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RESEARCH ARTICLE

ACTIVATION OF EGYPTIAN BENTONITE TO IMPROVE THEIR DRILLING FLUIDS PROPERTIES

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ABSTRACT

Bentonite clay is the main component of the water- based drilling fluid. In this study the Egyptian bentonite from (south Hammam) was tested for its potential use in drilling fluids. The drilling mud quality of the bentonite from (south Hammam) was significantly improved by using low molecular weight polyvinyl alcohol (PVA) as a viscosifier additive. The Egyptian bentonite was characterized by X-ray fluorescence analysis (XRF), X-ray diffraction (XRD), thermal gravimetric analysis (TGA) and transition electron microscopy (TEM). The treated Egyptian bentonite was evaluated as drilling mud for oil–well drilling. The evaluation involves the study of the rheological properties (apparent viscosity, plastic viscosity, yield point, gel strength and thixotropy), filtration properties before and after treatment with polyvinyl alcohol as viscosifier additives. Effect of temperature on the formulated mud at different temperatures was also studied and the results were compared with the commercial grade bentonite according to American Petroleum Institute specification (API) and O.C.M.A specification.

INTRODUCTION

Drilling fluid or mud is the fluid that is used in drilling operation in which that fluid is pumped from the surface down to the drill string through the bit and back to the surface via annulus. The fluids used to maintain pressure, cool drill bits and carry cuttings from the holes, there are two types of drilling fluids, aqueous drlling fluids (water based mud) and non aqueous-drilling fluids (oil based mud or synthetic mud) (Ryen Caenn and Chillingar George 1996; Morita *et al.*,1996 Lagaly *et al.*, 2006). Bentonite is the main constituent of the water based drilling fluid due to high viscosity, good swelling and low filter loss (Apaleke *et al.*, 2012). Bentonite is a smectite clay mineral and largely composed of >80% montmorillonite clay mineral and a smaller amount of other clay minerals as kaolanite or non clay minerals as quartz. Montmorillonite is a type of natural mineral clay and has a layered structure. It consists of stacked layer silicates including two silica tetrahedral sheets (two tetrahedral layers of silicon oxide $(SiO_4)_4^-$) sandwiching an edge-shared octahedral sheet of either aluminum or magnesium hydroxide or iron oxide. There are some hydrophilic cations residing in the gallery, such as Na or Ca ions, which can be exchanged by other cations. there are two types of Montmorillonite Na- Montmorillonite and Ca-Montmorillonite (Bol 1986; Luckham and Rossi 1999). Na-bentonite is characterized by its ability to absorb large amounts of water and form viscous, thixotropic suspensions while Ca-bentonite, which has Ca^{+2}as the dominant exchangeable cation, is characterized by its low water absorption and low swelling capabilities and its inability to stay suspended in water (Thaemlitz *et al.*, 1999). The properties of natural montmorillonite clay can be enhanced or modified by organic modification, due to the substitution of the exchangeable cations in the interlayered area or cation exchange capacity (CEP) (Christidis and Huff 2009; Güven and Bailey 1988; Eisenhour and Brown 2009; Guangming *et al.*, 1998; Nevin *et al.*, 2002; Sevim *et al.*, 2005).The surface modification of the clay minerals either by physical adsorption using polymers or grafting by using surfactant. The activation of bentonite by alkali or polymer is occurred for upgrading of the bentonite to meet the API specification and OCMA standard (Dardir *et al.*, 2011; API 1998). These activations typically employ various additives such as soda ash (Na_2CO_3), MgO,biopolymers like Xanthan gum (XG) which is the most common natural biopolymer used as a viscosifier in the oil industry due to its unique rheological properties and polymers like CMC (carboxyl methyl cellulose), HEC (hydoxy ethyl cellulose) as viscosity improvement agents. While soda and MgO improve the swelling or viscosity. CMC, HEC and soda hinder filtration losses. Modification of bentonite with polymers (soluble in water) and similar compounds has been studied by different investigators and outstanding rheological behaviors such as viscosity, thixotrophy etc. have been measured (Jhe 2004; Lemi *et al.*, 2005). The interaction of non-ionic polymers with the clay surface is possible through two types of polymers. The

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amount of water bound to the montmorillonite surface increases linearly with the concentration of the oxygen (or hydroxyls) there are two mechanisms (Eisenhour and Brown 2009; Darley and Gray1988). The *first involves ion-dipole interactions*, where the saturating cation on the clay surface serves as an adsorption site for polar non-ionic molecules. The *second type of interaction involves hydrogen bonding*, either by the direct interaction between the adsorbed polymer clay surface, and polymer (Zamora *et al.*, 2000). In the oil drilling field, the polymer is usually selected to reduce fluid loss, increase cutting carrying capacity, serve as emulsifiers and lubricants, especially as shale inhibition additives in water based drilling fluids (Lei Wang *et al.*, 2011). In this study low molecular weight polyvinyl alcohol (PVA) was used for modification of the local clay for its potential use in drilling fluids.

Experimental

Sample collection and preparation

Clay stone samples were taken from the exposed surface of three inconsiderable exploited quarries located at south Hamam, Egypt. The studied samples were designated as L_1, L_2 and L_3. The samples were taking from the fresh deposits using the representative sampling methods. The collected samples mainly brownish grey to yellowish green. Samples was crushed to finer particles and sun-dried for 5 days to ease pulverizing and sieving. The samples were then ground to powder with the aid of mortar and pastle, and then sieved with shaker to obtain 75μ fractions, to suit the API 13 A specification for bentonite L_1, L_2, L_3 samples (Karagüzel *et al.*, 2010) and commercial grade bentonite that is used as water based drilling fluids (R). Polyvinyl alcohol (PVA) commercial grade from (qualikems company) degree of polymerization from 1700-1800, used as viscosifier for treatment of the local Egyptian bentonite.

Mineralogical studies

Mineralogical studies for local bentonite samples (L_1,L_2 and L_3) before and after activation with PVA were determined by X-ray diffraction (XRD) using a philips X-ray diffraction equipment model Pw 710 with mono chromator, cu radiation (h= 1.542 Å) at 40 kV, 35 mA and scanning speed 0.02°/s. The reflection peaks between 2Θ =2° - 70°, corresponding spacing (d, Å) and the relative intensities (I/I°) were obtained (Moore and Reynolds 1997). The diffraction charts and the relative intensities were obtained and compared with ICDD files Table 1.

Table 1. X-ray diffraction analysis for local bentonite samples L_1, L_2.L_3

Constituents	L_1	L_2	L_3
Major const.	Na - Montmorillonite	Na - Montmorillonite	Na – Montmorillonite and Quartz
Minor const.	Calsite	calsite	kaolinite
Trace const.	Quartez	kaolinite	calsite

Chemical analysis by using X-ray fluorescence Spectrometry (XRF)

For the studied samples was carried out to determine the chemical composition by (XRF) spectrometer (Garcia-Romero and Suarez 2010) and the results were listed in Table (2)

Table 2. Chemical analysis for local-bentonite samples L_1, L_2.L_3

Elements	Local-bentonite %(L1)	Local-bentonite % (L2)	Local-bentonite %(L3)
SiO_2	54.91	54.45	61.16
TiO_2	1.53	1.70	0.59
Al_2O_3	19.01	16.42	19.44
Fe_2O_3	6.31	10.25	4.60
MnO	0.08	0.10	0.06
MgO	2.47	2.16	2.2
CaO	1.99	1.03	1.38
Na_2O	2.75	2.33	1.22
K_2O	1.03	1.12	2.05
P_2O_5	0.16	0.42	– –
Cl	1.20	0.79	– –
SO_3	0.48	0.15	0.50
L.O.I 250 °C	– –	– –	– –
L.O.I1000 °C	8.06	9.00	8.40

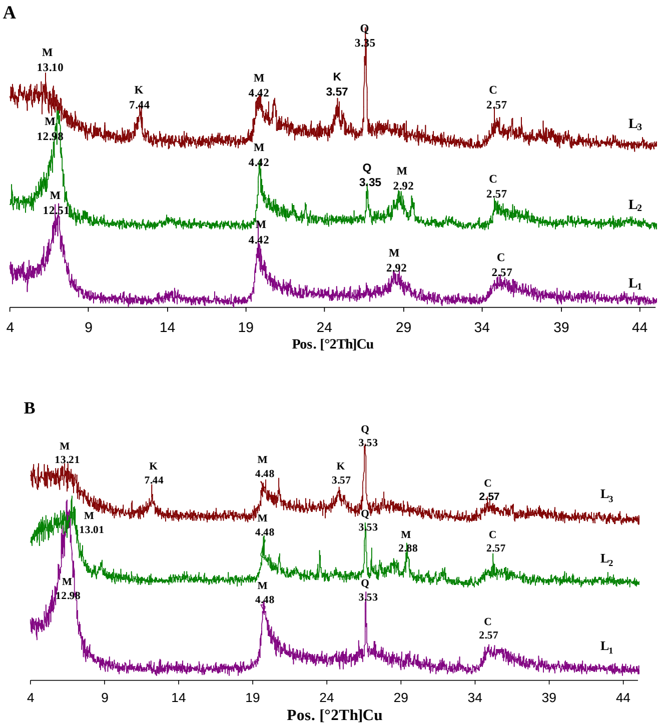

Figure 1. (A) X-ray diffraction analysis of untreated samples (L_1, L_2 and L_3) and (B) for treated bentonite samples (L_1, L_2 and L_3) with PVA(M=Montmorillonite, C= Calsite, K= Kaolinite and Q = Quartez)

Thermal analysis

Thermo gravimetric analysis (TGA) was done by means of a thermo gravimetric analyzer (TG–DSC TAQ600) (Beaufort *et al.*, 2001). The thermal behaviors of the samples were recorded in the chart Fig (2).

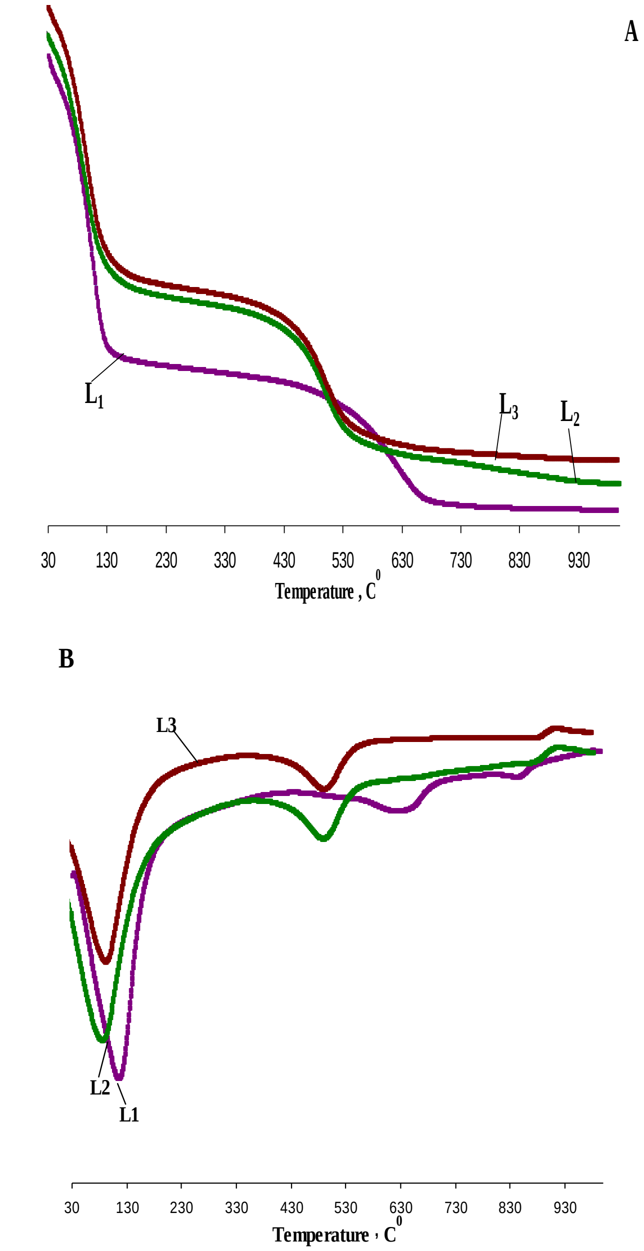

Figure 2. TGA for Fig. (A) and DTA for Fig. (B) For local untreated bentonite samples L_1, L_2 and L_3

FTIR measurements

FT-IR Spectrum analysis for the studied local bentonite samples L_1, L_2.L_3 was carried out by using NICOLET. FT-IR IS-10 (Gu¨ngo¨r and Karaogˇlan 2001; Tyagi *et al.*, 2006), as shown in Fig (3).

Transmission electron microscope

Transmission electron micrographs (TEM) for untreated bentonite L_1, L_2.L_3 samples before and after treatment with polyvinyl alcohol (PVA) were shown in Fig (4). The analysis was performed by a JEOL-GEM-2100 Japan, 200kv, 1.5 X electron microscope. The samples were prepared by dipping the prepared dispersion onto the copper-coated carbon film grids and left air-dried. (Tao Wan *et al.*, 2011)

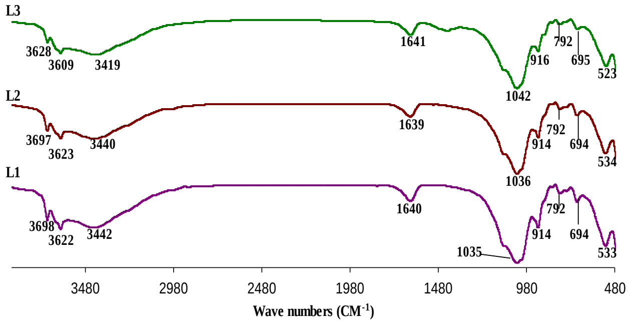

Figure 3. FTIR for untreated samples L_1, L_2 and L_3

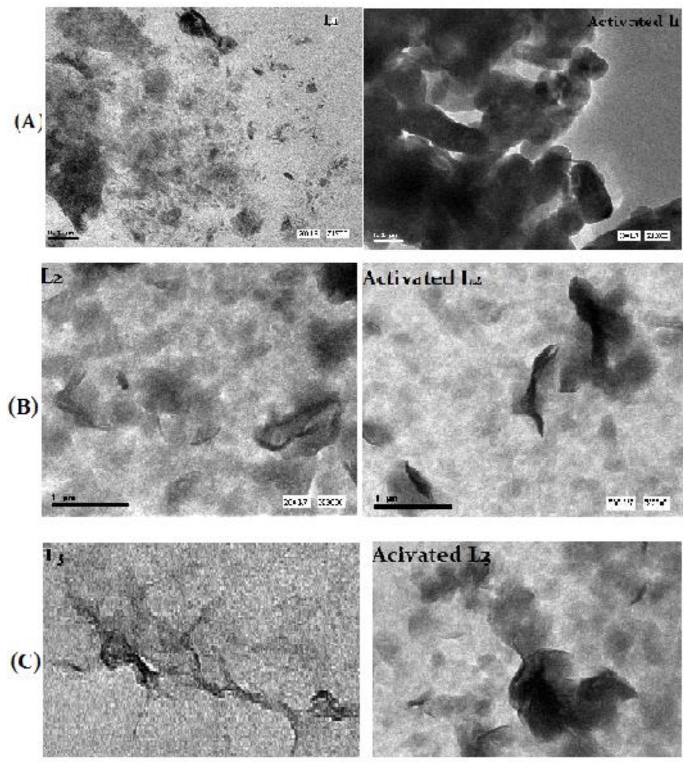

Figure 4. TEM micrograph of (A) row L_1 and activated L_1, (B) row L_2 and activated L_2 and (C) row L_3 and activated L_3

Swelling test

To 100 ml graduated cylinder filled with 100 ml distilled water add 2 g dried bentonite over period of 8 hours, after slowly spreading the bentonite the mixture was left for settling for 5–6 h recorded. 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 2 g bentonite placed in the graduated cylinder (Abdou *et al.*, 2013; ISO 13500 Petroleum and natural gas industries ISO 13500:2008).

Mud formulation

Non treated mud

The mud formulations consists of three mud batches for local non treated bentonite ML_1, ML_2, ML_3 and commercial grade bentonite MR. Each batch was prepared by adding 21 g bentonite to 350 ml fresh water as the following

1- The samples were mixed in a Hamiltan mixer for 20 minutes then cured overnight.
2- Each sample was stirred for 15 minutes, before the rheological and filtration properties were detremined.

Treated mud

1- Different concentrations from polyvinyl alcohol (PVA) were added to mud batches ML_1, ML_2, ML_3.
2- The samples were mixed for 20 minutes and cured overnight.
3- Each sample was stirred for 15 minutes, and then the rheological properties and filtration were measured.

So we have four mud batches ML_1, ML_2, ML_3 and MR

Mud testing

Rheological properties

Rheological properties for the samples before and after activation with polyvinyl alcohol were determined to asses their respons to the used as activator and consequently the capacity for using as drilling fluids. All rheological and filtration tests should follow the American Petrolum Institute (API) and Oil Companies Materials Association (OCMA) specifications. Apparent viscosities (AV), plastic viscosity (PV), yield point (YP), Gel strength and thixotropy were measured according to API specifications by using (Chandler Model 3500) viscometer. Apparent viscosity, CP = (600 rpm reading /2) Plastic Viscosity, CP = (600 rpm reading) - (300 rpm reading) Yield Point, lb/100 ft^2= (300 rpm reading) - (Plastic Viscosity)

Filtration test

API fluid loss test was carried out by using a standard filter (Ofite) press at 100 psi pressure for 30 minutes at room temperature. The final volume of filtrate after 30 min in ml was noted as API filtrate for all samples (ML_1, ML_2, ML_3 and MR) as illustrated in Table (4).

Table 3. Density and yield value for L_1, L_2, and L_3 samples compared with R

Sample Number	Density of 6% Bentonite lb/gal	Yield value (barrels per ton mud slurry)	
		Before activation	After activation
L_1	8.2	62.0	90.0
L_2	8.3	52.0	86.0
L_3	8.5	48.0	75.0
R	8.8	94.0	94.0

Table 4. Swelling ratio and PH value of L_1, L_2, and L_3 samples compared with R

Sample Number	Swelling Ratio %		PH value	
	Before activation	After activation	20 min	24 h
L_1	11	14	9.23	9.09
L_2	8	12	8.89	8.39
L_3	6.5	9.5	8.75	8.31
R	12	12	10.85	10.75

Yield

Bentonite yield was determined according to OCMA (Oil Companies Materials Association) specifications which corresponds to 90.0 barrels per ton mud slurry, as mentioned in Table (4)

PH and density

The pH values were measured for mud batches ML_1, ML_2, ML_3 and MR and illustrated in Table (4). Also density were measured for all samples by using Fann mud balance model 140 and illustrated in Table (3)

Effect of temperature on the rheological properties

By using Baroid 251-27 cup heater Formulated mud (ML_1) (21 gram bentonite+ 350 ml fresh water + 0.5 gram from polyvinyl alcohol (PVA)) was mixed for 20 minutes and cured overnight. The mud sample was stirred for 15 minutes, before the rheological properties were measured. Put the mud in cup heater and gradually raise the temperature from $25C^0$ to $90C^0$, the reological properties were measured at different temperatures and the results was compared with the commercial grade bentonite formulated mud (MR)

RESULTS AND DISCUSSION

Mineralogical studies

Mineralogical analysis for local bentonite samples L_1, L_2, L_3 showed that they are essentially montmorillonite. X-ray was used to determine the clay mineral types. The dominant clay mineral was found to be Na-montmorillonite for (L_1, L_2 and L_3) with minor amount of kaolinite and calsite. Quartz was always present in clay fraction. The Na- bentonite sample shows sharp and intense peak which corresponds to an interplanar distance $d_{(001)}$ of 12.51 Å for L_1, $d_{(001)}$ 12.98 Å for L_2 and $d_{(001)}$ 13.10 Å for L_3 fig (1A) while fig (1B) show that the basal spacing was shifted to 12.98 Å for L_1, 13.01 Å for L_2 and 13.21 Å for L_3 after treatment with PVA .The intensity and the line width of these (001) peaks indicates good crystallization which indicates good adsorption for polyvinyl alcohol on clay and this will improve the clay rheological properties (Falode *et al.*, 2008; Akc ay *et al.*, 1999)

Chemical analysis

By using X-ray fluorescence Spectrometry (XRF) values obtained in XRF analysis showed that the ratio Al_2O_3/SiO_2 ratios were 1/3 to 1/4 as expected for a montmorillonite which is the main component of bentonite samples (L_1, L_2 and L_3) under study. The ratio of (Na_2O+K2O) / (CaO+MgO) is 1.1 for L_1 and L_2 and 0.9 for L_3.confirming that all samples were Na-bentonite. Table (2) illustrate the chemical composition for local bentonite samples (L_1, L_2 and L_3)

2.3 Thermo gravimetric analysis (TGA)

The mass loss steps observed up to 200 °C were attributed to the desorption of physically adsorbed water, second mass loss step the dehydration of the hydrated exchangeable cations such as Na^+ and Ca^{+2}. Between 200 and 500 °C, (Hassan and Abdel-Khalek 1998). The weight loss of 11.55% was lost at 130 °C for L_1, 9.63 % for L_2 and 10.16 % for L_3.while 2.2% was lost at 525 °C for L_1,6.24 % for L_2 and 6.07 % for L_3. In addition, about 1.5 % was lost at temperature range of 600–1000 due to surface dehydroxylation and loss of non-chemically attached metal oxides. DTA (B) curve showed two endothermic peaks resulted from loss of adsorbed water and the second from loss of water of crystallinity. (Barick and Tripathy 2010; Hedley *et al.*, 2007)

Fourier transform infrared spectroscopy

The FT-IR spectra of the bentonite fractions were measured in transmittance between 480 cm^{-1} 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 535 cm^{-1} and 490 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 Clasite, band at 790 cm^{-1} for Mg–Fe– OH. The bands at 3698 cm^{-1} and 792 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 Fe3+.

TEM observations

Transmission electron micrographs for Na-bentonite samples of L_1, L_2 and L_3 are shown in Fig (4).On treatment the clay samples with PVA the dispersion was more this is better clear in fig (4(A))for sample L_1 which show more homogenous distribution of silicate layers without clay aggregates but in case of samples L_2 and L_3 fig. (4 (B&C)), after activation of PVA they show aggregates of layers with variable numbers of lamella

Mud evaluation

Rheology

Rheological properties of water-based mud formulated with local-bentonite samples ML_1, ML_2 and ML_3 were studied before and after treatment with polyvinyl alcohol (PVA) compared with water-based mud formulated with commercial grade bentonite MR as illustrated in Figs. (5), (6)

Testing of rheology indicate the following

A-Before activation

Apparent viscosity (AV): for local bentonite samples were 5 cp, 3 cp and 2 cp for ML_1, ML_2 and ML_3 which are lower than API standard.

Plastic viscosity (PV): for local bentonite mud batches 4 cp, 2cp and1 cp for ML_1, ML_2 and ML_3 which are also lower than API standard.

Yield point (YP): for local bentonite mud batches were 2 lb/100ft^2 for ML_1, ML_2 and ML_3. The yield point was low in value for prepared drilling fluid; it should be high enough to carry cuttings out of the borehole.

The previous results indicates poor rheological properties which means that the clay cannot be applied as drilling mud , but on the other hand on the activation of the clay with polyvinyl alcohol the results changed as illustrated in Fig(7)

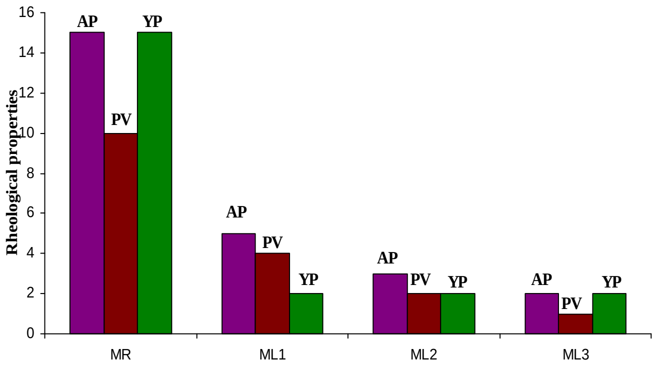

Fig 5. Rheological properties of ML_1, ML_2 and ML_3 before activation with PVA compared with MR

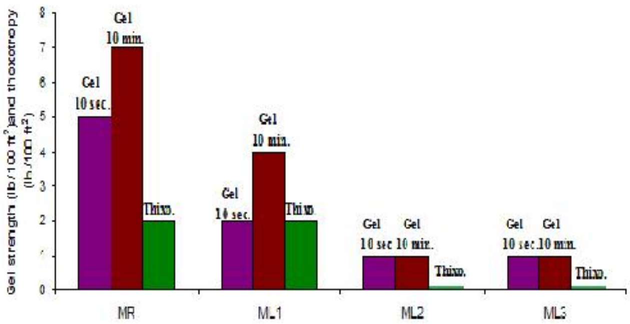

Fig 6. Gel strength after 10 sec. and after 10 min. and thixotrop for ML_1, M L_2 and ML_3 before activation with PVA compared with MR

B-**After activation** with polyvinyl alcohol: as the concentration of PVA increase the rheological properties increased .Apparent viscosity (AV) for local-bentonite increased from 5 to 26.5 cp after treatment with 0.7 g PVA for ML_1, increased from 3 to 19.5 cp for ML_2 and increased from 2 to 16.5 cp for ML_3 which is compatible with API standard Fig (7).

Plastic viscosity (PV) for local bentonite increased from 4 to 12 cp after treatment for ML_1, increased form 2 to 10 cp for ML_2 and increased from 1 to 8 cp for ML_3 which is satisfying the API standard Fig (7).

Yield point (YP) for local-bentonite varies from 2 to 29 lb/100ft^2 after activation with PVA for ML_1, varies from 2 to 19 lb/100ft^2 for ML_2 and varies from 2 to 17 lb/100ft^2 for ML_3 as illustrated in Fig (7).

So the activation of the clay samples with 0.70 g of PVA show good results for all mud batches but the best results for mud batch ML_1 which show rheological properties compatible with the API specification and OCMA standard when treated with 0.5 g of PVA.

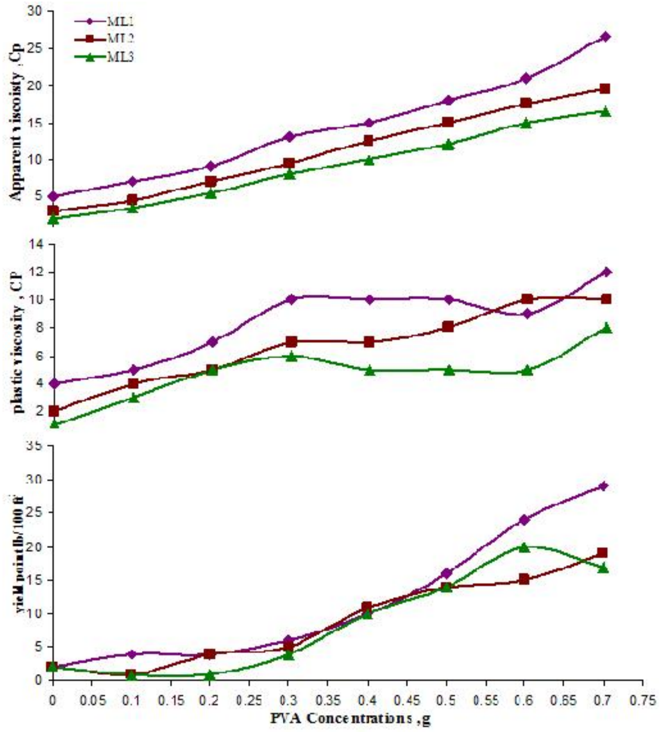

Figure 7. Rheological properties, concentration of PVA relationship for formulated mud batches (ML_1, ML_2 and ML_3)

Gel strength of water-based mud formulated with local bentonite (ML_1, ML_2 and ML_3) were studied after treatment and illustrated in Fig (8).

Testing results of gel strength indicated the following: G_{10sec} for local-bentonite varies was 11 lb/100ft^2 for ML_1, 4 lb/100ft^2 for ML_2 and 3 lb/100ft^2 for ML_3.

$G_{10\ mint}$ for local bentonite was 13 lb/100ft^2 for ML_1, 6 lb/100ft^2 for ML_2 and 5 lb/100ft^2 for ML_3.

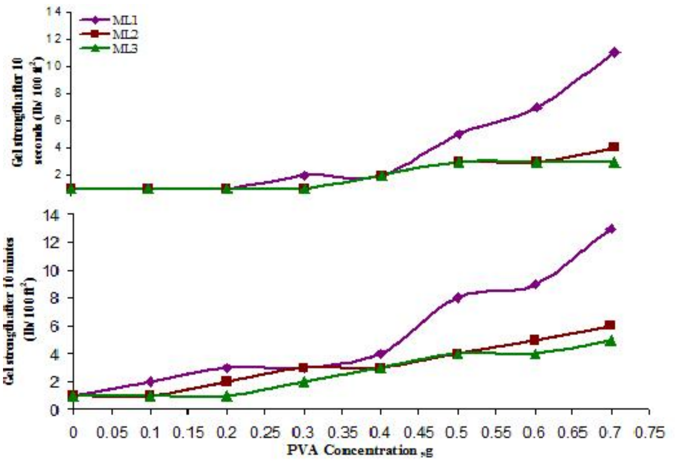

Figure 8. G_{10sec}, $G_{10\ mint}$ and concentration of PVA relationship of formulated muds (ML_1 , ML_2 and ML_3)

Thixotropy of local bentonite was 3 lb/100ft^2 for ML_1 and was 2 lb/100ft^2 for (ML_2 and ML_3) as illustrated in the Figure (9)

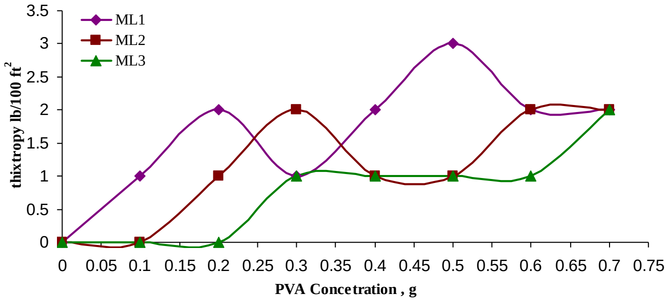

Figure 9. Concentration of PVA and Thixotropy of formulated muds (ML_1, ML_2 and ML_3)

The result of rheological properties after treatment with PVA at different concentrations indicated that PVA has direct effect on rheological properties of the mud formulated from local caly bentonite samples L_1.L_2 and L_3 as the cocentartion of PVA increased. The best local sample is L_1 with concentarion of 0.5 g PVA as viscosifier additive.

Yield

The yield for the local-bentonite for formulated mud batches 6% increased from 32.65 to 97.77 bbl/Ton slurry after treatment with 0.4 g of PVA at 6% bentonite which is acceptable value corresponding to API bentonite yield and OCMA.

Filtration test

Table (5) illustrate filter loss for formulated mud with local bentonite before and after treatment with PVA the results indicating that filter loss decreased by using PVA as a viscosifier agent.

Table 5. Filter loss (ml) and mud cake thickness (mm) for mud batches (ML_1, ML_2 ML_3) before and after treatment with PVA compared to MR

Mud type	Filtration , ml		Mud cake thickness , mm	
	Before activation by PVA	After activation by 0.4 g PVA	Before activation by PVA	After activation by 0.4 g PVA
ML_1	22	15	1.2	1.1
ML_2	21	20	1.4	1.3
ML_3	20	22	1.6	1.7
MR	15		1.9	

Swelling ratio

The swelling ratio for L_1 increased from 9 to 14 after addition of PVA, from 8 to 11 for L_2 and increased from 6.5 to 9.5 for L_3 while the swelling ratio for commercial grade bentonite is 11 as illustrated in Table 4

Effect of temperature on the rheological properties

Water-based mud formulated with 21g local-bentonite of sample L_1/350 ml H_2O and 0.5 gram PVA were subjected to heat from 25$c^0$to 90 c^0 and compared to MR mud as illustrated in Fig (10). Testing results of rheology indicated the following: Apparent viscosity (AV) for formulated mud ML_1 decreased from 15 to 12.5 cp, Plastic viscosity (PV) for formulated mud decreased from 10 to 6 cp and Yield point (YP) increased from 10 to 13 lb/100ft^2 as illustrated in Fig (10). Increasing in yield point value still in range as API specification.

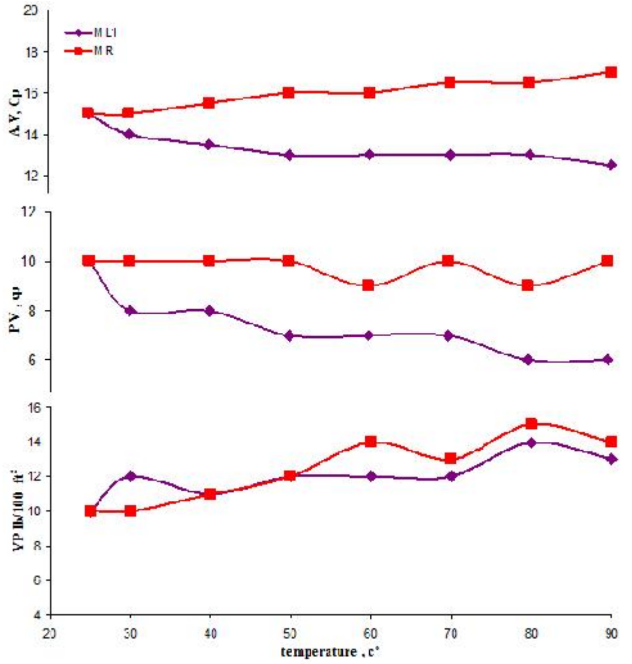

Figure10. Rheological properties of formulated mud ML_1 compared to MR at different temperatures

The effect of temperature on Gel strength of formulated mud ML_1 were studied compared to the MR

$G_{10\ sec}$: increased from 2 to 11 lb/100ft^2 while MR was 5 lb/100ft^2
$G_{10\ min}$: increased from 4 to 15 lb/100ft^2 while MR was decreased from 7 to 6 lb/100ft^2 as showed in Fig (11). Increasing in gel strength with increasing temperature still compatible with API specification.

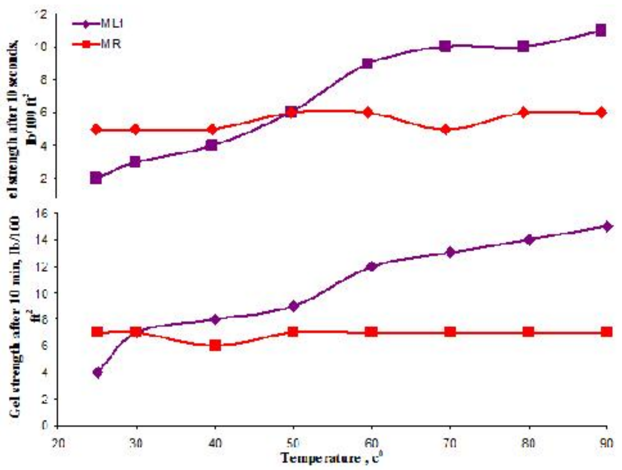

Figure 11. Temperature relationship, Gel strength of formulated mud ML_1 at 10 sec. and 10 min. compared to MR

Thixotropy of formulated mud ML_1 increased from 2 to 4 lb/100ft^2 while for MR decreased from 2 to 1 lb/100ft^2 as illustrated in the following Fig (12).

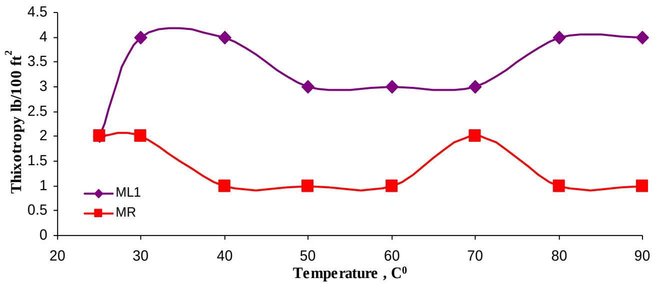

Figure 12. Temperature relationship, Thixtropy of formulated mud ML_1 compared to MR

Conclusion

The characterization of non activated clays collected from South Hamam , Egypt for the application as a drilling mud fluid (water-based mud) were investigated. Also the activation compatibility for these clays was explained as an attempt to minimize the important cost of the imported bentonite to Egypt by a local solution. The following main conclusion can be drawn from the present study.

1. Most of the studied samples were of bentonite clay, was mainly Na-montmorillonite.
2. The studied non – activated samples cannot be used as a drilling mud without activation.
3. The studied clay sample L_1 can be used after activation with PVA as a drilling fluid for its rheological properties satisfied the API standard and OCMA specification while clay samples L_2, L_3 cannot be used as a drilling fluid even after the activation with PVA.
4. The obtained results revealed that the activated samples may be expressed as medium grade bentonite clays and these grades of clays can be used as drilling fluids for shallow depth wells.

Nomenclatures

L: local untreated bentonite
R: commercial grade bentonite
ML: Mud formulated from local bentonite treated with PVA
MR: Mud formulated from commercial grade bentonite
YP: yield point (lb/100 ft^2)
AV: apparent viscosity (cp)
PV: plastic viscosity (cp)
PVA: polyvinyl alcohol
GS: gel strength (lb/100 ft^2)
TEM: transition electron microscope
TGA Thermo gravimetric analysis
DTA Differential thermal analysis
OCMA Oil Companies Materials Association

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