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Characterization of Nanostructure Sedimentary Clay Material of Netrokona District

T. M. S. A. Hossain[1]*, M. Hasanuzzam[2]

[1]Department of Mechatronics Engineering (MTE), World University of Bangladesh, Dhaka-1230

[2]Department of Nanomaterials and Ceramic Engineering (NCE), Bangladesh University of Engineering and Technology (BUET), Dhaka-1000, Bangladesh

ABSTRACT

Clay is an essential industrial mineral that often needs improvement for applications. Due to the vast demand in the ceramic industry, nanostructure clay materials can be modified to use as advanced materials such as porous filtration, heat resistance insulators, and other applications. The excellent alternating layered crystal sheet structure of clay minerals allows for comprehensively modifying its properties. In this research, local clay was chosen as potential material and its properties are thoroughly investigated. Chemical composition analysis by X-ray fluorescence revealed that the clay contains Al2O3 (26.67 %) and SiO2 (66.32 %) as major oxides and impurity oxides include mainly Fe2O3 (2.01%) and TiO2 (2.47 %) with the acceptable amount. A mineralogical study employing X-ray diffraction (XRD) exhibited mainly kaolinite and quartz phases with minor contents of illite and rutile phases. Field Emission Scanning Electron Microscopy (FE-SEM) displayed the morphological features of clay mineral structure. The particle size of kaolinite platelets varied from < 2 μm. In addition, the particle shape of clay structure was also discerned with size spans of 50−500 nm. EDX analysis also gives the elemental analysis of the nanostructure sheet. The local sedimentary clay resembled white clay-type characteristics though XRD contradicts the well-crystalline structure of kaolinite. Differential thermal analysis (DSC-TG) confirmed phase transition occurs with increasing temperature. The obtained results from various characterization techniques provide an in-depth understanding of its fundamental characteristics to suit it for applications.

KEYWORDS: Nanostructure Materials, Kaolinite, Advanced Applications, Minerals, Microstructure and Sedimentary clay

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1. INTRODUCTION

Clay nanostructured materials are a group of hydrous aluminum silicates with a repeating layered structure that has considerable demand as advanced materials in diversified applications. It is the expression of very small, crystalline particles dominated by planar arrays of [SiO4]4- structural units and many structural hydroxyls and water of the clay minerals, kaolinite has shown profound industrial applications such as ceramic raw materials, filler pigment, and coating for paper, filler for paint, formulation of medicine/cosmetics and catalyst manufacture. Kaolinite is composed mainly of silica sheets [Si2O5]2- linked to modified gibbsite sheets [Al2 (OH) 4]2- [1]. The silica sheet is called tetrahedral layer due to the tetrahedral shape of the [SiO4]4- group (each side is 0.26 nm). The oxygens/hydroxyls are arranged to form the corners of octahedra (each side is 0.29 nm) having six corners and eight faces in the gibbsite sheet. In the atomic lattices of kaolinite, one unit length (basal spacing) is 0.72 nm. In kaolinite, a gibbsite sheet is constructed directly over a silica sheet in such a manner that one in three of the

OH radicals is replaced by the unsaturated apical oxygens of the silica sheet. The apical oxygens thus form a bridge between the two sheets resulting in a composite unit layer of kaolinite, Al2Si2O5(OH)4. An outstanding feature of kaolinite is that in the gibbsite sheet only two out of three plausible sites are occupied by Al3+ keeping one vacant and the resulting structure is called dioctahedral. A kaolin crystal consists of many composite layers (typically 70 to 100 layers). The entire crystal is united by hydrogen bonds between OH- radicals of the dioctahedral layer and oxygen atoms of the adjacent tetrahedral layer. In kaolinite, the layers are stacked so that the atoms in one silicon layer are displaced regularly along the a-axis. The unit cell is triclinic with

α= 91.80, β= 104.50, γ= 90 and a = 5.16 Å, b = 8.94 Å and c = 7.37 Å [2].

Clays are broadly classified into two types: a) residual clay and 2) sedimentary clay. Residual clays (commonly known as China clays) are located near source-altered igneous rocks and are obtained in the purest state of the mineral kaolinite. In general, the mineralogical composition shows 80-95 % kaolinite and 5-15 % mica (muscovite). The remainder is quartz, paragonite, and a trace amount of montmorillonite, different oxides of magnesium, calcium, and iron. The content of silica and alumina is nearly 46.5 % and 39.5 %, respectively. The iron content (as Fe_2O_3) is between 0.5-1.2 % and the alkalis are less than 2 %. In general, the total impurities of China clay amount to about 5-15 %. A typical composition of China clay is SiO_2 (46 %), Al_2O_3 (38 %), Fe_2O_3 (0.3 %), TiO_2 (0.03 %), CaO (0.6 %), MgO (0.6 %), Na_2O (0.15 %), K_2O (0.5 %). In contrast, sedimentary clays are transported by natural agencies and thus impurities are picked up and retained during their transportation. Fine impurities present are sometimes difficult to remove. The main sedimentary clays are ball clay, fire clay, and brick clay. The chief mineral of sedimentary ball clay is disordered kaolinite with main impurities as quartz and mica (illite) and minor impurities as oxides of iron and titanium and organic matter. Various deposits of clays were identified by the Geological Survey of Bangladesh (GSB) namely Bijoypur in Maymenshing, Maddhyapara and Barapukuria in Dinajpur, Patia in Chittagong, and Sylhet [3]. However, no systematic complete study has been carried out to characterize these deposits for their suitability in applications as advanced materials such as porous fiber, structural heat insulation, etc. In this context, a detailed cutting-edge-technology-based characterization and analysis were conducted to probe the composition, mineralogical test, crystallinity, phase changes, and morphology (size and shape) of kaolinite nanoparticles.

2. EXPERIMENTAL

The Geological Survey of Bangladesh (GSB) indicated that Bangladesh has deposits of sedimentary types of clays [4]. Based on this report, several deposits across the country have been identified as the sources of various types of clay. Important deposits are Bijoypur in Mymensingh, Barapukuria and Maddhyapara in Dinajpur. In this research work, Durgapur (Bijoy-07) clay collected from the Netrokona district which is commonly known as Durgapur (Bijoy-07) clay in that area of deposits is used for characterization and beneficiation. Durgapur clay is of high quality and is used predominantly by the ceramic, paper, and rubber industry industries of the country. These regions are totally sediment areas. The clay deposits are found in different riverside areas. Generally, the clay is light greyish white to bluish white with light yellow and slightly soapy to feel, massive, and soft to medium hard. The color of Durgapur (Bijoy-07) clay was found to be whitish yellow. The clay was collected in the necessary amount and prepared for the characterization technique. The aim of the present research is the investigation the properties of clay and its thermal behavior. The identification and quantification of natural clay were conducted by Field Emission Scanning Electron Microscopy (FESEM), X-ray Fluorescence Spectroscopy (XRF), and X-ray Diffractometry (XRD). Clay minerals often adopt the form of a mixed-layer structure with various ratios of individual components. Thus, IR spectra and Differential Scanning Calorimetry (DSC)/Thermogravimetric (TG) analysis were also conducted.

3. RESULT AND DISCUSSIONS

In this research to expose Durgapur clay as a prospective advanced material, a detailed analysis was conducted employing several experimental tools. The morphology of clay was determined by employing Field Emission Scanning Electron Microscopy. X-ray fluorescence was used for the compositional analysis of clay materials. X-ray diffraction will lead to an understanding of the structural characteristics of clay minerals. Thermal behavior and weight loss of clay samples were studied by Differential Scanning Calorimetry to obtain information on phase changes. Obtained results are presented below under each technique.

3.1 Optical Microscopy

The theoretical resolution of optical microscopy is about 0.2 □m. The length scale of clay particles is below this value thereby rendering limited information from optical microscopy of clays. However, the overall size (and shape) of clay aggregates and their color can valuably be discerned from it. Fig. 1 shows two clay chunks, one of which is broken into two parts. The color of the clay was found to be whitish. A tint of iron-rich mineral has shown to exist along with this clay.



Fig. 1. Optical image of Bijoy-07 natural clay.

3.2 X-ray Fluorescence Analysis

To determine the percentage of oxides present, X-ray fluorescence (XRF) was employed as a widely used versatile tool for elemental and chemical analysis of clays. XRF emits characteristic secondary X-rays from a material that has been excited by bombarding with high- energy X-rays.

Table 1 shows the composition (major) of (Bijoy-07) clay. The clay is Al2O3 rich than other natural clays obtained from different areas.

TABLE 1: COMPOSITIONAL ANALYSIS OF CLAY

Oxide	SiO2	Al2O3	Fe2O3	TiO2
Weight (%)	66.32	26.67	2.01	2.47

The clay also contains a small amount of CaO (0.15 %), MgO (0.38 %), ZrO2 (0.23 %), P2O5 (0.06 %), Cr2O5 (0.05 %), and K2O (1.56 %). The presence of iron and titanium oxide bearing materials may impair the usefulness of clay [5].

3.3 X-ray Diffraction Analysis

To quantitatively analyze minerals, present in natural clays, X-ray diffraction (XRD) is an indispensable tool. The high-intensity X-ray beam (PAN ANALYTICAL EMPYREAN) was diffracted on clays within a scanning range (2□) from 10° to 70° with CuK□ □radiation. Fig. 2 presents an XRD pattern of natural clay. Kaolinite and quartz are found to be major mineral constituents of clay. Rutile was detected as the main titanium-bearing impurity along with a small amount of llite.

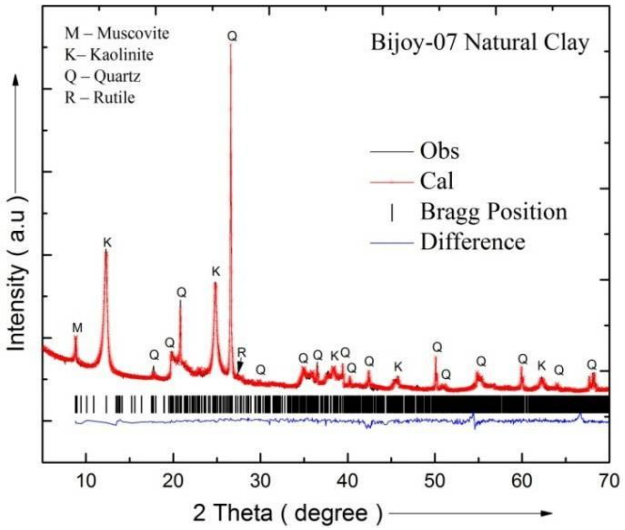

Fig. 2.XRD pattern of Bijoy-07 natural clay.

Obtained two strong reflections are well matched with the standard prominent basal reflections at 12.39° and 24.93°. The peaks were found to be sharp representing well-crystalline kaolinite [10-11]. To determine the quantitative percentage of these minerals Rietveld refinement was conducted using HIGH SCORE PLUS Software. Table 2 shows the quantitative mineralogical analysis of

major minerals. In addition to this list, calcium sodium aluminum silicate and trimagnesium dioxide dihydroxide were also detected as 4.6 % and 0.7 %, respectively.

TABLE 2: RIETVELT ANALYSIS

Minerals	Kaolinite	Quartz	Illite	Muscovite
Wt (%)	46.0	23.4	21.6	3.7

3.4 Secondary Electron Microscopy Analysis

To reveal the approximate size and shape of clay particles, Scanning Electron Microscopy (SEM) can be employed with its ability to obtain a very high-resolution and magnified image of individual grains of clay minerals. In this context, Field Emission Scanning Electron Microscopy (FESEM) (Model: JEOL JSM-7600F) was used.

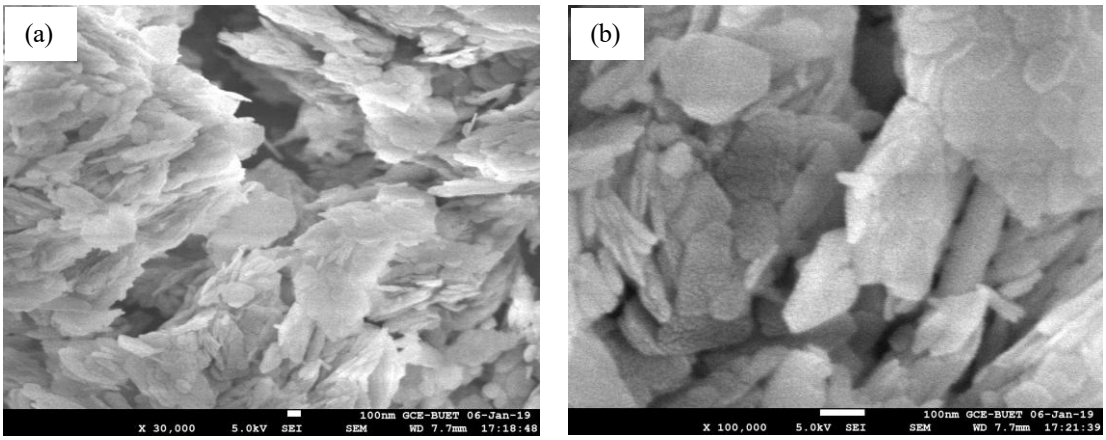

Fig. 3. SEM micrographs of Bijoy-07 natural clay showing: (a) kaolinite hexagonal sheet plates and (b) kaolinite particles stacking.

Samples were scanned for secondary electrons (SE) mode for morphological contrast and backscattered electrons (BSE) mode for phase contrast [9]. Fig. 3 shows the FESEM micrographs of nanoparticles of natural clay. High-magnified images revealed both plate (Fig. 3a) and hexagonal-shaped stacking (Fig. 3b) clay particles.

3.5 Energy Dispersive X-ray (EDX) Analysis

Energy Dispersive X-ray (EDX) can provide valuable information on major and minor elements allowing highly reliable compositional identification [6].

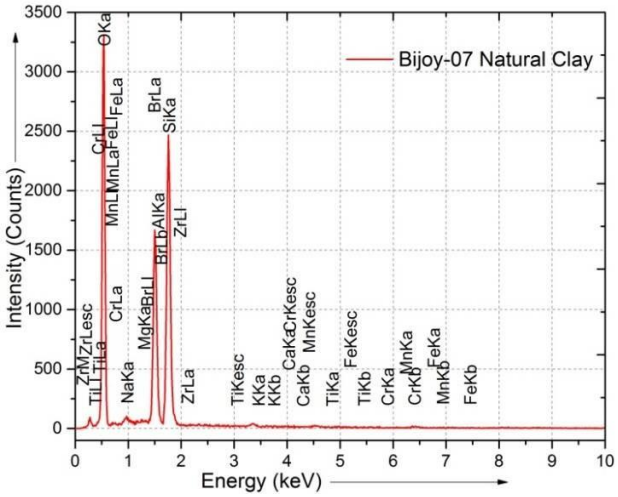

Fig. 4. EDX spectra of Bijoy-07 natural clay.

EDX is used for qualitative as well as quantitative (the percentage of the concentration of each element of the sample) analysis. Fig. 4 shows captured EDX spectra from the clay sample. Peaks of major elements namely Si and Al with allied oxygen were identified. Table 3 shows a summary of the elemental analysis of clay in atomic (and mass) percentage.

Al and Si contents were found to be higher in conformity with the compositional analysis of XRF (Table 1). Except for detected minor elements namely Mg (K), K (K), Ti (K), Cr (K), and Mn(K), the concentration of major elements is only presented. In

analyzed clay, the ratio of Al to Si is lower by around half. In addition, some minor elements namely Ti, Ca, Mg, Mn, Fe, K, Fe, and Cr are also identified in the clay sample.

TABLE 3: EDX ELEMENTAL ANALYSIS OF NATURAL CLAY

Element	O(K)	Al(K)	Si(K)	Fe(K)
Mass (%)	39.31	19.98	38.34	6.40
Atom (%)	53.26	16.05	29.59	0.40

3.6 FTIR Analysis

The structural differences of clay can be ascertained by vibration spectroscopic investigations yielding useful information about hydration characteristics, interlayer cations, and moisture content in clays. Fig. 5 shows infrared spectra of the typically associated minerals present in clays. The band at 533 cm−1 shows deformation vibration of Si–O and Si–O–Al whereas the band at 469 cm−1 indicates the presence of amorphous silica

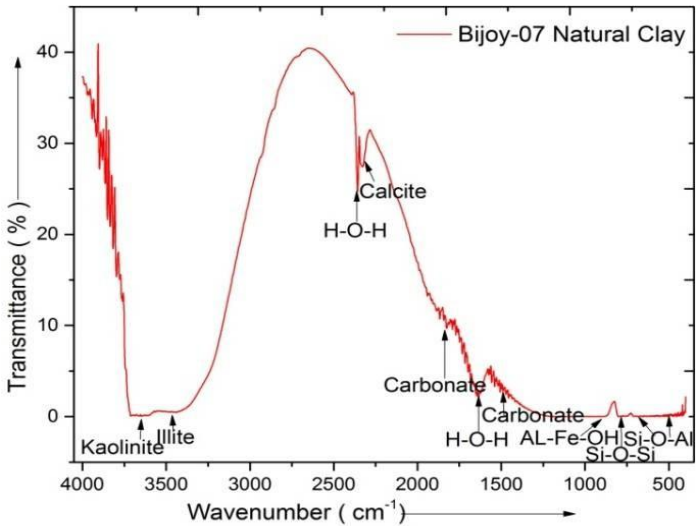

Fig. 5. FTIR spectrum of Bijoy-07 natural clay.

Bands observed at 780-798 cm-1 is due to Si-O-Si inter tetrahedral bridging bonds in SiO2. Bands at 1620 and 2642 cm–1 could be assigned to H-O-H bending of water, which is observed in almost all the natural hydrous silicates like illite minerals. The absorption bands observed at 1813 cm-1 is related to carbonate and at around 2358 cm-1 related to calcite. The bands between 3450 and 3670 cm–1 is attributed to the OH stretching mode [7-8]. The band at 3620 cm-1 may be ascribed to the inner hydroxyls and the other three characteristic bands are generally ascribed to vibrations of the external hydroxyls.

3.7 Differential Scanning Calorimetric (DSC) Analysis

Differential thermal analysis is an important method to detect reactions due to the dehydroxylation of clay minerals, decomposition of carbonates, loss of combined water, loss of sulfur, and decomposition of organic matter [12-13].

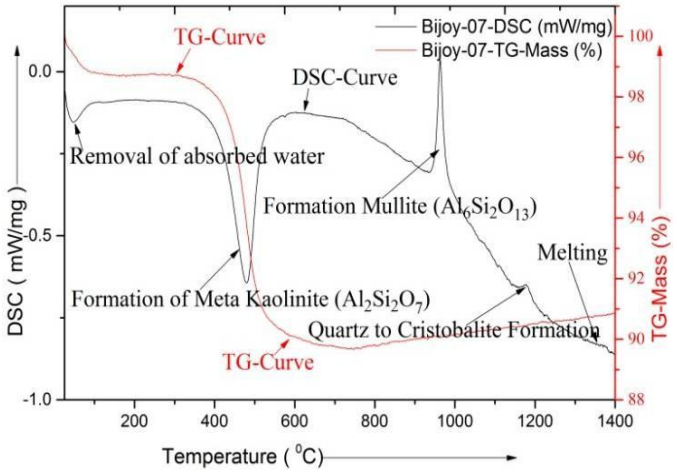

Fig. 6. DSC and TG curves ofBijoy-07 natural clay.

Fig. 6 shows DSC and TG curves of clay analyzed at room temperature to 1400oC depicting the effect of energy changes (endothermic or exothermic reactions) and weight changes in the sample. A first endothermic reaction was observed at a temperature below 100oC indicating desorption of surface H2O (e.g., H2O on exterior surfaces) and dehydration (e.g., interlayer H2O) at low temperatures. Kaolinite shows a weight loss starting at just above 400oC due to dehydroxylation, extending to about 650oC (TG-curve of Fig. 6). A significantly high endothermic peak observed at 456oC is related to the formation of meta-kaolinite (Al2Si2O7). Quartz transformation takes place at 573oC. An exothermic peak that appeared at 1000oC is related to the formation of the mullite (Al6Si2O13) phase. An allotropic phase transition from quartz to cristobalite was detected by a small exothermic peak at 1200oC.

4. CONCLUSION

The sedimentary deposit of local clay was characterized in this study. The investigated clay contains silica and alumina as predominant oxides. Iron, titanium, zirconium, calcium, potassium, and magnesium oxides are present as minor constituents. The major minerals include kaolinite, quartz, and illite with a trace of rutile mineral. The clay sample contains some key impurities namely iron and titanium oxides that may be removed by magnetic separation technique. The characteristic four bands of kaolinite were confirmed from IR spectroscopy. The nanosized kaolinite sheet plate and particles would allow this material to suit its properties for applications as an advanced material for porous ceramic filters and structural heat insulators.

5. ACKNOWLEDGMENT

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