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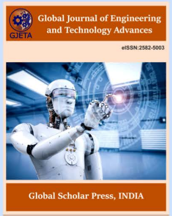

(RESEARCH ARTICLE)

Effects of acid treatment on physical and chemical properties of kaolinite clay from Mayo-Kebi, Garoua sedimentary basin, Cameroon

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Abstract

Kaolinite clay treated by Hydrochloric acid available at Mayo-Kebi, Garoua sedimentary Bassin, northern region of Cameroon was characterized by XRD, FTIR, TGA and Emission Scanning Electron Microscopy (ESEM). This research was performed to improve the purification of kaolinite with the goal of reducing the impurities of iron oxides. The experiments showed that treatment of kaolinite in dilute hydrochloric acid clean the clay minerals by removing iron oxides and other metallic compounds from the surface of the clay.

Keywords: Kaolinite; Hydrochloric Acid; Benoue Bassin; Cameroon

1. Introduction

Kaolinite is an inorganic aluminosilicate clay mineral with natural flaky shape [1]. The general chemical formula is articulated as $Al_2Si_2O_5(OH)_4$. The basal (d001) spacing of kaolinite is 7 Å. Kaolinite has a crystal structure composed 1:1 type of a two-layer structure established by an angle sharing [SiO_4] tetrahedral layer and an edge sharing [AlO_6] octahedral layer [2, 3]. Clays have a variable chemical composition that largely depends on parent rock from which they originated and the physical and chemical changes in the environment where they are found [4]. The industrial utilization of clay is closely related to its reactivity and surface properties and depends strongly on surface modification. Several ways are recommended in literature to enhance the properties of clay materials with thermo-chemical treatment or chemical activation [5]. The most common physical modification is thermal treatment which involves the alteration of chemical composition and/or crystalline structure by the effect of temperature. The structure and composition of clay minerals can be modified by heating at high temperature [6]. Studies by [7] use meta-kaolin to formulate mesoporous catalysts through acid treatment. The study of the effect of acid and alkaline activation on kaolin was also carried out by [8] and established that 6 M HCl activated under reflux states for 6 h will eliminate roughly 90 % of the octahedral Al^{3+} cations, followed by amorphous silica with greater surface area [8].

Acid treatment is one of the most common chemical treatments for clay minerals and has been used to increase the specific surface area and the number of acidic centers, modify the surface functional group and to obtain solids with high porosity. Numerous studies have been reported on the acid treatment of clays, especially on bentonite, vermiculite, kaolin, palygorskite-sepiolite and glauconite [9-10]. The various types of acids used for acid treatment including inorganic acids such as hydrochloric, phosphoric, sulfuric, nitric and organic acids such as acetic, citric, oxalic, and lactic. Among all of these, hydrochloric acid is probably the most widely used in acid activation, because it shows strong affection by the process parameters and superior results in specific surface area, porosity and adsorption capacity [11] This study was conducted with the aim of assessing the effect of treating kaolinite clays from Mayo-Kebi with different concentrations of hydrochloric acid on the elemental composition of clays.

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2. Geological setting

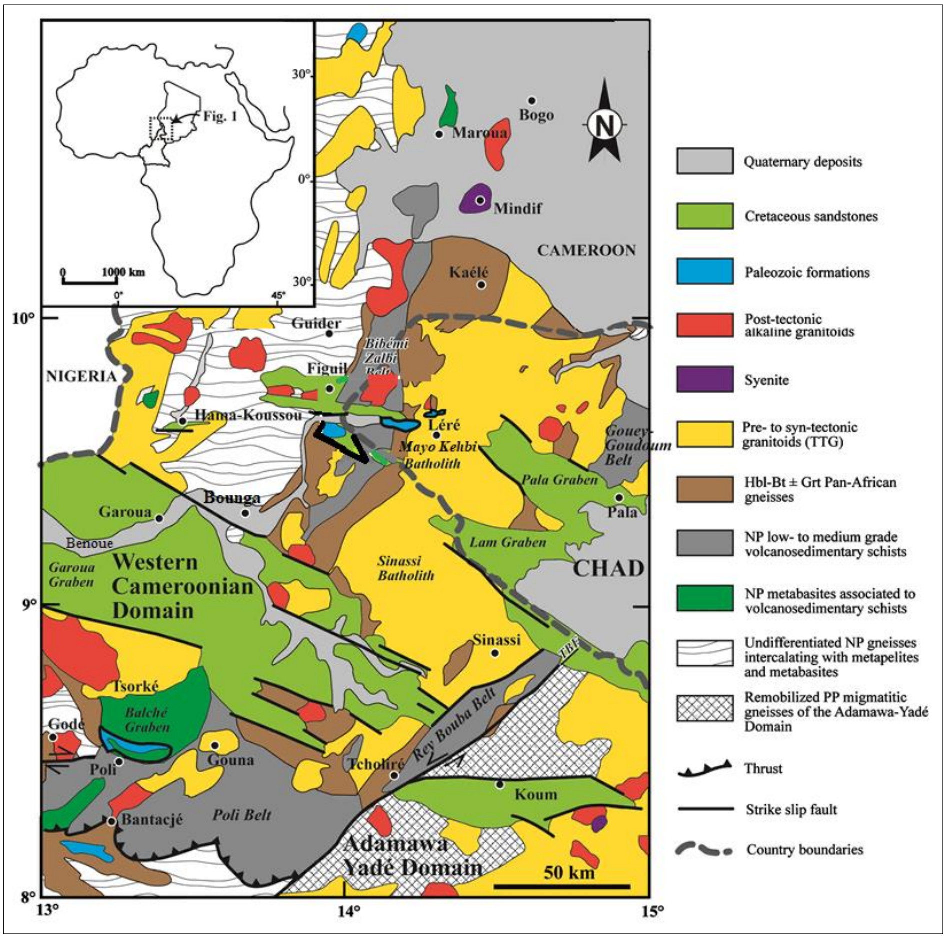


Figure 1 Location and Geological map of North Cameroon (redrawn from [12])

The Garoua sedimentary basin, like many basins of northern Cameroon, is part of the large Benue trough. This trough strikes approximately NE–SW and is about 1000 km long and 100 km wide (figure 1). The first stage of the formation of the trough is related to Permo–Triassic rifting which happened after the fracturing of Gondwana. This stage is followed by Neocomian–Aptian rifting, characterized by the opening of many troughs in west and central Africa, due probably to extensional processes [12]. Structural and geological studies by [13] and [14] show that the Garoua basin is an E–W to N120 trending trough infilled by Middle to Upper Cretaceous marine sandstones. These sediments have also been described by [15] and [16]. The Garoua sandstones are very similar to the Albian Bima sandstones dated in the neighbouring Upper Benue.

3. Experimental works

3.1. Materials

Fresh kaolinite clay was collected from Mayo-Kebi, Garoua sedimentary basin. The kaolinite clay samples were collected at one site (Bounga) in depths at 0.5 and 1.5 meter. From each sampling 5 kg samples were collected and packed in newly cleaned plastic buckets which were then covered with their lids. Hydrochloric acid were purchased from Sigma Aldrich Company.

Kaolinite clay used for this study crushed into powder and washed several times with distilled water to remove any particles attached to its surface. The powder was dried in an oven at 105 °C for 2 hours. Weighed 100 g of each sample was put in a beaker and 200 ml of hydrochloric acid of different molarities (0.1M, 0.25M, 0.5M, 1M, and 2M) was added to each clay sample and the reaction allowed to proceed for 24 hours. Each of the mixtures obtained was subjected to filtration and the residue washed using distilled water to remove any acid-matrix. Rinsed residues were oven-dried then later cooled. The cooled samples were prepared for analysis [4, 17].

3.2. Characterization Techniques

The treated clay was characterized by using different analytical equipment. The chemical composition of the samples was determined by using X-Ray Fluorescence spectroscopy (XRF). Fourier Transformed Infrared (FTIR) spectra were recorded on a Perkin-Elmer infrared spectrophotometer in the range of 400 - 4000 cm^{-1}. Thermogravimetric analysis (TGA) was carried out with a Mettler Toledo TGA/SDTA851. Emission Scanning Electron Microscopy (ESEM) was used to observe the surface morphology and analyze the microstructure of the clay.

4. Results and Discussion

4.1. XRF Characterization

Characterization of the untreated and treated sample with X-ray fluorescence showed that the clay consists mainly of silica and alumina, with metallic compounds like Fe_2O_3 and TiO_2 occurring as minor and other compounds including K_2O, CaO, MgO, MnO. Analysis gave SiO_2 (42.78% depth 0.5 and 55.37% depth 1.5), Al_2O_3 (17.02% depth 0.5 and 23.56% depth 1.5), Fe_2O_3 (19.98% depth 0.5 and 3.98% depth 1.5), TiO_2 (7.76% depth 0.5 and 0.71% depth 1.5), K_2O (0.97% depth 0.5 and 1.63% depth 1.5), MgO (0.08% depth 0.5 and 1.86% depth 1.5), MnO (0.08% depth 0.5 and 0.06% depth 1.5).

The results obtained by weight loss on ignition showed that the average percentage of 10.87% (depth 0.5) and 12.08% (depth 1.5). These values increased by acid treatment, is may be as a result of the presence of organic matter lost during the roasting and/or some non-metals like sulphur which could have been removed from the clay in the form of SO_2.

Once clay treated, it was observed that the composition changes significantly. SiO_2 content was increased with the increasing of acid strength. Also, Al_2O_3 content decreased in the treated sample caused by the leaching of Al^{3+} ions from the octahedral layer due to hydrolysis under acidic conditions [18]. At a very high strength (2M), most of the Al^{+3} and other ions are removed due to severe leaching of the clay structure.

Table 1 Variation of the chemical composition of clays with acid treatment

Depths	Acid conc.	SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	MgO	MnO	TiO_2	LOI
0.5 m	0,0M	42.78	17.02	19.98	0.61	0.97	0.02	0.08	7.76	10.87
	0.1M	45.31	16.73	18.29	0.54	0.95	0.02	0.08	7.24	11.47
	0.25M	50.29	15.56	14.56	0.43	0.93	0.02	0.07	6.17	12.17
	0.5M	51.18	15.26	13.31	0.38	0.88	0.01	0.05	5.89	13.09
	1M	53.23	14.98	9.37	0.35	0.86	0.01	0.04	5.72	15.51
	2M	55.43	13.58	7.94	0.31	0.82	ND	0.01	5.53	16.86
1.5 m	0,0M	55.37	23.56	3.98	0.79	1.63	1.86	0.06	0.71	12.08
	0.1M	56.39	22.38	3.83	0.53	1. 43	1.81	0.05	0.63	13.15
	0.25M	58.23	21.81	3.74	0.51	1.29	1.78	0.04	0.51	13.87
	0.5M	61.09	20.24	3.62	0.44	1.18	1.66	0.03	0.46	14.16
	1M	62.21	17.27	2.57	0.42	1.17	1.57	0.02	0.41	14.68
	2M	64.18	16.09	2.41	0.39	1.12	1.22	0.02	0.39	15.04

ND = Not Detected

4.2. FTIR Characterization

The Fourier Transformed Infrared (FTIR) is shown in Figure 2 and the corresponding band assignments are shown in Table 2. Comparing the graph in Figure 2, it is clear that there were some structural modifications on the clay after treated with different concentration of HCL. The untreated clay shows the absorption bands at 3689, 3621, 1114, 1027, 1003, 908,792, 684, 534 and 523 cm^{-1}. When the clay was treated with HCL, it showed some of the bands were removed or shifted and their intensities also changed. This indicates that there is a minimal damage to the crystal structure of the clay material [19]. During the acid treatment of the clay samples, the protons from the acid medium penetrate into the clay structures attacking the OH groups thereby causing the alteration in the adsorption bands attributed to the OH vibrations and octahedral cations [20].

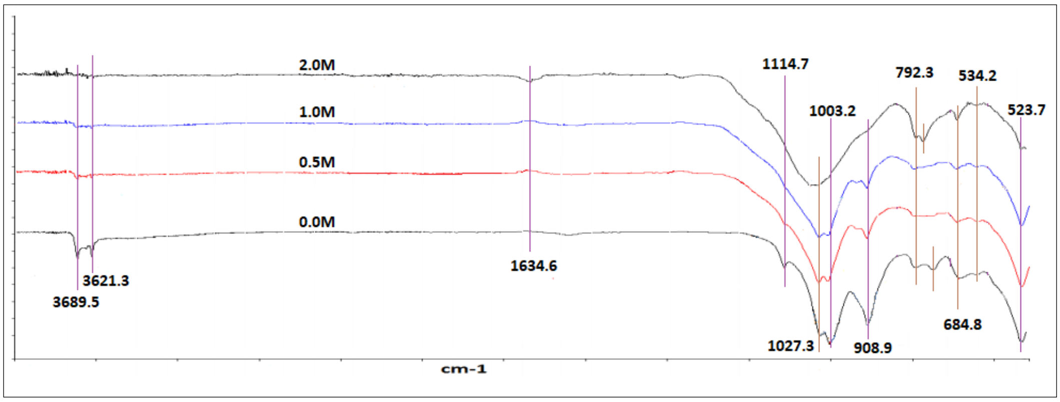


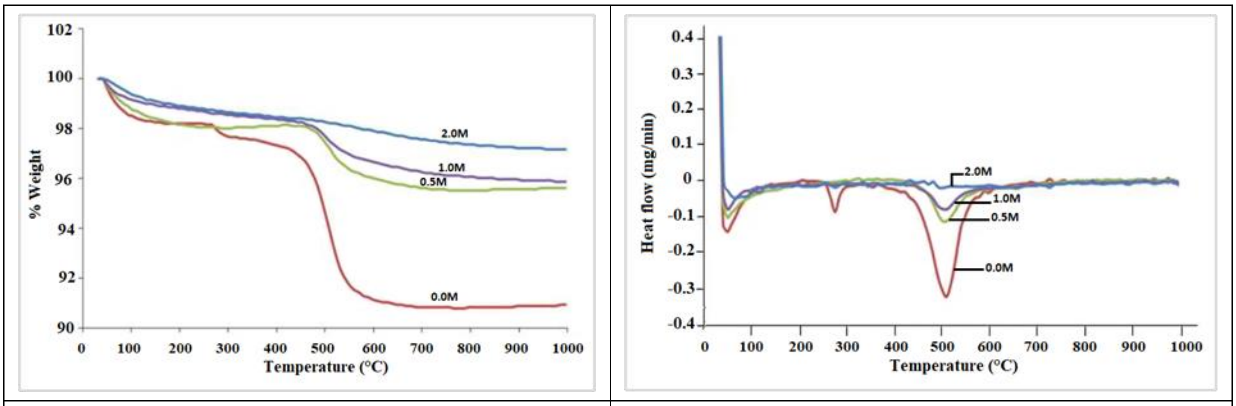
Figure 2 FTIR spectra of untreated and treated clay at different HCL concentration

Figure 2 illustrates the changes of untreated clay in the Si-O (at peaks 534, 684, 1003, 1027 and 1114 cm-1), Si-O-Al (at peak 792 cm-1), Al-Al-OH (at peak 908) and Al-OH (at peaks 3689 and 3621 cm-1) infrared vibration bands once treated with HCL under reflux conditions. The bands of the untreated clay become sharper after HCL treatment which gives the evidence that the kaolinite phase is more ordered after the acid treatment [16]. After treating with HCL, a new absorption band appeared at around 1621 cm-1 at 2M and increased as the acid strength increase. This peak is quite intense in which can be attributed to the bending vibration mode of physisorbed water on the surface of free silica produced due to the acid treatment.

In addition, another new peak was observed at 792 cm-1 for the acid treated sample and gained intensity with an increase in the acid strength. This new peak is due to the formation of free amorphous silica and/or quartz admixture [8]. Between 1200 cm-1 and 400 cm-1 bands corresponding silica was observed. The strong bands in the 1000-1120 cm-1 region are due to Si-O stretching in untreated Sayong kaolinite clay which changed in shape and position and finally diminished after 1M HCL treatment due to structural changes in the tetrahedral cations [17]. The bands 1114 cm-1 and 792 cm-1 were disappeared after the acid treatment whiles the band 684 cm-1 (Si-O) was increased as the acid concentration increase. The FTIR result is in clear agreement with the XRF study which indicates sequential degradation of the clay sheet upon acid treatment.

4.3. TGA-DTA Analysis

Figure 3 illustrates the TGA and DTA curves for untreated and treated Mayo-Kebi kaolinite clay samples. In general, clay materials contain three kinds of water molecules in their structure. The physisorbed and interlayer water is loosely bound and are mobile that they can be removed by heat treatment below 200˚C. The water molecule present in the first coordination sphere of the interlayer ions is strongly bonded and they require higher temperature in the range of 300-500 ˚C for their removal. Lastly the structural hydroxyl groups can condense and dehydrate in the temperature range of 500-800 ˚C. According to [19], the low temperature water loss can be assigned to the physisorbed water, whereas the high temperature weight loss is due to the dehydration and dehydroxylation of the clay sheet [1].



(a) (b)

Figure 3 TGA (a) and DTA (b) of untreated and treated Mayo-Kebi kaolinite clay

Comparing the TGA profiles in Figure 3a (for untreated and treated clay), it was observed that the acid treatment increased the amount of physisorbed water and it increased with increase in acid concentration. This is attributed to the fact that acid treatment increased the amount of amorphous silica and also surface area which made the water adsorption higher. This result is an agreement with XRF analysis. The DTA profiles of untreated and treated clay are represented in Figure 3b. The DTA curves of untreated clay showed three endothermic peaks. The strong endothermal peaks centered at 520˚C assigned to the loss of the structural water of the kaolinite caused by dehydroxylation of coordinated and structural water molecule. The other endothermic peaks centered at around 49˚C and 275˚C. When the sample treated with HCL, the physisorbed water increased as the acid strength increase. This effect is clearly related to the adsorbed water and the fact that the amount of amorphous silica increased in the clay which made the amount of adsorbed water higher during the acid treatment. In addition, the structural and coordinated water also decreased in treated samples at temperature above 275˚C.

4.4. ESEM Analysis

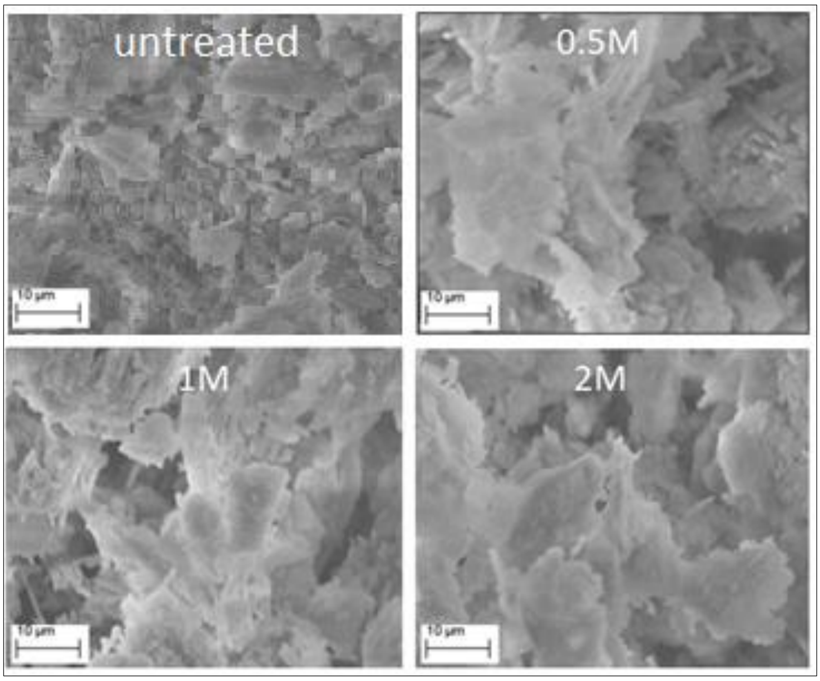


Figure 4 Micrographs of untreated and treated with 0.0M, 0.5M, 1M and 2M HCL at 5000x magnification

The morphological features of untreated and treated kaolinite clay were obtained by ESEM and shown in Figure 4. The micrograph of untreated clay shows the presence of a mixture of large plates that appeared to have been formed by several flaky particles stacked together in the form of agglomerates. The treated samples show the clay maintained some similarity of the structure with untreated clay. However, it was observed that the morphology of the treated clay showing a slight disorder in the plates and the appearance of some particles with fiber or needle shapes. Besides, the characteristic of the treated clay form of very porous and spongy silica particles, in agreement with the chemical composition and the surface area of the samples. The micrographs of 0.0M-2M (Figure 4b, c, d) treated clay indicate the disaggregation of the clay structure and the clay particles well-bonded aggregates rather than detached particles [8, 21].

5. Conclusion

In this work, the effect of hydrochloric acid on the Mayo-Kebi kaolinite clay has been successfully investigated. The XRF and ESEM study indicated clearly the leaching and disintegration of the clay sheet after treatment with acid. TGA profile of the parent and the acid treated clay shows that acid treatment increased the amount of physisorbed water and it increased with increased strength of the acid. FTIR spectrum of the acid treated kaolinite clay shows that there is not much variation in the peak pattern for acid treated kaolinite.

Compliance with ethical standards

Disclosure of conflict of interest

The authors declare no conflicts of interest regarding the publication of this paper.

References

[1] Olaremu, G., (2015). Physico-Chemical Characterization of Akoko Mined Kaolin Clay. Journal of Mineral and Materials Characterization and Engineering, 3(5), 353–361. doi:10.4236/jmmce.2015.35038

[2] Chen, X.-G., (2012). Effects of acid treatment on the physico-chemical and pore characteristics of halloysite. Colloids Surfaces a Physicochemical and Engineering Aspects, 396, 182–188. doi:10.1016/j.colsurfa.2011.12.067

[3] Kumar, S., Panda, A. K., and Singh, R. K. (2013). Preparation and Characterization of Acids and Alkali Treated Kaolin Clay. Bulletin of Chemical Reaction Engineering and Catalysis, 8(1), 61–69.

[4] Eze, K. A., Nwadiogbu, J. O., Nwankere, E. T., (2012). Effect of acid treatment on the physiochemical properties of Kaolin clay. Archives of Applied Science Research, 4(2): 792-794.

[5] Lomertwala, H. M., Njoroge, P. W., Opiyo, S. A., Ptoton, B. M., (2020). Effect of Acid Treatment on the Chemical Properties of Clays from Selected Sites in Murang'a and Nyeri counties, Kenya. Journal of Natural Sciences Research ISSN 2224-3186 (Paper) ISSN 2225-0921 (Online) Vol.11, No.22, 2020. doi:10.7176/JNSR/11-22-05

[6] Heller-Kallai, L., (2006). Developments in clay science: handbook of clay science, in: L. Heller-Kallai (Ed.), Thermally Modified Clay Minerals, Elsevier, Oxford.

[7] Lenarda, M., Storaro, L., Talon, A., Moretti, E., and Riello, P., (2007). Solid acid catalysts from clays: Preparation of mesoporous catalysts by chemical activation of metakaolin under acid conditions. Journal of Colloid and Interface Science, 311(2), 537–543. doi: 10.1016/j.jcis.2007.03.015

[8] Belver, C., Bañares Muñoz, M. A., and Vicente, M. A., (2002). Chemical Activation of a Kaolinite under Acid and Alkaline Conditions. Chemistry of Materials, 14(5), 2033–2043. doi:10.1021/cm0111736

[9] Foletto, E. L., Volzone, C., Porto, L.M., (2006). Clarification of cottonseed oil: how structural properties of treated bentonites by acid affect bleaching efficiency. Latin America Applied Research, 36: 37–40.

[10] Srasra, E., Trabelsi-Ayedi, M., (2000). Textural properties of acid activated glauconite. Applied Clay Science, 17: 71–84.

[11] Siddiqui, M.K.H., (1968). Bleaching Earths, 1st ed., Pergamon Press, London.

[12] Klamadji M. N., Dedzo M. G., Tchameni R., (2021). Fractional Crystallization and Crustal Contamination of Doleritic and Trachytic Dykes Crosscutting the Cretaceous Sedimentary Basins from Figuil (North Cameroon) and Léré (South-Western Chad): Geodynamic Implications. Journal of Geoscience and Environment Protection, Vol.9 No.12. DOI: 10.4236/gep.2021.912012

[13] ELF-SEREPCA (1981) Operational service, geological map of the Garoua basin (unpublished).

[14] Regnoult J.M., (1986). Geological synthesis of Cameroon, Yaoundé, Directorate of Mines and Geology of Cameroon.

[15] Roch E., Jeremine E., Faure and Muret A., (1953). Geological itinerary in Camroon en the southwest of the Chad territory, bulletin of thr mining service N°1.

[16] Schwoerer P., (1965).Reconnaissance geological map at a scale of 1:500 000 with notes on the Garoua Easth map, Yaounde, Directorate of mines and Geologiy of Cameroon.

[17] Muriithi, N. T., Karoki, K. B., Gachanja, A. N., (2012). Chemical and mineral analyses of Mwea Clays, International Journal of Physical Sciences, 17: 44.

[18] Panda, A. K., Mishra, B. G., Mishra, D. K., and Singh, R. K., (2010). Mineralogical, crystallographic and morphological characteristics of natural kaolins from the Ivory Coast (West Africa), Colloids and Surfaces: A Physicochemical Engineering Aspects 363, 98-104.

[19] Ajemba, R. O., and Onukwuli, O. D., (2012). Investigation of the effects of sulphuric acid modification on the structural and bleaching performance of Ukpor Clay, Journal of Basic and Applied Scientific Research 2 (9), 9438-9445.

[20] Ajemba, R. O., and Onukwuli, O. D., (2012). Studies of the effects of hydrochloric acid-leaching on the structural and adsorptive performance of Nteje (Nigeria) Clay, International Journal of Engineering Research and Technology 1 (3), 1-11.

[21] Mako, E.; Senkar, Z.; Kristof, J.; and Vagvolgyi, V., (2006). Surface modification of mechanochemically activated kaolinites by selective leaching, Journal of Colloid and Interface Science 294, 362-370.