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FULL LENGTH ARTICLE

Evaluation of Egyptian bentonite and nano-bentonite as drilling mud

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KEYWORDS

Drilling fluids;
Nano-bentonite;
Rheological properties

Abstract Nano-particles of bentonite have been prepared by nano-grinding. The nano-bentonite was characterized by X-ray fluorescence analysis (XRF), X-ray powder diffraction (XRD), thermal gravimetric analysis (TGA) and Transmission electron microscopy (TEM). The bentonite particles had been ground to the size ranging from 4 to 9 nm. Both natural and nano-bentonite were evaluated as drilling mud. The evaluation involved the study of the rheological properties, filtration and gel strength before and after treatment with viscosities and filter loss agent, and compared with the American Petroleum Institute API bentonite. With decreasing the grain size of bentonite to the nano-scale, the results were not satisfied to the API -standard.

1. Introduction

Drilling fluids are primary water–bentonite suspensions, they are important for the oil, gas and geothermal drilling industry because they perform many functions like transporting rock cuttings to surface, lubricating the drill bit, applying hydrostatic pressure in the well bore to ensure well safety and minimizing fluid loss across permeable formations by forming a filter cake on the wells of the well bore [1–15].

Bentonite is formed by the weathering of volcanic ash. The weathering process, by which the clay minerals are formed from the parent minerals is complex. The main factors are climate, topography vegetation, and time of exposure [16–20].

Bentonites are used worldwide as drilling fluid additives. Their main functions are the viscosities of the mud in order to reduce the fluid loss to the formation. A good quality bentonite should contain mainly montmorillonite [21–25]. In practice, bentonites often contain other clay minerals such as elites, kaolinites, chlorites and non-clay components such as quartz and feldspar in appreciable amounts. Because montmorillonitic clays have the highest swelling capacity (which is responsible for viscosity build up and formation of low permeability filter cake), the presence of other materials will have an adverse effect on bentonite quality. The type of exchangeable ions has a great effect on the swelling capacity of the montmorillonite. By far the best performance is obtained with sodium montmorillonite. If the mineral composition of bentonite is such that its viscosifying power is insufficient, various additives can be added. The additive can be either a salt or a polymer and enhances of the fluid by slightly flocculating the bentonite was suspension. It was shown that sufficient sodium is required

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to obtain a well dispersed bentonite suspension. Also it is found that the physical properties of bentonites are affected by the Na/Ca ratio and that bentonite swelling was readily improved by the addition of small amounts of sodium carbonate. In practice, magnesium oxide was commonly used to enhance the gel strength [26–30].

Different types of chemicals and polymers were used in designing the drilling mud to meet some functional requirements such as the appropriate mud rheology, density, mud activity, fluid loss control property [31–34].

The selection of additives must take account of both the technical and environmental factors. Poly acrylamide and modified starch were frequently used at the early stage of drilling for depths equivalent to 150 °C bottom hole temperature.

In this research local bentonite and nano-bentonite were studied before and after treatment compared to the API bentonite.

2. Experimental method

2.1. Sample collection and preparation

The field sampling exercise was carried out during the dry season. Fresh samples of the clay were collected. The samples were crushed to finer particles and sundried for 5 days to ease pulverizing and sieving. They then were ground to powder with the aid of mortar and pastle, and then sieved with a rota shaker to obtain 63 μ fractions to Suit API specification for local bentonite (B) [34,35]. Nano-bentonite (BN) was prepared by crushing the local bentonite with the aid of a planatery ball mill PM 400, see Fig. 1.

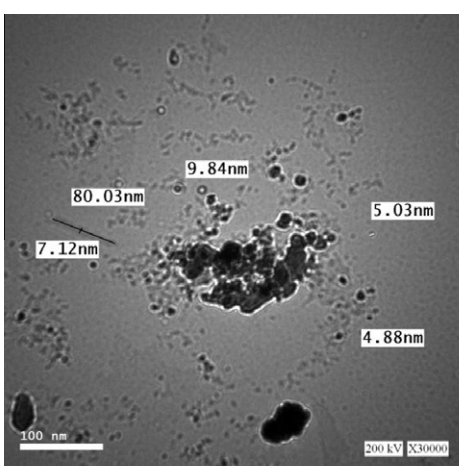

Figure 1 TEM for nano-bentonite.

Table 1 X-ray diffraction analysis for local bentonite and nano-bentonite.

Constituents	Local bentonite	Nano-bentonite
Major const.	Montmorillonite, Quartz	Montmorillonite, Quartz
Minor const.	Kaolinite	Kaolinite
Trace const.	–	–

2.2. Mineralogical studies

Mineralogical composition for both the local bentonite and nano-bentonite was determined by X-ray diffraction (XRD) using a philips X-ray diffraction equipment model Pw 710 with mono chromator, cu radiation (h = 1.542 A°) at 40 kV, 35 mA. and scanning speed 0.02°/s. The reflection peaks between 2ø = 2° and 60°, corresponding spacing (d, A°) and the relative intensities (I/I°) were obtained [36]. The diffraction charts and the relative intensities were obtained and compared with ICDD files Table 1.

2.3. Chemical analysis

X-ray fluorescence (XRF), for the studied samples was carried out to determine the chemical composition by (XRF) Spectrometry [37] and the results were listed in Table 2.

2.4. Thermal analysis

Thermo gravimetric analysis (TGA) was done by means of a thermo gravimetric analyzer (TG–DSC TAQ600) [38]. The thermal behaviors of the samples were recorded in the chart Figs. 2 and 3.

2.5. FT-IR spectrum analysis

FT-IR Spectrum analysis for the studied local bentonite and nano-bentonite was carried out by using NICOLET. FT-IR IS-10 [39,40], as shown in Figs. 4 and 5.

2.6. Mud formulation

2.6.1. Nontreated mud

The mud formulation consists of three mud patches for local nontreated bentonites (B) and nano bentonite (B_N) compared to the API bentonite (B_0). Each patch was prepared by adding 22.5 g bentonite to 350 ml water and mixed for 20 min in a mixer [41–43].

2.6.2. Treated mud

The mud formulation consists of three mud patches for local treated bentonites (B) and nano-bentonite (B_N) compared to the API bentonite (B_0). Each patch was prepared by adding

Table 2 Chemical analysis for local-bentonite and nano-bentonite.

Elements	Local-bentonite %	Nano-bentonite %
SiO_2	54.91	54.45
TiO_2	1.53	1.70
Al_2O_3	17.01	16.42
Fe_2O_3	9.31	10.25
MnO	0.08	0.10
MgO	2.47	2.16
CaO	0.99	1.03
Na_2O	2.75	2.33
K_2O	1.03	1.12
P_2O_5	0.16	0.42
Cl	1.20	0.79
SO_3	0.48	0.15
L.O.I 250 °C	–	–
L.O.I 1000 °C	8.06	9.00

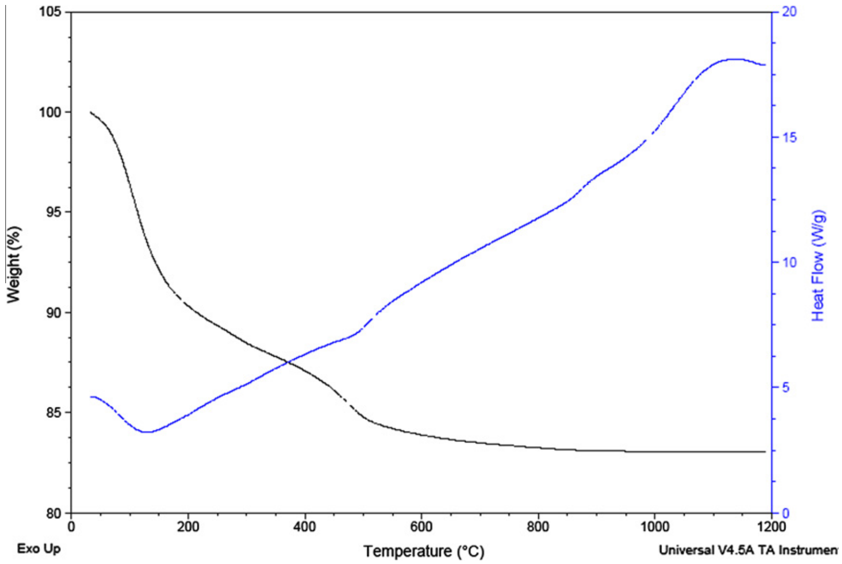

Figure 2 DSC TGA for local bentonite.

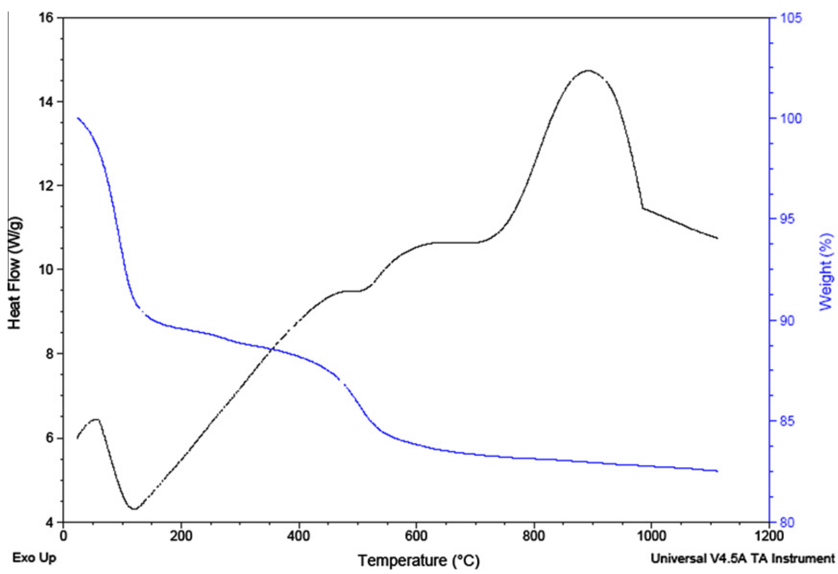

Figure 3 DSC TGA for nano-bentonite.

22.5 g bentonite to 350 ml water and 0.1% of poly anionic cellulose was mixed for 20 min in a mixer.

2.7. Mud testing

2.7.1. Rheological properties

Apparent viscosities (AV), plastic viscosity (PV), yield point (YP) and gel strength were measured according to API specifications (1998) by using the Fann V-G meter viscometer (Chan 35 Model 35 SA). A good bentonite for drilling purpose will have an apparent viscosity of at least 15 cp which is the viscometer dial reading/2 at 600 rpm, as shown in Figs. 6 and 7.

2.7.2. Filtration test

API fluid loss test was carried out using a filtration apparatus (API Filter press ofite apparatus) by placing the mud slurry (after 24 h) into a stainless steel chamber with an opening at the bottom. A filter paper was placed on the bottom. The slurry was exposed to 80–100 psi for 30 min and the amount of water was measured. A good bentonite for drilling purpose will have a fluid loss of 15 ml or less; as indicated in Figs. 8 and 9.

2.7.3. Yield

Bentonite yield was determined according to OCMA (Oil Companies Materials Association) specifications; API 15 cp considered apparent viscosity is measured to be an acceptable yield value which corresponds to 100.4 barrels per ton mud slurry, as mentioned in Table 4.

2.7.4. Swelling test

The swelling test consists of sprinkling 1.4 g of bentonite into a 100 ml graduated cylinder filled with distilled water over a per-

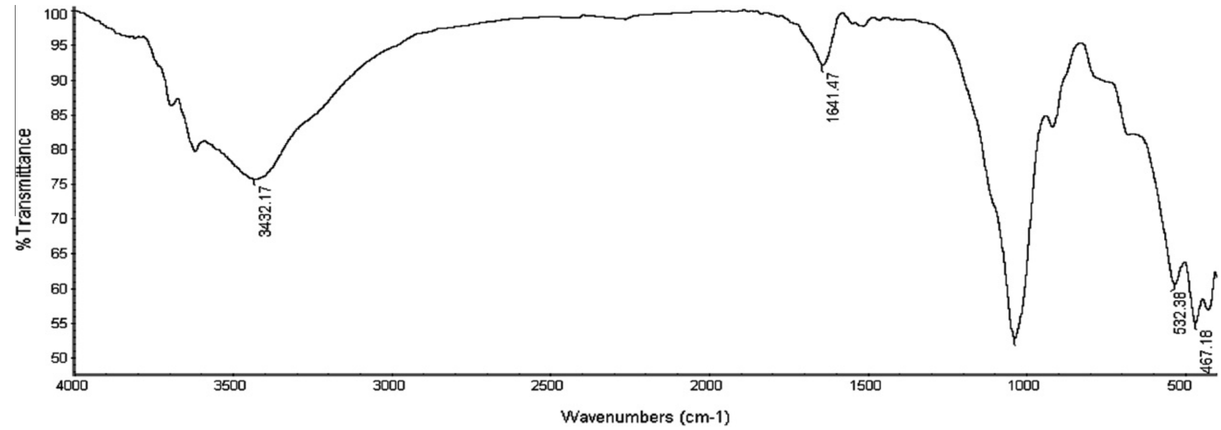

Figure 4 FT-IR for local-bentonite.

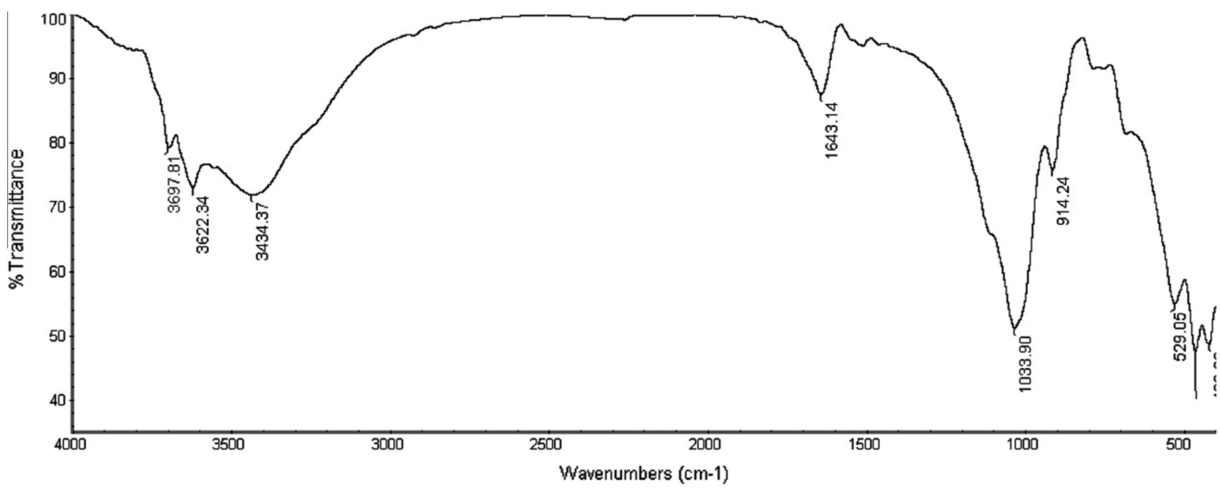

Figure 5 FT-IR for nano-bentonite.

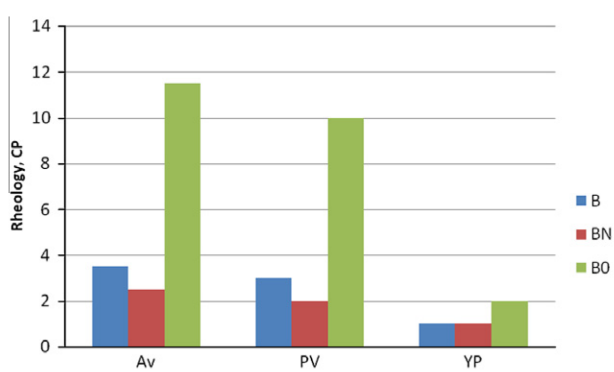

Figure 6 Rheology of local nontreated bentonite (B) and nano-bentonite (B_N) compared to the API bentonite (B_0).

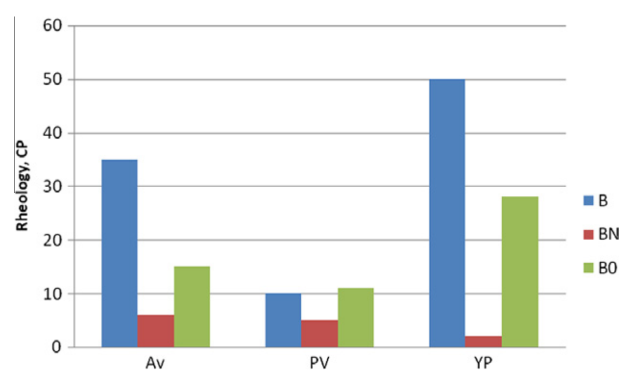

Figure 7 Rheology of local treated bentonite (B) and nano-bentonite (B_N) compared to the API bentonite (B_0).

iod of 8 h; the swollen bentonite was measured after 72 h. The degree of swelling or swelling ratio was obtained by dividing the total volume measuring after swelling test to the original volume of the bentonite. The original volume corresponded to the volume (free volume) of 1.4 g bentonite placed in the graduated cylinder, as shown in Table 4.

3. Result and discussion

3.1. Size characterization of bentonite

Transmission electron microscopy, TEM, provides a unique opportunity to directly visualize nano-particle morphology.

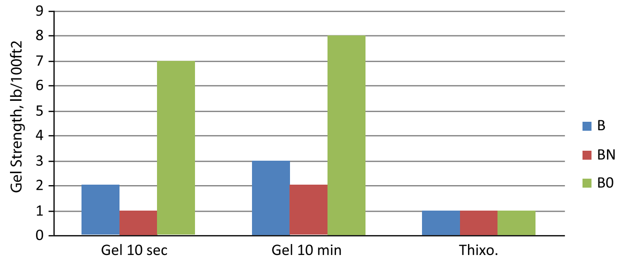

Figure 8 Gel strength of local nontreated bentonite (B) and nano-bentonite (B_N) compared to the API bentonite (B_0).

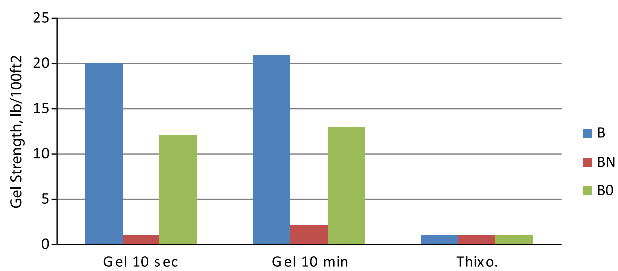

Figure 9 Gel strength of local treated bentonite (B) and nano-bentonite (B_N) compared to the API bentonite (B_0).

Despite requiring good skills and being labor intensive, various images were captured and this is done at various magnifications, these techniques can provide a representative perception of the morphology being analyzed. Therefore, the present study aims to take advantage of these techniques to better understand the dispersion mechanisms of layered bentonite in drilling mud, as well as to investigate the compatibilization effect of nano-bentonite on the physical properties of the mud fluids.

3.2. Mineralogical studies

Mineralogical analysis for local bentonite and nano-bentonite showed that they are essentially montmorillonite as expected [44].

XRD patterns of the samples in air dried state indicated that the main constituents are montmorillonite, Quartz and Kaolinite in decreasing order of their abundance as shown in Table 1.

3.3. Chemical analysis

XRF Values obtained in XRF analysis showed that the Al_2O_3/Sio_2 ratio was 1/3 as expected for montmorillonite which is the main component of bentonite under study. The ratio of {(Na_2O + K_2O)/(CaO + MgO)} for the two samples was found to be 1.08% confirming that the samples were Na-bentonite [45]. Table 2 illustrates the chemical composition for local bentonite and nano-bentonite.

3.4. Thermal analysis

3.4.1. Local bentonite

The TGA–DSC showed that the adsorbed water was lost from the clay up to about 130 °C. After this, physico-adsorbed loss occurs in two steps, the steps being below and above 500 °C. The weight loss of 10.2% was lost at 130 °C, while ~6% was lost at 525 °C. In addition, about 2% was lost at temperature range of 600–1200 due to surface dehydroxylation and loss of non-chemically attached metal oxides.

DSC curve showed two endothermic peaks resulted from loss of adsorbed water and the second from loss of water of crystallinity.

3.4.2. Nano-bentonite

The clay thermal stability was analyzed in an inert atmosphere under nitrogen condition at a heating rate of 10 °C/min^{-1} from ambient temperature to 1200 °C under flowing N_2 atmosphere on a Thermo gravimetric Analyzer (TG–DSC TAQ-600). A weight loss centered at 130 °C (~10%) caused by the loss of clay-adsorbed water and a weight loss centered at 450 °C, probably caused by self-dehydroxylation of –OH group on the surface of the clay. About 1% was observed in the range of 500–1200 °C due to the evaporation of adsorbed metal like MnO and MgO.

The TG curves of the clay samples can be divided into three parts according to the main substance that participated in the mass loss on the TGA curves: (1) temperature below 300 °C at which the clay adsorbed water was lost, (2) at temperatures between 300 and 800 °C, the released lattice water gas and the liberation of some SO_3 gases from clay were lost (3) in addition at the temperature between 500 and 1200 °C the metal oxides that were physically attached on the surface and on the cage clay surface particles were lost.

3.5. FT-IR data

The FT-IR spectra of the bentonite fractions were measured in transmittance between 500 and 4000 cm^{-1}. Two bands at 362 cm^{-1} and 914 cm^{-1} are corresponding to dioctahedral semectites. The band at 3697 cm^{-1} corresponds to semectite. The Al–Al–OH stretching vibration of the octahedral semectite is observed bands at 529 cm^{-1} and 440 cm^{-1} were observed for Si–O–Al and Si–O–Mg indicated tetrahedral bending modes. Al–Al–OH at 914 cm^{-1} and also OH bending vibrations of Kaolinite and illite, band at 790 cm^{-1} for Mg–Fe–OH. The bands at 3697 cm^{-1} and 790 cm^{-1} (Al–OH–Mg) bands and the weak band at 770 cm^{-1} (Fe^{+3}–OH–Mg) band indicate that the semectite was containing Mg and Fe^{3+}.

3.6. Mud evaluation

3.6.1. Rheological properties

Rheology of water-based mud formulated with local-bentonite (B), and nano-bentonite (B_N) compared to water-based mud formulated with standard (API) bentonite (**B_0**) were studied before and after treatment and illustrated in Figs. 6 and 7. Testing results of rheology indicated the following:

Apparent viscosity (AV) for local-bentonite increased from 3 to 35 cp after treatment which is higher than 15 cp as API requirements. In the case of nano-bentonite the apparent viscosity slightly increases from 2.5 to 6 cp which is lower than API standard.

Plastic viscosity (PV) for local bentonite increased from 3 to 10 cp after treatment which is satisfying the API standard. Whereas; nano-bentonite slightly increased from 2 to 5 cp which is lower than the API standard.

Yield point (YP) for local-bentonite varies from 1 to 50 lb/100ft^2 after treatment whereas; nano-bentonite slightly increased from 1 to 2 lb/100ft^2 which is lower than the API standard [46,47].

Gel strength of water-based mud formulated with local-bentonite (B), and nano-bentonite (B_N) compared to water-based mud formulated with standard (API) bentonite (B_0) were studied before and after treatment and illustrated in Figs. 8 and 9. Testing results of gel strength indicated the following:

Gel strength 10 s for local-bentonite varies from 2 to 20 lb/100ft^2 after treatment which is higher than 12 lb/100ft^2 as the API requirements. Gel strength 10 s of nano-bentonite does not change and equal to 1 lb/100ft^2 which is lower than the API standard.

Gel strength 10 min for local bentonite varies from 3 to 21 lb/100ft^2 after treatment which is higher than 13 lb/100ft^2 as the API requirements whereas; nano-bentonite was not changed and equal to 2 lb/100ft^2 which is lower than the API standard.

Thixotropy of local bentonite was not changed and equal to1 lb/100ft^2 before and after treatment whereas; thixotropy of nano-bentonite varies from 0 to 1 lb/100ft^2 which is acceptable to the API requirements [48].

3.6.2. Filtration

Table 3 shows the filter loss of local treated bentonite formulated in the water-based mud (B) decreased from 22 to 13 ml after treatment and complies with the international standard and less than the maximum acceptable filter loss of the API bentonite (B_0) which is 15 ml. The filter loss of nano-bentonite (B_N) was decreased from 240 to 54 ml after treatment which is still larger than the maximum acceptable filter loss of the API bentonite [49].

3.6.3. Bentonite yield

Table 4 shows that the yield for the local-bentonite (B) increased from 38.4 to 124.9 Bbl/Ton after treatment which is higher than the acceptable API bentonite yield 100.4 Bbl/Ton. Accordingly, the local treated bentonite classified as high yield bentonite. The yield for nano-bentonite (BN) slightly increased from 12.2 to 18.6 Bbl/Ton after treatment which is lower than the API bentonite yield (B0) 100.4 Bbl/Ton, accordingly the local treated nano-bentonite classified as low yield bentonite as native clay.

3.6.4. Swelling ratio

Testing results of the swelling ratio after 72 h were illustrated in Table 4 and indicated that the swelling ratio for local treated bentonite (B) which equals 4.5 was approximately near from the swelling ratio for the API Bentonite (BN) which reached to 5 and far from the swelling ratio for nano-bentonite (B0) which equals 1.5.

Table 3 Filter loss of local-bentonite (B) and nano-bentonite (B_N) compared to the API bentonite (B_0).

Sample	Filter loss (ml)	
	Nontreated	Treated
B	22	13
B_N	240	54
B_0	12.5	15

Table 4 Yield and swelling ratio of local bentonite (B) and Nano-bentonite (B_N) compared to the API bentonite (B_0).

Sample	Bentonite yield Bbl/Ton		Swelling ratio %
	Nontreated	Treated	
B	38.4	124.9	4.5
B_N	12.2	18.6	1.5
B_0	100.4	100.4	5.5

4. Conclusions

This experimental investigation is as an attempt to better characterize the rheological properties of the local-bentonite and nano-bentonite as drilling fluids. Rheological properties and filtration characteristics of local-bentonite and nano-bentonite muds without additives are not suitable for any drilling operations. Treated local-bentonite based slurries perform better as the concentration reaches full saturation. Yield and rheological properties of local-bentonite muds are substantially affected by a grain size variation of bentonite clay. The finer the grain sizes the more the surface area of particles that controls the rheological behavior of slurry. Increments on both plastic viscosity and yield point values at grain sizes of 200 mesh are postulated as a result of different physical interaction effect between the surface areas of clay particles and the liquid phase in the slurry after the grain sizes of local-bentonite clay are reduced to certain sizes.

Finer grain sizes concentration to nano scale local-bentonite before and after treatment decreases, swilling, yield, rheology and increasing filter lose. Extensive care should be taken into consideration when using the API Standards to formulate bentonite based drilling fluids. The effects of grain size must be evaluated based on the required residue >75 microns (US standard sieve No. 200) not to exceed 2.5% by weight. Additional requirement is that the amount of bentonite passing through a dry 100 mesh (150 micron) screen shall be at least 98% by weight.

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