Rheological challenges in bentonite-based fluids: a preliminary study

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ABSTRACT: Bentonite drilling fluids are essential in various geotechnical applications, where their rheological properties are crucial for excavation stability, material transport and lubrication. Despite international standards, discrepancies often arise between laboratory tests and on-site measurements. The preparation of these fluids involves variables such as bentonite concentration, mixing speed, and duration, which affect the final properties. This article investigates the characteristics of bentonite and drilling fluids, exploring how chemical composition influences preparation methods and rheological properties. The focus is on understanding how different types of bentonites (calcium and sodium) with varying mineralogical compositions affect fluid behavior. X-Ray diffraction and X-ray fluorescence analyses were used to characterize the bentonites, and clay properties were evaluated through swelling index and Atterberg's limits. Water-based bentonite fluids were prepared at different concentrations and characterized by Marsh viscosity. The study also examines the impact of carboxymethylcellulose (CMC) additives to enhance fluid performance. The study showed that sodium bentonite, due to its higher swelling capacity and liquid limit, significantly enhances viscosity when CMC is added, while calcium bentonite remains mostly unaffected by the same CMC concentration.

1 INTRODUCTION

Bentonite is a clay mineral primarily composed of montmorillonite, along with varying quantities of other minerals such as quartz calcium and sodium feldspar (Abu-Jdayil et al. 2014). Water-based bentonite fluids are extensively utilized in drilling due to their superior rheological properties, which are essential for effective drilling operations (Kunlin et al. 2016). These fluids display shear-thinning behavior, enhancing their pumpability and efficiency in transporting cuttings. To achieve the desired rheological and filtration characteristics, high concentrations of bentonite are typically required (around or above 6 wt.%; Abbas et al. 2022). However, excessive amounts of bentonite can lead to reduced drilling efficiency and cause several operational challenges, including differential sticking, poor borehole cleaning, increased torque and drag, and potential formation damage (Kunlin et al. 2016). Therefore, it is crucial to develop drilling fluids with lower solid content. In this context, polymers can be employed to formulate fluids with rheological and fluid loss properties similar to those of higher-bentonite-content fluids (Cui et al. 2023). Carboxymethylcellulose (CMC) is a prominent polymer in this regard. It is a water-soluble polymer derived from cellulose through a process involving sodium hydroxide and monochloroacetic acid. CMC is effective at dispersing clays and mitigating fluid loss in porous and permeable formations (Nobrega et al. 2019).

The effectiveness of CMC varies depending on the type of bentonite used, whether sodium or calcium. Research by Abu-Jdayil & Ghannam, as well as Benchabane & Bekkour, has investigated the impact of CMC on sodium and calcium bentonites, respectively, with a focus on their rheological properties (Abu-Jdayil et al. 2014; Benchabane et al. 2006). The first study found that adding CMC in concentrations ranging from 0.1 wt.% to 0.5 wt.% increases the viscosity of sodium bentonite dispersions, shifting the rheological behavior from a mixed Bingham/shear-thickening to a shear-thinning profile. In contrast, the second study, which examined various polymer additives including CMC, revealed that the rheological behavior of CMC-extended calcium bentonite aligns with that of CMC suspension alone, following a power-law model.

Given the differences in preparation conditions and analysis methods in these studies, direct comparison of the two types of fluids is challenging. To address this, the current study explores the impact of adding CMC to bentonite, focusing on both sodium-based and calcium-based types. The objective is to correlate changes in viscosity, as measured by Marsh viscosity, with the physicochemical properties of the bentonites. These properties include the liquid limit, swelling index, crystalline phases, chemical composition, loss on ignition and cation exchange capacity. To ensure a reliable comparison, the study will use consistent preparation methods and measurement conditions for both types of bentonite fluids. In particular, both types of bentonite were used to prepare drilling fluids at identical bentonite concentrations, and for fluids modified with CMC, the polymer was also added at the same concentration. These fluids were analyzed based on the previously mentioned measurements to examine how effectively CMC enhances the properties of the drilling fluids. This approach will try to put the basis for a clearer understanding of how CMC affects different bentonite types and help optimize the formulation of low-solid-content drilling fluids.

2 MATERIALS AND METHODS

2.1 *Materials*

Bentonite fluids were prepared using two different types of natural bentonites (calcium and sodium) at different concentrations in the range 0 wt.% -7 wt.% in tap water.

Technical-grade high-viscosity carboxymethylcellulose (CMC) was used as polymer modifiers at a concentration of 0.5 wt.% with respect to the bentonite powder amount. The names of formulations tested in this work are summarised in Table 1.

Table 1. Composition and name of the different powder mix formulations tested (percentages in wt.%).

ID	Details	Bentonite	CMC
Ben-Ca	Calcium bentonite	100%	0%
Ben-Na	Sodium bentonite	100%	0%
Ben-Ca-CMC	Calcium bentonite extended with CMC	99.5%	0.5%
Ben-Na-CMC	Sodium bentonite extended with CMC	99.5%	0.5%

The fluids were prepared by mixing the powder with tap water using a mechanical mixer Giorgio Bormac AM 20-D ARGOlab with a 70 mm diameter cowless rotating stirrer positioned about 2 cm from the bottom of a 5 L plastic can with a 17 cm diameter at 1500 rpm for 5 minutes. The amount of water used was 2000 L. It is well known that the water composition is a key factor on the bentonites slurry viscosity (Ece et al., 1999). Therefore, it was decided to operate with water with well-defined characteristics that were kept as constant as possible over time (Table 2).

Table 2. Acceptance value for the water used in this study.

ID	Unit	Range/Value
pH		7 - 8
Hardness	°F	10 - 20
Salinity	%	0
Conductivity	µS/cm	150 – 950

2.2 *Methods*

2.2.1 *Bentonite powder characterization*

The bentonite underwent characterization, including evaluations of Atterberg limits, water content, dry residue over 75 µm, swelling index, and mineralogical composition.

Liquid limit values were determined with the Casagrande apparatus, following the ASTM D4318 (ASTM-D4318-17, 2017), in duplicate.

The water content of the powder was measured using a thermobalance Gibertini mod. crystaltherm, at 105°C until constant weight of the dry sample. The dry residue over 75 μm was determined on the dry powder weighting the residue retained over a standard sieve of 75 micron (200 Mesh), and calculated as (1):

$$dry\ residue\ \% = \frac{\mathrm{W_{residue}}}{\mathrm{W_0}} \cdot 100 \tag{1}$$

where $\mathrm{W_{residue}}$ = residue weight; $\mathrm{W_0}$ = initial weight.

The wet residue was determined on a 5% solution of bentonite, passed through a standard sieve of 200 Mesh. The wet residue (2) was dried overnight at 105°C and weighted to obtain an indication on the insoluble minerals fraction, using the following formula:

$$wet\ residue\ \% = \frac{\mathrm{W_{dried\ residue}}}{\mathrm{W_0}} \cdot 100 \tag{2}$$

where $\mathrm{W_{dried\ residue}}$ = dried residue weight; $\mathrm{W_0}$ = initial weight.

The swelling index was determined following the standard ASTM D5890 (ASTM-D5890, 2019).

The X-ray powder diffraction (XRD) pattern of the natural Na-bentonite powder and Ca-bentonite powder was recorded on a Philips X'Pert diffractometer (PANalytical B.V.). The diffractometer operated at 40 kV and 40 mA in a continuous scan mode in the 2θ range from 5° to 90°, with a step size of 0.02° and counting time of 2 s. The monochromatic radiation adopted was CuKα1. The crystalline phases in the resulting diffractograms were identified through the COD database (S. Graulis et al. 2009). X-ray fluorescence (XRF) characterization was conducted to determine the oxides composition of the samples (SiO_2, Al_2O_3, Fe_2O_3, CaO, MgO, Na_2O, K_2O, SO_3). The elemental content of the clay samples was obtained through XRF analysis. The analysis was carried out using an Energy Dispersive X-ray Spectroscopy (EDS) system, model OXFORD, connected to a Scanning Electron Microscope (SEM).

The Loss on Ignition (LOI) value was determined by heating at 1000°C in a muffle furnace for 6 hours. Ceramic crucibles were used, and weighing was performed both before and after the process. Approximately 0.5 g of ground powder was used for the analysis. The cation exchange capacity (CEC) was measured according to D.M.185/99 (D.M. 185,1999), with barium chloride and triethanolamine.

2.2.2 *Bentonite fluids characterization*

The bentonite fluids, prepared at different bentonite concentrations, were characterized through the Marsh cone test immediately following the active mixing phase (T0) (it was noted, although the data is not shown here, that this value remained nearly constant over 24 hours of monitoring for both bentonites, with only a slight increase of Marsh viscosity of less than 5%). This procedure was designed to evaluate the impact of varying mixing parameters on the hydration behavior of the bentonite slurry.

3 RESULTS AND DISCUSSION

3.1 *Bentonite characterization*

Table 3 summarizes the characteristics of the bentonite powders utilized in this study for fluid preparation. As anticipated, the elevated sodium content in Ben-Na accounts for its increased water absorption capacity, resulting in a higher swelling index compared to Ben-Ca. This difference arises because Ben-Ca contains higher concentrations of calcium and magnesium, which are less inclined to absorb water compared to sodium (Muhammad et al. 2021). Consequently, the measured liquid limits of Ben-Na and Ben-Ca reflect these typical behaviors associated with sodium and calcium bentonites, respectively (Dananaj et al. 2005).

Additionally, the presence and concentration of other elements in the bentonite powders vary based on their geological origin (Table 4). These additional elements, which can include trace metals and other minerals, are influenced by the specific source of the bentonite and its formation conditions. This variability in elemental composition can impact the overall properties and performance of the bentonite in different applications. For example, sodium bentonites are essential for drilling and water treatment, thanks to their high water retention capacity, while calcium bentonites are key in clarifying wines and fruit juices or in foundry processes. However, iron-rich bentonites are unsuitable for ceramics due to their brownish hue, and quartz-rich bentonites may not meet the requirements of cosmetics or pharmaceuticals industry.

Table 3. Characteristics of the two natural bentonite samples.

	Unit	Ben-Na	Ben-Ca
Type of bentonite		Sodium	Calcium
Physical state		Powder	Powder
Colour		Brown	White
Water content	%	12.3	13.7
Dry residue over 75 microns	%	16.4	6.1
Wet residue over 75 microns	%	3.8	3.0
Swelling index	mL/2g	25	5
Liquid limit	%	565	120
Loss on ignition	%	14.6	14.0

Table 4. Elemental analysis of the two natural bentonite samples.

	Unit	Ben-Na	Ben-Ca
SiO_2	%	47.7	64.1
TiO_2	%	0.9	0.2
Al2O_3	%	14.7	13.6
Fe_2O_3	%	11.5	1.8
MnO	%	0.2	0.1
MgO	%	3.4	2.6
CaO	%	2.8	1.9
Na_2O	%	3.2	0.6
K_2O	%	0.5	0.6
SO_3	%	0.3	0.4
Cl	%	0.2	0.1
Si/Al	%	3.2	4.7

The results shown in Table 4 represent the typical oxide composition of bentonite powders, with the Na^+/Ca^{2+} ratio specifically confirming the classification of Ben-Na as sodium-based (ratio of 1.14) and Ben-Ca as calcium-based (ratio of 0.32) (Abu-Jdayil et al. 2011).

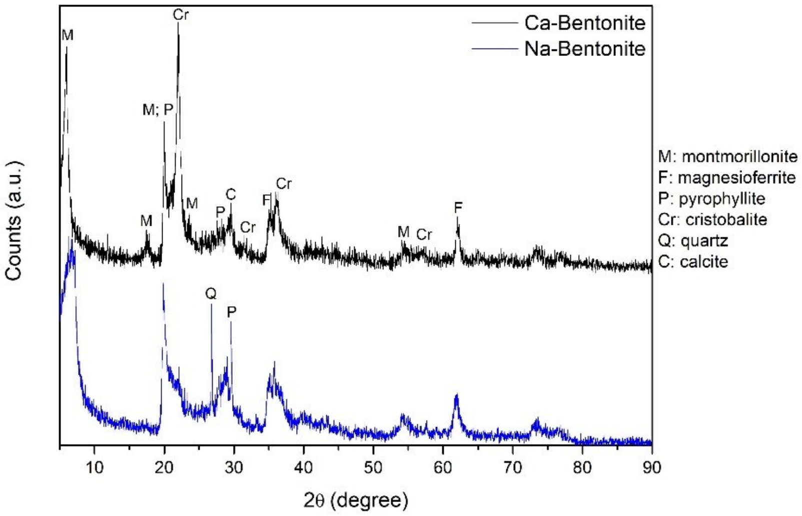

Figure 1. XRD of bentonite powders.

The XRD pattern (Figure 1) revealed that the main clay mineral in the bentonite samples is montmorillonite $[(Na,Ca)_{0,3}(Al,Mg)_2Si_4O_{10}(OH)_2\cdot n(H_2O)]$. Specifically, in Ca-bentonite, the reflections at 5.88°, 17.72° and 19.8° were attributed to montmorillonite containing calcium as the exchangeable cation $[Al_2CaO_{12}Si_4]$, while in Na-bentonite a shift in these characteristics peaks occurred due to the substitution of the interlayer cation with Na. Additionally, cristobalite $[SiO_2]$, pyrophyllite [$Al_2Si_4O_{10}(OH)_2$], magnesioferrite [$MgFe_2O_4$], and calcite [$CaCO_3$] were identified. However, in the Na-Bentonite sample, quartz [SiO_2] was detected, while no significant presence of calcite was observed. In Ca-montmorillonite, swelling is limited to the crystalline phase because the interlayer forces remain strong enough to prevent osmotic swelling. However, in Na-montmorillonite, both crystalline and osmotic swelling occur, as the weaker interlayer attraction after crystalline swelling allows osmotic swelling to take place. As a result, montmorillonite with a higher sodium content in its exchangeable complex exhibits greater swelling capacity compared to those with a higher calcium content. Therefore, bentonites with greater water absorption capability necessitate a smaller quantity of bentonite powder in the water mixture to attain a specific viscosity. Nonetheless, pure bentonites with exceptionally high montmorillonite content are rare and costly. As Madsen & Muller-Vonmoos (Madsen et al. 1989) documented, the card-like structure of bentonite sol requires time to expand, and the particles need more time to disperse and swell. Consequently, the increase in viscosity occurs gradually over time.

3.2 *Effect of the additive extension on Marsh viscosity*

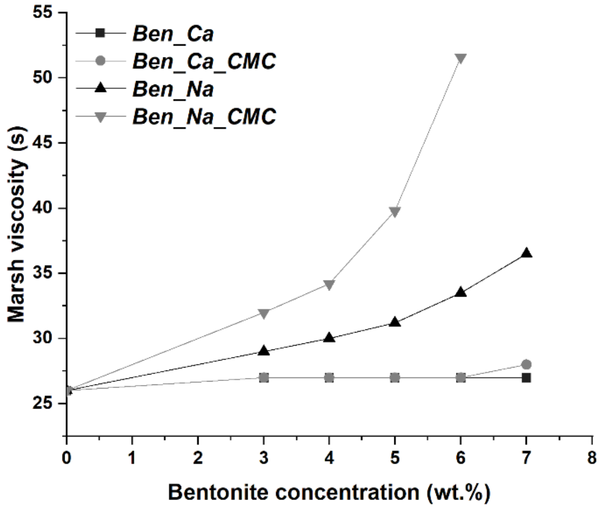

Figure 2. Marsh viscosity of bentonite fluids with and without 0.5 wt.% of CMC.

Figure 2 presents the Marsh viscosity results, illustrating viscosity trends at the different bentonite concentration (wt.%) measured at T0. As previously mentioned and consistent with the characteristics discussed earlier, the analysis of the "pure" bentonite powders shows different flow behaviors, with viscosity increasing as the bentonite concentration rises. Specifically, Ben-Ca demonstrates a class I trend (pseudo-constant), while Ben-Na shows a class III trend (exponential), based on the classification by Besq, which utilizes the Marsh funnel test to categorize bentonites according to their hydrodynamic and structural properties (Besq et al. 2003).

A notable finding is observed when comparing these trends to those of formulations containing CMC. The addition of CMC to the bentonite fluids leads to an increase in Marsh viscosity, due to CMC chains interacting with and opening the interlayers between bentonite particles by adhering to their surfaces (Feddersen et al.1993). This effect is evident in the Ben-Na formulation, consistent with existing literature that shows a concentration of CMC (0.5 wt.%) forming a colloidal network that significantly enhances shear stress and viscosity (Abu-Jdayil et al. 2014).

In contrast, this effect is not observed with Ben-Ca. The amount of CMC added to Ben-Ca is insufficient to alter Marsh viscosity values, which can be attributed to the significantly different properties of the two types of bentonites. Specifically, Ben-Ca exhibits a swelling index and liquid limit that are 80% and 79% lower, respectively, compared to Ben-Na, as shown in Table 2. This suggests that in the calcium bentonite, the polymer chains are unable to penetrate the clay interlayers effectively (Benchabane et al. 2006). The limited interaction between CMC and the clay platelets in Ben-Ca restricts the enhancement of viscosity, highlighting the importance of bentonite type in determining the effectiveness of polymer additives.

4 CONCLUSIONS

Various experimental analyses were conducted to assess the effectiveness of polymer additions in bentonite fluids and to compare the performance of sodium and calcium bentonite fluids under identical preparation and testing conditions. The results indicated significant differences between the two types of bentonite in terms of swelling index and liquid limit. These parameters are useful for predicting how the addition of CMC polymer might enhance viscosity characteristics.

In this study, it was observed that for calcium bentonite, which has a swelling index of 5 mL/2g and a liquid limit of 120%, the Marsh viscosity trend remains pseudo-constant. Here, a 0.5 wt.% concentration of CMC is insufficient to produce a noticeable increase in viscosity. Conversely, sodium bentonite, with a swelling index of 25 mL/2g and a liquid limit of 565%, exhibits an exponential Marsh viscosity trend. For this type of bentonite, the same amount of CMC significantly improves viscosity. This enhancement is attributed to both the chemical nature of the bentonite and its ability to interact with the CMC, facilitating polymer penetration into the clay interlayers. The differences observed are explained by the varying chemical properties of the bentonites, which influence how effectively CMC can interact and modify the fluid characteristics. Moving forward, future research will focus on exploring different types of sodium bentonite with varying chemical compositions and investigating the impact of different CMC concentrations. This will provide a deeper understanding of how the CMC dosage and the properties of bentonite powders influence the rheological properties of bentonite fluids.

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