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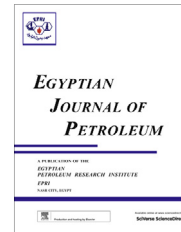
FULL LENGTH ARTICLE

The composition and activation aspects of El-Fayoum clays for using as a drilling fluid

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Abstract The local consumption and the high importation cost of imported bentonite to Egypt led to find local substitute solutions for economical aspects. Hence, this paper presents the composition and activation capability of clays collected from the claystone quarries in Kom Oshim area at El-Fayoum province, Egypt for their application as a drilling fluid (water-based mud). It is an attempt to minimize the importation cost of the imported bentonites to Egypt by a local solution and maximize the usage of the studied clay sediments. The physical, chemical, thermal and mineralogical characteristics of five Egyptian clay samples were studied. The rheological properties of the prepared slurries from these samples and their activation characterization during studying the influence of NaOH and POLYPAC-R polymer as activators were investigated. It was found that the non-activated samples cannot be used as a drilling mud but it could be after the activation process where some significant changes in the rheological properties were observed. The experimental results revealed that the activated fitted samples may express as medium grade bentonitic clay and this type of clay can be used as a drilling fluid for shallow depth wells.
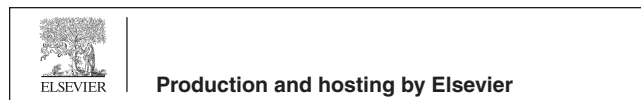
1. Introduction

The name bentonite is given to a particular type of clay that was first noted in about 1980 in Upper Cretaceous tuff near Fort Benton Wyoming, USA. The main constituent, which is

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the determinant factor in the clay's properties, is the clay mineral montmorillonite. This in turn, derived its name from a deposit at Montmorillon, south France. Montmorillonite is a layered silicate mineral that belongs to smectite group of clay minerals. The crystals of montmorillonite consist of three layers: an aluminum octahedron sheet sandwiched between silicon tetrahedron sheets. In montmorillonite, some of the Al ions may be substituted by magnesium ions, resulting in a negative charge on the two large surfaces of montmorillonite platelets. This negative charge is neutralized by interlayer cations, such as, Na^+, K^+, and Ca^{2+}, which separate adjacent layers of platelets. This structure made montmorillonite chemically stable. In industry, montmorillonite is generally classified

as sodium (Na) or calcium (Ca) types, depending on which exchangeable cation is dominant on montmorillonite surface.

Bentonite often contained more than 75% montmorillonite. It had a wide range of industrial applications, such as bleaching earth, cat litter, foundry, construction and civil engineering but it was widely used in oil drilling fields as a mud additive for viscosity and filtration control. Bentonite swelled considerably when exposed to water, making it ideal for protecting formations from invasion by drilling fluids. Sodium bentonite met the drilling-mud and civil engineering specifications due to its excellent colloidal and rheological properties [1].

On the other hand, Egypt imports annually huge amounts of bentonite for oil well drilling and other industrial purposes. Normally bentonite deposits are exploited by surface quarrying. Bentonitic clays have been found in Egypt with promising reserves mainly in sex regions according to previous studies [2]. The major problems facing the utilization of the Egyptian clays are their low concentration of smectite, high level of impurities and inconsistent composition. Also, the response of the Egyptian clays to Na-exchange is poor and their hydration, plastic and rheological properties are inferior to those of the commercial bentonites [3].

Furthermore, most Egyptian clays without beneficiation were unsuitable for the majority of industrial applications [4,5]. The activation processes inculcate the addition of the common salts and polymers [6–8].

The present paper deals with the characterization and upgrading of the clay in Kom Oshim area at El-Fayoum province, Egypt for its application as a drilling fluid (water-based mud). It is an attempt to minimize the cost of the imported bentonite to Egypt by a local solution and to maximize its profiting in a useful application.

2. Geology

The samples used in this study were taken from the claystone quarries located at Kom Oshim area, El-Fayoum province, Egypt. These samples belonged to Qasr El-Sagha Formation of the Upper Eocene age [9]. The studied quarries are situated 30 km north of El-Fayoum city between latitude 29 31′ and 29 35′ north and longitude 30 50′ and 30 59′ east (Fig. 1). The stratigraphy of El-Fayoum province had been dealt by many authors [10]. Qasr El-Sagha Formation is divided into five units as follows [11]:

Top 5- Sandy limestone (1.5 m).

- 4- Loose sands (19.0 m).
- 3- Clay, calcareous sandstone and limestone intercalations (28.0 m).
- 2- Interbedded purple clay with fine sand interbeds (19.0 m).

Base 1- Gypsiferous sandy clay with fossiliferous calcareous sandstone (91.0 m).

The lowest two units attaining a total thickness of about 110 m represented the bulk of the claystone-siltstone interbeds. Some of these had been exploited at Gebel Qasr El-Sagha as bentonitic clays [12] or around Kom Oshim village as bentonite [13]. The Qasr El-Sagha Formation differed widely in thickness and composition from one area to another in El-Fayoum province. The formation is greatly minimized in thickness from about 180 m at Gebel Qasr El-Sagha to about 40 m at Qaret El-Faras, north of El-Fayoum depression, with slight change in facies [14].

3. Experimental procedures

3.1. Materials

Five claystone samples were taken from the exposed surface of five inconsiderable exploited quarries located at Kom Oshim area, El-Fayoum province, Egypt. In an abbreviated notation, the studied samples were designated as q9, q10, q11, q35 and q124. The samples were taken from the fresh deposits using the representative sampling method. The collected samples were mainly brownish gray to dark yellow, massive claystone.

Also in the present study, two types of activators were used which are caustic soda (1.5% by wt.) and synthetic polymer (POLYPAC-R, 0.5% by wt.) as an attempt to upgrade the clay samples by the activation process and hence improve their rheological properties.

3.2. Raw samples characterization

All collected claystone samples were dried at 60 °C for 48 h and then crushed using a jaw crusher to 100% below 5 mm size. A representative batch of each sample was further ground to 100% pass through 200 μm sieve diameter using a porcelain mill to avoid sample contamination. These sieved samples were used for mineralogical, chemical and dehydration analyses. These analyses were performed using X-ray diffraction (XRD), X-ray fluorescence (XRF) and thermal differential and gravimetric (DTA and TGA) investigations. In addition, international standards were used also in the study as mentioned later.

The used XRD apparatus was a X'Pert PRO PW3040/60 (PANalytical) diffractometer equipped with a Cu-Kα radiation source. The test was run at 40 kV and 40 mA. A continuous mode was used for collecting the data in the 2θ range from 3 to 30 at a scanning speed of 2 /m. The acquired data were analyzed using X'Pert high score software works with PDF-2 database. The used XRF was an Axios sequential spectrometer manufactured by PANalytical, Netherlands. Also, a traditional manual method prescribed by the American Society for Testing and Materials (ASTM) for the determination of the loss on ignition [15], (LOI), was followed. The methods described in the ASTM [16] were used for the determination of soluble cations and anions (Na^+, K^+, Cl^- and SO_4^-). Also, the method recorded in a scientific paper [17] was used for the pH determination. It was measured at 22 C by a Jenway 3510 electronic pH meter, UK. The thermal analyses were carried out using a computerized DT.50 thermal analyzer (Shimadzu Co., Japan). The heating rate was 20 °C/min for the thermo-analytical instruments and the nitrogen atmosphere (30 ml/min) was used. Textural analysis of the studied samples was conducted using laser diffraction grain size analyzer of Horiba model LA-930, France. Also, the textural classification was followed as reported in [18]. Physical properties of the studied samples were determined such as Atterberg limits [19] and free swell test [20]. Results of the free swell test were also explained [21]. Also, the activity ratio of clay and its classes was calculated [22]. To measure the rheological properties of the clay samples, viscometer instrument model 35A (USA)
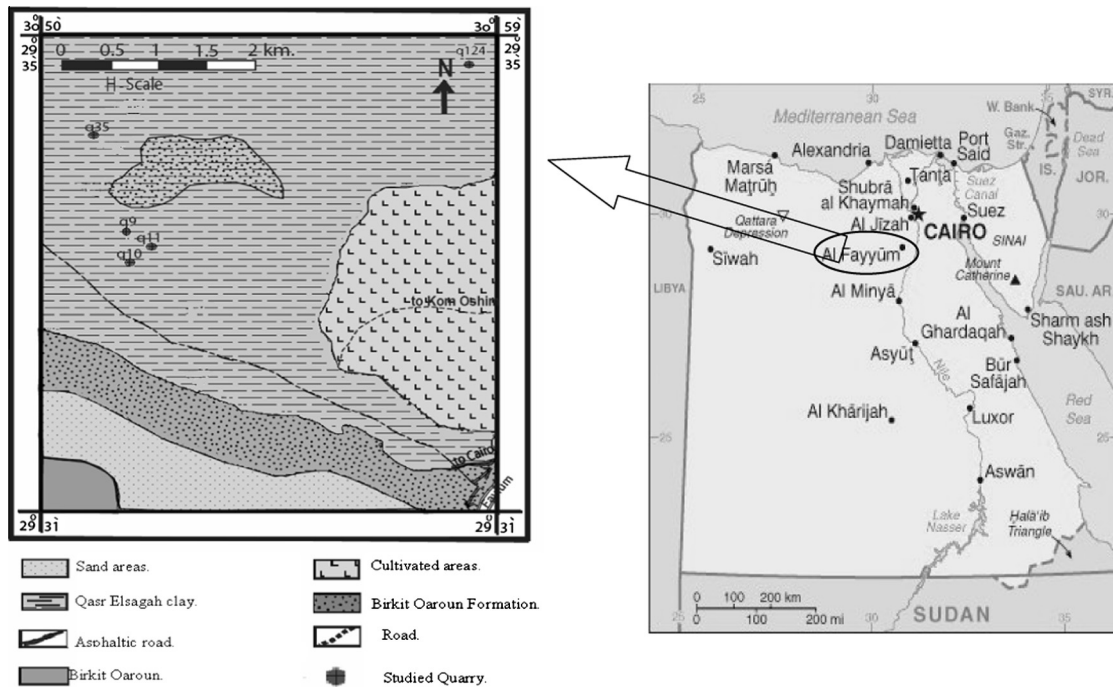
Figure 1 Location map shows the studied claystone quarries at Kom Oshim area, El-Fayoum province, Egypt.

was used which was also used to measure viscosity and gel strength parameters. Also, the ASTM D5891-02 (2009) [23] test method was used to determine the filtrate loss of the clay slurry.

3.3. Rheological tests

Rheological properties for the sample slurry before and after activation process were determined to assess their response to the used activators and consequently the capability for using as a drilling fluid. Generally, the bentonite used in drilling fluids should follow the Oil Company Materials Association (OCMA) or the American Petroleum Institute (API) specifications. In the present study, plastic viscosity (PV), apparent viscosity (AV), yield point (YP) and filtrate loss properties were determined [24].

3.4. Activation method

To determine the rheological properties of the studied samples, slurry of each sample (q9, q10, q11, q35 and q124) with distilled water was prepared at different concentrations from 4.5% up to 7.5 with an increment of 1%.

For making alkaline and polymer activated sample, a slurry of 22.5 g from each sample with 350 ml of distilled water was prepared and mixed in a blender with the activators 1.5% by weight caustic soda (obtained from Misr chemical industries, Egypt) and 0.5% by weight POLYPAC-R (polyanionic cellulose water soluble polymer). The used POLYPAC-R is a white powder, pH of 7 and specific gravity of 1.5. It is common and designed to control fluid loss and increase viscosity in water-based mud.

The prepared slurry was stirred for 20 min at 600 rpm then it was aged overnight [25]. All the activators used were of industrial grade. The used concentration of the activators was added in the clay concentration slurry of only 6.5% taking into consideration achieving somewhat apparent viscosity of 15cP (centipoise) which was stated in the process of clay activation purpose [5] and traditionally used.

4. Results and discussion

4.1. Characterizations of raw samples

4.1.1. Mineralogical composition

XRD patterns of the studied samples are illustrated in Fig. 2. The mineralogical analysis showed that all samples were essentially montmorillonite, kaolinite and illite as clay minerals in a descending order of abundance. Quartz and calcite minerals were also found in minor amounts as non clay components. The montmorillonite mineral was represented by a basal reflection (0 0 1) at 14–15 Å in the air dried state which corresponded to montmorillonite with predominantly Ca as interlayer cation. After heating of the oriented samples at 550 C for 2 h, montmorillonite main peak changed to 9.5 Å, kaolinite mineral disappeared and illite did not show considerable changes. With glycolation of the oriented samples for 4 h, montmorillonite peaks expanded and exhibited a change to 17–18 Å, kaolinite and illite peaks did not show any changes in their positions. Table 1 showed the semi-quantitative percentage of detected clay minerals with respect to all fraction sizes of the studied samples. It varied from 47 (q124) to 75% (q35) for montmorillonite, from 11 (q35) to 18% (q124) for kaolinite and from 2% (q35 and q10) to 5% (q124) for illite minerals.

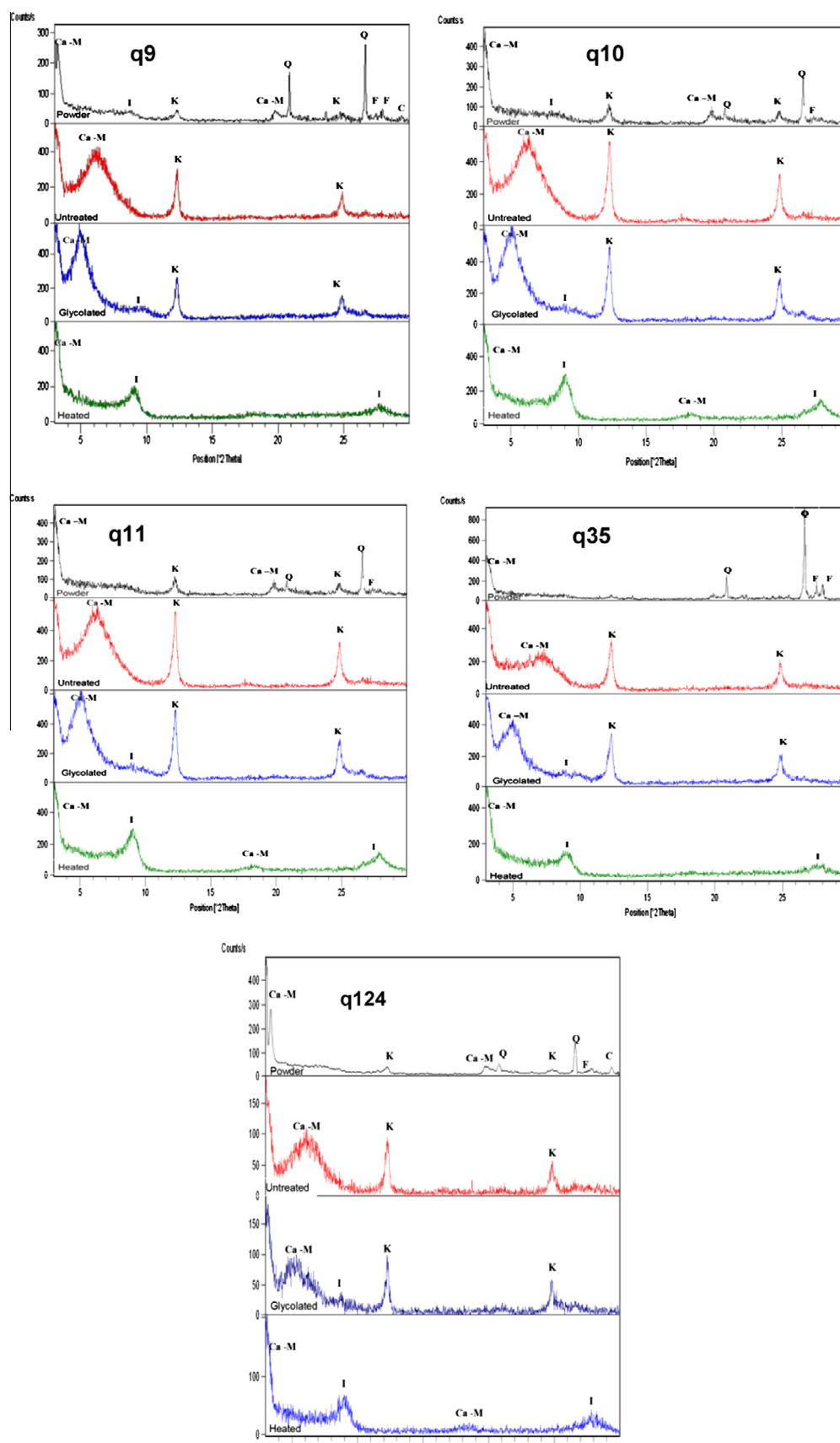Ca-M: Calcic montmorillonite I: Illite K: Kaolinite Q: Quartz F: Feldspar C: Calcite.

Figure 2   X-ray diffractograms of the powder and treated clay fractions of different samples (q9, q10, q11, q35 and q124).

Table 1 Semiquantitative percentage of the clay minerals in clay fractions of the studied samples.

Sample no.	Montmorillonite, %	Kaolinite,%	Llite, %
q9	65	13	4
q10	66	14	2
q11	63	12	4
q35	75	11	2
q124	47	18	5

4.1.2. Thermal analyses

DTA and TGA thermo-grams of the studied samples (Fig. 3) showed low temperature endothermic peaks at about 76–97 C which revealed that the absorbed and exchangeable cations coordinate water were released. The variation in the peak sizes maybe due to the variation in the montmorillonite content in the samples [26]. The peaks at about 270–268 C may relate to the dehydration of goethite mineral [FeO (OH)]. Otherwise, these peaks represented the loss of the interlayer water of both montmorillonite and illite structures [27]. The presence of iron mineral was confirmed by the field observation whereas yellow and purple patches were enclosed in claystones. Endothermic peaks at about 511–523 °C represented both of the starting removals of the structural OH^- from montmorillonite, illite and kaolinite lattices and the production of amorphous phase for kaolinite (disordered metakaolin). The relatively low peak temperature of dehydroxylation of the samples may be attributed to the presence of excess structural iron in the octahedral layer of the smectite. The small curves at high temperatures (890–941 C) indicated by the S-shaped endothermic–exothermic reaction peaks referred mainly to the final structural breakdown of the montmorillonite and to the recrystallization of its components [28]. These system peaks were not accompanied by any loss of weight since they are structural peaks. All the detected endothermic peaks of the samples were accompanied by a corresponding total loss of weight (TGA) at a range of 10.8 (q35) to 12.5% (q124).

4.1.3. Chemical composition, soluble salts and pH value

Table 2 shows the chemical composition of the studied samples. As expected, all the examined samples consisted mainly of SiO_2, Al_2O_3 and Fe_2O_3 in a descending order of abundance. A minor to trance amounts of CaO, MgO, Na_2O, K_2O, TiO_2, SO_3 and P_2O_5 were also detected. The percentage of the main oxides indicated the presence of smectite clay. The small percentage of CaO was attributed to the presence of lime concentration, as detected by the XRD analysis. The loss on ignition values primary were referred to the removal of hygroscopic water and loss of the interlayer water in the structure of clay minerals. Also, results of soluble salt content exhibited trace amounts (<2%) of Na^+, K^+, Cl^- and SO_4^{2-}. Moreover, the pH of the examined clay samples showed alkaline medium. The values were between 8.35 (q9) and 10.17 (q124). The rocks containing montmorillonite mineral produced an alkaline pH [29]. The results of the SiO_2 and Al_2O_3 were in agreement with the relationship between the pH value and the solubility of these oxides. It was well known that both silica and alumina are increasingly soluble at higher pH values [30].

4.1.4. Particle size distribution

Results of the particle size distribution by the laser diffraction grain-size analyzer are listed in Table 3. All the studied samples were of a well-sorted class of the argillaceous sediments (rock contains more than 90% silt and clay). The samples Nos. q9, q10 and q35 showed a very fine-grained claystone class. The clay size fraction varied from 82.23 (q9) to 89.60% (q35). The q11 and q124 had appreciable amount of the silt fraction. These samples achieved a silty claystone category of fine-grained and very fine-grained classes, respectively. It is worthy of note that the samples q9 and q10 provided the highest content of the clay fraction (<2 μm) of about 82%. This result was confirmed by the XRD analysis and given in Table 1.

4.1.5. Atterberg limits, free swell test and activity ratio

Results of the physical properties of the studied samples are summarized in Table 4. It was found that all the samples were inorganic clays of high plasticity (CH). The liquid limit (LL) varied from 74 (q124) to 185 (q10) with a corresponding plastic limit (PL) from 30 (q124) to 64 (q10). Liquid limit greater than 50% referred to the presence of montmorillonite [31]. The Atterberg limits (LL, PL and PI) were in good agreement with the XRD results. The higher the clay as well as the montmorillonite contents, the higher the Atterberg limits. Results of the free swell test showed a range from 140 (q124) to 600% (q10) indicating the samples of moderate (q124) to extremely high swelling potentials (q9, q10, q11 and q35). The results explained that as the montmorillonite content increased the free swelling increased.

Results of the activity pointed out that most of the samples showed high (q124) to very high potentials (q9, q10, q11 and q35) for swelling [32]. Also, The sample nos q9, q10 and q11 were clays of the active class, q35 of normal class and q124 of inactive class. The obtained results from the activity ratios denoted the presence of montmorillonite in calcic form with a strong potential for swelling.

As a comparison between the results of mineralogical analysis, clay activity and the free swell test, it was found that a strong relationship exists between the high content of montmorillonite, the high free swell and the potential affinity for swelling.

4.2. Rheological properties of the slurry before activation

Results of the studied rheological tests of the non-activated samples are demonstrated in Tables 5–7. The incremental concentration of the samples led to increasing of the viscosity, yield point, gel strength and decreasing of the filtrate loss. Most of the obtained results were out of the recommended standard limits of OCMA and API specifications (Table 8) which means that the clays cannot be applied as a drilling mud without activation. The viscosity was increased by decreasing of silt fractions or by decreasing the particle surface area (clay fraction) [5]. The high content of silt (29.3%), as a non-swelling-fraction, and lowest content of the clay particles (70.7%) as well as montmorillonite content (65%) provided poor properties for q124. Q11 and q35 showed good results for the filtrate loss according to OCMA specification (16 ml max.) but with only high clay concentration (6.5 and 7.5%). The YP should be high enough to carry cuttings out of the borehole but not so large as to create excessive pump pressure when starting the mud flow. The samples that matched requirements of the OCMA specification for the filtrate loss will reduce the cake formation and prevent the hole packed off. These will run simultaneously with the cost reduction.

Figure 3   DTA and TGA thermo-graphs of the studied samples.
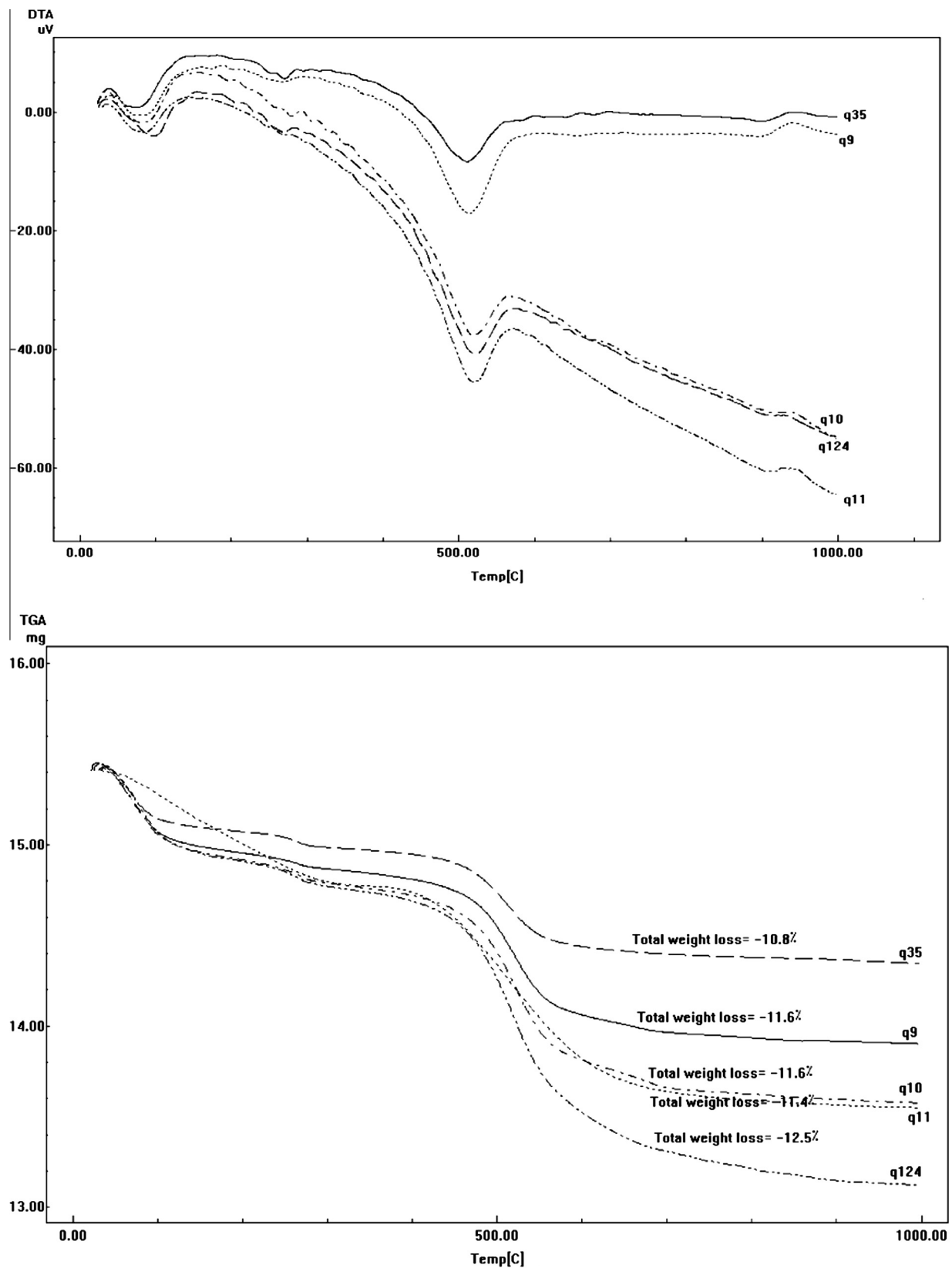
Overall, the presence of the montmorillonite in calcic form achieves unsatisfactory rheological results for the samples. To reduce the calcium content in the montmorillonite, the research directed to add Na activator materials (activation process). The Na ions are expected to replace Ca ions leading to the increase of repulsive attractive forces between the particles

Table 2 Soluble salts, oxides content, LOI and pH values of the studied samples.

sample no.	Cations and anions of soluble salts, %				Oxide content, %										L.O.I.%	pH
	Na^+	K^+	Cl^-	SO_4^-	SiO_2	Al_2O_3	Fe_2O_3	TiO_2	CaO	MgO	Na_2O	K_2O	SO_3	P_2O_5		
q9	0.54	0.03	0.10	0.48	48.16	26.99	7.79	1.29	0.50	1.60	1.41	1.60	0.02	0.01	8.35	12.35
q10	0.99	0.15	0.02	0.33	48.86	19.40	13.17	0.31	1.88	2.03	2.62	1.66	0.06	0.01	9.75	12.11
q11	0.88	0.15	0.037	0.44	51.31	21.94	9.93	0.72	1.41	2.08	2.78	1.87	0.05	0.01	9.21	11.95
q35	0.27	0.03	0.15	0.54	48.56	26.18	8.02	1.37	0.81	1.80	1.74	1.65	0.01	0.01	8.63	11.22
q124	1.63	0.18	1.80	0.82	45.55	17.32	10.49	0.04	1.84	2.01	2.87	0.20	0.20	0.77	10.17	13.83

Table 3 Data of the grain size analysis in weight percentage of the studied samples.

Sample no.	Size fraction (microns)								Clay	Total
	Sand				Silt				$<2\,m\mu$	
	Coarse	Medium	Fine	Very fine	Coarse	Medium	Fine	Very fine		
q9	0.00	0.00	0.00	0.00	0.00	0.1 0	0.15	17.61	82.23	100.00
q10	0.00	0.00	0.00	0.00	0.00	0.10	0.11	17.35	82.23	100.00
q11	0.00	0.00	0.00	0.00	0.00	0.00	3.11	18.27	78.62	100.00
q35	0.00	0.00	0.00	0.00	0.00	0.00	0.00	10.40	89.60	100.00
q124	0.00	0.00	0.00	0.00	0.00	0.26	5.94	23.10	70.70	100.00

Table 4 Results of the Atterberg limits, free swell (%) and activity of the studied samples.

| Sample no.	LL	PL	P I	Free swell (%)	Activity (A)
q9	166	59	107	500	1.30
q10	185	64	121	600	1.46
q11	140	55	85	270	1.25
q35	150	56	94	550	1.04
q124	74	30	44	140	0.60

and hence, improve the technological properties. On the other hand, the activation of bentonite may affect bentonite properties from two aspects by changing the water adsorption capacity and so swelling of bentonites, and the separation of the bentonite plates.

4.3. Rheological properties of the slurry after activation

Results of the studied rheological tests of the activated samples are illustrated in Table 9 and Fig. 4(a and b). It was observed that the added activators on the clay slurry increased the viscometer dial reading at 600 and 300 rpm. Addition of 1.5% caustic soda led to enhance the rheological properties in spite of which most of the samples still did not satisfy both OCMA and API specifications in one or more parameter values. Addition of 0.5% POLYPAC-R provided a remarkable enhancement in the rheological properties of the samples. Also, the decrease of the gel strength values with activators except for sample q124 was noted. Also, the decreasing was effective with the caustic soda addition. Generally, if the mud has a high gel strength, it will create high pump pressure in order to break circulation after the mud is static for long time. Furthermore, increasing gel strength indicates a buildup of ultra fine solid. Therefore, the mud must be treated by adding chemicals or diluting with fresh base fluid.

All samples except q124 met the specifications but may need a little amount of filtrate reducer to minimize the filter cake thickness, such as starch or carboxymethyl cellulose (CMC), particularly in case of NaOH. Moreover, the addition of both of the used activators did not provide good properties for the q124 to achieve the drilling fluid specifications. This could be attributed to the low content of montmorillonite and the presence of high proportion of non-swelling fraction. This sample may need a high concentration of the used activators or other types of activators should be used. Overall, this sample is inactive as revealed from the result of activity ratios (0.60). It is worth mentioning that the plastic viscosity of the activated clay samples using both activators varied between 7 (q11: NaOH,q 35: POLYPAC-R) and 10cP (q9: NaOH and q124: POLYPAC-R). It satisfied the oil grade bentonite requirements of 5.0cP for Meycogel and relatively near to 11.0cP for the Wyoming bentonites. Also, the sample response to the activators by the addition of POLYPAC-R was better than those of NaOH. Moreover, NaOH reacted with a calcium bentonite as follows [33]:

$$Ca\text{-bentonite} + NaOH \rightarrow Na\text{-bentonite} + Ca(OH)_2.$$
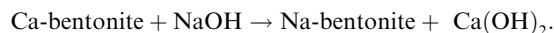
The positive properties obtained by the addition of the activators were attributed to the function of the materials added. Caustic soda is used in most water-base muds to increase and maintain pH and alkalinity of drilling fluids. Increasing the pH led to the increase of fluid viscosity due to the action of hydroxyl ions (OH^-). It is expected that NaOH is able to produce a high amount of the hydroxide ion (OH^-) which imparts a higher pH and viscosity of the clay suspension.

The used POLYPAC-R is a polyanionic cellulosic polymer used essentially for controlling water loss in fresh and sea waters, stabilizing the bore-hole and improving viscosity of drilling. The polymer formed a thin, resilient, low permeability filter cake that minimized the potential for differences between sticking and invasion of filtrate and mud solids into permeable formation. On the other hand, polymers in the bentonite suspension interact with clay particles according to their ionic and non-ionic character. The ionic polymers induced electrostatic interactions but the non-ionic ones were adsorbed on

Table 5 Results of the rheological properties of the clay slurry for the samples nos. q9 and q10 at different concentrations.

	600 RPM	300 RPM	AV. cP.	PV. cP.	Yp. Ib/100 ft^2	Filtrate loss ml/30 min.
Concentration % sample no.q9						
4.5	15	10.0	7.5	5.0	5.0	24.0
5.5	18	13.0	9.0	5.0	8.0	21.0
6.5	21	15.0	10.5	6.0	9.0	20.0
7.5	28	22.0	14.0	6.0	16.0	18.0
Concentration % sample no.q10						
4.5	16.0	10.0	8.0	6.0	4.0	23.0
5.5	23.0	16.0	11.5	7.0	9.0	22.0
6.5	28.0	21.0	14.0	7.0	14.0	20.0
7.5	32.0	25.0	16.0	7.0	18.0	18.5

Table 6 Results of the rheological properties of the clay slurry for the samples nos. q11, q35 and q124 at different concentrations.

	600 RPM	300 RPM	AV. cP.	PV. cP.	Yp. Ib/100 ft^2	Filtrate loss ml/30 min
Concentration % sample no. q11						
4.5	18.0	9.0	9.0	9.0	1.0	20.0
5.5	22.0	12.0	11.0	10.0	2.0	18.0
6.5	25.0	17.0	11.5	8.0	9.0	16.0
7.5	32.0	22.0	16.0	10.0	12.0	15.0
Concentration % sample no. q35						
4.5	19.0	8.0	9.5	11.0	3.0	19.0
5.5	23.0	10.0	11.5	13.0	3.0	17.0
6.5	28.0	17.0	14.0	11.0	6.0	15.0
7.5	31.0	21.0	15.5	10.0	11.0	13.0
Concentration % sample no. q124						
4.5	3.0	2.0	1.5	1.0	1.0	62.0
5.5	4.0	3.0	2.0	1.0	2.0	51.0
6.5	6.0	5.0	3.0	1.0	2.0	45.0
7.5	7.0	6.0	3.5	1.0	2.5	35.0

Table 7 Results of the gel strength of the samples nos. q9, q10, q11, q35 and q124 at different concentrations.

Sample no.	q9		q10		q11		q35		q124	
Concentration %	Gel strength, Ib/100 ft^2									
	10^-	10^-	10^-	10^-	10^-	10^-	10^-	10^-	10^-	10^-
4.5	4.0	8.0	5.0	8.0	10.0	12.0	7.0	11.0	1.0	2.0
5.5	5.0	10.0	6.0	10.0	11.0	13.0	9.0	13.0	2.0	3.0
6.5	8.0	15.0	11.0	15.0	15.0	16.0	11.0	14.0	2.0	3.0
7.5	13.0	19.0	13.0	23.0	17.0	18.0	15.0	17.0	3.0	5.0

the surface of clay minerals by steric interactions (repulsive and attractive forces) [34].

When water-soluble polymers were added to bentonite suspensions, they dispersed in the liquid phase (water), their functional groups dissociated and the polymeric chain assumed a straight configuration. The chains were adsorbed on clay particle surfaces according to their ionic nature [34].

The flocculation of water-bentonite system can be achieved and is explained by (i) the formation of bridges, i.e., lateral interactions between hydrophobic groups of polymers that approached the clay particles; (ii) segments of the same polymeric chains that are adsorbed on different clay particles linked to each other and interacting by attraction; and (iii)

Table 8 Limits of the studied rheological specifications of API and OCMA grade.

	Requirement
API Parameter	
Viscometer dial reading at 600 rpm	30.0 (min)
Yield point/plastic viscosity ratio	3.0 (max)
Filtrate (fluid loss)	13.0 ml (max)
OCMA Parameter	
Viscometer dial reading at 600 rpm	30.0 (min)
Yield point/plastic viscosity ratio	6.0 (max)
Filtrate (fluid loss)	16.0 ml (max)

Table 9 Results of the gel strength of the clay slurries at concentration of 6.5% of the studied samples activated by 1.5% caustic soda and 0.5% POLYPAC-R activators.

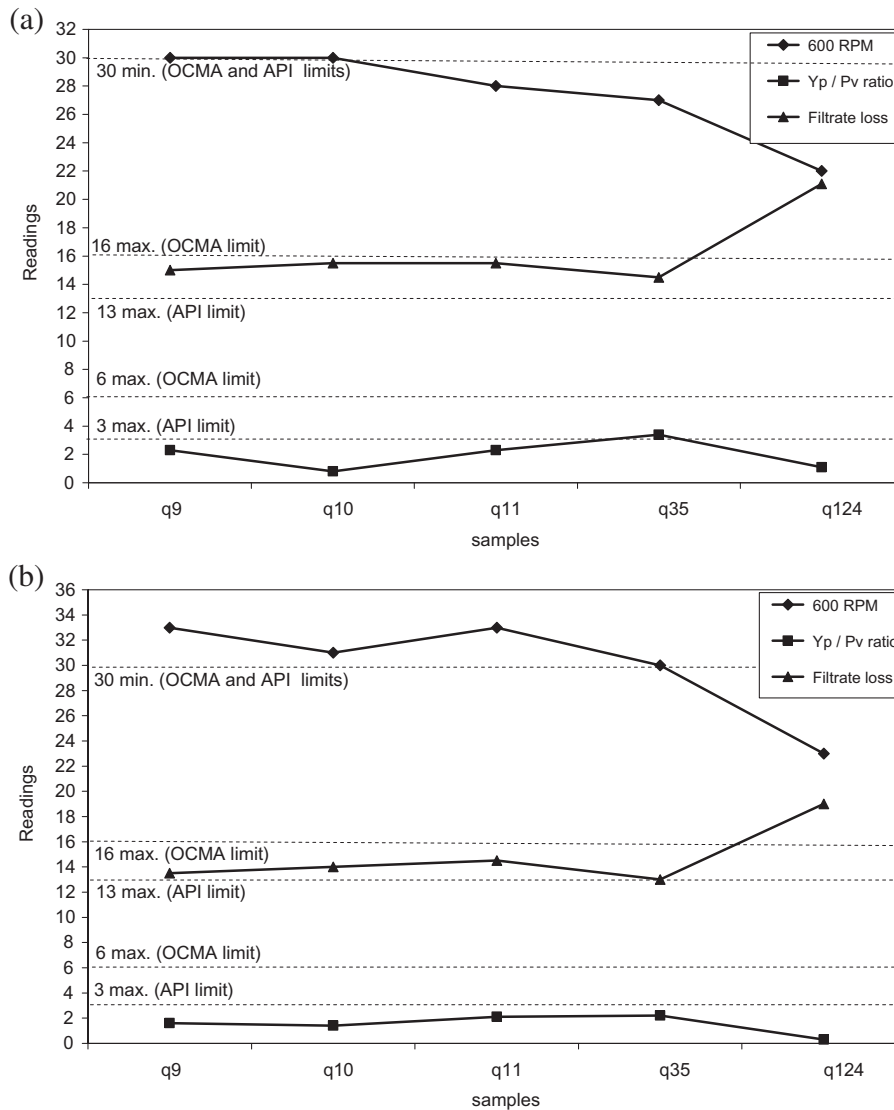| Sample no.	q9		q10		q11		q35		q124	
Activator %	Gel strength, Ib/100 ft^2									
	10^-	10^-	10^-	10^-	10^-	10^-	10^-	10^-	10^-	10^-
caustic soda, 1.5	11.0	12.0	13.0	14.0	12.0	12.0	11.5	12.2	7.0	9.0
POLYPAC-R, 0.5	12.0	13.5	13.0	1.4	12.0	14.0	12.5	13.0	9.0	11.0

Figure 4 (a) Variation of the rheological properties of the clay slurries at concentration of 6.5% activated by caustic soda activator (1.5% by wt.). (b) Variation of the rheological properties of the clay slurries at concentration of 6.5% activated by POLYPAC-R activator (0.5% by wt.).

the encapsulation phenomenon, i.e., the formation of large flocs of particles or clay particle agglomerates [34].

It was stated that any additive, when added into the system, changed the electrical double layers on the surrounding clay particles; consequently, electrostatic behavior between clay particles and rheological properties will be changed [34].

5. Conclusions

The characterization of non-activated clays collected from quarries located in Kom Oshim area, El-Fayoum province, Egypt for the application as a drilling fluid (water-based

mud) were investigated. Also, the activation capability for these clays was explained as an attempt to minimize the importation cost of the imported bentonite to Egypt by a local solution and maximize the usage of the studied clay sediments. The following main conclusions can be drawn from the present study.

(1) Most of the studied samples were of clay-like bentonite of calcic form and exhibited different physical and rheological properties.
(2) The studied non-activated samples cannot be used as a drilling mud without activation.
(3) All the studied clay samples, except q124 (inactive), can be used after activation as a drilling fluid where their rheological properties satisfied the OCMA and API requirements.
(4) The technological properties of the activated samples using POLYPAC-R were significantly improved than those obtained by NaOH activator.
(5) The obtained results revealed that most of the activated samples may be expressed as medium grade bentonitic clays and these grades of clays can be used as a drilling fluid for shallow depth wells.

Acknowledgments

References

[1] F. Lebedenko, D. Plēe, Appl. Clay Sci. 3 (1988) 1–10.
[2] I. Ibrahim, N. Felix, A. Ismail, Ind. Appl. Clays. Mineral. Soc. Egypt (1994).
[3] Inglethorpe, S., Bloodworh, A., Razak, M. Evaluation of clays from the environs of Cairo, Egypt for brick manufacture and use as bentonites. Br. Geol. Surv., WG/90/38R Tech. Report (1993).
[4] M. Hassan, N. Abdel-Khalek, Appl. Clay Sci. 13 (1998) 99–115.
[5] Fahmy, F. Evaluation of some shales in Egypt as drilling fluid base. Ph. D Thesis, Fac. Sci., Al Azhar Univ. Cairo, Egypt, 2004.
[6] L. Amorim, I. Maria, L. Helio, C. Heber, Mater. Res. 10 (2007) 53–56.
[7] C. Karagüzel, T. Cetinel, F. Boylu, K. Cinku, M. Celik, Appl. Clay Sci. 48 (2010) 398–404.
[8] F. Boylu, Appl. Clay Sci. 52 (2011) 104–108.
[9] A. Elewa, A. Omar, A. Dakrory, Egypt. J. Geol. 42 (1998) 439–469.
[10] Abd El-Aziz, S. Stratigraphy of the Eocene sequences of Fayoum-Minia district, Egypt. Ph.D Thesis, Fac. Sci., Cairo Univ. Egypt, 2002.
[11] H. Swedan, Ann. Geol. Surv. Egypt 22 (1992) 157–166.
[12] E. Basta, M. El-Kadi, M. Abdel Maksoud, Bull. Fac. Sci. Cairo Univ. 43 (1971) 271–284.
[13] K. El-barrawy, B. Girgis, N. Felix, Bull. NRC 15 (1990) 177–186.
[14] M. El-Anbaawy, A. Dardir, E. Meabid, Ann. Geol. Surv. Egypt 16 (1989) 45–49.
[15] ASTM D7348. Standard test methods for loss on ignition (LOI) of solid combustion residues. Standard Annual Book of ASTM Standards, USA. 05.06 (2008).
[16] ASTM C114. Standard test methods for chemical analysis of hydraulic cement. Standard Annual Book of ASTM Standards, USA. 04.01 (2009).
[17] A. Al-Zahrani, S. Al-Shahrani, Y. Al-Tawil, J. King Saud Univ. 13 (2000) 193–203.
[18] M.P. Picard, Sedi. Petrol. 41 (1971) 179–195.
[19] ASTM D4318. Standard test methods for liquid limit, plastic limit and plasticity index of soils. Standard Annual Book of ASTM, USA. 04.08 (2010).
[20] W. Holtz, H. Gibbs, Trans. Am. Soc. Civil Eng. 121 (1956) 577–641.
[21] A. Ahmed, Civil engineer guide for the designing and enforcement of concrete buildings, part 9: Soil mechanics and foundation designing of the shallow foundations and piles, Egypt. Lib, Cairo, Egypt, 2003.
[22] Skempton, A. The colloidal activity of clays. Proc. 3rd Int. Conf. Soil Mech. Found. Eng. 1 (1953) 57–61.
[23] ASTM D5891. Standard test method for fluid loss of clay component of geosynthetic clay liners. Book of ASTM Standards, USA. 04.13 (2009).
[24] B. Erdogan, S. Demirci, Appl. Clay Sci. 10 (1996) 401–410.
[25] I. Odom, AIME Trans. 282 (1987) 1893–1901.
[26] R. Grim, W. Bradley, G. Brown, Mineral. Soc. Great Brit. Monog. 8 (1951) 138–172.
[27] R. Grim, Applied clay mineralogy, McGraw-Hill, New York, 1962.
[28] J. Zussman, Physical methods in determinative mineralogy, 2nd ed., ACD Press, London, United Kigdom, 1977, 631–636.
[29] M. Vander, M. Merwe, M. Christl, Aust. J. Soil Res. 40 (2002) 115–120.
[30] Mason, B. Principles of geochemistry.3rd Ed. John Wiley and Sons, New York, 1966.
[31] B. Means, J. Parcher, Physical properties of soils, Charles E. Merrill Publishing, Columbus, Ohio, 1963.
[32] A. Saidi, M. Shamanian, M. Barati, K. Azari, Hyperactivation of bentonite in pelletizing process, Int. J. ISSI 1 (2004) 38–41.
[33] Amorim, L., Melhoria, V. Proteção e recuperação de fluidos hidroargilosos para uso na perfuração de poços de petróleo. Ph. D. Thesis, Campina Grande: Federal University of Campina Grande, 2003.
[34] L. Amorim, M. Barbosa, H. Lira, H. Ferreira, J. Mater. Sci. 40 (2005) 171–177.