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Beneficiation and Technological Properties of Clays from Sabha South East of Libya

Alhadi M Ali Shiwa
University of Zawia
Faculty of Oil, Gas and Renewable Energy Engineering
Department of Geological and Geophysical Engineering
Email: a.shiwa@zu.edu.ly

Abstract

This research paper gives attention to the mineralogical and physical characterization of Sabha clays from Libya. Apart from that, the impending of the Sabha clays to the industrial application was also evaluated. The Sabha clays are geologically linked to Messak Formation of Jarmah Member and the industrial significance was not tested yet. Mineral recognition and characterization studies were accomplished using X-ray diffraction (XRD), differential thermal analyses (DTA), thermogravimetric analyses (TGA) and scanning electron microscopy with an energy dispersive X-ray spectrum (SEM-EDX) technique. Physical tests such as particle size, BET surface area, rheology, zeta potential, plasticity, colour, brightness, pH, cation exchange capacity and water content (W.C) was also performed. Based on the analyses, the Sabha clays are composed of kaolinite as its major phyllosilicate, whereas quartz occurs in minor amounts and could be used for industrial application.

Kay word: Jarmah Member, Kaolinite, Sabha, Libya.

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1. Introduction

Ceramic is obtained by releasing natural clay containing various materials. The main minerals and the most commonly used are kaolin. Kaolin is a soft, light, often chalk-like sedimentary rock that has an earthy odour. In addition to kaolinite, kaolin usually contains quartz and mica as well as, often, feldspar, illite, montmorillonite, ilmenite, anatase, hematite, bauxite, zircon, rutile, kyanite, Sillimanitesillimanite, graphite, attapulgite and halloysite. Kaolinite, a major component of kaolin considered as silicate aluminium silicate. It is white, grey-white, or slightly coloured and mainly consists of degradation of feldspar (potassium feldspar), granite, and aluminium silicate. The kaolin is developed through the Kaolinization process. It has a stable chemical structure and good physical properties for ceramic production. It is plastic, low dry shrinkage during the stage of processing and has the melting point of 1750 ° C. Calcination of kaolin will transform its original appearance to pure white colour (Verga 2007) the main character, which determines the utility of the clay for various applications, is its purity. Pure kaolinite ($Al_2O_3.2SiO_2.2H_2O$) is white in colour and its chemical composition is 46.54% SiO_2, 39.50% Al_2O_3 and 13.96% H_2O. The mined kaolin is usually associated with various impurities such as quartz, anatase, rutile, pyrite, siderite, feldspar, etc., depending on the origin and depositional environment (Grimshaw 1971). This paper aims to contribute to the study of the physico-chemical behaviour of Sabha clays from Libya and its effect on calcination process.

2. Geology and characterization of Sabha clays

Sabha clays are unconformably overlying the Carboniferous strata in the SE and S sectors of Sabha area. This sedimentary rock formation is the part of the continental Mesozoic sequence that composes of sandstones, siltstones, claystones and conglomerates. It was previously assigned to the Continental Post- Tassilian Group or to the Nubian Sandstone (Burollet et al. 1960, Goudarzi 1970, etc.). The term Messak Sandstone was proposed for the unit by Klitzsch (1963), who later described its type section on the western flank of the Murzuq basin (Klitzsch 1972). The Messak Formation was divided into the lower Jarmah Member and the upper Awbari Member (Strojexport 1980). This lithostratigraphic division is mainly based on the presence of claystones in the Jarmah Member; the Awbari Member is almost completely built of sandstone. The type section of the Jarmah Member was described by Klitzsch and Baird (1969) from the area of Awbari sheet. This section can also be considered as the type section for the Awbari Member. A further subdivision of the Jarmah Member into Irawan beds and Sabha beds, and Lower and Upper Sabha beds (Strojexport 1980). These informal units are regarded as being only of local importance and used for detailed investigation of clay deposits in the vicinity of Sabha. Fig.1 showed the location of Kaolinite in Sabha.

The type section was named Germa beds by Klitzsch and Baird (1969) after the village of Jarmah (Germa). A typical feature of the Jarmah Member is its beds, which consist of up to several meters thick of claystone and siltstone, overlying the basal layer of sandstone and conglomerate. The latter is insufficiently exposed in the area; its thickness is estimated at about 10 m. The nature of the boundary between the Lower Carboniferous and Jarmah Member can only be studied in the northern part of Az Zíghan. Basal conglomerates consist of rounded quartz pebbles, probably coming from the basement. Basal beds in the area between Sabha, Tamanhint, Samnu and Az Zíghan are overlain by a sequence of kaolinic claystones and sporadic siltstones with intercalations of kaolinic sandstone. Unlike the underlying Marár Formation, the Jarmah Member contains better-crystallized kaolinite (Strojexport 1980). The sequence contains ferruginous layers and in places abundant plant fossils. The admixture of muscovite varies. The thickness of the sequence ranges from about 20 to 25 m. Sedimentation continued upwards with a thick sandstone complex with infrequent intercalations of siltstone and claystone, sometimes containing an admixture of sand. The percentage of claystones and siltstones is 35 to 37 % at a locality 18 km west of Sabha, approx., 25 % NE of Sabha and close to zero (4 %) 9 km SW of Samnu. The prevalent rock type is quartzose sandstone, often laminated. Clastic quartz grains (with acicular rutile: sagenite) and sporadic polyaggregate grains are subrounded, poorly to medium-sorted and prevalently medium sized. The matrix is usually clayey/silty, forming streaks and laminae. Common is a cement of intergranular quartz particles. Calcareous impregnations are sporadic (Strojexport 1980).

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Fig. 1 Location Map of Sabha and the nearby

3. Materials and analytical methods

For this study about five representative Sabha clays samples (1-2kg each) were collected from Samnu, Tamanhint and Sabha. All this locality is located in the South Eastern part of Libya(Fig.1) The mineralogical study of Sabha clay was measured by X-ray Diffraction (XRD) techniques, and Optical Microscopy (OM. Fig. 2 shows the XRD pattern fractionated to +63 µm, sieved, and Ni-filtered under Cu-Kα radiation at a 2θ/θ range of 0.040° at 25°C (room temperature). The primary properties of Sabha kaolinite such as pH, electrical Conductivity (EC) by (μ_s), brightness test (BR), plastic index (PI) and water content (W. C) by (%), were evaluated in this study and all results shown in Table No.1. Physical properties such as particle size, BET surface area, zeta potential and rheology are also tested. The geochemical behaviour of Sabha clays is investigated by X-Ray Fluorescence (XRF) method and the Cation Exchange Capacity (CEC) analysis is measured by Atomic Absorption Spectrometer (AAS). The micromorphology parameters of Sabha clays are revealed by Scanning Electron Microscopy (SEM Philips 20.00 KX) - Energy Dispersive X-Ray Spectroscopy (EDX).

4. Results

4.1 Mineralogical Analysis

The petrographic analysis and X-ray Diffraction (XRD) methods are performed on the thin section of the Sabha clays. The results indicate a similarity in kaolinite/quartz ratio and trace percentage of heavy minerals; Fig.3 shows the peaks of XRD diagram and it is in agreement with the petrology study by thin section in Fig.2.

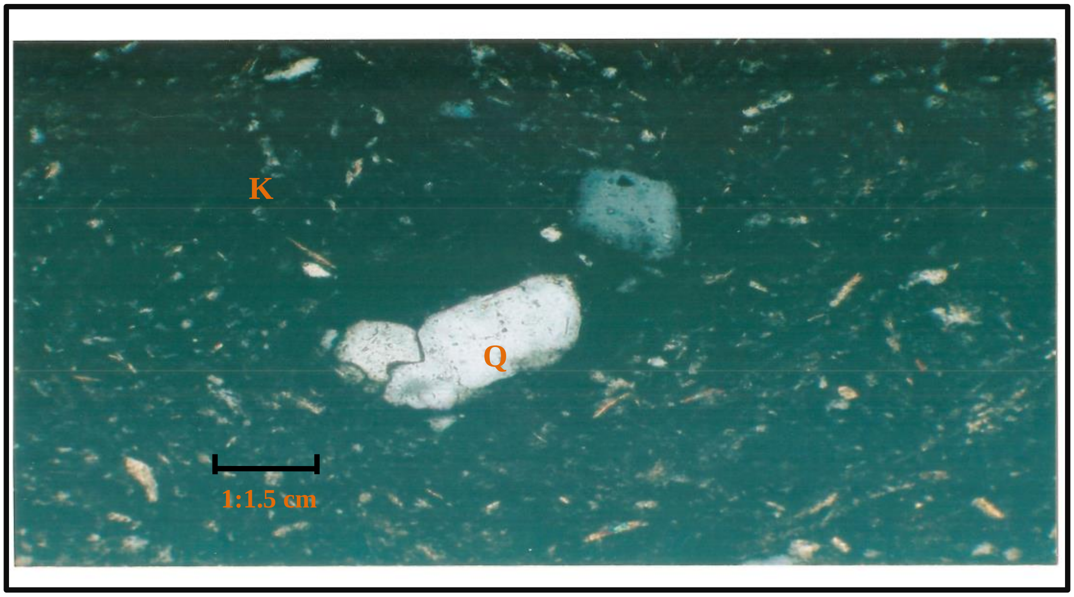

Fig.2: Petrographic features Sabha clays. Kaolinite occurs as matrix mass, whereas the anhedral quartz is considered as impurities (, K: kaolinite and Q: quartz)

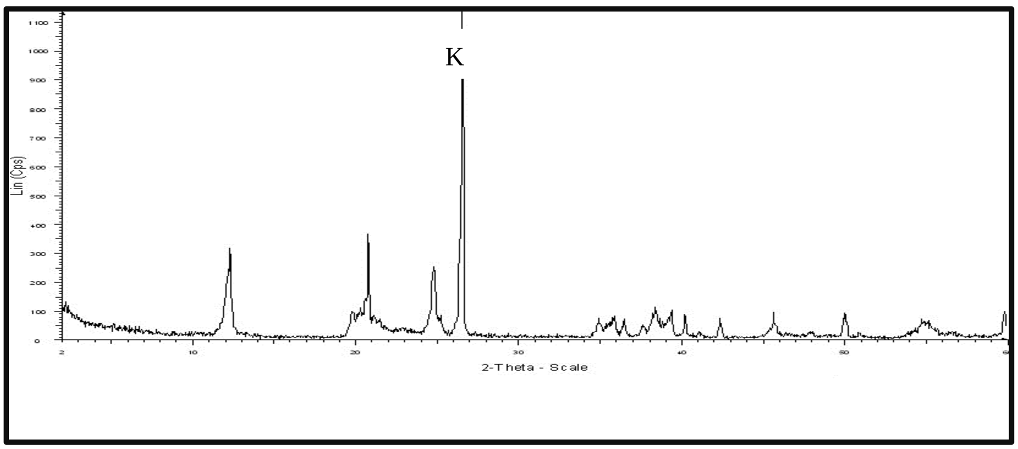

Fig.3 the XRD patt Q Sabha clays K: kaolinite and Q: quartz.

4.2 Physical properties

All results of the physical parameters of Sabha clays were measure and are simplified in Table No.1, the brightness value of all samples seem to be in the range (81.15 and 82.71). This value indicates that from the Sabha clays is potentially economic. The brightness always related to the impurities content and the highest always considered as high quality The petrographic analysis also reveal that the Sabha clays composed with kaolinite and quartz in the ratio of 50:50% in volume. The findings agree with Ramadan et al, 2008 whom stated that the to qua South Eastern of Libya, has numerous clay resources that dispersed along a great part of, mainly in the Sabha zone, Wadi Shatti and surroundings (Ramadan, et al, 2008), as shown in fig.1.

Table No.1 the physical properties of kaolinite from Jarmah Member, Sabha, Libya.

Sample No.	PH	EC	BR	PI	W. C (%)
1	6.93	70.80	81.15	35	37.38
2	8.04	95.40	82.19	49	53.91
3	8.09	110.02	82.32	45	48.83
4	9.01	93.25	82.71	43	45.93
5	7.30	94.25	81.09	31	34.06

4.2.1 Particle Size

The particle size distribution of kaolin is evaluated using a laser particle size analyzer (Malvern, Mastersizer 2000) with a fraction less than 63µm.Particle Size distribution and surface characteristics are important properties affecting the transport of particles and thus also the trace elements associated with them. However, determination of particle size distributions in natural, heterogeneous particle populations is subject to artefacts arising not only from the limitation of the techniques used by (Hassellöv et al. 2001).

The results indicate that Sabha clays composed of very fine colloidal particles. More than 95% of the particles having a mean size in the range of 22 - 47µm indicating that there were little amounts of as impurity minerals associated with tested kaolin.

Sabha clays composed of particles with size range varying from silts (<50 – 2µm) to submicron clays (<1 µm). The results from the curve (0.35 - 8 µm) it showed non-uniform properties. Results of particle size distribution of Sabha clays is presented in a cumulative curve. Fig. 4.

Table 2 the specific surface area characterizations of the studied Libyan kaolin.

Sample No.	**1**	**2**	**3**	**4**	**5**	**6**
Surface Area (m^2/g)						
Single point surface area at P/Po	1.41	11.25	3.56	9.01	9.37	9.62
BET Surface Area	1.58	11.57	3.68	9.37	9.86	9.93

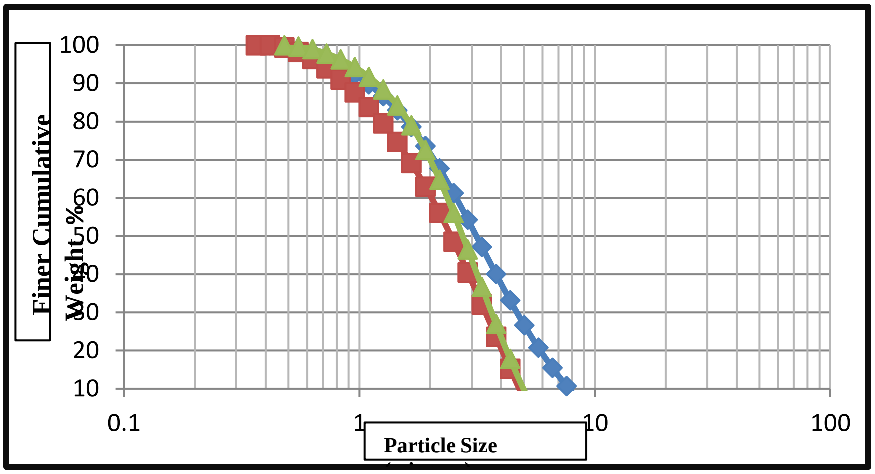

Fig. 4 Particle size distribution of Sabah kaolin, Libya.

4.2.2 Specific Surface Area

The specific surface area for the studied kaolin was examined using Brunauer, Emmett and Teller (BET) analysis. The BET analysis on the tested samples was done by a single point surface area and single point desorption pores at a relative pressure at (P/P0).

Table 2 shows the results of the surface area properties of the studied kaolin retrieved from the BET analysis. The results revealed that the maximum value was 9.93 (m^2/g) for sample No.6 with Single point surface area at P/Po value 9.62 (m2/g).

Figure 5 shows the BET surface area plot of the studied kaolin; the analysis conditions were set at the bath temperature 195.85 °C. It noted, that all point were matching and closed, this indicating, the chemical and mineral composition was homogenized.

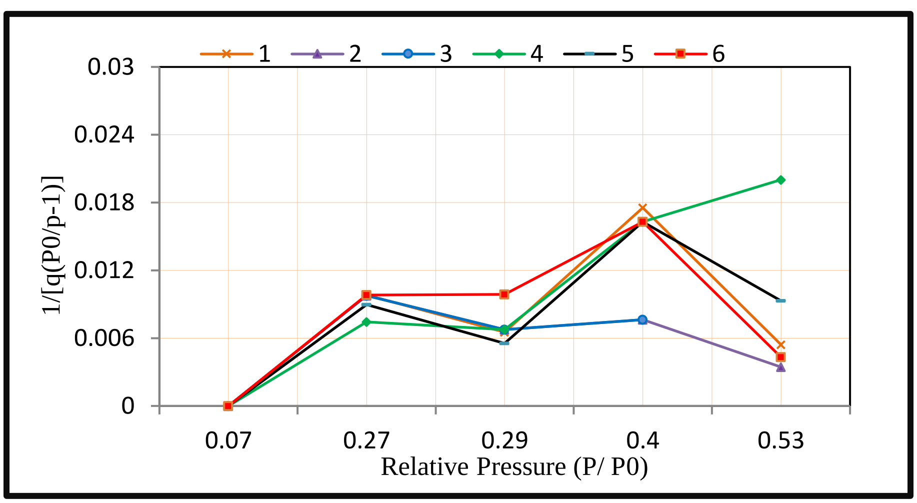

Fig. 5 shows the BET surface area plot studied kaolin

4.2.3 Rheology Analysis

Figure 6 reveals that collected sample No.1 has shear rates (1 /S) at 10-40 This indicates that the material responded to the increasing rate of the twist of samples, monitored with the increased amplitude shear stress and strain at a constant temperature. The value of viscosity is decreased with the increase of the amplitude of the shear rate (1 / S). Studied kaolin has shown good viscosity as it was in the range of (0.00 X 10-3 1.2.0 x10-3 pas). Thus, Sabha clay is seen as efficient adsorbents because they sustain the geometry form of tetrahedral.

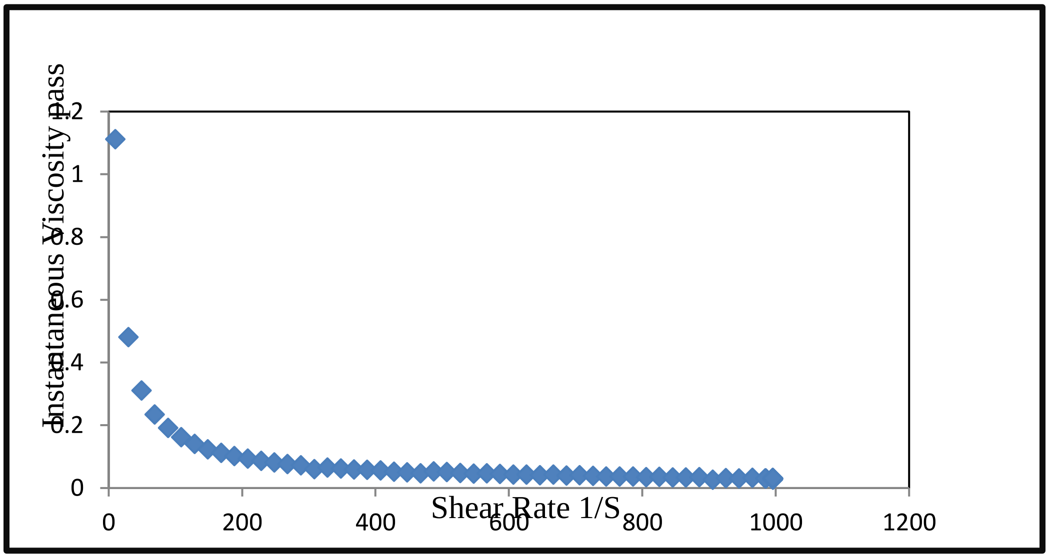

Fig. 6 Instantaneous viscosity of studied Libyan kaolin No.1

The results of the viscosity of samples No.2 and 3 are plotted in Figures 7 and 8. Both samples have shear rates (1 /S) at 5-35, indicating that the material responded to the increasing rate of the deformation of samples, monitored with the increased amplitude shear stress and strain. However, the value of viscosity is decreased with the increase of the amplitude of the shear rate (1 / S). Both studied clays have shown good viscosity as it was in the range of (0.00 X 10-3 to 3.00 pas) and (0.00 X 10 -3 -to 1.00 pas) respectively because they are rich with Si: Al (Johnson et al. 1998). This means that the efficient adsorbents because they maintain the geometry form of tetrahedral. It notable, that matching between all studied kaolin.

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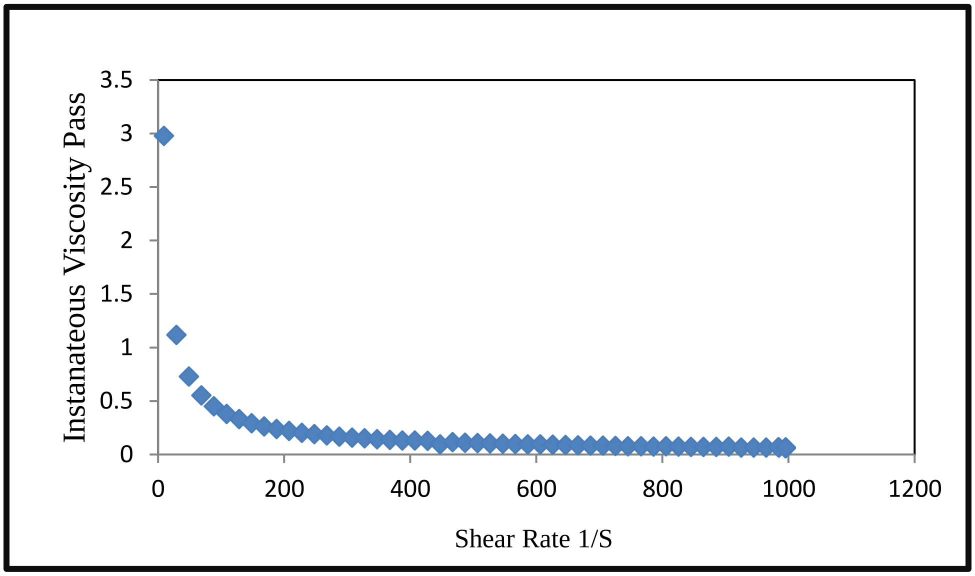

Fig. 7 Instantaneous viscosity of the studied clays (Sample No.2)

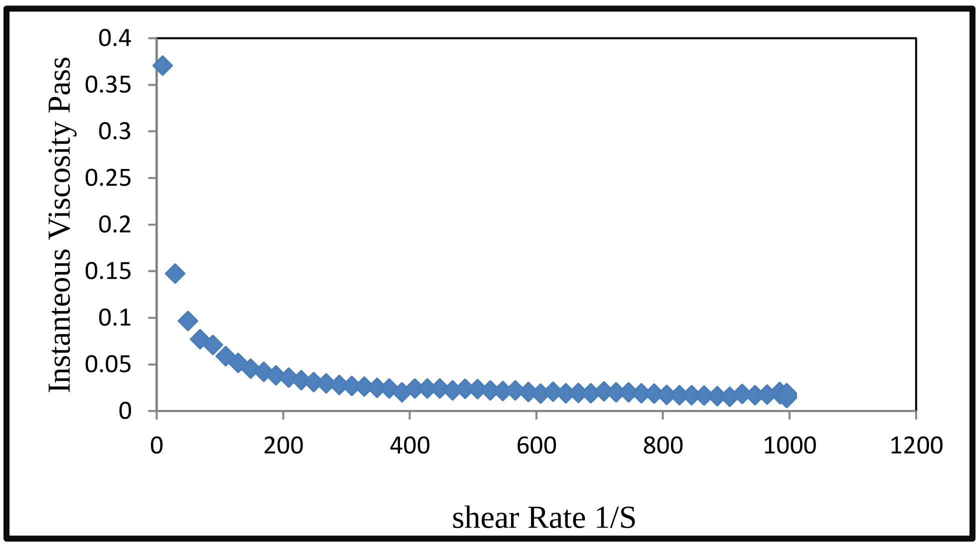

Fig. 8 Instantaneous viscosity of studied clays (SampleNo.3)

4.2.4 Zeta Potential Analysis

From the results of zeta potential for the selected sample is listed in Table 3 and plotted in Figure 9 with the measurement time typically of 1 to 5 minutes. It shows two types of zeta potential values for the selected samples: positive value for samples No. 4, 5, 1and 6 in the range of 0-40 mV and negative value for samples No. 1, 2 and 3 from -20 to -60 (mV) The zeta value of the studied clays significantly showed a similarity with the zeta potential data reported in the literature of kaolin from somewhere else as reported (Pek-Ing Au & Yee-Kwong Leong 2013) by having the zeta potential value from -20 to -65 (mV) with a higher pH value (0-10).

Table 3 Zeta potential (mV) of Sabha clays

Sample ID	Zeta potential (mV)
1	-54
2	-26
3	-32
4	39
5	3.4
6	10.2

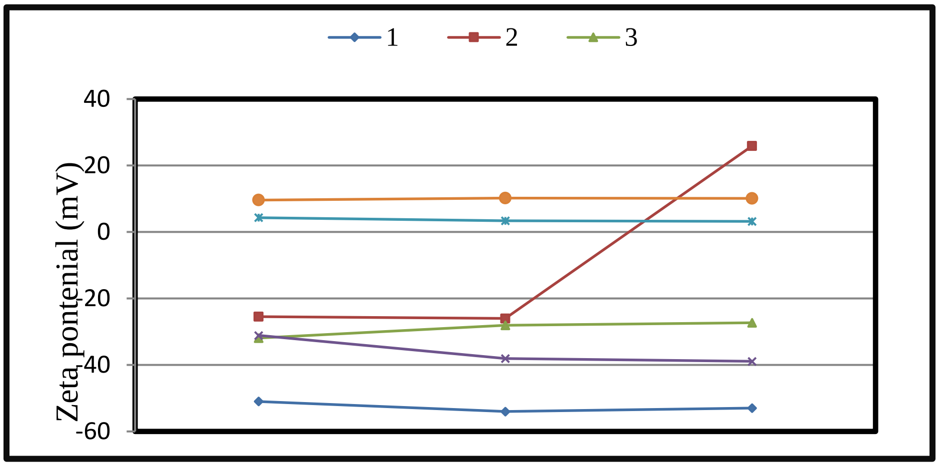

Fig. 9 Zeta potential (mV) of studied Libyan kaolin

4.3 Chemical properties

The results of chemical composition by (%) of Sabha clays are shown in Table No.4., the huge ratio of most of the elements in selected samples was for SiO_2 and Al_2O_3, of which it is in the range of 44.26- 52.24% for $SiO_2$and 35.38 – 4046% for Al_2O_3. According to Grimshaw 1971, he concluded that purity of the clay is one of the utmost characteristic, which determines the utility of the clay for various applications. Pure kaolinite ($Al_2O_3.2SiO_2.2H_2O$) is white in colour and its chemical composition is 46.54% for SiO_2, 39.50% for Al_2O_3, so this values for SiO_2 and Al_2O_3 are the similar Toth value of Grimshaw 1971. Because Basal beds in the area between Sabha, Tamanhint, Samnu and Az Zíghan are overlain by a sequence of kaolinic claystones and sporadic siltstones with intercalations of kaolinite sandstone (Strojexport 1980), we found the high percentage of Fe_2O_3 compared with others (1.07–1.49%), the ratio SiO_2 to Al_2O_3 of Jarmah kaolinite is closed with Mayouom sandy kaolins commercially values averaging 1.01 and 1.30 respectively (Nkoumou et, al, 2009).

Table No. 4 the major elements (%) and loss on ignition of Sabha kaolinite from Libya

Elements (%)	**S1**	**S2**	**S3**	**S4**	**S5**
SiO_2	56.29	50.69	53.07	48.99	56.07
Al_2O_3	38.12	42.97	40.52	44.79	38.44
CaO	0.09	0.08	0.08	0.09	0.06
Fe_2O_3	1.19	1.53	1.21	1.64	1.15
K_2O	0.14	0.25	0.3	0.29	0.14
MgO	0.25	0.28	0.27	0.29	0.24
MnO	0.01	0.01	0.01	0.01	0.01
Na_2O	0.14	0.13	0.13	0.12	0.12
P_2O_5	0.09	0.09	0.1	0.09	0.09
Ti_2O	3.63	3.91	4.26	3.63	3.63
Total	99.95	99.94	99.95	99.94	99.95
SiO_2 / Al_2O_3	1.47	1.17	1.3	1.09	1.45
LOI	4.72	6.517	5.117	5.83	3.989

LOI = Loss on Ignition

Table No.5 and Fig. 10 show us the values of Cation Exchange Capacity (CEC) for Sabha clays, Na, K has the highest value in the samples under t normal dilution because the chemical composition of the minerals which included those samples was kaolinite and quartz. Sabha clay has the excellent degree of sorted and arrangement of kaolinite crystals

Table No. 5 Cation Exchange Capacity (CEC) values (meq /100gm) of Sabha clays

			CEC (meq /100g)		
Sample No.	Na	K	Ca	Mg	Pb
1	5.14	6.85	21.51	46.99	1.15
2	5.15	5.28	14.07	38.51	0.99
3	5.24	6.08	11.08	34.31	1.06
4	5.55	7.03	10.13	23.04	1.07
5	3.81	5.19	17.92	51.84	1.05
Mean	45.80	78.02	40.78	24.31	82.00
Dilution	10	10	200	100	10

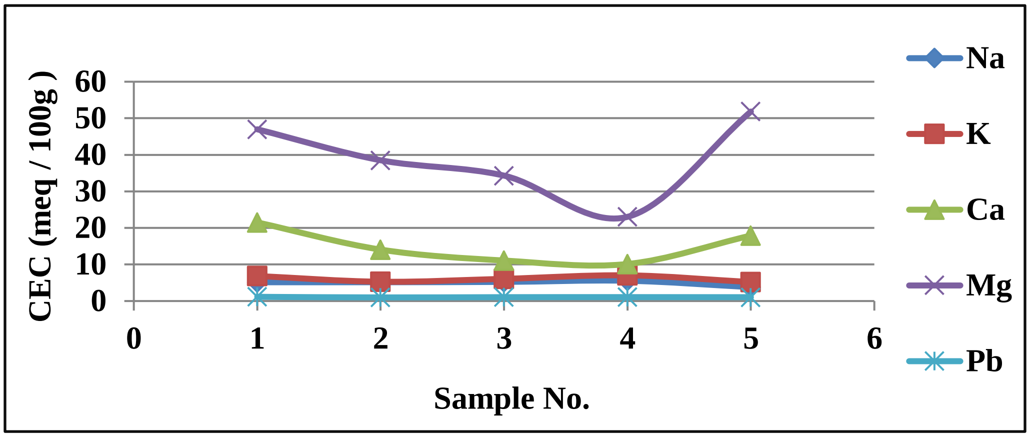

Fig.10 the cation exchange capacity CEC (meq /100gm) of Sabha clays

4.4 Micrographical Properties

Fig. 11 Shown the SEM photography of Sabha clays at a drying temperature (25°C). Fig. 11(A) shows a very well crystallized of kaolinite were found in sample No.1, Fig. 11(b).The crystals of kaolinite in Sabha clays in sample No.2 is shown the arrangement of faces of crystals (fig. 11(c)). It shows the excellent crystallized kaolinite that features the open booklets and well arrangement of platelets. Kaolinite in sample No. 5, indicates the high quality (purity, grain size and efficiency), so the koalinite from Sabha, which is located in a South-Eastern part of Libya Achieve the required specifications for ceramic industrial, which is based on the purity of grain size and the amount of Al_2O_3.

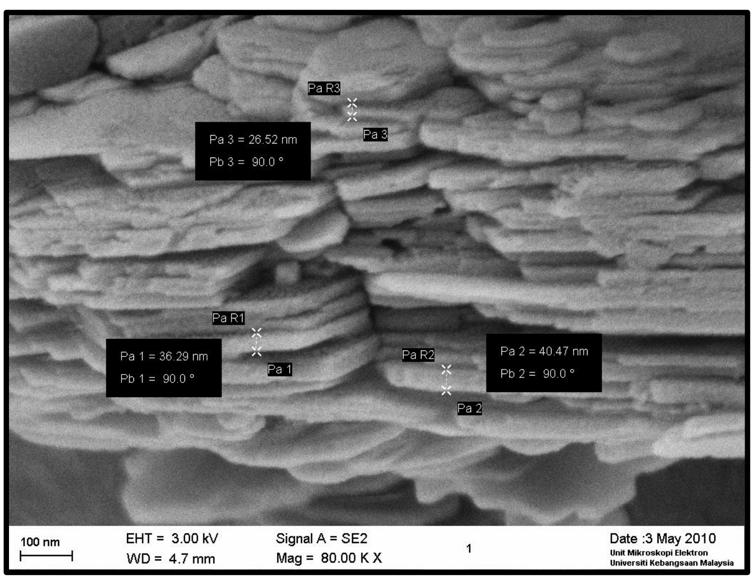

Fig. 11(A) Scanning Electron Micrographs showing well arrangement of platelets of kaolinite

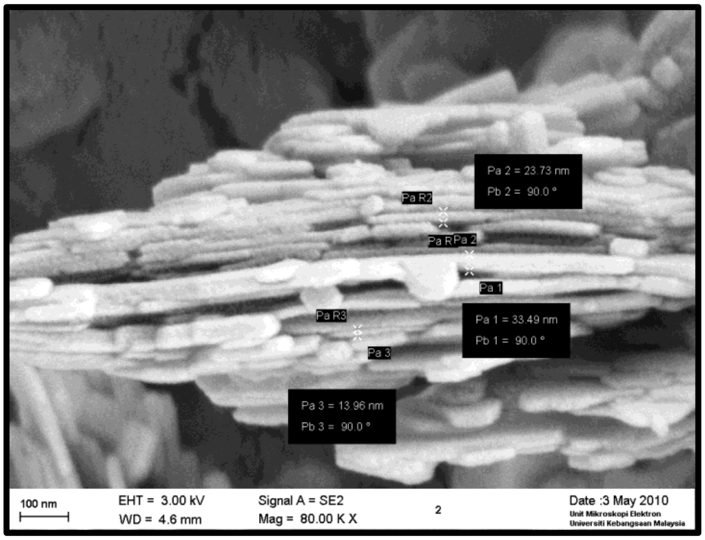

Fig.11(B) Scanning Electron Micrographs showing the distance between the platelets of kaolinite crystals

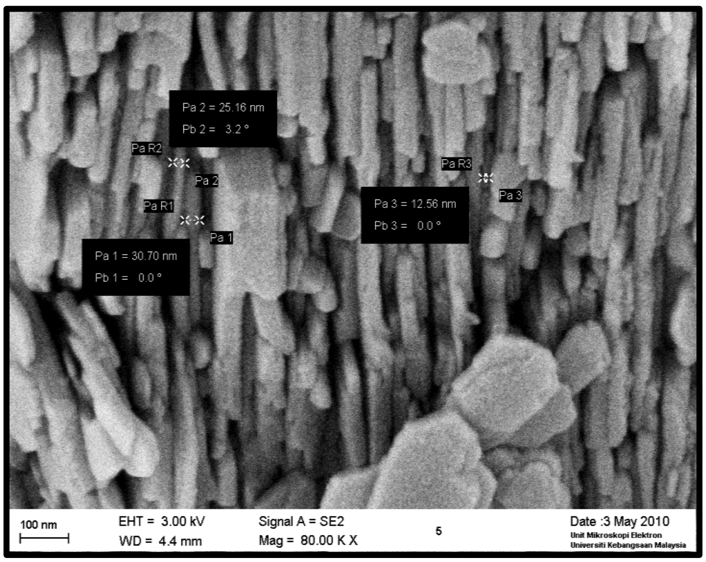

Fig. 11(C) Scanning Electron Micrographs showing the platelets of kaolinite as with angle 90º

5. Conclusion

Sabha clays contain kaolinite, quartz as the major minerals with a minor amount of heavy minerals. The findings were supported by the XRD results and petrography study. It is concluded that the high percentage of Al_2O_3, SiO_2 and CEC value of Sabha clays can be considered as an important deposit for the ceramic industry. Further improvement in physical and chemical properties is needed in order to improvise the quality of Sabha clays for any other industries

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