

# Study on the activation of Qara Tappah Iraqi bentonite and its test as gelling agent

Fadhil A.Rasin<sup>1</sup> and Entesar A. Hamad<sup>2</sup>

<sup>1</sup>Engineering of materials, University of technology, Baghdad, Iraq

<sup>2</sup>Department of physics, College of Science, Al-Mustansiriyah University, Baghdad, Iraq

## ABSTRACT

*The aim of this research is to study the activation conditions of Qara Tappah bentonite at various ratios for converting it to the gelling form. Soda ash is used in the activation process with ratios (2.5%, 5%, 7.5%). The best result for obtaining the gelling form in the limit of experimental work was found at the activation ratio (7.5%).*

**Keyword:** Bentonite, Activation, Gel formation.

## 1. INTRODUCTION

Bentonite is a smectite clay, the major mineral in it is montmorillonite. Chemically, montmorillonite is described as a hydrous aluminum silicate containing small amounts of alkali and alkaline earth metals. It is made of two building blocks, the aluminum octahedral sheet and the silica tetrahedral sheet (1,2,3). The montmorillonite lattice is negative in charge, owing primarily to isomorphous replacements of ions within the structure. This negative character is balanced by cations which are held on the surface of the flakes and clay can be readily exchanged, the cations most commonly found in nature are sodium and calcium (4,5). The edge of the clay particle is assumed to possess an electrical double layer of a different nature to that of the flat surface, a positive double layer is created on the edge surfaces owing to the exposed alumina sheet, whereby it may become (more) positive with decreasing PH and its sign may be reversed with increasing PH (6). Because of differences in structure and rheological properties between calcium bentonite and sodium bentonite, Na-bentonite is preferred for most of the industrial applications (7), therefore the Ca-bentonites are treated with some inorganic chemicals like  $\text{Na}_2\text{CO}_3$  to develop the rheological properties. In a suspension with a low solid content, the gel structures build up slowly with time. The concentration of clay present in the system and the salt content are decisive factors in the length of time required for a gel to attain maximum strength (8,9). A clay gel is a transitional hydrous (saturated) phase that exists between the plastic phase and fluid phase (10). The property of gel is present when the main exchangeable cation is  $\text{Na}^+$  in suspension with a low solid content, since the particles orientate themselves negative to positive and form a gel, which seems to consist of polymeric chains of individual montmorillonite layers (11,12,13). Because of this property, bentonite is sometimes used in clarifying turbid liquids, and used as carrier for a number of cosmetic preparations, tooth pastes, creams and other pharmaceutical industry (14). Our study concerns on Iraqi bentonite to be prepared as a gel and study their properties.

## 2. MATERIALS AND METHODS

Fractions smaller than  $38\mu\text{m}$  of Iraqi Qara Tappah Ca-bentonite were used. The chemical analysis of this bentonite is given in table(1). The X-ray powder diagrams of the original and activated bentonite were obtained with XRD unit model (7000), target Cu, ( $\lambda$ ) =  $1.5405 \text{ \AA}$ , 40 (kV), 30 (mA), also the cation exchange capacity (CEC) for all samples was measured by methylene blue adsorption method (ASTM C 837-81) (15).

**Table-1:** chemical analysis of Ca-Bentonite sample

Material oxide	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	Na <sub>2</sub> O	MgO	SO <sub>3</sub>	L.O.I
Bentonite (wt%)	58.58	15.8	2.5	2.77	0.7	6.1	0.1	12.48

Qara Tappah bentonite was activated with  $\text{Na}_2\text{CO}_3$  at various mass ratios (soda ratio: 2.5, 5, 7.5%) labeled (Q2.5, Q5, Q7.5). Bentonite and  $\text{Na}_2\text{CO}_3$  were added to distilled water until we get a slurry, the slurry was heated and

mixed for 60 min by using mechanical stirrer ,then cooled with water quickly ,left for 24h, they mixed with a suitable amount of Ethanol(1:4 Ethanol: Water), and centrifuged (4000 rpm, 2 min) by using (LABOFUGE-HERAEUSE) , and dried in an oven at 100°C . Then the dried samples were ground by ball mill to reduce the particle size. The samples as defined above after these process of preparation, were tested by adding a distilled water at 5% (w/v), this test for samples Q5 and Q7.5 shows a clear structure gel formation. It was found that the time is an important parameter for the formation.

#### **Determination of gel strength:**

Gel strength is a measure of the ability of a colloid to form gels and is related to inter particle forces of the mud. it is measured under static conditions. Gel strength measurements are normally taken as initial gel strength (zero quiescent time) and final gel strength (ten-minute quiescent time ). The following procedure was employed to determine two values- the initial gel-strength ( $g_0$ ) and the 10 minute gel- strength ( $g_{10}$ )[by using U.S.A viscometer, model 800, Ofite].

1)The mud sample (22.5 g of bentonite: 350 ml distilled water) was stirred at(6000 rpm)for 10 sec. and then allowed to stand undisturbed for 10 sec. followed by rotating the instrument at (3 rpm) and the maximum reading attained after the start of the rotation was recorded . this maximum reading was the initial gel strength in (Pa).

2) The mud was re-stirred at high speed (i.e. 600 rpm) for ten second and then allowed to stand undisturbed for ten minutes, followed by rotating the instrument at 3 (rpm) and the maximum reading was recorded , this maximum reading was the ten- minute gel in (Pa).

#### **Determination of gel formation index:**

By mixing 6g of prepared samples with 0.3g of light magnesium oxide, then this mixture was added in step wise of addition to 200ml of water contained in a 500-ml stoppered measuring cylinder, agitated thoroughly for one hour , then transferd 100 ml to a 100-ml measuring cylinder and allowed to stand for 24 hour , and the supernatant liquid was measured.

### **3. RESULTS AND DISCUSSION**

X-ray diffraction patterns of original and  $\text{Na}_2\text{CO}_3$  activated samples are illustrated in figure (1).The main montmorillonite hkl diffraction reflections are present in all samples. The lower  $d(001)$  values were found for the Na-Bentonite obtained by sod activation at (7.5%), which is about (11.84 °A) than in the original Ca-B( 14.8°A) . The CEC results is shown in the figure (2), which is increased with the increasing of soda ratio, also it was reached the optimum value at soda ratio (7.5%) which is about 178 meq/100 g bentonite. As a result, the ratios of CEC still increase and did not reach a constant value at the range of soda ratios in our study. This means that the reaction did not reach the balance and the net negative charge on the bentonite particles still needs more ratio of soda to compensate the deficiency of the positive charge.

These results show that the original Qara-Tappah bentonite was converted to Na- bentonite . when a soduim bentonite is dispersed in water, highly stable colloidal suspension is formed with high viscosity and thixotropy, this suspension begins to take on the characteristics of a gel, so the maximum rheological properties were observed for Na-bentonite (Q7.5) which show the maximum value of gel strength , as shown in the figure (3), and gel formation iudex as shown in the figure (4) . this means that the repulsive force between the sodium bentonite plates surfaces is high enough to prevent the aggregation of the plates, so the plates intercept free amount of water from the suspension between them, and then they were oriented positive edge to negative surface ((flocculation) to form the card- house structure(2). In addition,  $\text{Na}^+$  ions which are adsorbed onto one clay surface, pass through into the solution when they interact with water and easily leave out the clay minerals, so the water molecules easily moved into the layers of the clay, Here, hydrogen bridge bonds are formed by the hydrogen atoms contained in the water molecules. Platelets become isolated from each other, while bonded through interposition water. When left still, a mesh is formed which causes an increasing in the viscosity and leads to form gel(16). As shown in the figure (5).

### **4. CONCLUSIONS**

The conclusion behind this study shows the Q.T. bentonite have an ability to activation at 7.5 % soda ratio as an excellent result to conversion to the soduim form and shows a gel behavior which is an important results for industrial applications.

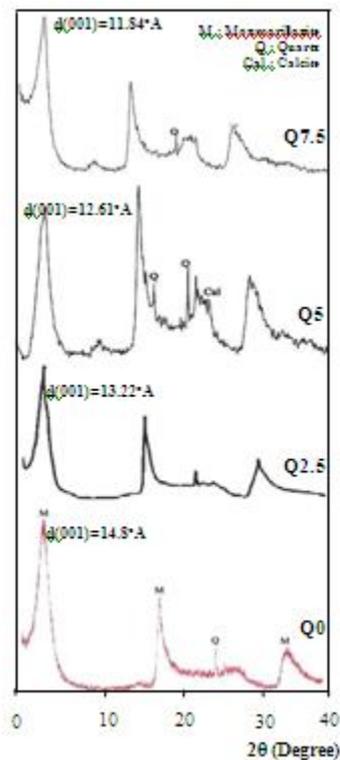


Figure 1.XRD patterns of the original and activated bentonite

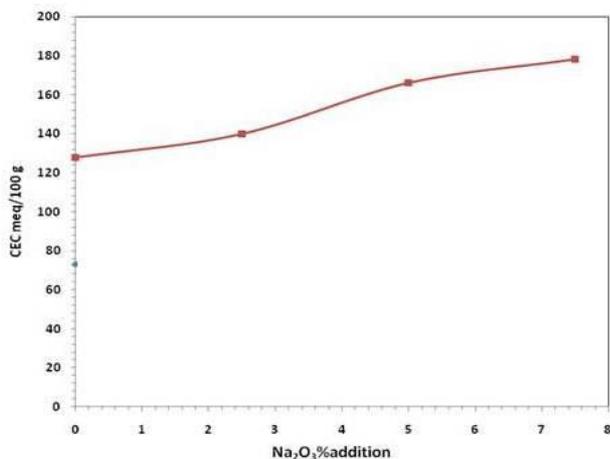


Figure 2 Plot of the CEC versus Na<sub>2</sub>CO<sub>3</sub> %, where CEC (Q) refers to CEC of Qara Tappah activated bentonite

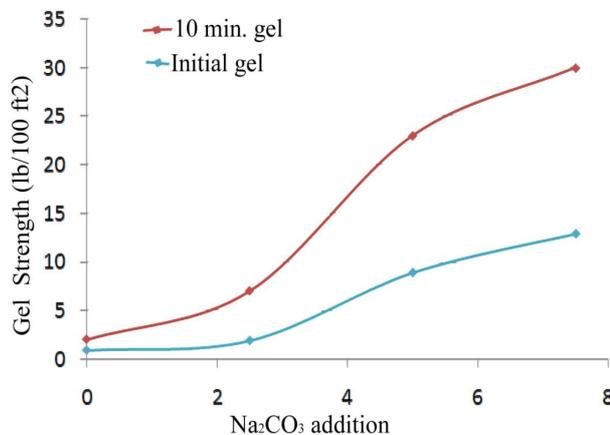


Figure 3.Gel strength at various ratios of soda addition



Figure 4.Gel formation index for the activated samples



**Figure 5.** Gel formation of Iraqi bentonite

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