

Experimental study of rheological characteristics of bentonite-based drilling fluids

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ABSTRACT: Bentonite and polymer-based drilling slurries are widely used in numerous geotechnical applications such as Slurry Shield TBM tunnelling, MicroTunnelling, Horizontal Directional Drilling and other trenchless technologies, piles and diaphragms constructions. In all cases, the rheological characteristics of the fluids must be correctly adjusted to ensure the stability of the excavation, proper lubrication and transportability of the excavated material. To date, there are several international standards and many best practices, empirical abacuses and indications for the definition of rheological properties of drilling fluids. However, there is often a lack of correlation between the results of different tests or measurements that can be carried out in laboratories using rheometers, viscometers and other sensitive and accurate equipment, and the rapid measurements that are widely used on site. The study presented in this paper involves the systematic performance of laboratory tests and rapid measurements on a natural bentonite used in the construction of tunnels and other underground works. The aim is to provide a broad overview of the rheological characteristics of drilling fluids prepared at different bentonite dosages, a series of correlations between the results of laboratory tests carried out using different mixing speed and mixing time. The results of the study support a better selection of the correct properties during drilling, an informed choice of the most appropriate measuring instruments and a more accurate measurement and control of the rheological properties of drilling fluids.

Keywords: Excavation, Viscosity, Marsh, Mixing, Rheology, Slurry

1 INTRODUCTION

Bentonite and polymer-based drilling slurries are used extensively in various geotechnical applications such as Slurry Shield TBM tunnelling, Micro-tunnelling, Horizontal Directional Drilling and other trenchless technologies, piles and diaphragms constructions. The crucial aspect in all these applications is to appropriately adjust the rheological properties of the fluids. This adjustment is necessary to guarantee excavation stability, adequate lubrication and the efficient transportation of the excavated materials.

Drilling fluids can be oil-based, synthetic-based and water-based: such fluids have different characteristics but, among them, water-based drilling fluids

are environmentally friendlier and cheaper than oil-based and synthetic ones (Liu et al., 2022).

Bentonite fluids are a particular type of water-based drilling fluids obtained by mixing water and bentonite, a natural volcanic clay. These fluids are commonly employed in tasks involving soil excavation as they are able to stabilize the hole walls by creating a protective barrier and ensuring the transportability of the excavated material (Chiarelli, 2014).

The essential element in the preparation of these fluids lies in the interactions between clay and water. When dry clay, which is composed of stacked plate-like structures, comes into contact with water, it undergoes a series of transformations. These transformations involve various stages, including hydration, dispersion, gelation,

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aggregation, and flocculation, as explained by Muller-Vonmoos et al. (1989).

Furthermore, these fluids typically exhibit pseudoplastic behaviour, although there are exceptions. Pseudoplasticity means that they require the application of a certain level of stress, known as the yield point or yield stress (represented as τ_0), to initiate flow. Below the yield stress the sample does not flow but behaves elastically (Steffe, 1996). When forces exceed the shear stress, the behaviour of the material can exhibit either pseudoplastic or Newtonian characteristics, as illustrated in Figure 1. Pseudoplasticity can be better understood by considering that the system is composed of molecules that can arrange themselves into lattice structures. To disrupt or “break” these structures, stress levels equal to or exceeding the yield point are required. This means that the material can transition between a solid-like and a liquid-like state depending on the applied stress. Whether this plasticity is reversible or not depends on the material capacity to re-establish the lattice structure after deformation (Luckham and Rossi, 1999).

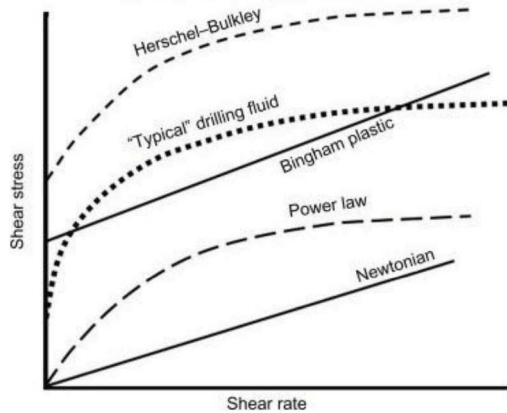


Figure 1. Shear stress vs shear rate and main rheological behaviour of fluids (modified after Braun and Rosen, 1999).

The concentration of bentonite in fluid formulations is restricted because excessive amounts of powder can lead to issues such as the creation of a thick filter cake and formation damage. To address these challenges, the inclusion of polymers and other additives can be beneficial (Saboori et al., 2019). These additives serve multiple purposes, including the achievement of a thinner filter cake on the wellbore wall, reducing the potential for damage and fluid loss, and improving the rheological properties of the fluid (Song et al., 2016).

The intricate interaction between clay and water is of the utmost importance in the formulation of bentonite fluids. Its understanding plays a crucial role in tailoring these fluids for diverse applications, ranging from their use as drilling fluids in the oil industry to soil stabilization in civil engineering projects. The literature contains some information, including results from experimental tests and theoretical explanations,

that shed light on the rheological properties of bentonite drilling fluids (Etehadhi et al., 2022).

This study aims to offer supplementary insights into the rheological properties of bentonite drilling fluids. In particular the main objectives are: i) the investigation of the influence that mixing parameters adopted on site, such as time and rpm (round per minute), have on the properties of bentonite drilling fluids and ii) the definition of a correlation between the apparent viscosity and the results of easily accessible tests such as the Marsh cone, offering a valuable way to swiftly assess the parameter without resorting to more advanced instruments like rotational rheometers.

2 MATERIALS AND METHODS

To investigate the rheological properties, a natural bentonite was selected and different fluid samples were prepared by mixing it in varying concentrations with water.

The first step was the bentonite characterization in terms of Atterberg limits, water content, dry residue over 75 μm and swelling index; then different fluids were prepared adopting different mixing conditions, varying mixing speed and mixing time, and tested in terms of rheological parameters such as Marsh viscosity, API filtrate and filtercake. Also, a rheological analysis with a rotational rheometer was performed.

In the following paragraphs the testing methods and the models used are described.

2.1 Mixing and concentration

2.1.1 Bentonite sample characterization

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 200 Mesh, and calculated as (1):

$$\text{dry residue \%} = \frac{W_{\text{residue}}}{W_0} \cdot 100 \quad (1)$$

where W_0 is the initial weight of the powder, and W_{residue} is the residue retained after the sieving.

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:

$$\text{wet residue \%} = \frac{W_{\text{driedresidue}}}{W_0} \cdot 100 \quad (2)$$

where W_0 is the initial weight of the powder, and $W_{\text{dried residue}}$ is the residue retained after the sieving of the slurry and dried overnight.

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

The pH of the bentonite slurry was determined using a pH test strip.

2.1.2 Bentonite slurries preparation

Different bentonite fluids were prepared at different rpm and mixing times using an orbital mixer Giorgio Bormac AM 20-D ARGOLab with a three-blade rotating stirrer positioned about 2 cm from the bottom of a 5 L plastic can with a 17 cm diameter. Every sample was prepared filling the can with tap water weighing 2500 g. The study can be divided into two main phases. The two different phases involved different concentration of bentonite as well as mixing parameters. In the first phase it was investigated the effect of different mixing parameter (Table 1) on the bentonite slurries properties described from the American Petroleum Institute (API-13B-RP1, 2015).

Table 1. Mixing parameters.

Bentonite weight %		5% wt	6% wt	7% wt
Fixed time: 15 min	Mixing speed	750	✓	✓
	(rpm)	1000	✓	✓
		2000	✓	✓
Fixed rpm: 1500	Mixing time	5	✓	✓
	(min)	10	✓	✓
		15	✓	✓

For the second phase the mixing parameters were fixed, (Table 2) varying only the bentonite concentration.

Table 2. Second phase mixing parameters.

Bentonite weight %		6.0% wt	6.5% wt	7.0% wt	7.5% wt
Fixed time: 20 min	Fixed rpm: 1500	✓	✓	✓	✓

2.2 Rheological tests

The bentonite slurries obtained at different dosages were subjected to the Marsh cone test at $t = 0$ h (immediately after the active mixing phase, T_0) after 1 hour, 3 hour and 24 hours of steady state. This assessment aimed to observe any distinct effects on the hydration phase of the bentonite slurry due to the different mixing parameters specified. The Marsh cone test was conducted following the guidelines outlined in API 13B-RP1 specifications. The retention capacity of the bentonite slurries was determined

by assessing the fluid loss both immediately after T_0 and after a 24-hour period, following the guidelines outlined in API 13B-RP1 specifications.

The rheological characteristics of bentonite fluids have been analysed using a rotational rheometer. A rotational rheometer is an instrument used to study the rheological properties of various types of fluids. These fluids are placed between a rotor and a stator, which can have different geometries. The fluid placed between the rotor and stator is set in motion by the rotation of the rotor. A second device measures the torque required to keep the plate in motion, and from this measurement, the applied stress on the fluid (shear stress, τ) can be determined. Meanwhile, the rotational speed provides information about the shear rate, $\dot{\gamma}$ (Sartori, 2013).

2.3 Common yield stress models for drilling fluids

Aqueous suspensions of clay that possess a relatively high particle concentration have traditionally been described according to Bingham's theory. The Bingham model is the simplest constitutive equation for viscoplastic fluids. Another model extensively adopted to describe the behavior of bentonite fluid viscosity is the the Herschel-Bulkley (H-B) model. This model was used in this study to describe the rheological behaviour of the bentonite slurry.

Typically, the H-B model, described by eq. (3) and (4), is used for many suspensions since Newtonian plastic, shear thinning, shear-thickening and Bingham can be considered as a special case of the model.

$$\tau = \tau_0 + K \cdot \dot{\gamma}^n \quad (3)$$

$$\eta = \frac{K \cdot (\dot{\gamma})^n + \tau_0}{\dot{\gamma}} \quad (4)$$

Where τ is the shear stress; τ_0 is the yield stress or yield point; η is the viscosity; $\dot{\gamma}$ is the shear rate; n is the flow behaviour index and n is the consistency coefficient.

3 RESULTS AND DISCUSSION

3.1 Bentonite characterization

In the first part of the study, we characterized bentonite powder (Table 3), identifying its water content, residue at 75 microns both dry and wet. Regarding the water absorption properties of this bentonite, the liquidity limit and swelling index were analysed. The liquid limit is quite high, standing at around 430%.

The bentonite selected in this study can be categorized as a natural sodium bentonite. Generally, the higher the content of montmorillonite in the natural bentonite, the more the card-like structure can expand, resulting in a higher liquid limit (while the plastic limit remains relatively unchanged), thereby increasing the plasticity index (the range between the liquid limit and

Table 3. Bentonite characterization.

Type	Natural Sodium Bentonite	
Physical state	Powder	
Colour	Yellow	
Water Content	%	13.0
Dry residue over 75 microns	%	16.8
Wet residue over 75 microns	%	3.3
Swelling index	mL	25
Liquid limit	%	430
pH sol. 5% @25°C	9	

the plastic limit). Consequently, bentonite with a higher capacity for water absorption requires less bentonite powder in the water mixture to achieve a specific viscosity. However, pure bentonites (with an exceptionally high montmorillonite content) are rare and expensive. It is well known (Madsen and Muller-Vonmoos, 1989) that due to their card-like structure, bentonites sol takes time to expand, and the particles require more time to disperse and swell. As a result, the viscosity increase over time is slow.

3.2 Mixing optimization

The preparation of a bentonite-based fluid initially involved the adoption of three different dosages of bentonite powder. In Figure 2 the results of Marsh viscosity are reported and the same behaviour on the API filtrate analysis can be observed.

Furthermore, it's important to note that extending the mixing time did not impact either the fluid loss or the thickness of the cake (Figure 3).

On the other hand, as rpm changes (mixing energy), there is a slight decrease in viscosity. The decreasing trend of viscosity with the increase of rpm adopted is in opposition to what expected (Figure 4) probably because the natural bentonite could be characterized by a low Na/Ca ratio as pointed out by the swelling analysis (25 mL). By enhancing the mixing efficiency there is a greater availability of Ca ions into solution. Such ions are recognized to have an adverse effect on the swelling and rheological properties of the bentonite fluid.

Using a typical electric motor curve as an example for a generic mixer, we generated a curve for the laboratory agitator based on the available data. To assess the laboratory findings, we compared them to the standard mixing speed and duration used in the field, taking into account the associated energy consumption taking as reference the previous part. Three scenarios were considered. The first scenario, representing the typical values used for on-site preparation, involved mixing at 1200 rpm for 20 minutes and re-mixing for 5 minutes. In the second scenario, mixing occurred at 1400 rpm for 10 minutes, followed by re-mixing for 2 minutes and 30 seconds.

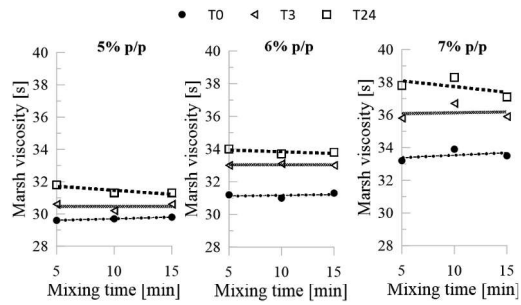


Figure 2. Marsh viscosity comparison immediately after mixing, hydrated 3 hours and 24 hours.

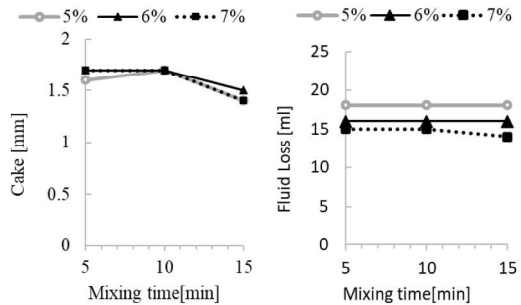


Figure 3. Fluid loss and cake thickness comparison for different %wt, immediately after mixing.

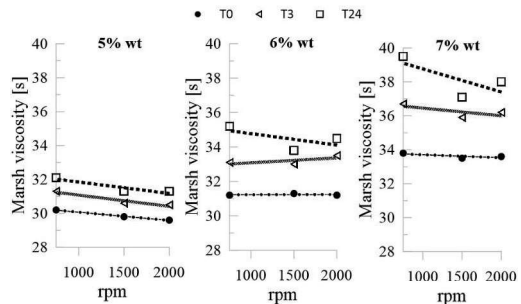


Figure 4. Marsh viscosity comparison immediately after mixing, hydrated 3 hours and 24 hours.

The third scenario involved mixing at 1600 rpm for 7 minutes and 30 seconds, with a subsequent re-mixing time of 2 minutes. It's worth noting that in Italy, the electricity cost for businesses typically falls within the range of 8 to 9 cents per kWh. The resulting percentage savings compared to the on-site preparation procedure are presented in Table 4.

The results show that it is possible to reduce the energy consumption optimising mixing time and mixing speed. It's important to notice that these results are obtained from laboratory apparatuses and they need to be adjusted if compared to field ones, taking into account specific field conditions.

Table 4. Cost options and savings.

	rpm	Time mix [min]	Remix time [min]	Cost [€]	Save [%]
1 st possibility	1200	20	5	0.15-0.17	0
2 nd possibility	1400	10	2.5	0.08-0.09	45%
3 rd possibility	1600	7.5	2	0.06-0.07	58%

3.3 Rheological measurement

To investigate the rheological characteristics of bentonite fluids they were subjected to progressively escalating shear rates, spanning from 250 s^{-1} to 2000 s^{-1} . The primary objective was to analyse the transformation of these fluids, transitioning from a gel-like state to a more liquid state, which occurs when the stress level exceeds the threshold causing the breakdown of the fluid’s network structure, ultimately resulting in a more gel-like consistency. In this instance, the aim was to attain a Marsh viscosity goal of 40 seconds Figure 5.

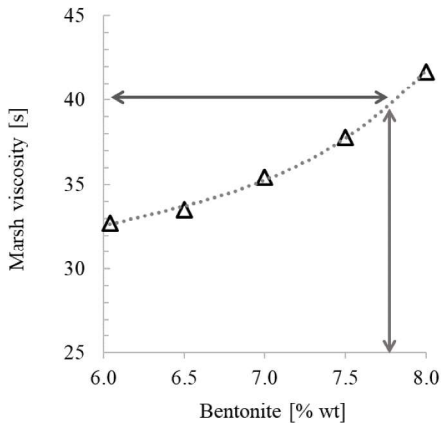


Figure 5. Individuation of the concentration to a marsh viscosity target of 40s.

The Herschel-Bulkley model was chosen as the best fit for characterizing the rheological properties of the studied bentonite fluids. This decision was based on minimizing errors between model-calculated and observed viscosities in experimental data. The suitability of this model is evident when analysing graphs of shear rate changes from 0 to 100 s^{-1} and back to 0 for various bentonite concentrations. The study examined different concentrations to understand how the thixotropic loop varies with increased bentonite powder content. During the decreasing shear rate phase, the Herschel-Bulkley model, supported by several studies (Yang, et al.) (Ren, et al., 2021) (Mellak, et al., 2014), effectively describes the fluids behaviour.

The fitting results, displayed in the Figure 6, demonstrate a strong alignment between the Herschel-Bulkley model and the experimental rheometer data.

An n value of less than 1 reveals the existence of pseudoplastic behaviour, signifying that viscosity rises as the shear rate diminishes.

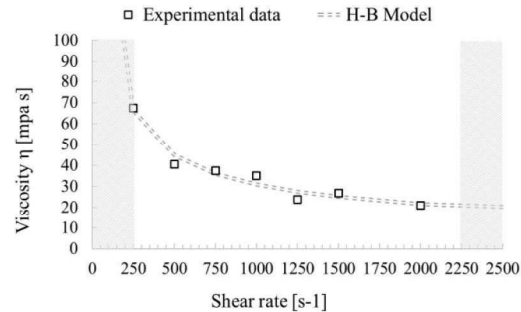


Figure 6. H-B model applied to the experimental data.

Studying bentonite fluid behaviour beyond the initial gel structure breakdown is essential for understanding its thixotropic rheological properties. Traditional models like the Herschel-Bulkley model are insufficient to capture the complex behaviour observed when shear rates are gradually increased and then decreased in thixotropic fluids, leading to the creation of a “thixotropic loop” in shear stress-shear rate graphs (Franceschini, et al., 2022).

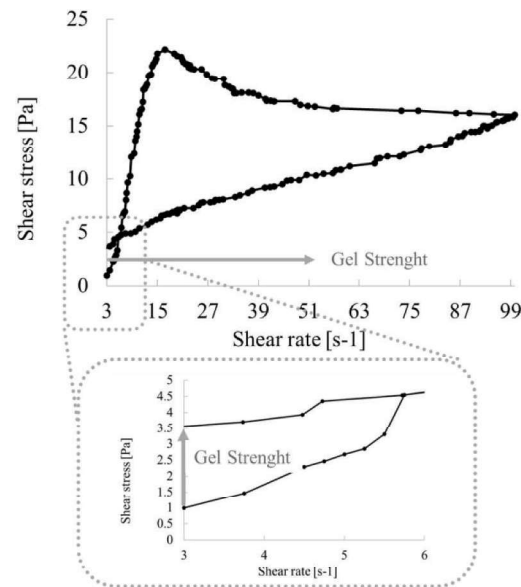


Figure 7. Hysteresis shear rate/shear stress cycle.

In the region of decreasing shear rates, the Herschel-Bulkley model is commonly used and referred to as the “steady state flow” zone. However, when examining the area of increasing shear rates, known as the “transient flow” zone, a different trend emerges (Franceschini, et al., 2022). Here, shear stress initially

risers linearly with shear rate, but as the shear rate continues to increase, shear stress decreases before returning to the steady state flow zone. This phenomenon is more pronounced with higher bentonite concentrations. The combination of transient flow and steady state flow forms the thixotropic loop, and its size grows with increasing bentonite content, indicating the materials degree of thixotropy (Yang, et al.) (Ren, et al., 2021). This degree of thixotropy is a measure of the energy required for bond dissociation per unit time and volume. The magnitude of the hysteresis area in the loop indicates whether the material has a gel-like structure under static conditions, resulting in increased rheological parameters and resistance to creep. In the case of bentonite fluids, the gel structure is related to swelling due to the insertion of water molecules between montmorillonite trilayer elements and particle interactions based on the electrical double layer theory (İşçia, et al.). At higher concentrations, particle aggregation into clusters occurs, forming a more complex network called a flocculate. The arrangements of particles contributing to the gel structure include edge-to-face and edge-edge arrangements, driven by electrostatic attractions or repulsions within the double layer. The transient flow zone is characterized by a “stress overshoot” peak, representing the elongation and resistance of the microstructure before its rupture due to increased shear rates (İşçia, et al.). Unlike the steady state flow zone, the transient flow zone is complex and challenging to describe with models like the Herschel-Bulkley model. It’s worth noting that the fluid cannot fully recover its original characteristics after being subjected to increased and then decreased shear rates, leading to changes in the shear force required for new flow. The hysteresis area increases as the bentonite content in the fluid rises, indicating higher thixotropy (Yang, et al.). Regarding the hysteresis areas (Table 5), as anticipated, their value increases as the bentonite content in the fluid increases because the material is more thixotropic.

Table 5. Values of hysteresis (thixotropy) areas for different concentrations of the Bentonite N.

Concentration	%	6.5	7.0	7.5
Area	J/m ³ s	551	644	651

After conducting a study on the rheological behaviour of bentonite fluids at different concentrations, an attempt was made to establish a correlation between the viscosity values measured with the Marsh cone (expressed in seconds) and the apparent viscosity values obtained with a rheometer. Based on literature studies (Pitt, 2000), it was established that the viscosity measured with the Marsh cone can be considered as the effective viscosity of a fluid subjected to a shear rate on the order of 2000 s⁻¹ (ranging from 1500 to 2500 s⁻¹). Therefore, all four

types of bentonite fluids at varying concentrations were subjected to a rotational rheometer at shear rates of 1000, 1500, and 2000 s⁻¹ to obtain measurements of apparent viscosity (Figure 8).

Subsequently, the obtained data points were analysed to establish a correlation with the Marsh cone times obtained in the previous phase of the study. The theoretical background for seeking a linear correlation is based on existing literature, which suggests that Marsh cone viscosity values expressed in seconds can be transformed into viscosity values (referred to as “effective viscosity”) expressed in cP using the following empirical expression:

$$\mu = \rho (t - 25) \quad (5)$$

Where μ effective viscosity in Centipoise [cP = mPa·s], ρ bentonite fluid density [g/cm³], t time to Marsh cone [s], 25 is a value that refers to the seconds it takes for a fluid with negligible viscosity at any density.

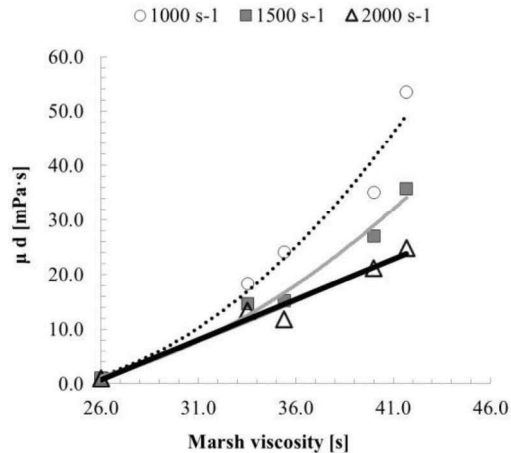


Figure 8. Correlation between Marsh viscosity and effective viscosity (μ) at different shear rate.

The expression suggests a linear relationship between viscosity and Marsh cone time. Table 6 presents the values obtained through rotational rheometer analysis of the optimally concentrated bentonite fluid, subjected to shear rates of 1000 s⁻¹, 1500 s⁻¹, and 2000 s⁻¹, resulting in apparent viscosity measurements.

Table 6. Apparent viscosity and Marsh time results for natural bentonite.

Shear rate [s ⁻¹]	Apparent viscosity [mPa s]	Marsh viscosity [s]
1000	35	40.0
1500	27	40.0
2000	21	40.0

4 CONCLUSIONS

The primary aim of this study was to further the body of knowledge related to the rheological properties of bentonite-based drilling fluids.

First, it was evaluated how operative conditions adopted during the mixing step as time and rpm influence the properties of bentonite drilling fluids. The main outcomes were that Marsh viscosity was almost constant at the increase of mixing time, similarly to the results of the fluid loss and the cake thickness. This allows to reduce the energy consumption associated with prolonged mixing operations.

Secondly, a good fit between the Herschel-Bulkley model and the experimental rheometer data was obtained. The n value lower than 1 revealed the occurrence of pseudoplastic behaviour as signifying that viscosity rises as the shear rate diminishes. The hysteresis area increased as the bentonite content in the fluid raised, indicating higher thixotropy of the materials.

Finally, a correlation between the apparent viscosity and the results of Marsh cone tests was investigated, offering a valuable way to swiftly assess the parameter with easily accessible tools.

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