ChemSearch Journal 8(2): 16 – 21, December, 2017
Publication of Chemical Society of Nigeria, Kano Chapter

Received: 30/05/2017 Accepted: 13/08/2017

Physicochemical Evaluation of Industrial Potentialities of Getso Kaolin

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ABSTRACT

Fossil fuel depletion has prompted the need for alternative sources of energy and income generation. Kaolin is an abundant solid mineral found in many parts of Nigeria, whose economy is over dependent on oil. However, lack of detailed study, characterization and analysis made it difficult for investors to show interest in this manufacturing sector. In this study kaolin from Getso, Kano state, northern part of Nigeria was characterized using pH measurement, X-Ray Fluorescence Analysis (XRF), Thermogravimetric-Derivative Thermal Analysis (TG-DTA), X-Ray Diffraction (XRD) and Fourier Transform Infra-Red Analysis (FTIR) to establish its potentialities for industrial applications. The XRF analysis indicated that Getso kaolin consists mainly of silica, 47.07% and alumina, 39.20% with other metal oxides like iron (III) oxide as minor component, while the XRD and FTIR results indicated that the main mineral present in the sample is kaolinite with small amounts of mica and quartz. The pH was found to be 7.2, which qualified it for very good industrial applications. The chemical composition of Getso kaolin was found to be comparable to the theoretical composition and that of commercial kaolin making it suitable for some industrial applications like paper, adhesives, plastics, fiberglass, rubber and paints.

Keywords: Getso Kaolin, Characterization, Theoretical kaolinite

INTRODUCTION

Nigerian economy is largely dependent on petroleum for revenue generation, neglecting other equally important resources like solid minerals and agriculture. The mining of minerals in Nigeria accounts for only 0.3% of its gross domestic product (GDP), due to the influence of vast oil resources. Globally, the mining industry has been a close rival to the petroleum industry yet, mining industry generates a paltry $89 million per annum for Nigeria (Online Nigeria Daily News, 2014).

Kaolin occurs in most of the countries in the world, however very few deposits are of good quality. The grade of kaolin will determine its price and suitable industrial applications. The properties considered include white colour, softness, small particle size and chemical inertness (Ekosse, 2010). Kaolin is a naturally occurring mineral of the clay family comprising largely of one of the kaolin group of minerals; halloysite, dictkite, nacrite and kaolinite. Kaolinite is however, the most common kaolin mineral with most versatile and wide industrial applications due to its physical and chemical properties, crystal structure, and surface chemistry (Prasad *et al.*, 1991).

Kaolin is commonly a complex mixture of different minerals composed of fine-grained and plate-like particles formed as a result of intense weathering or hydrothermal alteration of aluminosilicates that are found in feldspar-rich rock, like granite. The hard feldspar is converted into soft matrix found in kaolin pits through a process known as kaolinisation, while the quartz and mica of the granite remain relatively unchanged. Smectite may also form in small quantities in some deposits (Murray, 1991; Zegeye *et al.*, 2013; Xu *et al.*, 2015). Kaolin deposits can be sedimentary, residual, or hydrothermal leading to different properties for different kaolins, necessitating the need to fully test and evaluate it to determine its utilizations. The uses of kaolin depend on several factors including but not limited to the geological conditions under which the kaolin formed, the total mineralogical compositions of the kaolin deposits, and the physical and chemical properties (Murray, 2006).

World production of kaolin is dominated by United State, United Kingdom and Russia, with countries like Brazil, Czecholovakia, Germany, France and South Korea also producing in large scale (Murray, 2006). Approximately 50 million tons of clay materials valued at $1.1 billion are used annually in US, signifying the contribution it is making to the economy and welfare of the US (Murray, 1991).

Geological survey of Nigeria confirmed occurrence of kaolin in significant amount in several states of the country (Liew *et al.*, 2012). An estimated reserve of 3 billion tonnes of good kaolinitic clays has been identified in various states of Nigeria from more than 40 recorded kaolin deposit and occurrences (Ekosse, 2010). The industrial demands of kaolin in Nigeria was estimated to be over 360,000 metric tons annually, this is partly achieved from local production and foreign import, leaving a supply gap of over 250,000 metric tons annually (Murray, 2006). According to the research jointly conducted by Nigerian Mining Corporation and Gold and Based Metal Mines Limited, there is an estimated 4 million tons of kaolin deposit in Karaye, Gwarzo (Getso), Shanono, Tsanyawa axis of Kano state (Omobus Village, 2011).

Although kaolin is found abundantly in Nigeria yet detailed study, characterization and analysis of this mineral are still limited. This makes it difficult for the investors to show interest in this sector. Additionally, since different kaolin has different properties due to the influence of the factors that governed kaolin formation earlier mentioned, the primary focus of this study is to analyze the mineralogical and chemical composition of the kaolin obtained from Getso town in Kano state, Nigeria with the aim of finding suitable industrial applications for it through comparison with some known industrial kaolin. The characterization techniques include pH, XRF, TGA, XRD and FTIR analyses. It is expected that the findings of this study will encourage government and private investors to reconsider the current exploitation and application practices of kaolin deposits. Study is still underway on the physical and mechanical properties of Getso kaolin with the aim of further ascertaining the findings in this work and suggesting more applications.

MATERIALS AND METHODS

Description of Getso Town

Getso is small rural town with mainly farming population, located in Gwarzo local government area, Kano state, North-Western Nigeria. It is located on flat land at geographical coordinates with latitude 11^o 53’ 0” north and longitude 7^o 58’ 0” east.

Sampling and Sample Pretreatment

Samples were obtained by digging six pits uniformly distributed within the deposit at a distance of about 30 m apart using random sampling method. The pits were about 1m in diameter and up to 10 m deep. The samples were quartered into 1 kg specimens for laboratory analysis. After this the sample is known as Getso kaolin. The Getso kaolin sample was dried in a well-ventilated room, grounded and sieved through 200 mesh (75μ) sieve.

Characterization of Getso Kaolin

The ignition loss was determined by calcination of the kaolin sample at 600 ^oC for 3 hours. The pH was measured using EUTECH INSTRUMENT pH Tutor (Singapore). The chemical composition was determined using Energy Dispersive X-Ray Fluorescence Spectrometer NEXCG (USA). Structural phase analysis was carried out on a Bruker D8 having Siemens Diffractometer D5000 with Cu-Kα radiation (40 kV, 40 mA, λ = 1.5406Å) (USA). The TG-DTA analysis was carried out using Perkin Elmer Simultaneous Thermal Analyzer (STA 8000) (USA) in the temperature range of 50 ^oC to 1200 ^oC and heating rate of 10 ^oC/min. Perkin Elmer 1650 Infra-Red Spectrometer (USA) was used for FTIR analysis of samples in the range of 4000 cm^{-1} to 400 cm^{-1} (Manoharan *et al.*, 2012; Pan *et al.*, 2013; Yacob *et al.*, 2016).

RESULTS AND DISCUSSION

Combination of techniques such as TG-DTA, FTIR and XRD are suitable for the characterization of clays and their derivatives (Manoharan *et al.*, 2012). In the present work, the mentioned techniques in addition to XRF were used for the characterization of the Getso kaolin sample and the results obtained are as presented herein.

X-Ray Fluorescence (XRF)

The XRF analysis was carried out to estimate the chemical composition of the Getso kaolin, and the result is as presented in Table 1. It consists mainly of silica and alumina, with metallic oxides like Fe_2O_3 and TiO_2 occurring as minor. The loss on ignition was 11.62% which is as a result of organic matter lost and/or some non-metals like sulphur which could have been removed from the clay in the form of SO_2 (Eze *et al.*, 2012).

Table 1 Chemical Composition of Getso Kaolin

Compounds	Composition (%)
SiO_2	47.07
Al_2O_3	39.20
Fe_2O_3	1.27
K_2O	0.74
MnO_2	0.060
TiO_2	0.054
CaO	0.046
LOI	11.62

Thermogravimetric-Derivative Thermal Analysis (TG-DTA)

Five main changes are normally observed from TG-DTA curves of kaolin; i) release of absorbed water in pores or on the surface, at temperature below 100 ^{o}C, ii) pre-dehydration process as a result of weight loss due to the reorganization in the octahedral layer occurring at the -OH of the surface, between 100 to 400 ^{o}C, iii) formation of metakaolinite as a result of dehydroxylation of kaolinite, which takes place between 400 and 650 ^{o}C, iv) decomposition of alunite, at 500 to 900 ^{o}C and v) formation of mullite indicated by an exothermic peak around 1000 ^{o}C (Cristóbal *et al.*, 2010; Mohsen, 2010; Panda *et al.*, 2010; Ptáček *et al.*, 2010; Vizcayno *et al.*, 2010; Manoharan *et al.*, 2012).

The TG-DTA curve of Getso kaolin presented in Figure 1 is similar to a typical kaolinite (Yang *et al.*, 2010). The DTA curve exhibited two endothermic peaks at 80.53 ^{o}C and 526.52 ^{o}C due to the removal of absorbed water and dehydroxylation of kaolinite, respectively. The formation of metakaolinite is according to equation (1);

$$Al_2Si_2O_5(OH)_4 \longrightarrow Al_2Si_2O_7 + 2H_2O \qquad (1)$$

And the third peak is an exothermic peak around 1200 ^{o}C due to the formation of α-Al_2O_3 or nucleation of mullite.

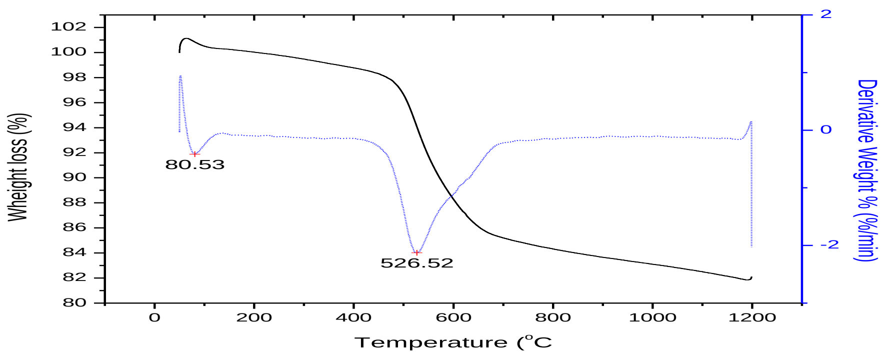

Figure 1: TG-DTA curve of Getso kaolin

X-Ray Diffraction (XRD)

The characteristic peaks of the Getso kaolin sample from the diffractogram in Figure 2 were ascribed to kaolinite [$Al_2Si_2O_5(OH)_4$] (JCPDS Card no. 58-2005), the peaks were associated with the triclinic structure of kaolin (a = 5.15560, b = 8.93970 and c = 7.40730). The peaks appeared at 2θ positions of 12, 20, 25, 27, 35, 38, 45, 55, 62, 70, 72, 73 and 77^{o}. The typical characteristic peaks of kaolinite were the well-defined reflections at 2θ value of 12^{o} and 25^{o}, while the other peaks corresponding to the 2θ value of 34-36^{o}, 38-42^{o}, 45-50^{o}, and 54-63^{o} differ for different kaolinites (Panda *et al.*, 2010). The XRD result corroborates the observation from TG-DTA analysis indicating the sample under study is kaolin. The crystallite size was calculated using two most intense peaks at 12^{o} and 25^{o}, it was found to be 10.36 and 10.69 nm respectively, with an average of 10.53 nm.

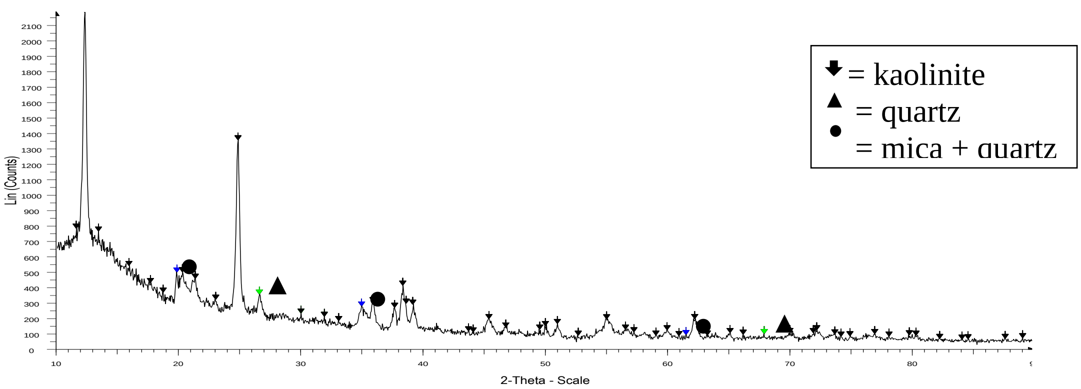

Figure 2: XRD pattern of Getso kaolin

Fourier Transform Infra-Red (FTIR)

Figure 3 depicts the Infra-Red spectra of the Getso kaolin, the sample exhibits the following bands that are typical of kaolinites: 3694, 3654, 3620, 1106, 1008, 913, 794, 754, 694, 537, 469 and 430 cm^{-1}. The bands at 3694, 3654 and 3620 cm^{-1} were due to Al-OH stretching vibrations indicating the presence of mica; the strong band at 3694 cm^{-1}

was related to in phase symmetric stretching, the weak band at 3654 cm^{-1} was assigned to out-of-plane stretching vibration, and the band at 3620 cm^{-1} was attributed to the inner hydroxyl groups lying between the tetrahedral and octahedral sheets. Meanwhile, the bands around 3440 and 1640 cm^{-1} were due to physisorbed water on the surface of the clay and the band at 913 cm^{-1} was assigned to the Al-O-H bending vibration. The presence of quartz can be explained by double peaks at 794 and 754 cm^{-1} due Si-O-Si symmetrical stretching vibrations. Additionally, the bands around 694 and 469 cm^{-1} were assigned to Si-O symmetrical bending vibration, and the band at 537 cm^{-1} was assigned to $Al^{(VI)}$-O-Si present in kaolinite. The final bands at 2926 and 2855 cm^{-1} were due to the C-H stretching vibrations from organic matter (Cristóbal *et al.*, 2010; Vizcayno *et al.*, 2010; Manoharan *et al.*, 2012; Fitos *et al.*, 2015). The appearance of peaks due to mica and quartz in the FTIR spectra is supported by the observation of these peaks in the XRD diffractogram of the Getso kaolin. Furthermore, the FTIR analysis confirmed the TGA and XRD results that the sample is kaolin.

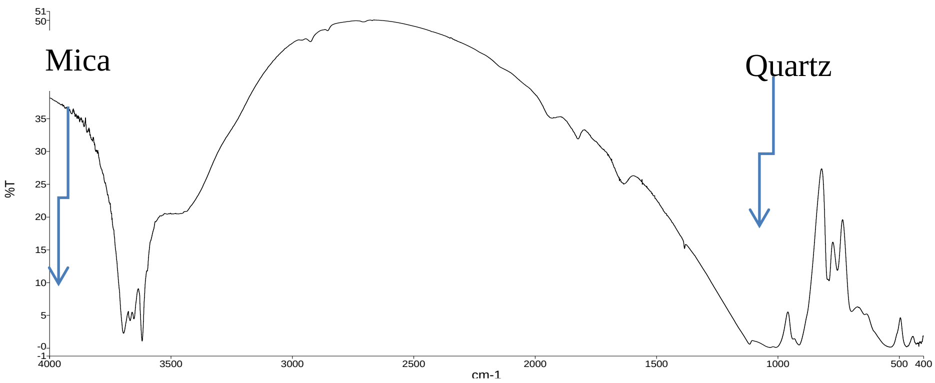

Figure 3: FTIR spectrum of Getso kaolin

Suggestive Industrial Applications of Getso Kaolin

Nigerian kaolin is reported to have been widely used for the manufacture of bricks, paints, refractories and ceramics, and sold locally or exported to the neighbouring countries, even though it could have promising applications in several clay-based industries. The major uses of kaolin are directly related to its physical and chemical properties (Ekosse, 2010).

The basic requirements of kaolin for most industrial applications is to have a chemical composition that is comparatively closer to its theoretical value, however obtaining pure kaolin in nature is very difficult as it is usually associated with other minerals (Ekosse, 2010). To trace its grade and probable industrial applications, the chemical composition of Getso kaolin is compared with that of a theoretical kaolinite and commercial kaolin including two well-known commercial kaolins from Cornwell deposits in the UK; English China Clay (ECC) and Sekeharman Kaolin Deposit (SKD) as shown in Table 2. ECC is used in paper, paint, pharmaceutical, cosmetics and other high quality industrial applications and SKD is used for sanitary ware and silico aluminous refractory ceramics (Konta, 1999).

As reported in Table 2, Getso kaolin is remarkably close in composition to theoretical kaolin. The major chemical variations are in the iron and potassium values. This can be connected to the fact that Getso kaolin is primary and also contained mica and quartz. Iron can range from as low as 0.2% to as high as 1.0% and titania, from as low as 1% to as high as 2.2% and still make a good quality product (Murray, 1991). The composition of Getso kaolin is equally close to that of ECC kaolin, differing only in iron content. This further qualifies the suitability of this kaolin for high quality industrial applications.

Table 2: Comparison of the elemental composition of Getso kaolin with theoretical and commercial kaolin.

Compounds	Chemical Composition (%)				Composition for Industrial Application (%)			
	Getso kaolin	ECC	SKD	Theoretical kaolinite	Paper Coating	Paper Filler	Ceramics	Pharmaceutics & Cosmetics
SiO_2	47.07	47	50	46.3	45-47	46-48	48-50	44.6-46.4
Al_2O_3	39.20	38	32.9	39.8	37-38	37-38	36-37	38.1-39.5
Fe_2O_3	1.27	0.39	1.2	-	0.5-1.0	0.5-1.0	0.6-1.0	0.1-0.2
CaO	0.046	0.03	0.2	-	-	-	-	0.1-0.2
Na_2O	-	0.15	0.2	-	-	-	-	0-0.1
K_2O	0.74	0.8	1.6	-	-	-	1.2-2.7	0-0.2
TiO_2	0.054	0.03	1	-	0.5-1.3	0.04-1.5	0.02-0.1	0-1.4
LOI	11.62	13	12.6	13.9	13.9-14.3	12.3-13.7	11.2-12.5	13.8-13.9

Source: Nkoumbou *et al.*, 2009; Fitos *et al.*, 2015

Paper industry is the largest consumer of kaolin, filler and coating clay are the two basic type of clays used in paper industry. Chemical composition of the Getso kaolin is very close to kaolin specification for paper industry, except for the Fe_2O_3 content, which can be beneficiated using chemical or magnetic separation followed by delamination. The pH of coating clays is in the range of 6.5-7.5, and the pH of the Getso kaolin was found to be 7.2, qualifying it to suitable for industrial applications. High pH values indicated the presence of soluble salts and this hindered certain industrial applications (Murray, 2006). Getso kaolin can also serve as a raw material for manufacture of ceramics, as can be seen from its chemical composition in Table 2. Currently other properties of the kaolin are been tested including firing characteristics to support this suggestion.

Another suggestive industrial application of Getso kaolin is as filler in the plastics, adhesives and fiberglass industries. The characteristic requirements of perfect filler in these manufacturing sectors are low cost, good availability, low oil absorption, small and uniform particle size, good dispersion, low density, good chemical resistance, light colour and low free-moisture levels (Murray, 2006).

Other important areas that Getso kaolin might find application are rubber and paint industries. It can also be used for the production of alumina and synthetic zeolite, as it has high composition of aluminium and silica as can be seen from Table 1. Based on this quality Getso kaolin was used in the synthesis of mesoporous alumina and the results are published elsewhere (Yacob *et al.,* 2016; Bello *et al.,* 2017). A relatively small end-use of kaolin is in the agricultural industry for the production of fertilizer, pesticides and animal feeds this is connected with the flat particle shape that helps to improve adhesion of the pesticide to the sprayed plant. Generally all the marketed kaolin worldwide were obtained after processing of low grade material through screening and separation of iron and titanium minerals (Murray, 2006).

CONCLUSION

The present research characterized Getso kaolin and suggests probable industrial applications. The XRF analysis revealed the main chemical composition as silica (47.07%) and alumina (39.2%) with other metal oxides including iron (III) oxide as minor components. From the XRD and FTIR results, the main mineral present in Getso kaolin is kaolinite with small amounts of mica and quartz, while the pH was found to be at 7.2. The findings of the research qualified Getso kaolin as high grade with composition comparable to the theoretical kaolinite. Based on these findings, the research has suggested suitable industrial applications for Getso kaolin in the paper, adhesive, plastic, fiberglass, rubber, and paint industries among others. Furthermore as most of the inhabitants of Getso are farmers, agricultural industry will be very suitable for consumption of the kaolin.

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