

# EFFECT OF FAST-SWELLING POROUS HYDROGEL ON THE REDUCTION OF FILTER LOSS FOR WATER BASED-DRILLING FLUIDS

FAWZIA I. EL-DIB<sup>a</sup>, M. M. DARDIR<sup>\*,b</sup>, Gh. ESHAQ<sup>a</sup> and M. M. FAYAD<sup>b</sup>

 <sup>a</sup>Egyptian Petroleum Research Institute (EPRI), Petrochemical Department, Surfactant Laboratory, CAIRO, EGYPT
 <sup>b</sup>Egyptian Petroleum Research Institute (EPRI), Production Department, Drilling Fluids Laboratory, CAIRO, EGYPT

# ABSTRACT

Filtration is one of the desired properties of a drilling mud that it should minimize fluid loss from the well bore into the surrounding. Fluid loss is the measurement of filtration passing from the drilling fluid into a porous permeable formation. Low fluid loss is a characteristic of good drilling fluid and is the key to borehole integrity. In this study two samples of fast swelling highly porous superabsorbent hydrogel (HPSHs) were synthesized via rapid solution polymerization of partially neutralized acrylic acid in presence of N, N'-methylenebisacrylamide (MBA) as cross-linking agent under atmospheric conditions, ammonium persulfate (APS) as initiator and (acetone and sodium bicarbonate) were used as a porosity generators to create highly porous structures with highly rate swelling. Another sample of superabsorbent hydrogel (SH) was synthesized via rapid solution polymerization, with the same ingredients but without using (acetone and NaHCO<sub>3</sub>). And the results showed that (HPSHs) have more swelling capacity than (SH). The new prepared (SPHPs) and (SH) were investigated as viscosifiers and filter loss additives for water-based drilling fluids formulated from local Egyptian clay (Na-bentonite) to improve the rheological and filtration properties. The study of rheological and filtration properties of water based mud treated with the new (SPHPs) and (SH) indicated that (SPHPs) have the best results for improving of the rheological properties and reducing filter loss and mud cake of water-based mud than the corresponding reference water-based mud treated with the conventional filter loss (carboxymethylcellulose). The results of mineralogical studies and scanning electro microscopy (SEM) for the water based mud treated with (SH) and (SPHPs) indicated that both of the new prepared (SH) and (SPHPs) were intercalated and attached strongly to the local Na-bentonite clay.

Key words: Water-based mud, Rheological properties, Filtration properties, Super absorbent, Hydrogel.

<sup>\*</sup>Author for correspondence; E-mail: monamdardir@yahoo.com

# INTRODUCTION

Drilling fluid or mud is a specially designed fluid that is circulated through a wellbore as the wellbore is being drilled to facilitate the drilling operation. Drilling fluids facilitate the removal of drill cutting from the wellbore, cool and aid in supporting both of drill pipe and drill bit and provide a hydrostatic head to maintain the integrity of the wellbore walls thereby minimizing the potential for well blowouts<sup>1-4</sup>. Oil-producing formations are generally porous layers having varying degrees of permeability to the flow of fluids such as oil, water, or gas<sup>5,6</sup>. Specific drilling fluid systems are selected to optimize a drilling operation in accordance with the characteristics of a particular geological formation.

In many wells drilled with aqueous drilling fluids suffer serious decreases in permeability of oil bearing zones as result of water blocking of the pore spaces and swelling of anhydrous clays within the pore space<sup>7,8</sup>.

Consequently, the rate of oil production is largely determined by the rate of flow, through these permeable formations which in turn is dependent upon the porosity of permeability of the sand or stone present in drilling through such a porous layer, it is desirable to employ a drilling fluid having such characteristics that excessive amounts of liquids or solids are prevented from penetrating through the porous formation. The ability of the drilling fluids to prevent excessive formation fluid penetration is called filtration control<sup>9,10</sup>.

Two types of drilling fluids or muds are used for drilling for oil and gas, aqueous based mud (water–based mud) WBM and non aqueous-based mud (oil-based mud) OBM and synthetic based mud (SBM). (WBM) are more economically, safety and environmentally friendly. although oil-based mud and synthetic based mud are considered the preferred drilling fluids than (WBM) when drilling water-sensitive shale formations and other difficulty properties and they also provide excellent borehole stability, thermal stability, corrosion inhabitation, ease in engineering and fluid maintenance, better fluid loss control and excellent filter cake quality<sup>11-16</sup>.

Rheological and filtration properties are the most important parameters of drilling fluids that can affect the drilling operation, fluid loss is the measurement of the filtrate passing from the drilling fluid into a porous permeable formation. Low fluid loss with thin mud cake is a characteristic of good drilling fluid and is the key to borehole integrity. So, one of the main function of good drilling fluids is to create a thin mud cake on the sides of borehole to prevent the excessive loss of fluid into the formation. Higher filter loss with thick mud cake can cause many problems like stuck pipe and lost circulation<sup>17-23</sup>. Sodium

bentonite is added to fresh water mud to increase hole cleaning capability, reduce filtration into permeable low permeability and promote hole stability. So, it gives the proper viscosity and filtration control properties. Some water based mud has inability to meet up the API specification and has a high filter loss with a thick filter cake. Carboxymethylcellulose, starch and xanthan gum are the most effective additives for minimizing filter loss and increasing viscosity that has been used to treatment the water- based but on using these additives the rheological properties will have a high value with high mud weight and the filtrate will be reduced but it still have a thick mud cake due to high mud weight. The high mud weight value can cause damage to sub-surface formation, plugging of production zones, hole erosion, decreased penetration rate, pipe failures, stuck pipe and lost circulation<sup>24-29</sup>. So in order to decrease the probability of such problems we have to use new materials which don't increase the mud weight and viscosity to high values. Superabsorbent hydrogels are slightly cross-linked hydrophilic polymer, which are able to absorb, swell and retain thousand times of to hundreds times of their own weight. swelling rate of superabsorbent hydrogels can be enhanced through porosity creation in the hydrogel structure porous structure is formed by using foaming agents, such as sod-bicarbonate, potassium carbonate and calcium carbonate<sup>30-32</sup> and organic solvent such as methanol, acetone, pentane and hexane<sup>33</sup> owing to their high water affinity and biocompatibility, hydrogen based on poly acrylic acid have found extensive applications in controlled drug delivery system various areas such as pharmaceutical<sup>34</sup>, hygienic, a niculatural<sup>35,36</sup>, coal dewatering<sup>37</sup>, additives for concrete and drilling additives.

In this work, we extend our previous study work<sup>29</sup> where an Egyptian untreated Nabentonite and super absorbent hydrogel were being added to improve the rheological properties, reduce filter loss and reduce the mud cake.

The objective of this study was to synthesis both highly porous superabsorbent hydrogel (HPSHs) and superabsorbent hydrogel (SH) based on acrylic acid to be used as filter loss reducer and viscosifiers for water based drilling fluids.

# EXPERIMENTAL

#### Materials

Acrylic acid (AA) monomer, ammonium persulphate (APS) N N'-methylene disacrylamide (MBA) crosslinker, sodium metalisulfite hydroxide (KOH) and acetone supplied by fluka were used as obtained. Untreated local bentonite B<sup>o</sup> (Egyptian local benonite extend our previous study work) and carboxymethylcellulose (CMC) was purchased from Baroid company to use as reference mud sample.

#### Preparation of super absorbent hydrogel polyacrylic acid (SH)

Acrylic acid monomer (3g) was partially neutralized with the KOH (1.8 g KOH, 1.5 ml H<sub>2</sub>O) then the cross linker solution (0.002 g MBA + 0.4 ml H<sub>2</sub>O) and (0.007 of APS, 0.2 ml H<sub>2</sub>O) as initiator were added to the above mixture the reaction mixture was vigorously shaken using magnetic stirrer. In this procedure, polymerization was allowed to start after addition of metabiuslfite solution (0.006 g SMBS, 0.2 mL H<sub>2</sub>O) the products were allowed to dry in oven at 60°C for 3 days, then (SH) was stored in our tight container until further use. The product obtained was super absorbent hydrogel polyacrylic acid (B1).

## Preparation of highly porous super absorbent hydrogel polyacrylic acid (HPSHs)

The mixture obtained in step 2-2 after addition of sodiumbisulphite solution the temperature as well as, the viscosity of the reaction mixture were rapidly increased in a 20-50 second just before gelation point sodium bicarbonate (0.2 g SBC) or sodium bicarbonate (0.2 g SBC and acetone 1 mL) were added very quickly to the solution to get products B2 and B3. The viscosity of the reaction mixture was rapidly increased and the bubbles were trapped within the highly viscous mixture.

#### Swelling measurements

#### Equilibrium swelling capacity was determined by gravimetric method

0.050 g of dry samples were immersed in 200 mL distilled water and allowed to swell for 24 hrs. The water content was calculated on the bases of the weight difference of the hydrogel samples before and after swelling.

Water content = 
$$\frac{W_s - W_d}{W_d}$$
 (36)

where  $(w_s)$  is the weight of the swollen hydrogel and  $(w_d)$  is the weight of the dry sample.

#### Testing for water-based mud

The prepared super absorbent hydrogel (B1) and highly super absorbent hydrogel (B2 and B3) were evaluated as viscosifiers and filter loss additives for treatment of the untreated Na-bentonite Bo. All water-based mud and Na-bentonite tests were determined according to the API (American Petroleum Institute) ANSI/API 13B-1<sup>38</sup> and OCMA specification No. DFCP-4<sup>39</sup>.

#### Mud formulation

The mud batches of the various concentrations of untreated Na- bentonite B<sup>o</sup> using fresh water were treated by the new additives B1, B2 and B3 and prepared as follows:

- 1. Mixing 6% of the untreated Na- bentonite (B°) in 500 mL fresh water. The samples were mixed in a Hamilton mixer for 20 min then cured overnight.
- 2. Each sample was stirred for 15 min, before the rheological and filtration properties were determined.
- 3. Different concentrations from the prepared super absorbent hydrogel B1 and highly super absorbent hydrogel (B2 and B3) were added to the mud batches (0.01-0.1) g/500 mL.
- 4. The samples were mixed for 20 min and cured overnight.
- 5. Each sample was stirred for 15 min, and then the rheological and filtration properties were determined.
- Formulation of MR the reference mud batches was carried out by mixing 6% of the untreated Na- bentonite in 500 mL fresh water then carrying out steps 2 and 3 for the mud batch by adding 0.5 g of carboxymethylcellulose (CMC) as a viscosifiers and filter loss additives, so we have 5 mud batches :

MB°: mud batch formulated from untreated Na-bentonite

MR: reference mud batch formulated from untreated Na-bentonite + CMC

MB1, MB2 and MB3, three mud batches that formulated from untreated Nabentonite and treated with the new prepared hydrogel B1, B2 and B3, respectively

After addition of wide range of concentration of the new prepared porous hydrogel (B1) and supperpourous hydrogel (B2 and B3) (0.01-0.1) g/500 mL, we could conclude that the optimum concentrations that gave the best mud formulation were 0.1 g for MB1, 0.07 g for MB2 and 0.03 g for MB3.

#### **Rheological properties**

Rheological properties for the samples before and after treatment with the prepared hydrogel were determined to assess their response to the used activator and consequently the ability to be used as drilling fluids. All rheological and filtration tests conducted in this study are based on the American Petroleum Institute (API) and Oil Companies Materials Association (OCMA) specifications. Apparent viscosities (AV), plastic viscosity (PV), yield point (YP) were measured according to API specifications using (Fann viscometer model 35 SA) and determined by calculating the relationship between shear rate and shear stress, where shear stress was taken from a dial reading that was in the degrees of a circle according to equations (1-4).

Shear rate $(s^{-1}) = rpm \times 1.7034$	(1)
Apparent viscosity, $cp = (600 \text{ rpm reading}/2)$	(2)
Plastic Viscosity, cp = (600 rpm reading) - (300 rpm reading)	(3)
Yield Point, $lb/100 \text{ ft}^2 = (300 \text{ rpm reading}) - (Plastic viscosity)$	(4)

# Determination of gel strength and thixotropy of the mud

The gel strength was taken after agitation of the mud in the cup for 10 sec., and then after the mud has rested in the cup for 10 min. Thixotropy is the difference between the low readings after 10 s and 10 min.

# Effect of temperature on the rheological properties procedure

By using Fann cup heater formulated mud (MB1 , MB2 and MB3 ) (21 gram of untreated Na- bentonite + 500 ml fresh water + the optimum concentration for each prepared composite were mixed for 20 minutes and cured overnight.

- 1. The mud sample was stirred for 15 min, before the rheological properties were measured.
- 2. Put the mud in cup heater and gradually raise the temperature from 25 to  $90^{\circ}$ C.
- 3. The rheological properties were measured at different temperatures and the results were compared with the reference mud sample (MR).

# **Filtration test**

API fluid loss test was carried out by using a standard filter (Ofite) press at 100 psi pressure for 30 min at room temperature. The final volume of filtrate after 30 min in ml was noted as API filtrate and filter cake description for all samples were studied.

# Mud weight (density)

The mud weight (density) in pounds per gallon 1b/gal for the mud batches MB1,

MB2 and MB3 were determined by using mud balance model (140) and compared to the mud weight of MB0 and MR.

#### **X-Ray diffraction**

X-ray diffraction test was carried out to the untreated Na-bentonite clay B<sup>o</sup> before and after treatment with the new prepared hydrogel B1, B2 and B3. Determination and identification of different crystalline phases of samples can be carried out 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  $0.02^{\circ}$ /s. The reflection peaks between  $2\Theta = 2^{\circ}$  and  $70^{\circ}$ , corresponding spacing (d, Å) and the relative intensities (I/Io) were obtained. The diffraction charts and the relative intensities were obtained and compared with ICDD file. XRD was carried out at Egyptian Petroleum Research Institute (EPRI).

#### Scanning electro microscope

The morphology of local Egyptian untreated Na-bentonite before and after treatment with the new prepared hydrogel B1, B2 and B3 were examined by scanning electron microscope model JMS 5300 Jeol in egyptian petroleum research institute (EPRI).

## **RESULTS AND DISCUSSION**

# Preparation of super absorbent hydrogel (SH) and highly super absorbent hydrogel (PHSHs)

Hydrogel are cross-linked hydrophilic polymers with a network structure, which are able to absorb large amounts of water<sup>40,41</sup>.

In the synthesis procedure of superabsorbent hydrogel (SH) B1, acrylic acid is the monomer, methylene bisacrylamide is used as across linker, ammonium persulfate (APS) as initiator and sodium metabisulfite (NaHSo<sub>3</sub>) as accelerator. After addition of NaHSO<sub>3</sub>, gelation point was observed during 20-50-s at 32-35 c. After the gelation point the exothermic polymerization proceeded very fast, and the temperature rose to 90°C in less than 2 min.

Recently, many efforts have been made to improve the swelling rate of superabsorbent hydrogel (SH) for improving applicability. So, the swelling rate of superabsorbent hydrogels can be enhanced through the creation of porosity structure. Herein the foam technique have been used to create the porous structure of hydrogel. Therefore, in the synthesis of highly porous superabsorbent hydrogels (HPSHs) B2<sup>42</sup>, all ingredients were

used as in synthesis process of (SH). In addition, both  $NaHCO_3$  and acetone as porous generators were used to procedure B3 or, NaHCO<sub>3</sub> alone to procedure B2. In the polymerization process, the important factors that influence synthesis of (HPSHs) was the PH of the acrylic acid monomer solution. At the pH 5.0 (HPSHs) with well-distributed pours were produced because of the stability and the proper formation rate of the foam. Also, the sequence addition of the components as {APS -(SBC)or(-SMBS-acetone) has significant importance. The SBC was added to the polymerization mixture. It reacted with the nonneutralized acidic groups of the acrylic acid monomer and carbon dioxide gas bubbles were evolved, then (SMBS) was added rapidly. Gelation point was observed in 20-50-s periods. Carbon dioxide bubbles could not escape from the very viscous mixture and thus they were trapped and consequently created a foamy structure. In case of both foaming agents  $(NaHCO_3 and acetone)$ . Before the gelation point carbon dioxide bubbles produced as mentioned above, the gas bubbles could not escape easily from the highly viscous mixture after gelation and subsequently caused foam formation. At the same time the exothermic polymerization rose the temperature in few seconds caused the acetone to consequently produce hydrogel with highly porous structure.

#### Swelling equilibrium

The swelling equilibrium were measured for samples B1, B2 and B3. Table 1 showed that the more porous structure cause the more swelling capacity, which confirms the superiority of the two–foaming agent.

Hydrogel -	Porous ge	Swelling capacity	
	Acetone (mL)	NaHCO <sub>3</sub> /g	(g/g)
B1	-	-	120
B2	-	0.2	255
B3	1	0.2	297

Table 1: Swelling equilibrium of the prepared hydrogel

#### **Rheological properties**

# Rheological properties of water-based mud treated with prepared (SH) and (SPHAs) composite

The new prepared hydrogel B1, B2 and B3 were evaluated as a viscosifiers and filter loss reducer additives for water – based mud. The three mud batches MB1, MB2 and MB3

were formulated from untreated Na-bentonite and treated with different concentration of the new prepared hydrogel B1, B2 and B3, respectively.

Table 2, 3 and 4 showed the treatment of water – based mud MB1, MB2 and MB3.

Table 2: The rheological properties of water based-mud formulated of untreated<br/>Na-bentonite with difference concentration of the new prepared super<br/>absorbent hydrogel (B1)

Mud type	Conc.	AV (aP)	PV	YP (1b/100£ <sup>2</sup> )	Gel s (1b/1	Thixtropy	
	$01 S_1(g)$	(cr)	(cr)	(10/10010)	G10 (sec)	G10 (min)	- (1b/100 ft <sup>-</sup> )
6% of untreated bentonite		8.5	6	6	0	6	0
	+0.01	8.5	6	5	6	6	0
	+0.02	9	5	8	8	7	0
	+0.03	9.5	5	9	8	8	0
	+0.04	10	5	10	8	9	1
MB1	+0.05	11	6	11	8	9	1
	+0.06	12	7	10	10	12	2
	+0.07	13	8	10	11	12	1
	+0.08	13.5	8	11	12	14	2
	+0.09	14.5	9	11	13	15	2
	+0.1	16	10	12	15	17	2
	+0.11	17	10	14	16	18	2
	+0.12	19	11	16	17	19	2

From the above results showed in Table 2, we can conclude that water-based muds which consist of 6% non-treated Na-bentonites and different concentrations of the prepared B1 ranged from 0.01 to 0.12 g. showed rheological properties : Apparent viscosity (AV) increased from (8.5 to 19 cP), Plastic viscosity (PV) increased from (6 to 11 cP).

Yield point (YP) increased from (5 to 16 1b/100ft<sup>2</sup>) as the concentrations of the prepared (PHPC) B1 was increased from (0.01 to 0.12 g).

The gel strength  $G10_{sec}$  ranged between (6 to 17 1b/100ft<sup>2</sup>) while gel strength  $G10_{min}$ . ranged between (6 to 19 1b/100ft<sup>2</sup>) and thixotropy was increased from 0 to 2 1b/100ft<sup>2</sup>

From the above result, we consider that (0.1 g) of prepared super absorbent hydrogel (B1) as a selected concentration. It represents the concentration at which the rheological properties of the mud compatible with API specification.

The rheological properties of the water-based mud formulated of 6% non-treated Na-bentonite and different concentrations of the prepared B2 ranged from 0.01 to 0.1 g were illustrated in Table 3.

Mud type	Conc. of	AV (aP)	PV	YP (1b/1006 <sup>2</sup> )	Gel st (1b/1	Thixtropy		
	<b>S</b> <sub>2</sub> ( <b>g</b> )	(cr)	(cr)	(10/10010)	G10 (sec)	G10 (min)	(1D/1001t <sup>-</sup> )	
60% of bentonite		8.5	6	5	6	6	0	
	0.01	9.5	6	7	6	6	0	
	0.02	10	7	6	6	7	1	
	0.03	11	7	8	8	10	2	
MB2	0.04	12	8	8	9	10	1	
	0.05	13	8	10	10	11	1	
	0.06	14	8	12	12	13	1	
	0.07	17.5	6	23	15	16	1	
	0.08	20	6	28	16	18	2	
	0.09	21.5	9	21	20	22	2	
	0.1	22.5	10	25	23	25	2	

 Table 3: The rheological properties of water based-mud formulated of untreated Na-bentonite with difference concentration of the new prepared highly super absorbent hydrogel (B2)

From the above results, we can conclude that water-based muds which consist of 6% Local non-treated Na-bentonites and different concentrations of the prepared B2 ranged from 0.01 to 0.1 g. The rheological properties: Apparent viscosity (AV) increased from (8.5 to 22.5 cP), plastic viscosity (PV) was increased from (6 to 10 cP).

Yield point (YP) increased from (5 to 25 1b/100ft<sup>2</sup>) as the concentrations of the

prepared (B2) was increased from (0.01 to 0.1 g). The gel strength  $G10_{sec}$ . ranged between (6 to 23 1b/100ft<sup>2</sup>) while gel strength  $G10_{min}$ . Ranged between (6 to 25 1b/100ft<sup>2</sup>) and thixotropy was increased from (0 to 2 1b/100ft<sup>2</sup>). From the above result we consider that (0.7 g) of the prepared B2 as a selected concentration. It represents the concentration at which the rheological properties of the water based mud compatible with API specification.

The rheological properties of the water-based mud formulated of 6% non-treated Na-bentonites and different concentrations of the prepared (B3) ranged from 0.01 to 0.05 g (B3) were illustrated in Table 4.

Mud type	Conc. of	AV (aP)	PV (cP)	YP (1b/100& <sup>2</sup> )	Gel str (1b/10	Thixtropy	
	<b>S</b> <sub>3</sub> ( <b>g</b> )	(()	((1)	(10/10010)	G10 (min)	G10 (sec)	(1b/100ft <sup>-</sup> )
6% of untreated bentonite		8.5	6	5	6	6	0
	0.01	10	6	8	7	8	1
	0.02	13	6	14	9	11	2
MB3	0.03	19	6	22	15	16	1
	0.04	21	6	30	15	17	2
	0.05	28	8	40	17	19	2

 Table 4: The rheological properties of water based-mud formulated of the untreated

 Na-bentonite with difference concentration of the new prepared highly super

 absorbent hydrogel (B3)

From the above results, we can conclude that water-based mud which consist of 6% non-treated Na-bentonite and different concentrations of the prepared hydrogel (B3) ranged from 0.01 to 0.05 g. the rheological properties : Apparent viscosity (AV) increased from (8.5 to 28 cP), and Plastic viscosity (PV) was increased from (6 to 8 cP). Yield point (YP) increased from (5 to 40 1b/100ft<sup>2</sup>) as the concentrations of the prepared composite (3) increased from (0.01 to 0.05 g). The gel strength  $G10_{sec}$ . ranged between (6 to 17 1b/100ft<sup>2</sup>) while gel strength  $G10_{min}$ . ranged between (6 to 19 1b/100ft<sup>2</sup>) and thixotropy changed from 0 to 2 1b/100ft<sup>2</sup>. From the above result, we consider that (0.03 g) of prepared (B3) as a selected concentration. It represents the concentration at which the rheological properties of the mud compatible with API specification

Figs. 1 and 2 illustrated the rheological properties of water-based mud MB1, MB2

and MB3 that treated with the optimum concentration of the new prepared hydrogel B1,B2 and B3, respectively compared to the water based mud of non treated Na-bentonite MBo and reference mud sample MR at  $60^{\circ}$ F.



Fig. 1: Rheological properties of water-based mud MB1, MB2, MB3 compared to MBo and MR



Fig. 2: Gel strength G10 sec and G10 min and Thixtropy of MB1, MB2, MB3 compared to MBo and MR

# Effect of temperature on the rheological properties of water-based mud

In our study, the rheological properties of water-based mud changes with increasing temperature ranging between 60°F and 200°F. For water-based mud consist of untreated Na-bentonite and treated with the new prepared hydrogel B1, B2 and B3 we selected 0.1 g

of B1, 0.07 g of B2 and 0.03 g of B3 as optimum concentration for the corresponding waterbased mud formulation MB1, MB2 and MB3 and then study the effect of temperature on the rheological properties as the temperature increasing ranging between 60°F and 200°F. Comparing the result with MR mud batch Fig. 3 showed that:

For  $M_R$ : The apparent viscosity increased from 17.5 cP to 32.5 cP while the plastic viscosity increased from 5 cP to 23 cP and the yield point also decreased from 25 lb/100 ft<sup>2</sup> to 19 lb/100 ft<sup>2</sup>.

The increase in the rheological properties (apparent viscosity and plastic viscosity will lead to increase in mud weight and this will lead to a thick filter cake).

**MB1**: The apparent viscosity showed slightly increased from 16 cp to 18 cp as the temperature increased from  $60^{\circ}$  F to  $140^{\circ}$  F, then decreased to 16 cp as the temperature increased up to  $200^{\circ}$  F.

Plastic viscosity (pv) kept its value constant (8 cp) as the temperature increased from  $60^{\circ}$  F to  $140^{\circ}$  F and it increased to 10 cp as the temperature raised to  $200^{\circ}$ F.

The yield point (yp) increased from 16 1b/100 ft<sup>2</sup> to 20 1b/100 ft<sup>2</sup> as the temperature increased from 60°F to 140°F and as the temperature increased up to 200°F, it caused a decrease of (yp) up to 12 1b/100 ft<sup>2</sup>

**MB2**: Apparent viscosity (AV) increased from 17.5 cp to 21 cp as the temperature increases from  $60^{\circ}$ F to  $160^{\circ}$ F and it showed slightly decrease up to 17.5 cp at  $200^{\circ}$ F

Plastic viscosity (PV): kept its value constant (8 cp) as the temperature increased from  $60^{\circ}$ F to  $200^{\circ}$ F.

Yield point (YP): the yield point increased from 23 1b/100 ft<sup>2</sup> to 25 1b/100 ft<sup>2</sup> as the temperature increased from  $60^{\circ}$ F to  $160^{\circ}$ F then it showed a slight decrease to 25 1b/100 ft<sup>2</sup> at  $200^{\circ}$ F.

**MB3**: Apparent viscosity (AV) increased from 19 cp to 21.5 cp as the temperature increases from  $60^{\circ}$ F to 140 ° F and it showed gradually decrease up to 19 cp at  $200^{\circ}$ F.

Plastic viscosity (PV): Ranged between 8 cp and 7 cp as the temperature increased from  $60^{\circ}$ F to  $200^{\circ}$ F.

Yield point (YP): The yield point increased from 22 1b/100 ft<sup>2</sup> to 26 1b/100 ft<sup>2</sup> as the temperature increased from 60°F to 160 ° F then it showed a slight decreased up to 22 1b/100 ft<sup>2</sup> at  $200^{\circ}$ F.

From the above result, we conclude that on treating the water based mud with carboxymethylcellulose (MR) the rheological properties increased with increased temperature and this led to high mud weight that led to thick mud cake but on treatment the water-based mud with the new prepared super absorbent hydrogel B1 and highly super absorbent hydrogel B2 and B3 the increased in the temperature didn't affect the rheological properties to get high values, so, the mud treated with the new hydrogel B1, B2 and B3 keep their mud weight constant and this lead to formation of thin mud cake.



Fig. 3: Rheological properties of formulated water-based mud MB<sub>1</sub>, MB<sub>2</sub> and MB<sub>3</sub> at varied temperatures compared to MR

#### Effect of temperature on gel strength and thixotropy

Water-based mud treated with the convention viscosifiers and filter loss additives (CMC) (MR) Fig. (4) showed that :

MR: G10 sec increased from 12 1b/100 ft<sup>2</sup> to 22 1b/100 ft<sup>2</sup>.

G10 min increased from 14 1b/100 ft<sup>2</sup> to 34 1b/100 ft<sup>2</sup>.

**Thixtropy:** Increased from 2 1b/100 ft<sup>2</sup> to 12 1b /100 ft<sup>2</sup> as the temperature increased from  $60^{\circ}$ F to  $200^{\circ}$ F.

**MB1**: G10 sec. increased from 6 1b/100 ft<sup>2</sup> to 10 1b/100 ft<sup>2</sup> as the temperature increased from 60°F to 140°F then it decreased to 9 1b/100 ft<sup>2</sup> at 200°F.

G10 min increased from 6 1b/100 ft<sup>2</sup> to 12 1b/100 ft<sup>2</sup> as the temperature increased from  $60^{\circ}$ F to  $140^{\circ}$ F then it decreased to 10 1b/100 ft<sup>2</sup> at  $200^{\circ}$ F.

**Thixtropy:** As the temperature increased from  $60^{\circ}$ F to  $200^{\circ}$ F the thixtropy intravels between 0 1b/100 ft<sup>2</sup> to 1 1b/100 ft<sup>2</sup>.

**MB2**: G10 sec increased from 6 1b/100 ft<sup>2</sup> to 8 1b/100 ft<sup>2</sup> as the temperature increased from 60°F to 140°F then it decreased again up to 7 1b/100 ft<sup>2</sup> at  $200^{\circ}$ F

G10 min increased from 7 1b/100 ft<sup>2</sup> to 10 1b/100 ft<sup>2</sup> as the temperature increased from  $60^{\circ}$ F to  $140^{\circ}$ F then it decreased to 8 1b/100 ft<sup>2</sup> at  $200^{\circ}$ F

**Thixtropy:** As the temperature increased from  $60^{\circ}$ F to  $200^{\circ}$ F the thixtropy in travels between 1 1b/100 ft<sup>2</sup> to 2 1b/100 ft<sup>2</sup>.

**MB3**: G10 sec increased from 7 1b/100 ft<sup>2</sup> to 9 1b/100 ft<sup>2</sup> as the temperature increased from 60°F to 140°F then it decreased again up to 7 1b/100 ft<sup>2</sup> at  $200^{\circ}$ F.

G10 min increased from 8 1b/100 ft<sup>2</sup> to 11 1b/100 ft<sup>2</sup> as the temperature increased from  $60^{\circ}$ F to  $140^{\circ}$ F then it decreased to 8 1b/100 ft<sup>2</sup> at 200 °F.

**Thixtropy:** As the temperature increased from  $60^{\circ}$ F to  $200^{\circ}$ F the thixtropy intravels between 1 1b/100 ft<sup>2</sup> to 2 1b/100 ft<sup>2</sup>.

High gel strength of MR can cause many problems and a thick filter cake formed as a result of high gel strength at high temperature while in the water based mud treated with super absorbent hydrogel B1 and highly super absorbent hydrogel (B1, B2 and B3) as the temperature increased the gel strength don't increase to high values. It almost kept their values constant with different temperature





#### Relationship between shear stress and shear rate of water-based mud

Share rate-shear stress relationship for water-based mud  $MB_1$ ,  $MB_2$  and  $MB_3$  compared to (MR) Fig. 5 showed that the share rate shear stress Figs. have a non linear ship this proved that the mud obeyed the power low model for non-Newtonian fluid.

Also, the non linear relationship between share rate and shear stress indicated that the muds are pseudoplastic.



Fig. 5: Shear rate-shear stress relationship of MB<sub>1</sub>, MB<sub>2</sub> and MB<sub>3</sub> at 60°F, 120°F and 180°F compared to MR

#### Effective viscosity of water-based mud

Plotting viscosity of mud versus the value of shear rate are illustrated in Fig. 6. The testing result showed that : the effective viscosity of the mud decreased with increased shear rate. These results are similar to field mud ( $M_R$ ) formulated with the reference mud from the Fig. 6. We conclude that: the values of the effective viscosity verses the shear rate of waterbased mud treated with the new prepared super absorbent hydrogel B1 and highly super absorbent hydrogel B2 and B3 are extremely shear thining<sup>43</sup>.



Fig. 6: Effective viscosity of of MB<sub>1</sub>, MB<sub>2</sub> and MB<sub>3</sub> at 60°F, 120°F and 180°F compared to MR

## **Filtration properties**

The filter loss of water mud was measured under the effects of pressure (100 PSI) after 30 min. Table 5 illustrated the standard filter press at 100 PSI after 30 min and the correct filter loss determined for the mud batches compared to the reference mud batch (MR). The results showed that the formulated mud batches (MB<sub>1</sub>, MB<sub>2</sub>, MB<sub>3</sub>) showed lower filter loss than that of the reference and untreated Na-bentonite mud batches (MR and MB<sub>0</sub>). So, it can be concluded that the prepared hydrogel provide protection against water loss through binding of free water, blocking pores and forming a tight filter cake. Both the prepared hydrogel and the Na-bentonite have the ability to chemically bind water to either the polar sites on the clay platelets or to the composite molecules and form a tight impermeable layer. By binding the water, the viscosity of the mud increases, so it becomes

more resistant to flow into the porous formation. The filter cake of the mud batches  $MB_1$ ,  $MB_2$  and  $MB_3$  were of lower thickness than MR and MBo. pH value of the filtrate was of order  $MB_3 > MB_2 > MB_1 > MR > MB_0$ . The mud weight (density) of  $MB_1$ ,  $MB_2$  and  $MB_3$  was 8.6 1b/gal, which is equal to the density of  $MB_0$  mud batch while the density of MR treated with the (CMC) increased to 10 1b/gal so the thickness of filter cake increased.

Mud type	Filtrate loss mL after 30 min	Filter cake description	Mud weight (density) 1b/gal
Мо	21 mL	Filter cake thickness (mm) = 1.9 mm	8.6
		Texture of f.c = porous, non comprisable, elastic	
		Flexibility of $f.c = flexible$	
		Hardness of $f.c = soft$	
		Filtration pH value = $7.5$	
MR	15 mL	Filter cake thickness $(mm) = 2.5 mm$	10
		Texture of f.c = porous, non comprisable, elastic	
		Flexibility of $f.c = flexible$	
		Hardness of $f.c = soft$	
		Filtration pH value = 8.5	
MB1	11 mL	Filter cake thickness $(mm) = 1.2 mm$	8.6
		Texture of f.c = porous, non comprisable, elastic	
		Hardness of $f.c = semi soft$	
		Filtration pH value = 8.5	
MB2	10 mL	Filter cake thickness $(mm) = 0.9 mm$	8.6
		Texture of f.c = porous, non comprisable, elastic	
		Hardness of f.c =semi soft	
		Filtration pH value = 9.5	
MB3	9 mL	Filter cake thickness (mm) = $0.7 \text{ mm}$	8.6
		Texture of f.c = porous, non comprisable, elastic	
		Hardness of $f.c = semi soft$	
		Filtration pH value = 10.4	

Table 5:	Filter	loss (n	nL), :	filter	cake	(mm)	descriptions	and	mud	weight	for	mud
	batche	es MB <sub>1</sub> ,	MB <sub>2</sub>	and I	MB <sub>3</sub> c	ompar	ed to the mu	d bat	ches M	IR and I	MBo	

# **X-Ray diffraction**

Fig. 7 represents the XRD pattern of intercalation of Na montomorillonite clay with the new prepared (SH) and (HPSHs), which are simplified to (B1, B2, B3). The observed basel spacing (d001) of B1 –MMT, B2 – MMT and B3 – MMT had the values 13.32, 13.89 and 14.99 Å, respectively, which is greater than the basel spacing (d001) for NaMMT clay (12.98Å). The intercalation of B3 into the clay is greater than that of B2 and B1. This showed that B3 was strongly attached to MMT because of the higher van der waals and electro static interactions with the negatively silicate layers.



# Fig. 7: X-ray diffraction analysis of MB<sub>1</sub>-MMT, MB<sub>2</sub>-MMT and MB<sub>3</sub>-MMT compared to M<sub>0</sub>-MMT, (M = Montmorillonite, C = Calcite, K = Kaolinite and Q = Quartz)

Table 6 showed that a significant diffraction pattern for Na montmorollonite at  $2\theta = 6.9$  corresponding to a layer spacing of 12.55 Å was calculated using Bragg's law: n  $\lambda = 2d\sin\theta$ 

where n is the whole number of wavelengths,  $\lambda$  is the wavelength of the X-ray (for Cu-target used here, the value is 1.54 Å), d is the interspace distance, and  $\theta$  is the angle of incident radiation<sup>44</sup>.

For the Na-MMT with the new prepared hydrogel B1, B2 and B3, this peak is shifted to lower angles with an increase of the d-spacing of the galleries of the treated Na MMT. The XRD data also indicate that the new hydrogel were arranged as a flat lying molecule in monolayers into the interlayer space of clay.

Material	Basal spacing region d <sub>001</sub> (Å)	Position (20°)
Mo-MMT	12.98	6.84
MB <sub>1</sub> -MMT	13.32	6.49
M <sub>B2</sub> -MMT	13.89	6.15
MB <sub>3</sub> -MMT	14.99	5.98

Table 6: Basal spacing values  $(d_{001})$  and diffraction angel of the untreated Na-bentonite and MB<sub>1</sub>, MB<sub>2</sub> and MB<sub>3</sub>

# Scanning electronic microscope (SEM)

Fig. 8 shows SEM micrograph of Na-MMT, B1 – MMT, B2 –MMT and B3- MMT (A, B, C and D), respectively. The samples Na-MMT, B1-MMT presented a compact structure and no porosity is absorbed.



Fig. 8: SEM micrographs of (A) Na-MMT, (B) B<sub>1</sub>-MMT, (C) B<sub>2</sub>-MMT and (D) B<sub>3</sub>-MMT

Fig. (A and B) in contrast a porous structure was clearly observed in the samples B2 –MMT and B3-MMT (Fig. C and D), but the porous for B3–MMT is more dense and uniform than B2-MMT. Moreover the highly porous superabsorbent hydrogel (HPSHs) B3 showed higher swelling capacity than (HPSHs) B2 (see section 3-2), which conforms that (actone and NaHCo<sub>3</sub>) are the superior pore-foaming agent than (NaHCO<sub>3</sub>) alone. Therefore, incorporating the HPSHs in the mud cake increase the system porosity and subsequently caused filtration reduction.

#### Application of the power law model to the rotary viscometer data

The flow behavior index (n) and consistency index (K) values are calculated according to the power law model Herschel-Buckley model and is given by –

$$\tau = \tau_0 + K.\gamma^{n\,45}$$

 $\tau$  = Shear stress

 $\tau_0$  = Yield stress or yield point

K = The consistency factor (viscosity in case of a newtonian or bingham fluid)

 $\Gamma$  = Share rate

n = The power of low exponent (rate index) or flow behavior.

Table 7 illustrated the flow behavior index (n) and the consistency index (k) of Mc and  $M_{\text{R}}$ 

	Conc. of	Dial	Dial	Power law model			
Mud type	viscosifier (g/350 cm <sup>3</sup> base fluid)	iscosifier reading at r z/350 cm <sup>3</sup> 600 rpm 3 ase fluid) (R <sub>600</sub> )		Flow behavior index ( <i>n</i> ) dimensionless	Consistency factor (k) lb.sec <sup>n</sup> /ft <sup>2</sup>		
MR	-	35	30	0.222	7.505		
$MB_1$	0.	32	24	0.415	1.808		
$MB_2$	0.	35	29	0.271	5.349		
$MB_3$	0.	38	30	0.341	3.584		

Table 7: The flow behavior index (n) and consistency index (K)

## CONCLUSION

- (i) Highly porous superabsorbent hydrogels (B2 and B3) were successfully synthesized in aqueous medium at room temperature under normal atmospheric conditions. The porosity was induced through different foaming agents (acetone and NaHCO<sub>3</sub>)
- (ii) The comparable non-porous hydrogel (B1) was prepared through a similar procedure with the porous hydrogels except without the addition of any foaming agent (acetone and NaHCO<sub>3</sub>).
- (iii) The study of rheological and filtration properties of water based mud treated with the new (SPHPs) and (SH) indicated that (SPHPs) have the best results for improving of the rheological properties and reducing filter loss and mud cake of water-based mud than the corresponding reference water-based mud treated with the conventional filter loss (carboxymethylcellulose).
- (iv) The results of mineralogical studies and scanning electro microscopy (SEM) for the water based mud treated with (SH) and (SPHPs) indicated that both of the new prepared (SH) and (SPHPs) were intercalated and attached strongly to the local Na-bentonite clay.
- (v) The highly porous superabsorbent hydrogel B3 has the highest performance properties at cocnteration (0.06 g/L).
- (vi) Gel formation of the prepared hydrogel compound can block the pores and decrease the permeability.

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