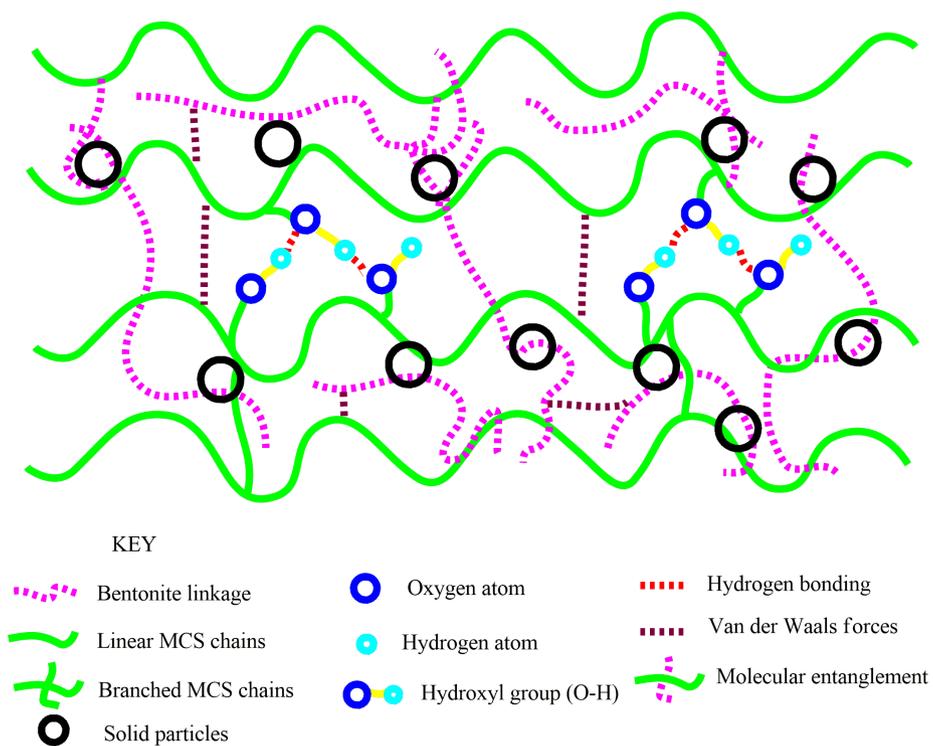


Fig. 14—Schematic depiction of MCS molecular interactions in the mud system.

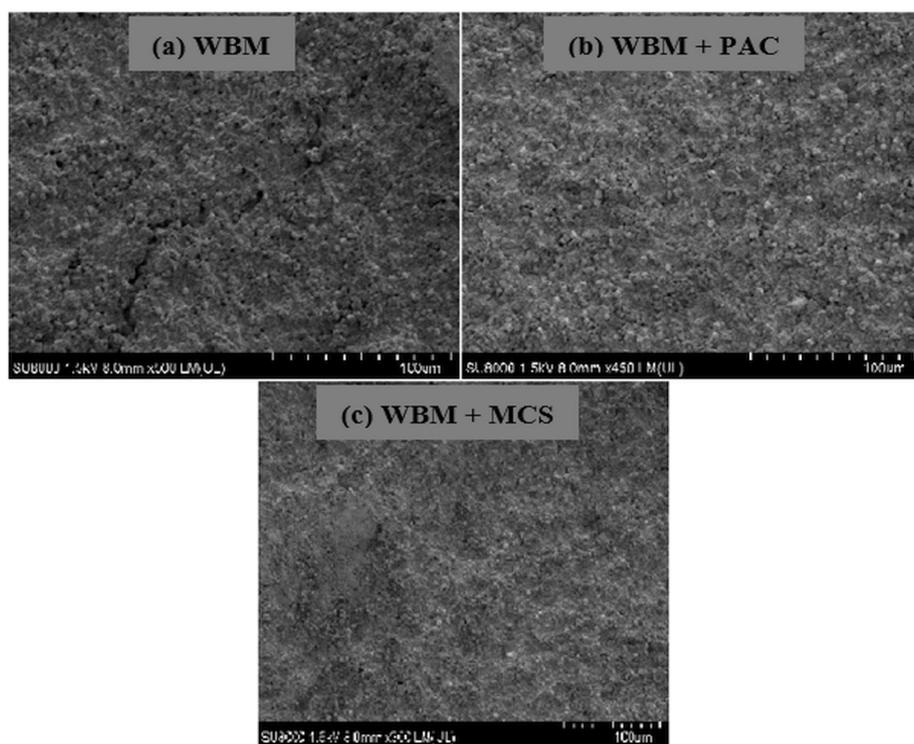


Fig. 15—SEM micrographs of filter cakes obtained at 220°C.

summary, MCS is biodegradable because it exhibits an acceptable value of BOD₅/COD ratio (Pereira et al. 2018) and displays low extent pollution to the environment.

Material	COD (mg/L)	BOD ₅ (mg/L)	BOD ₅ /COD
MCS	77 ± 2.08	35.343 ± 0.584	0.461 ± 0.019
CS	137 ± 3.61	71.518 ± 0.505	0.522 ± 0.017

Table 6—Experimental results on biodegradability of MCS and CS.

Conclusion

This work disclosed a potential novel drilling fluid additive (abbreviated as MCS) modified from environmentally friendly and readily available CS. The modification of CS was successfully achieved through etherification and, subsequently, treatment of the resulted intermediate with aqueous sodium hydroxide solution.

- The methodical investigations proved the MCS to perform excellently than PAC, which is extensively used as a drilling mud additive. With the addition of 0.3 wt% of MCS, both rheological and filtration loss properties were improved markedly.
- The rheological properties (PV, YP, AV, and gel strength) of the formulated drilling mud increased largely because of the addition of MCS. The filtration loss property of the formulated mud decreased noticeably owing to the inclusion of MCS in the mud formulation.
- The incorporation of MCS not only improved the filtration loss and rheological properties of WBM but also augmented its temperature resistance.
- The exceptional performance of MCS was owing to the strong hydrogen bonding originated because of the addition of OH functionalities.
- Moreover, the Herschel-Bulkley model satisfactorily predicted the rheological properties of the formulated drilling muds with and without PAC or MCS based on the linear correlation coefficient (R^2) and RMSE.
- Besides, MCS is an environmentally friendly rheology and fluid loss controlling agent that exhibits acceptable degradability properties.

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