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SODIUM ACTIVATION OF IRAQI HIGH GRADE MONTMORILLONITE CLAYSTONE BY DRY METHOD

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ABSTRACT

High grade calcium montmorillonite claystone (calcium bentonite) sample from Wadi Bashira region, Western Desert, Iraq, was activated with sodium by a dry grinding procedure and subsequently evaluated for use as drilling fluids to conform API specifications. Sodium carbonate (Na_2CO_3) was used to do the activation and convert calcium bentonite to its sodium form. This ion exchange was demonstrated by X-Ray diffraction patterns, wherein the main Ca-montmorillonite d (001) peak changed from 13.6 Å to 11.7 Å. The rheological properties of the activated sample were highly improved, but the sample failed to fulfill API drilling mud requirements. It was observed that certain additives (organic sodium salt and/ or Na-CMC) when added with Na_2CO_3 can improve the rheological properties of the activated bentonite sample so as it can be used as a drilling mud. The amounts of these additives and that of Na_2CO_3 were optimized and a bentonite having all API specifications was produced. Experimental results showed that an amount, not exceeding 1 wt.% of these additives with 6 wt.% Na_2CO_3 is optimal. Accordingly, it can be claimed that the application of this method is economically viable compared to wet activation as it saves water, energy and time.

التنشيط بالصوديوم لأطيان المونتمورولونايت العراقية ذات النوعية العالية بالطريقة الجافة

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المستخلص

نموذج من أطيان الكالسيوم مونتمورلونايت (الكالسيوم بنتونايت) من منطقة وادي بشيرة في الصحراء الغربية للعراق تم تنشيطه بالصوديوم بطريقة الطحن الجاف وتقييمه لأغراض سوائل الحفر وفق مواصفات API. استخدمت مادة كاربونات الصوديوم (Na_2CO_3) بنجاح لإنجاز التنشيط وتحويل البنتونايت الطبيعي، الذي هو من نوع الكالسيوم الى نوع الصوديوم. تم ملاحظة هذا التبادل الأيوني من مخطط الأشعة السينية الحائدة، حيث تغيرت قيمة القمة الرئيسية للكالسيوم مونتمورلونايت (001) d من 13.6 Å الى 11.7 Å ولقد كان هناك تحسن كبير في الخواص التيارية للبنتونايت المنشط، إلا أنها لم ترتقي لمواصفات API المطلوبة لحفر الآبار النفطية. ولكن لوحظ أن بعض المضافات مثل أملاح الصوديوم العضوية مع أو بدون Na-CMC عند أضافتها مع Na_2CO_3 يمكنها تحسين الخواص التيارية للبنتونايت المنشط وبالتالي يمكن استخدامه في أطيان الحفر. وعليه فقد تم تحديد الكميات المثلى والأنسب من هذه المضافات بالإضافة الى Na_2CO_3 لإنتاج بنتونايت يلبي متطلبات API. وقد بينت نتائج التجارب إن كمية المضافات بنسبة لا تزيد عن 1% وزناً مع 6% وزناً من كاربونات الصوديوم هي أكثر ملائمة لذلك. وبناءاً على ذلك يمكن القول بأن استخدام هذه الطريقة له مردود اقتصادي عالي بالمقارنة مع طريقة التنشيط الرطب، حيث إنها تؤدي الى ادخار الماء والطاقة والزمن.

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INTRODUCTION

Drilling fluid, or drilling mud, used in the petroleum industry for the drilling of boreholes and construction of oil wells, is generally a slurry of clayey materials, which exhibits a number of functions, the most important of which are: **a)** removing formation cuttings from the well, **b)** sealing off permeable formations of gas, oil, or water which may be encountered during the drilling of the well, **c)** cooling and lubricating the drilling tools and drilling pipes, and **d)** holding the cuttings in suspension and minimizing fluid loss across permeable formations by forming a filter cake on the wall of the well bore (Al-Hiti *et al*., 1988 and Mihalakis *et al*., 2004). An ideal drilling fluid is a thixotropic fluid, i.e. a fluid whose viscosity decreases as the degree of agitation or shear increases (Ainsworth, 1994). Bentonite is the most commonly used clay for the preparation of drilling muds (Mihalakis *et al*., 2004). Bentonite is a naturally occurring clay, composed predominantly of the clay mineral montmorillonite, which gives bentonite its properties (Grim, 1968). Montmorillonite is composed of layers, or platelets (crystals), with exchangeable cations occurring between them. The types of the exchangeable cations have a great influence on the technological properties of the bentonite. Bentonite is either the sodium (Na) or the calcium (Ca) type. Sodium bentonite (where Na^+ is the main exchangeable cation) has very high swelling capacity and thixotropic properties, with relatively stable colloidal properties when added to water, so it is highly valuable in the preparation of drilling fluids. Calcium bentonite, however, has no value for such activities. It has a little swelling ability and flocculation and settling times are rapid, resulting in much high filter (or fluid) loss than the sodium bentonite (Keren, 1988 and Bowyer and Moine, 2008). Calcium bentonite, on other hand can be converted to sodium bentonite by treatment with sodium carbonate (a process called sodium activation). This activation, if conducted properly, can result in a bentonite that exhibits many of the characteristics of natural sodium bentonite.

In Iraq, high grade bentonite claystone of the calcium type, averaging 80% montmorillonite, was discovered in the Western Desert (Wadi Bashira region) by a detailed exploration work (Al-Bassam *et al*., 1989). This claystone was initially subjected to a laboratory sodium activation using a wet method (Al-Ajeel *et al*., 1990). The rheological results of the activated bentonite, however, were inconclusive and hence, oil well drilling experiments were conducted using mud prepared on site (during the time of the experiments) by mixing a slurry of the calcium bentonite with sodium carbonate (the preparation was done according to the optimum conditions obtained from the aforementioned laboratory activation experiments). The drilling test was conducted in Al-Zubair (wells No.127 and 128) oil fields at Al-Basrah city. The depth of the drilled wells was about 3400 m. The result of this experiment was successful, the curing and additives used to produce the mud were resembled to natural sodium bentonite mud (Al-Egaely *et al*., 1992). With the success of this experiment, it was concluded that Wadi Bashira high grade bentonite can be used as a drilling mud if activated on site. Petroleum companies are currently demanding powdered activated bentonite. Obviously, this needed a new method to convert Wadi Bashira high grade Ca-bentonite to the sodium type. Extensive studies have been published related to the treatment of calcium bentonite with sodium carbonate, but it was obvious from these publications that there is no general rule (or route) that can be used for such treatments (Bleifuss, 1973; Erdogan and Demirci, 1996; Yildiez and Calimli, 2002; Besq *et al*., 2003; Lucian *et al*., 2004 and Al-Maghrabi and Aboushook, 2008). In fact, the mineralogical characteristics of the montmorillonite and the association of clay and non-clay minerals have a significant effect on the rheological properties of the activated bentonite (Besq *et al*., 2003 and Lucian *et al*., 2004).

This paper presents the work that was undertaken to investigate the potential of dry activation of Wadi Bashira high grade calcium bentonite to produce bentonite that met API specifications for use in drilling muds.

MATERIALS AND METHODS

The starting bentonite sample used in the activation experiments was a high grade bentonite (montmorillonite claystone) from Wadi Bashira region, Western Desert, supplied by the Bentonite Grinding Mill in Fallujah (about 50 Km west of Baghdad). Sodium carbonate (Na_2CO_3) along with additives such as Na-CMC (sodium carboxymethyl cellulose), and an organic sodium salt were used to do the activation of the bentonite sample and control its rheological properties. Raw bentonite sample was crushed and subsequently pulverized using common laboratory crushing and grinding equipment. Representative specimens were then taken for chemical, mineralogical, and cation exchange capacity (CEC) determinations. The chemical composition was determined by X-ray fluorescence analyses (Shimadzu-1800), whereas the minerals present were identified by XRD using a Shimadzu-7000 diffractometer. The generator settings were 40 kV and 30 mA, and the wavelength (λ) was 1.54 Å, using Cu-kα radiation. The CEC was determined using a methylene blue method (Schenning, 2004).

The activation was carried out by dry grinding, ground bentonite sample along with the other additives in a laboratory ball mill for a predetermined time, using steel balls as grinding media. The charge was then removed from the mill and the rheological properties of the bentonite sample were measured. In the course of the activation experiments, the effect of the activation time, amounts of sodium carbonate, organic sodium salt, and Na-CMC, were investigated. All additives were added in weight percentage of the dry weight of the bentonite sample. The rheological properties of the activated sample were measured according to API (American Petroleum Institute) specifications (1995) in the Central Laboratories Department, Research and Development Section Division, GEOSURV, Ministry of Industry and Minerals, as well as in the laboratories of the Research and Development Center of the Oil Ministry. The viscosities were measured using an OFITE viscometer model 800, 8-speed system. Slurry of 22.5 gram of bentonite and 350 ml of deionized water were prepared and mixed for 20 minutes on a Hamilton Beach Mixer. The suspension was aged for 16 hours and after subsequent mixing for about 5 minutes viscosity readings were obtained at both the 600 and the 300 rpm settings of the viscometer. From these results, apparent viscosity (AV), plastic viscosity (PV) and yield point were calculated. The API fluid loss (FL) test was carried out by placing the bentonite slurry in a stainless steel chamber with an opening in the center bottom. A filter paper was placed on the bottom. The slurry was exposed to 100 psi pressure for 30 minutes, and the amount of water that escaped was collected in a graduated cylinder and measured. The API rheological specifications for oil drilling fluids or mud (1995) are given in Table (1).

Table 1: API rheological specifications (API, 1995)

Requirements	Specification
Apparent viscosity (AV)	15 (cp) minimum
Yield point/ plastic viscosity (YP/ PV) ratio	3 maximum
Filter or fluids loss (FL)	15 (ml) maximum

RESULTS AND DISCUSSION

▪ Chemical and Mineralogical Composition

The chemical and mineralogical compositions of untreated duplicate bentonite samples were analyzed to evaluate and qualify the homogeneity of the bentonite material. Results of the analyses are given in Table (2) and the X-Ray diffraction pattern is shown in Fig. (1).

The theoretical chemical composition of pure Wadi Bashira bentonite claystone has been calculated by Al-Bassam *et al*. (2008), which showed that the CaO content is about 1.6%. Whereas, the CaO content of the raw bentonite sample under study is 5.7%. This difference is obviously due to the presence of CaO bearing minerals impurities, such as calcite ($CaCO_3$) and gypsum ($CaSO_4.2H_2O$), which are associated with the deposit (Table 2 and Fig.1). Also, the SiO_2/Al_2O_3 ratio of the raw bentonite is about 4, which is higher than the SiO_2/Al_2O_3 ratio 3 of the aforementioned pure bentonite, due to the presence of free silica in the form of quartz, as seen clearly in Fig. (1). The chemical analysis (Table 2) shows the presence of 3.2% MgO, which is related to some extent to the presence of palygorskite (Fig.1). Furthermore, the chemical analysis shows that the raw bentonite contains about 0.94% Cl with 1.3% Na_2O, indicating the presence of halite (NaCl). However, excluding the clay mineral palygorskite, it became clear that calcite, gypsum, quartz, and halite are the main non-clay minerals impurities associated with the predominant montmorillonite clay mineral that constitutes the raw bentonite sample. The X-Ray diffraction d (001) value, however, of the raw bentonite according to the ASTM (2008) was 13.62 Å (Fig.1) demonstrating that the montmorillonite is a calcium based. The d (001) value was shifted to 11.7 Å after sodium activation of the bentonite indicating that Na^+ ions replaced Ca^{+2} in the interlayers and formed Na-bentonite, as can be seen in the XRD pattern presented in Fig. (2). The high CEC (82 meq/100g) content of the raw bentonite could have a significant effect that facilitates the ions exchange process.

Table 2: Chemical and mineralogical composition of duplicate raw bentonite samples, as well as their CEC values

wt.%											CEC (meq/100g)
SiO_2	Fe_2O_3	Al_2O_3	TiO_2	CaO	MgO	SO_3	L.O.I	Na_2O	K_2O	Cl	
55.9	6.1	13.3	0.80	5.7	3.2	0.4	12.0	1.3	0.5	0.94	82
55.87	6.07	13.28	0.90	5.65	3.30	0.50	11.90	1.27	0.48	0.90	82
Mineralogical composition Montmorillonite, Calcite, Quartz, Palygorskite and Gypsum											

▪ Rheological Behavior of Activated Bentonite

Table (3) shows the rheological properties of raw bentonite after activation for various times using 4% Na_2CO_3. The results revealed that dry grinding markedly induced the activation of the bentonite sample. This can be seen clearly from the significant increase in the AV and YP/PV, and reduction in filter loss (FL), compared with the rheological properties of the raw bentonite (inactivated) sample (Table 3). However, these results did not conform to the API specifications shown in Table (2).

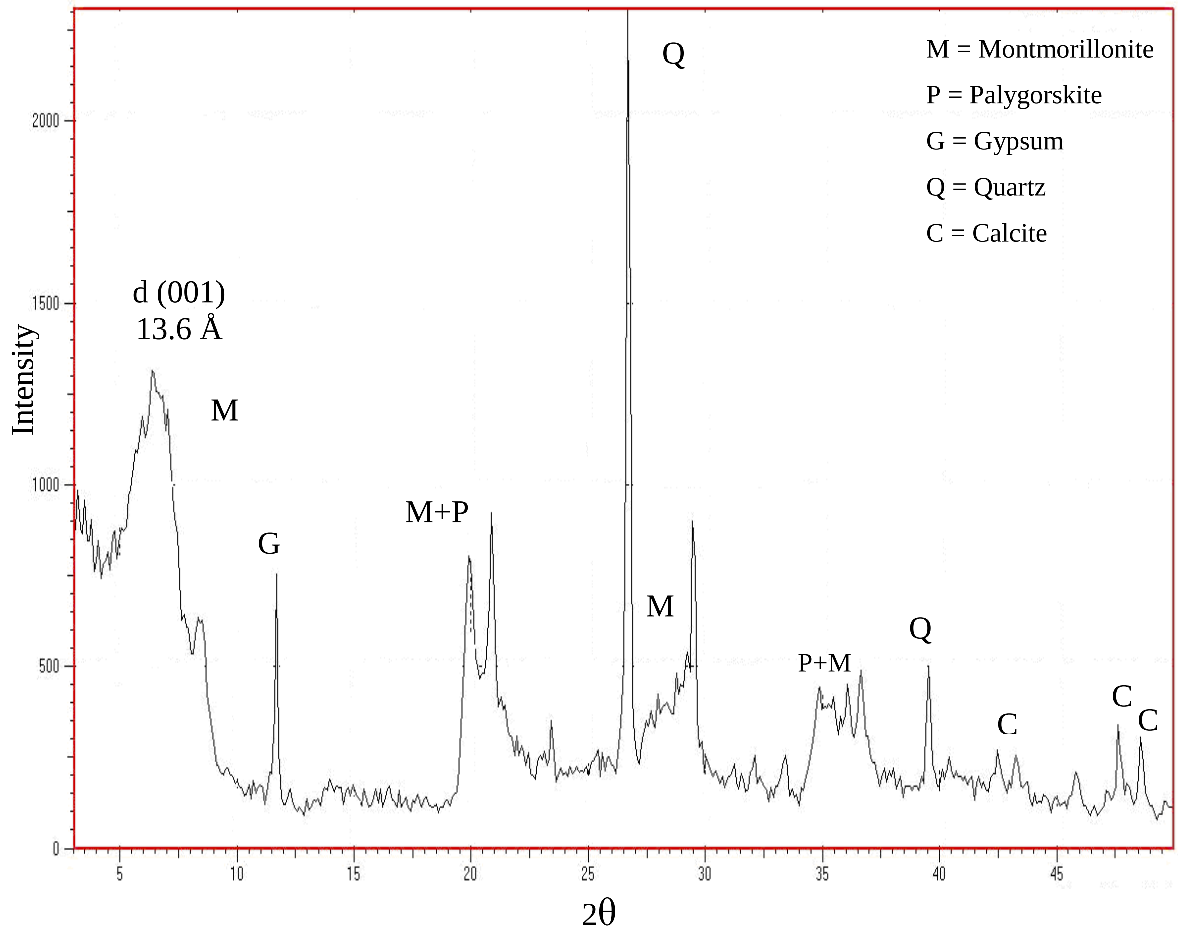

Fig.1: XRD pattern of raw bentonite sample

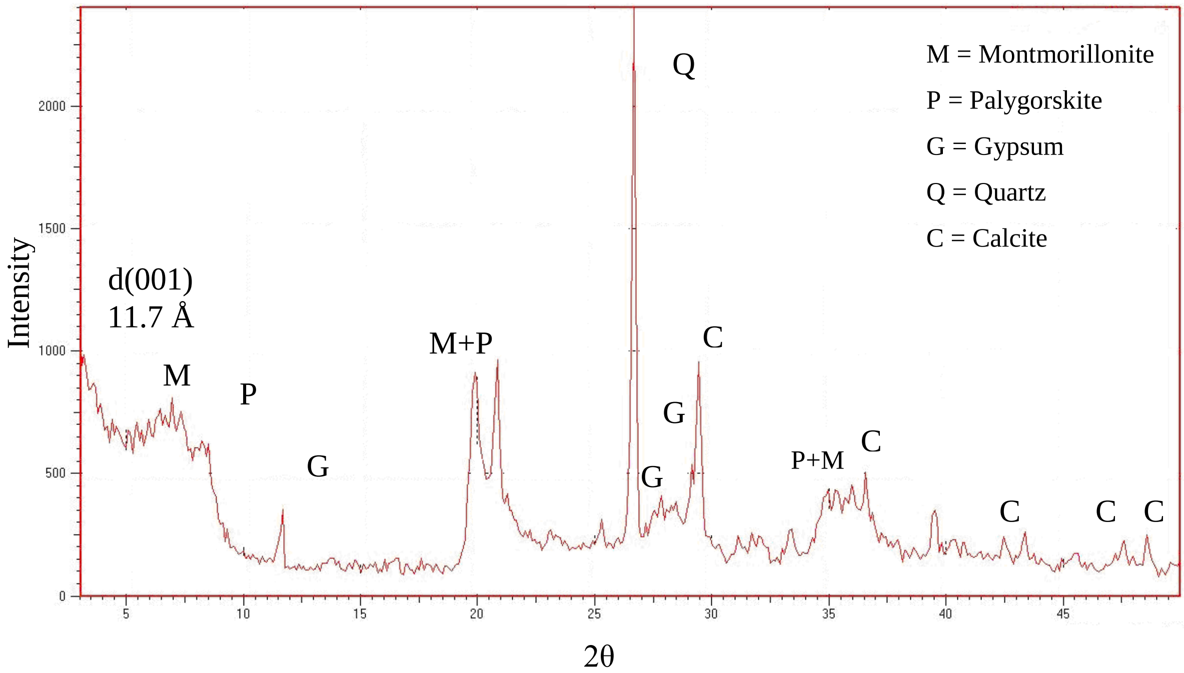

Fig.2: XRD pattern of Na-activated bentonite sample

Table 3: Rheological properties results of the raw bentonite and that activated with 4% Na_2CO_3 at different times

Test Run No.	**Time (min)**	**Rheological Properties**		
		AV (cp)	**YP/PV**	**FL (ml)**
TR1	15	20.5	18.5	30
TR2	30	20	5.2	32
TR3	45	19	5.2	31
TR4	60	17	5.2	31.6
Raw bentonite (inactivated)		3	1	47

The proposed interpretation of the above results is that grinding distorts the plate stacking of the montmorillonite and this delaminating process (that occurs in conjunction with the shear force imparted by the grinding) may account for the ease of activation (exchanging of Ca by Na ions). Another obvious effect of grinding is that the colloidal content of the clay increases, which results in increasing of the cation exchange capacity (Mingelgrin *et al*., 1978). Also, it has been claimed (Petrovic *et al*., 2002), that the reactivity of the bentonite clay and its CEC can be markedly increased by grinding. This in turn could also enhance the activation process. In the light of the above results, an activation time of 30 minutes was chosen for the next experiments. In this course, the effect of adding organic sodium salt (OSS) and/ or Na-CMC during the activation process on the rheological properties of the bentonite was presented.

Table (4) shows the rheological properties of a set of activated bentonite samples using 4 wt.% sodium carbonate with the addition of different amounts of organic sodium salt (1 – 4 wt.%). These results showed no significant difference in rheological properties (except the YP/PV ratio of run TR5) between the tested samples, yet the results still did not conform to API specifications. However, these results indicated that the addition of organic sodium salt (OSS) led to a significant decrease in the apparent viscosity (AV), as compared with that gained from using only sodium carbonate in the activation process (Table 3). Although the fluid loss (FL) remained high, a noticeable reduction in the FL was observed with increasing (OSS) concentrations (Table 4).

Table (5) shows the rheological results of a set of activation tests carried out by using 4 wt.% of each of sodium carbonate and OSS, with the addition of different amounts of Na-CMC (1, 1.5 and 2 wt.%). The results reveal that the YP/PV ratio and FL of the activated samples were enhanced and conformed to API specifications. However, the values of apparent viscosity (AV) remained lower than those stated in the API specifications of 15 cp minimum.

Table 4: Rheological properties of the activated samples with 4 wt.% Na_2CO_3 and addition of different amounts of organic sodium salt

Test Run No.	**Organic Sodium Salt (wt.%)**	**Rheological Properties**		
		AV (cp)	**YP/PV**	**FL (ml)**
TR5	1	12	10	28.3
TR6	2	11	5.3	26.5
TR7	3	10	4.66	24.5
TR8	4	10	4.6	22.5

Table 5: Results of activation tests using 4 wt.% Na_2CO_3 and 4 wt.% organic sodium salt (OSS) with addition of different amounts of Na-CMC

Test Run No.	Na-CMC (wt.%)	Rheological Properties		
		AV (cp)	YP/PV	FL (ml)
TR9	1	11.5	1.3	16.0
TR10	1.5	11.5	1.8	11.8
TR11	2	12	1.4	10.5

Considering the YP/PV ratio and FL values shown in Table (5), it can be seen that the best results obtained are those of test runs TR10 and TR11 where 1.5 and 2 wt.% Na-CMC were used. The results in Table (5) also show that, a low value of the YP/PV ratio was obtained from test run TR9, wherein 1 wt.% Na-CMC was used. Based on the results given in Tables (4 and 5), further activation tests were conducted by using different amounts (4, 5 and 6 wt.%) of sodium carbonate, with the addition of 2 wt.% OSS, and 1 wt.% Na-CMC. The results obtained (Table 6), indicated that using 6 wt.% of sodium carbonate along with the aforementioned additives, resulted in a bentonite having rheological properties conform to API specifications for oil well drilling mud.

Table 6: Results of activation tests using different amounts of Na_2CO_3 with addition of 2 wt.% organic sodium salt and 1 wt.% Na-CMC

Test Run No.	Na_2CO_3 (wt.%)	Rheological Properties		
		AV (cp)	YP/PV	FL (ml)
TR12	4	12	1.4	10.5
TR13	5	13.5	1.9	17.8
TR14	6	16	2.5	15

Based on the results of test run TR14 where 2 wt.% OSS and 1 wt.% Na-CMC were used, further tests were performed to investigate the effect of reducing the amount of the OSS and Na-CMC on the rheological properties of the final product. Table (7) shows the results of a set of activation tests carried out using 6 wt.% sodium carbonate with variable amounts of OSS and Na-CMC. It is clear from these results, that the rheological properties of both TR15 and TR16 tests, where 2 wt.% and 1 wt.% OSS with 0.5 wt.% and 1 wt.% Na-CMC were used respectively, very closely conform to API specification as far as the value of the YP/PV ratio is concerned. The resulting values, however, are slightly higher than API specifications for YP/PV (3 max.). Yet, according to previous reports (Al-Egaely *et al*., 1992), the activated bentonite produced by the conditions given for tests TR15 and TR16 can be successfully used as a drilling mud. The data of Table (6) also reveal that the YP/PV ratio is highly improved by eliminating OSS and using only Na-CMC (1 wt.%) in the activation process (test run TR17). The rheological properties of the activated bentonite obtained from this activation test are well within API specifications. On the other hand, the rheological properties (particularly, the AV and FL) deviate from API specification when 1 wt.% OSS with 0.5 wt.% Na-CMC were used in the activation process (test run TR18).

Table 7: Results of activation tests using 6 wt.% Na_2CO_3 with addition of different amounts of sodium salt and Na-CMC

Test Run No.	Additives		Rheological Properties		
	Organic sodium salt (wt.%)	**CMC (wt.%)**	**AV (cp)**	**YP/PV**	**FL (ml)**
TR15	2	0.5	15	3.60	13.6
TR16	1	1	19	3.4	14.6
TR17	_	1	16.5	2.1	14.2
TR18	1	0.5	11.5	2.2	18

Optimal activation conditions whereby the activated bentonites exhibit rheological properties that fulfill, or closely conform, to API specification are summarized in Table (8).

Table 8: Summary of optimum amounts of the materials needed in the activation process led to rheological properties that fit with API specification

Test Run No.	Additives			Rheological Properties		
	Na_2CO_3 (wt.%)	**OSS (wt.%)**	**CMC (wt.%)**	**AV. (cp)**	**YP/PV**	**FL (ml)**
TR14	6	2	1	16	2.5	15
TR15	6	2	0.5	15	1.7	13.4
TR16	6	1	1	19	3.4	14.6
TR17	6	0	1	16.5	2.1	14.2

CONCLUSIONS

According to the laboratory activation experiments, and the results of rheological properties of the activated bentonite samples, the following conclusions can be highlighted:

- High grade calcium bentonite from Wadi Bashira can be converted to sodium bentonite by treating with sodium carbonate using a dry grinding process.
- Additives such as organic sodium salt, and/ or Na-CMC, are highly effective in improving and controling the rheological properties of the activated bentonite to achieve API specifications for oil wells drilling muds.
- The optimum amounts of the additives needed in the activation process at which activated bentonite can fulfill API specifications are as follows:

Sodium Carbonate (wt.%)	Organic Sodium Salt (wt.%)	Na-CMC (wt.%)
6	1	1
6	2	0.5
6	2	1
6	0	1

- Mixing and homogenization of the raw bentonite is vital for the activation process in order to achieve consistent results.
- The dry activation method is highly economical as compared with the wet activation method. It dose not require additional water to do the activation and it eliminates the need for subsequent drying and grinding of the activated bentonite.

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