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# Activation Iraqi bentonite for using as drilling mud

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**Abstract.** Drilling fluid is very important in drilling a well because it plays a vital role in all drilling operations. Drilling fluid should have popular properties that simplify safe and easy completion of the well. Because of the big cost of transporting and imported clays that used as drilling fluid, Iraqi bentonite must be enhanced to use as drilling fluids due to its poor rheological and filtration properties, and stability. Iraqi bentonite has a limited usage in oil field because it is Ca- bentonite. The research has studied the usage of bentonite and its activation by NaOH in drilling fluid. The results show that Iraqi Ca-bentonite reaches to API specifications while the indirect activation includes activated Ca-bentonite by NaOH with different concentrations, HCL then NaOH without temperature and HCL then NaOH with temperature. Then activated bentonite is used as drilling mud. The results show that the indirect activated bentonite does not reach API specification, where XRF analysis show that an ion exchange between Ca and Na is not enough to convert Ca- bentonite to Na- bentonite.

## 1. Introduction

Drilling fluids 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 insure well safety and minimizing fluid loss across permeable formations by forming a filter cake on the wall of the well bore [1]. Bentonite is the main constituent of water- based mud due to high viscosity, good swelling and lower filter loss [2]. Bentonite is a smectite clay [3], because montmorillonite clay has 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. Bentonite often contains clay minerals such as montmorillonite, illites, kaolinites, chlorites and non-clay components such as quartz and feldspar in appreciable amounts. A good quality bentonite should contain mainly montmorillonite [4]. There are two types of bentonites: sodium bentonite (Na-bentonite), which has a high swelling capacity, and calcium bentonite, which is non-swelling clay and bentonite spontaneously forms colloidal dispersion in water. Bentonite is mainly composed of layers including silica and alumina sheets linked together and arranged on top of each other. Bentonite lamellar platelets are packed together by electrochemical forces and form aggregates that contain interposition water. The platelets are composed of a central octahedral alumina ( $\text{Al}_2\text{O}_3$ ) layers and two tetrahedral silica ( $\text{SiO}_2$ ) layers [5].

Iraqi bentonite exhibits high filtration loss and does not develop sufficient viscosity so, It cannot meet the API (15 cp minimum viscosity at 600rpm, 15cm<sup>3</sup> filtration losses) standards. By alkali activation or by



introducing some polymers additives, it is possible to upgrade raw bentonite to meet the above standard and thus require appropriate activation formulation. This application typically employs various additives such as inorganic salts, MgO salts; the inorganic salts improve the swelling or viscosity [2]. The chemical compositions which determine properties of the clays i.e. layer charge, cation exchange capacity,

adsorption capacity and morphology, can be varied depending upon their origins. These factors play a significant role in the modification of the natural clays. A number of methods for modifying clay minerals have been studied with a view to enhancing their adsorption capacities, including acid activation, treatment with cationic surfactants, clay-rubber composite, thermal activation, polymer addition by interparticle polymerization, binding of inorganic and organic anions, and grafting of organic compounds. Among these, acid activation is one of the most commonly used modification techniques because it is a simple and low-cost process [6].

The aims of this work are to enhance the quality of Iraqi bentonite by decreasing non-clay minerals, and activate it by Na ions to enhance the rheological and filtration properties to be used as drilling fluid.

## **2. Iraqi Bentonite Characterization**

### *2.1 Mineral Composition Analysis*

Mineral composition of Iraqi bentonite was measured by X-Ray diffraction (XRD) using SHIMADZU XRD-7000.

### *2.2 Chemical Composition Analysis*

Chemical composition for blank bentonite and activated bentonite were determined by (XRF) Spectrometry using SHIMADZU XRF-1800, the analyses for Iraqi bentonite are shown in Table 3, Table 6 and Table 12 respectively

### *2.3 Specific Surface Area (SSA) and Particle Size Distribution (PSD)*

The technique selected for establishing the correlations in the determination of the specific surface area is wet laser diffraction technique. Wet laser diffraction allows the particle size distribution of the test sample to be determined as direct parameter. The distribution was determined using MALVERN MASTERSIZER 2000 laser diffraction instrument. In this instrument, the particles cross a laser light beam and the scattered light is collected at 52 detectors [7].

## **3. Experimental Works**

### *3.1 Preparing Iraqi bentonite*

Iraqi Bentonite taken from Trefawi formation/AL-Anbar region/ Iraq, was grinded to powder and sieved into size  $<75\mu\text{m}$ , after that it was dried in oven at  $180^\circ\text{C}$  for 12 hr. Then, 22.5 g of Iraqi bentonite was mixed with 350 ml of distilled water for 20 minutes and left for 24 hr to hydrate, then the mud properties were measured.

### *3.2 Activation Iraqi Bentonite*

#### *3.2.1 Direct Activation*

Iraqi bentonite was activated by adding 0.8, 1.6, 2.4, 3.2 and 4 g of NaOH directly to 22.5 g of Iraqi bentonite and 350 ml of distilled water.

#### *3.2.2 Indirect Activation*

Many processes (heating at different heating temperature, mixing at different hours and addition of inorganic materials) were made for Iraqi bentonite before API standards mud (22.5 g bentonite with 350ml distilled water for 20 minutes) to make a good bentonite that can be used directly by mixing with distilled water without any addition and give it to oil companies for using it instead of commercial bentonite.

##### *3.2.2.1 Alkali Activation*

10 g of prepared Iraqi bentonite was mixed with 1, 3, 5, 7 and 9 g of NaOH dissolved in 100 ml distilled water with mixing time 2, 4 & 6 hours. The mixture was dried in at  $105^\circ\text{C}$ . Then, 22.5 g of the dried mixture was mixed with 350 ml distilled water for 20 minutes and left for 24 hours in a closed container to hydrate. After that, the mud properties were measured.

#### 3.2.2.2 Alkali Activation and Thermal Activation

10 g of prepared Iraqi bentonite was mixed with 1, 3, 5, 7 and 9 g of NaOH dissolved in 100 ml distilled water with mixing time 2, 4 & 6 hours and mixing temperature 70 & 100 °C. The mixture was dried in Electrical Oven at 105 °C, 22.5 g of the dried mixture was mixed with 350 ml distilled water for 20 minutes and left for 24 hours in a closed container to hydrate. Then, the mud properties of the mixture were measured.

#### 3.2.2.3 Alkali Activation and Acid Activation

10 g of prepared Iraqi bentonite was mixed with 2 ml (0.5 M) of HCl solution for 30 minutes. The acid to bentonite ratio was 2:10 (ml/g). Then, the mixture was washed several times until removing  $\text{Cl}^-$  ions and pH approximately reached 7. After that, the mixture was mixed with 1, 3, 5, 7 and 9 g of NaOH dissolved in 100 ml distilled water for 2 hours at room temperature (35 °C). The mixture was dried for 105 °C. 22.5 g of the dried mixture was mixed with 350 ml distilled water for 20 minutes and left 24 hours in a closed container to age [8]. The mud properties of the mixture were measured.

### 4. Mud Testing

#### 4.1 Rheological Properties

Rheological properties were measured using OFFITE Viscometer (Model 900). Rheological properties were used to indicate solids buildups flocculation or deflocculation of solids, lifting and suspension capabilities, and to calculate hydraulics of a drilling fluid [9]. Rheology is the study of how matter deforms and flows, it is primarily concerned with the relationship of shear stress and shear rate and the impact these have on flow characteristics inside pipes and annular spaces. Viscosity is a resistance to flow. The apparent viscosity is as half of the viscometer reading at 600RPM [10]. Plastic Viscosity (PV) is defined as the 600RPM minus the 300RPM shear stress reading. The plastic viscosity depends mainly on the friction between particles. Yield Point (YP) is defined as the 300RPM shear stress reading minus the plastic viscosity. Yield point is a measure of the attractive forces between clay particles in the mud under flowing conditions. It is also a measure of hole cleaning capabilities of the mud [11]. Gel Strength denoted the thixotropic properties of the mud and is a measurement of the attractive forces of the mud while at rest or under static conditions.

#### 4.2 Mud Density

Density is defined as weight per unit volume. It is expressed in pounds per gallon (lb/gal), pounds per cubic foot (lb/ft<sup>3</sup>), and kilograms per cubic meter (kg/m<sup>3</sup>), or compared to the weight of an equal volume of water, as specific gravity (SG). The pressure exerted by a static mud column depends on both the density and the depth; therefore it is convenient to express density in terms of pounds per square inch per foot (psi/ft). To prevent the inflow of formation fluids in the wellbore, the pressure of the mud column must exceed the pore pressure, which is pressure exerted by the fluids in the pores of the formation. Mud density was measured using mud balance.

#### 4.3 pH

pH is a value representing the hydrogen ion concentration in a liquid. The pH indicates acidity or alkalinity of a drilling fluid. The pH of a drilling fluid can affect its performance and is important for corrosion control. Low pH (Acidic) fluid is undesirable because it can corrode the drill string. Normal drilling fluid range between 9.5 to 10.5, higher values are not common [12].

#### 4.4 Filtration properties

There are two types of filtration: Static and dynamic. Static filtration test is used to indicate filter cake quality and filtrate volume loss for a drilling mud under specific test conditions. Filtration characteristics are affected by the types and quantities of solids and their physical and chemical interactions. Temperature and pressure further affect these solids and their interaction [13].

API fluid loss test was carried out using a static filtration apparatus (API Filter press offite apparatus) by placing the mud into a stainless steel chamber with an opening at the bottom. A filter paper was placed on the bottom. The mud was exposed to 100 psi for 7.5 min. and the amount of filtrate volume was measured.

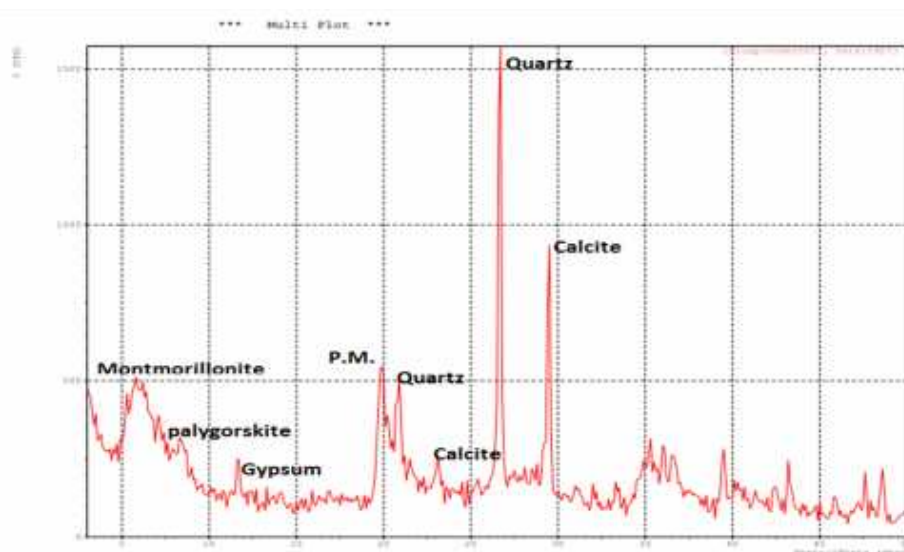
#### 4.5 Stability

Stability of drilling mud was measured by placing drilling mud in a 250 ml glass cylinder and checked the settlement after 2hrs, 4hrs and 24hrs.

### 5. Results and Discussions

#### 5.1 Mineral Composition of Iraqi Bentonite

XRD shows that Iraqi bentonite contains montmorillonite and palygorskite as main minerals and quartz and calcite as impurities. By using special process of grinding, quartz was removed and montmorillonite was increased.



**Figure 1.** XRD of Iraqi bentonite

#### 5.2 Chemical Composition of Iraqi Bentonite

XRF analysis is shown in Table 1, the ratio of  $\text{Al}_2\text{O}_3/\text{SiO}_2$  was 1/3 as expected for montmorillonite which is the main component of bentonite. The ratio of  $[(\text{Na}_2\text{O}+\text{K}_2\text{O})/(\text{CaO}+\text{MgO})]$  for Iraqi bentonite was 0.143 confirming that Iraqi bentonite was Ca-bentonite.

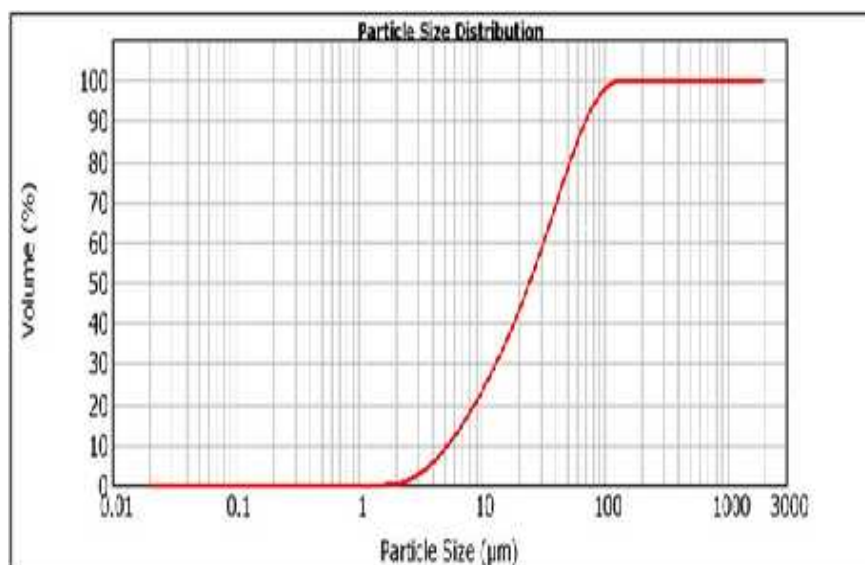
**Table 1.** XRF Analysis of Iraqi bentonite.

$\text{SiO}_2$	$\text{Fe}_2\text{O}_3$	$\text{Al}_2\text{O}_3$	$\text{CaO}$	$\text{MgO}$	$\text{Na}_2\text{O}$	$\text{K}_2\text{O}$
%	%	%	%	%	%	%
52.1	5.71	15.91	7.06	1.79	0.60	0.67

#### 5.3 Particle Size Distribution and Specific Surface Area

Particle size distribution (PSD) analysis is shown in figure 2, 10% of the particle size was smaller than  $5.354\mu\text{m}$ , 50% of the particle size was smaller than  $24.512\mu\text{m}$  and 90% of the particle size was smaller than  $69.96\mu\text{m}$ . the average particle size of Iraqi bentonite inside the powder was  $36.0825\mu\text{m}$ .

The specific surface area (SSA) of Iraqi bentonite diffraction was  $0.449\text{m}^2/\text{g}$ . The technique that the wet laser diffraction was used to measure the specific surface area depends mainly on particle size.



**Figure 2.** Particle size distribution for Iraqi bentonite

#### 5.4 Properties of Iraqi Bentonite

22.5g of prepared Iraqi bentonite was mixed with 350ml distilled water for 20 minutes and left for 24hr to hydrate. Then, Iraqi bentonite properties was measured. The results were shown in Table 2.

The rheological properties of Iraqi bentonite and gel strength were very bad in compared with API specifications. The filtration loss of Iraqi bentonite was very high and the stability is very low. The bad result that Iraqi bentonite has is because of the Ca in the composition of Ib. Iraqi bentonite was undesired clay because it minimized the adsorption and minimized the swelling of Iraqi bentonite. Therefore, many processes of activation were carried out to enhance its properties.

**Table2.** Properties of Iraqi bentonite

AV,cp	2.27
PV,cp	2.35
YP,(lb/100ft <sup>2</sup> )	0.73
YP/PV,(lb/100ft <sup>2</sup> /cp)	0.31
10s gel,(lb/100ft <sup>2</sup> )	0.8
10m gel,(lb/100ft <sup>2</sup> )	1.9
,ppg	8.6
pH	7.1
V <sub>7.5</sub> ,ml	27
V <sub>30m</sub> ,ml	54
Tmc,mm	2.13
Stability,2hr,%	76
Stability,4hr,%	67.2
Stability,24hr,%	50.8

#### 5.5 Activation Iraqi Bentonite

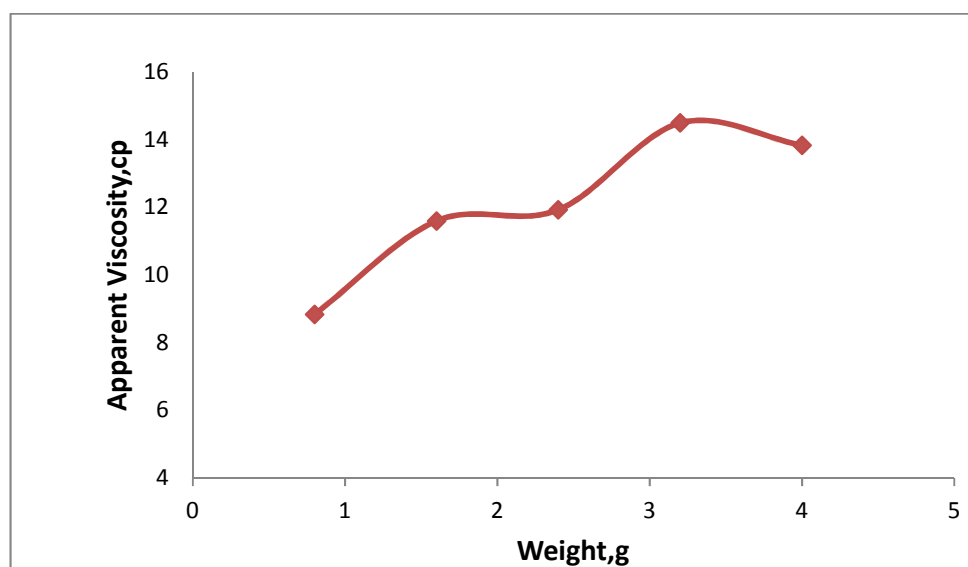
Two Processes of activation were used; direct activation and indirect activation

##### 5.5.1 Direct Activation

Iraqi bentonite was activated with NaoH. 0.8, 1.6, 2.4, 3.2 and 4 g of NaoH added to the API mixture of 22.5g Iraqi bentonite and 350ml distilled water in order to enhance the rheological properties.

**Table 3.** Direct Activation Iraqi bentonite by NaoH

W,g	AV,cp	PV,cp	YP, (lb/100ft <sup>2</sup> )	YP/PV, (lb/100ft <sup>2</sup> /cp)	Gel Strength, (lb/100ft <sup>2</sup> )	
					10,sec	10,min
0.8	8.84	3.94	9.79	2.48	5.9	6.2
1.6	11.6	4.68	13.85	2.96	7.7	6.6
2.4	11.93	4.05	15.76	3.89	7.8	6.9
3.2	14.5	5.67	17.65	3.11	8.6	7.9
4	13.84	5.85	15.98	2.73	8.3	7.3

**Figure 3.** Effect of Direct Activation Iraqi bentonite by NaoH on Apparent Viscosity

By adding NaoH, the viscosity of mud increased and reached the API specifications at 3.2g NaoH. From Figure 3, the viscosity increased when the weight of NaoH increased until adding 4g of NaoH, the viscosity becomes decreasing because there was foam in the mixture. The foam contains many bubbles which disappear causing the mud loss and decreasing in viscosity. other rheological properties such as plastic viscosity, yield point, yield point to plastic viscosity ratio and gel strength were enhanced and approximately accepted compared with API specification.

**Table 4.** Stability Direct Activation Iraqi Bentonite

W,g	Stability %		
	1hr	2hr	24hr
0.8	78.4	73.6	59.2
1.6	98.8	98	88
2.4	100	100	100
3.2	100	100	100
4	100	100	100

When adding 2.4, 3.2 and 4 g of NaoH, the stability has become 100%. The effect of NaoH on stability was very good and very big enhancement in comparison with Iraqi bentonite before activation, which Na ion is interacted with chemical composition of bentonite and Na inters to the composition and becomes Na-bentonite, and Ca was interacted with OH<sup>-1</sup> ion and became Ca(OH)<sub>2</sub>. There is cation exchange capacity between bentonite and NaoH.

**Table 5.** Density, pH and Filtration of direct activation Iraqi Bentonite

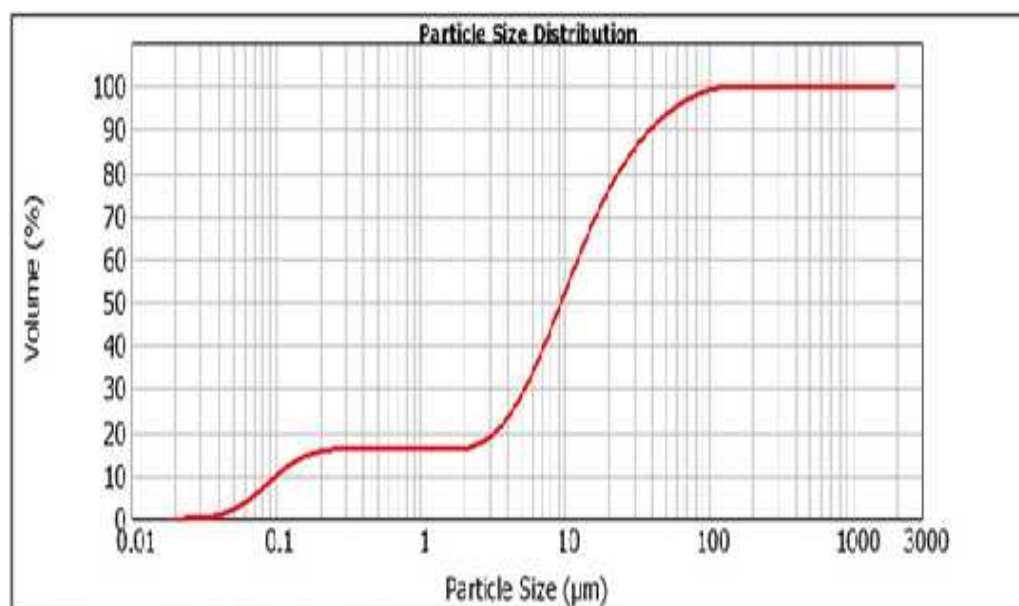
W,g	,ppg	pH	V <sub>7.5</sub> ,ml	V <sub>θ</sub> ,ml	Tmc,mm
0.8	8.6	12.5	65.5	131	5.13
1.6	8.58	12.7	57	114	5.66
2.4	8.55	12.8	50.2	100.4	6.28
3.2	8.53	13	43	86	7.46
4	8.5	13.4	35.4	70.8	8.4

The density of activated Iraqi bentonite by NaoH is accepted while the pH value is approximately high, the filtration loss is very high and must use filtration treatment if activated bentonite could be used as drilling fluid. CMC-LV or polyacrylamide or starch could be added in a small weight to minimize the filter loss.

XRF, particle size distribution and specific surface area were carried out for 3.2 g NaoH add to Iraqi bentonite as the best result of activation as shown in Table 6 and Figure 4 respectively.

**Table 6.** XRF Analysis of activated Iraqi bentonite by NaoH

SiO <sub>2</sub>	Fe <sub>2</sub> O <sub>3</sub>	Al <sub>2</sub> O <sub>3</sub>	CaO	MgO	Na <sub>2</sub> O	K <sub>2</sub> O
%	%	%	%	%	%	%
38..3	4.56	8.34	8.3	1.89	5.41	0.42

**Figure 4.** PSD of Activated Iraqi Bentonite by NaoH

XRF of direct activation Iraqi bentonite by 3.2g NaoH shows a big increase in the Na ion compared with Iraqi bentonite before activation, and the ratio of  $[(Na_2O+K_2O)/(CaO+MgO)]$  was found to be 0.57 confirming that Iraqi bentonite, composition. By XRF, The value of Na<sub>2</sub>O before activation was 0.6 and the value after adding 3.2g NaoH directly was 5.41, indicating that the NaoH interacted with ca-bentonite.

Ca-bentonite+NaoH → Na-bentonite+Ca(OH)<sub>2</sub>

Most of times, the composite Ca(OH)<sub>2</sub> was undesired in the drilling mud. The activated bentonite can be used in formations that have rich calcium or formations that was not affected by calcium.

By PSD analysis, it is shown that the particle size of activated bentonite decreased, where 10% volume of particle size inside bentonite was smaller than 0.103μm, 50% volume of particle size inside bentonite was smaller than 0.103μm, 50% volume of particle size inside bentonite was smaller than 9.433μm, and 90% volume of particle size inside bentonite was smaller than 39.255μm.



The specific surface area of activated bentonite was 13.5m<sup>2</sup>/g. Because the particle size decreased, the specific surface area increased. The increasing of specific surface area gives good indication of high adsorption and high swelling index of activated bentonite.

### 5.5.2 Indirect Activation

The aim of this activation is to make a good bentonite by enhancing its rheological properties. This process was made before the standard API mud (22.5g bentonite and 350ml of distilled water). There were three work activation processes as shown below.

#### 5.5.2.1 Alkali Activation

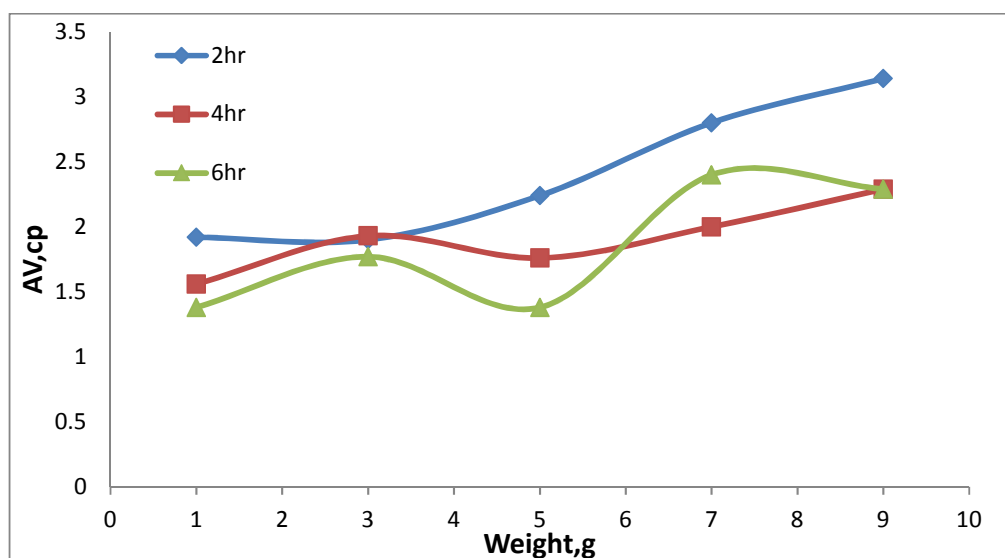
The process includes adding 1,3, 5, 7 and 9 g of NaOH dissolved in 100ml distilled water to 10g lb and mixing for 2, 4 and 6hr to maintain good interaction between molecules. Then, the mixture was washed several times to remove Ca(OH)<sub>2</sub> and decrease high PH resulting from add NaOH. After the pH had become less 12, the mixture dried in oven at 105<sup>0</sup>C and then 22.5g of dried mixture was mixed with 350 of distilled water to test mud properties.

The previous research shown that there is no effect on bentonite properties after 6 hr mixing time<sup>[14]</sup>. Rheological properties of activated Iraqi bentonite by alkali activation are shown in Table 6.below.

**Table 7.** Rheological Properties of Alkali Activation Iraqi bentonite

M	W,g	AV,cp	YP,(lb/100ft <sup>2</sup> )	PV,cp	YP/PV, (lb/100ft <sup>2</sup> /cp)	Gel Strength, (lb/100ft <sup>2</sup> )	
						10 sec	10 min
2	1	1.92	0.85	1.49	0.57	0.2	0.5
	3	1.9	0.9	1.45	0.62	0.8	0.4
	5	2.24	1.27	1.6	0.79	0.5	0.6
	7	2.8	1.2	2.2	0.56	0.7	1.1
	9	3.14	0.74	2.77	0.44	0.5	0.4
4	1	1.56	0.31	1.5	0.21	0.3	0.5
	3	1.93	0.66	1.6	0.41	0.6	0.9
	5	1.76	0.53	1.49	0.35	1.3	0.4
	7	2	0.4	1.8	0.22	1.1	1.4
	9	2.29	0.32	2.13	0.15	1.4	0.9
6	1	1.38	0.84	0.96	0.88	0.1	0.2
	3	1.77	0.9	1.32	0.68	0.8	1.3
	5	1.38	0.21	1.28	0.16	0.4	0.6
	7	2.4	1.4	1.7	0.82	0.9	0.9
	9	2.29	1.6	1.49	1.07	1	0.7

This process failed to activate Iraqi bentonite, the rheological properties were very bad and did not reach the API specification. From the results above, Plastic Viscosity (PV) was very low that mean there was no resistance to flow caused by mechanical friction. From the results above, Yield Point (YP) was very low because there were no attractive forces in muds under flow conditions.



**Figure 5.** Apparent Viscosity of Alkali Activation Iraqi Bentonite

The apparent viscosity has compound effect between plastic viscosity (PV) and yield point (YP). Because of the small values of plastic viscosity and yield point, the apparent viscosity became very low small values. But from figure above, the apparent viscosity was decreased when mixing time was increased, that led to fix the mixing time for 2 hr in alkali and thermal activation, and alkali and acid activation.

**Table 8.** Density, pH and Filtration of Alkali Activation Iraqi Bentonite

S	W,g	,ppg	pH	V <sub>7.5</sub> ,ml	V <sub>0</sub> ,ml	Tmc,mm
2	1	8.55	11.5	19.9	39.8	0.79
	3	8.6	10.7	22.1	44.2	1.1
	5	8.55	11.4	28.6	57.2	1.24
	7	8.5	10.8	22.9	45.8	1.4
	9	8.6	11.66	27.3	54.6	1.17
4	1	8.6	11.34	17.4	34.8	1.22
	3	8.6	11.2	25.2	50.4	0.8
	5	8.55	10.61	22.9	45.8	0.82
	7	8.5	10.9	29.5		0.99
	9	8.55	10.93	34.8	69.6	1.3
6	1	8.55	11.22	17.6	35.2	1.29
	3	8.6	10.7	20.3	40.6	1.22
	5	8.55	10.79	25.3	50.6	1.3
	7	8.5	10.9	27.4	54.8	1.17
	9	8.55	10.54	32.7	65.4	1.08

The filtration loss was very high because the NaOH has no any effect to filtration loss.

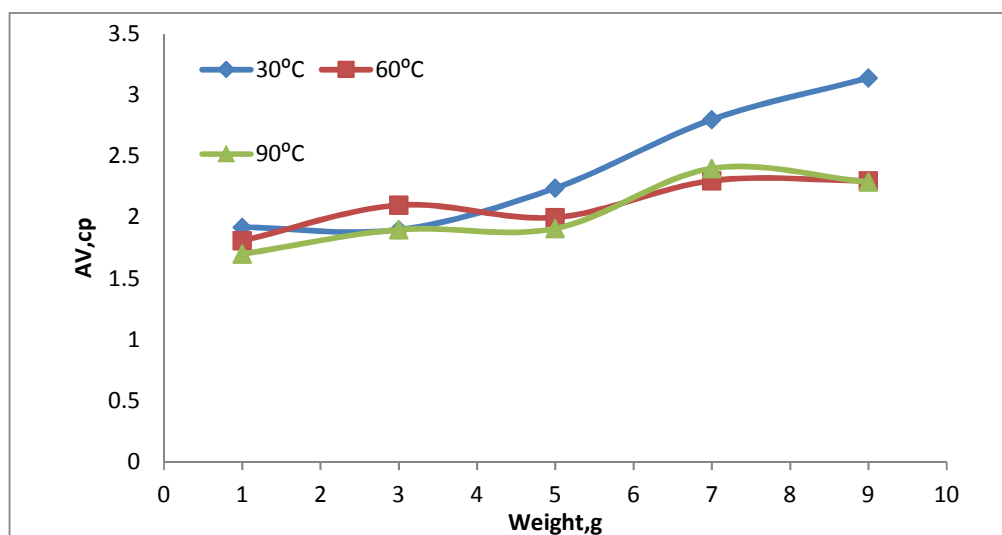
#### 5.5.2.2 Alkali Activation and Thermal Activation

By repeating the alkali activation processes and fixing the mixing time for 2hr, in addition to use the heating in mixing. The heat that was used in mixing was 60°C and 90°C and brought the first results that it was for 2hr without heating in mixing (at room temperature 30°C) just for comparison. The rheological properties of activation Iraqi bentonite are shown in Table 8.

**Table 9.** Rheological Properties of Alkali and Thermal Activation Iraqi Bentonite

T	W,g	AV,cp	PV,vp	YP, (lb/100ft <sup>2</sup> )	YP/PV, (lb/100ft <sup>2</sup> /cp)	Gel Strength, (lb/100ft <sup>2</sup> )	
						10 sec	10 min
30	1	1.92	0.85	1.49	0.57	0.2	0.5
	3	1.9	0.9	1.45	0.62	0.8	0.4
	5	2.24	1.27	1.6	0.79	0.5	0.4
	7	2.8	1.2	2.2	0.56	0.7	1.1
	9	3.14	0.74	2.77	0.44	0.5	0.4
60	1	1.81	1.7	0.22	0.13	0.3	0.2
	3	2.1	1.8	0.6	0.33	0.4	0.7
	5	2	1.7	0.6	0.35	0.4	0.3
	7	2.3	1.8	1	0.55	1.1	0.9
	9	2.3	2.4	1	0.43	0.6	1.1
90	1	1.7	1.5	0.2	0.34	0.3	0.4
	3	1.9	1.58	0.64	0.41	1.2	0.8
	5	1.91	1.7	0.43	0.25	1.3	0.4
	7	2.4	1.8	1.2	0.67	1.2	1.1
	9	2.29	1.49	1.6	1.07	1	0.7

The benefit of heating the mud is to increase the velocity by increasing the interaction between molecules and capacity of ion exchange. Because the rheological properties was not enhanced and reached the API standards, the activation failed. The Plastic viscosity values and yield point values were very low. The adsorption was very low because there was no attractive between the molecules of bentonite.

**Figure 6.** Apparent viscosity of alkali and thermal activation Iraqi bentonite

The Apparent Viscosity was not enhanced by alkali and thermal activation and did not reach the API specification. Apparent Viscosity was depended on plastic viscosity and yield point. Therefore the plastic viscosity and yield point had very small values, the apparent viscosity had low values. Figure 6 shows that the apparent viscosity decreased when the heating was increased.

**Table 10.** Density, pH and Filtration of Alkali and Thermal Activation Iraqi Bentonite

T	W,g	,ppg	pH	V <sub>7.5</sub> ,ml	V <sub>θ</sub> ,ml	Tmc,mm
30	1	8.55	11.5	19.9	39.8	0.79
	3	8.6	10.7	22.1	44.2	1.1
	5	8.55	11.4	28.6	57.2	1.24
	7	8.5	10.8	22.9	45.8	1.41
	9	8.6	11.66	27.3	54.6	1.17
60	1	8.55	10.76	18.4	36.8	0.82
	3	8.6	10.8	22.8	45.6	1.42
	5	8.55	11.61	26.3	52.6	1.71
	7	8.5	11.11	28.7	57.4	1.53
	9	8.5	11.1	33.8	67.4	1.03
90	1	8.55	11.22	31.5	63	1.45
	3	8.5	10.72	25.1	50.2	1.2
	5	8.6	10.86	31.2	62.4	1.32
	7	8.6	10.82	33.3	66.6	1.3
	9	8.55	11.4	45.8	90.6	1.65

Filtration losses are very high because Iraqi bentonite did not make an adsorption with NaOH molecules. Table 9 shows the thermal activation effect on density, pH and filtration properties of Iraqi bentonite.

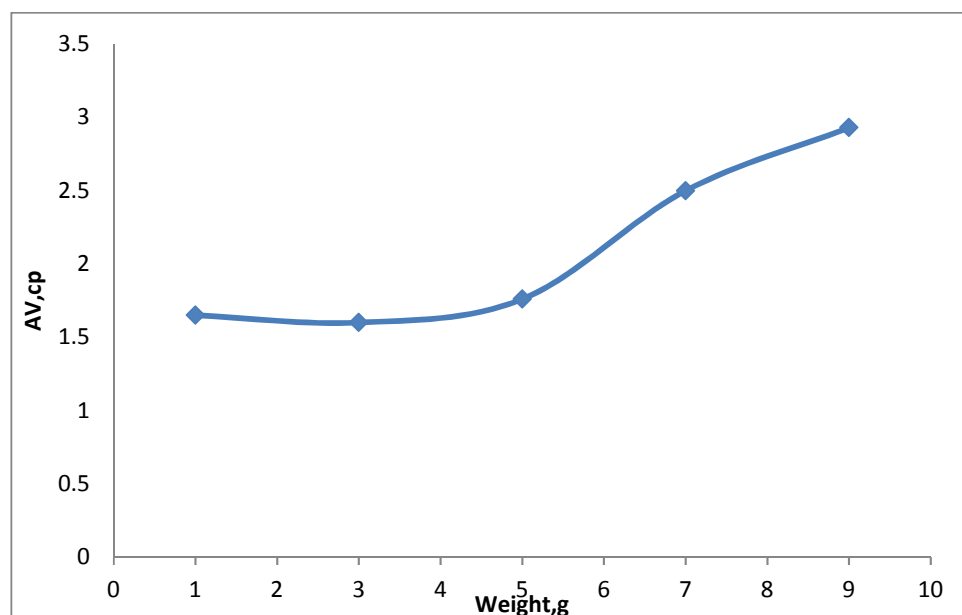
#### 5.5.2.3 Alkali and Acid Activation

HCl is very important in activation processes. By HCl, impurities such as calcite can be removed and by washing the solution of bentonite and HCl, the  $\text{Cl}^{-1}$  is removed and increased the value of PH<sup>[15]</sup>. Iraqi bentonite was treated with HCl first (Acid Activation) and then treated with NaOH (Alkali Activation). Rheological properties of alkali and acid activation Iraqi bentonite are shown in Table 10.

**Table 11.** Rheological Properties of Alkali and Acid Activation Iraqi Bentonite

W,g	AV,cp	PV,cp	YP, (lb/100ft <sup>2</sup> )	YP/PV, (lb/100ft <sup>2</sup> /cp)	Gel Strength,(lb/100ft <sup>2</sup> )	
					10 sec	10 min
1	1.65	1.49	0.32	0.21	0.2	0.3
3	1.6	1.4	0.4	0.29	0.8	0.9
5	1.76	1.6	0.32	0.2	0.4	0.6
7	2.5	1.94	1.12	0.58	0.5	1.1
9	2.93	2.13	0.23	0.11	0.5	1.2

The rheological properties of alkali and thermal activation for Iraqi bentonite failed to be enhanced and unable to reach API specifications. Because of acid activation by HCl, there was an enhancement in mineral composition of Iraqi bentonite by removing calcite and increasing the percentage of montmorillonite. . In spite of the mineral enhancement, it could not be used as drilling fluids because of the bad values of rheological properties.



**Figure 7.** Apparent Viscosity of Alkali and Acid Activation Iraqi Bentonite

Apparent Viscosity had very low values and did not reach the API specifications. Figure 7, shown an increase in weight of NaOH, may increase the apparent viscosity.

**Table 12.** Density, pH and Filtration of Alkali and Acid Activation Iraqi Bentonite

W,g	,ppg	pH	V <sub>7.5</sub> ,ml	V <sub>8</sub> ,ml	Tmc,mm
5	8.55	11.4	14.1	28.2	0.96
	8.5	11.23	28.3	58.6	1.43
	8.55	11.6	37.3	74.6	1.86
	8.6	11.81	31.3	62.6	2.12
9	8.6	11.7	26.4	52.8	2.4

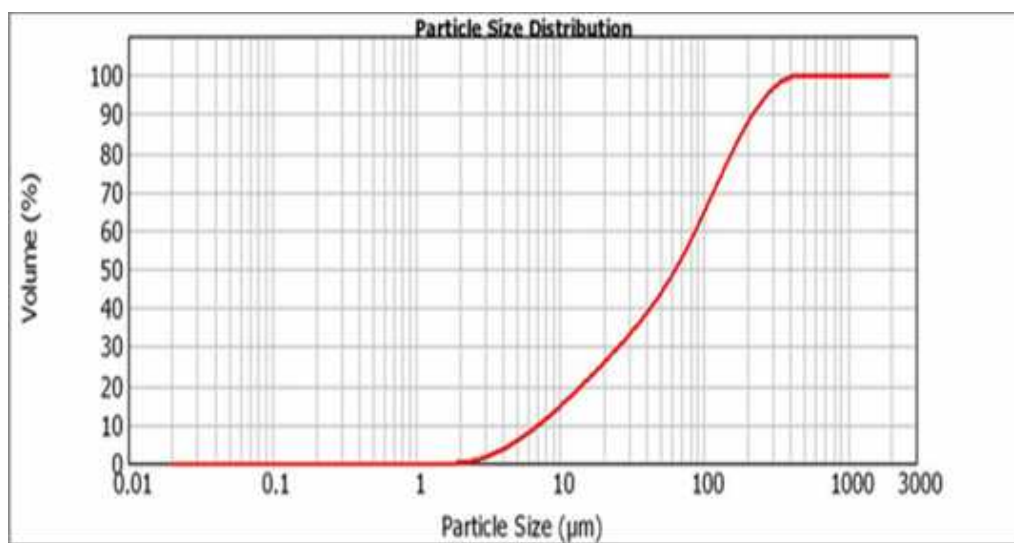
The filtration loss was very high and it was not fulfill API standards.

By direct and indirect activation process, results improve that NaOH could not work on filtration enhancement.

The XRF, SSA and PSD were carried out to the addition of 2 ml of HCl (0.5M) and 1g of NaOH (sample of alkali and acid activation Iraqi bentonite) and the results are shown in Table 12 and Figure 8.

**Table 13.** XRF of Alkali and Acid Activation Sample Iraqi Bentonite (2ml HCl and 1g NaOH)

SiO <sub>2</sub> %	Fe <sub>2</sub> O <sub>3</sub> %	Al <sub>2</sub> O <sub>3</sub> %	CaO %	MgO %	Na <sub>2</sub> O %	K <sub>2</sub> O %
53.39	6.27	14.50	3.26	3.18	1.48	0.49



**Figure 8.** PSD of Alkali and Acid Activation Iraqi Bentonite Sample (2ml HCl and 1g NaOH)

The XRF analysis shows that the ratio of  $[(Na_2O+K_2O)/(CaO+MgO)]$  of activated Iraqi bentonite was found 0.31, confirming that bentonite was Ca-bentonite and there was no difference in comparison with Iraqi bentonite before activation. The particle size distribution of activated Iraqi bentonite shown that there was 10% volume of particle sizes under  $7.198\mu m$ , 50% volume of particle sizes under  $63.245\mu m$ , and 90% volume of particle size  $217.771\mu m$ . The value of specific surface area was  $0.289(m^2/g)$ . This value was smaller than the value of Iraqi bentonite before activation. Because the particle size was increased, the value of specific surface area was decreased.

To search about why the indirect activation Iraqi bentonite failed, the first reason was that the solution of the mixture after mixing time before first wash out, was taken and made XRF analysis in Varian AA240FS Atomic Absorption. The analysis shows that the concentration of Ca in the solution is 2.49 ppm and the concentration of Na in the solution is 118709 ppm. The Na did not interact with Iraqi bentonite but it was removed in washing. The washing process was incorrect and failed the activation.

Another reason was that when taking picture to the three samples of three indirect activation processes and analyzing them in OLYMPUSVBX51M Microscope, the microscope picture shown that there is  $Ca(OH)_2$  in the composition of activated bentonite. The microscope pictures of three indirect activation processes are shown in Figure 9, 10 and 11.



**Figure 9.** Microscope Picture of Alkali Activation Iraqi Bentonite sample after drying



**Figure 10.**Microscope Picture of Alkali and Thermal Activation Iraqi Bentonite after drying



**Figure 11.**Microscope Picture of Alkali and Acid Activation Iraqi Bentonite Sample after drying

## 6. Conclusions

Iraqi bentonite obtained from Trefawi Formation/ Al-Anbar Region/ Iraq was characterized using XRF, PSD and SSA, and measured its properties. The results shown that Iraqi bentonite is calcium type and cannot use as drilling fluid because of its bad rheological properties and has highly percentage of impurities.

Iraqi bentonite contains quartz and calcite as non-clay minerals or impurities. Quartz was removed by using special method of grinding.

By direct Activation for Iraqi bentonite when 3.2 g NaoH was added, Ib was activated and enhanced its properties except the filtration. The Filtration can be enhanced by adding CMC-LV or PAC or starch and then activating directly Iraqi bentonite by 3.2 g NaoH can be used as drilling fluids.

In direct activation processes failed to activate Iraqi Ca-bentonite, so It cannot be used as drilling fluid.

## Nomenclature

Symbol	Description	Unit
W	Weight of sample	g
AV	Apparent viscosity	cp
PV	Plastic viscosity	cp
YP	Yield point	lb/100ft <sup>2</sup>
YP/PV	Yield point to plastic	lb/100ft <sup>2</sup> /cp

	viscosity	
	Density of the mud	ppg
V <sub>7.5</sub>	Filtrate volume at 7.5min	ml
V <sub>30</sub>	Filtrate volume at 30min	ml
T <sub>mc</sub>	Thickness of mud cake	mm
H	Temperature	°C
M	Mixing Time	hours

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