PHYSICOCHEMICAL CHARACTERIZATION OF NATURAL KAOLIN FROM JABOI INDONESIA

R. Dewi^{1,3}, H. Agusnar^{2,*}, Z. Alfian² and Tamrin²

¹Postgraduate School, Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Sumatera Utara, Medan, 20155, Indonesia

²Department of Chemistry, Faculty of Mathematics and Natural Sciences, Universitas Sumatera Utara, Medan, 20155, Indonesia

³Chemical Engineering Department, Lhokseumawe State Polytechnics, Lhokseumawe, Indonesia

*E-mail: harryagusnar@yahoo.com

ABSTRACT

In this study studied the physical and chemical properties of natural kaolin originating from Jaboi village, Sukajaya district, Sabang City, Aceh province, Indonesia. Natural kaolin deposits in this province are quite large and can be used as raw materials in various industries such as paints, ceramics, rubber medicines and others. Therefore, the study of the physical and chemical properties of natural kaolin through characteristic tests is very necessary so that its utilization in related industries can be optimized. The characteristic tests carried out include XRF, XRD, FTIR, SEM EDX and DSC/TGA where the results of the analysis obtained will be compared with the characteristics of commercial kaolin. Based on the XRF test, the main composition of Jaboi kaolin was obtained as follows: SiO₂ (silica) and Al₂O₃ (alumina) respectively at 84.8% and 2.96%, while the remaining impurities with concentrations below 1%. XRD diffractograms identify quartz as the main mineral followed by kaolinite and cristobalite. These results are in accordance with the functional groups of FTIR spectra. The SEM test obtained the typical morphology of Jaboi kaolin, in the form of a heterogeneous group of layered hexagonal sheets and EDX results showing the composition of Jaboi kaolin elements consisting of 31.94% silicon, 1.33% aluminum 14.61% carbon and 50.78% oxygen. Based on the derivatogram in thermal analysis, there is no visible crystallization of the Jaboi kaolin. The results of all the characterizations of Jaboi kaolin show that Jaboi kaolin is very suitable for use in the refractory and ceramics industries.

Keywords: DSC/TGA, FTIR, Jaboi, Kaolin, Physicochemical, XRF, SEM EDX, XRD.

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INTRODUCTION

Kaolin is composed of the mineral kaolinite as the main constituent which has the empirical formula Al₂Si₂O₅(OH)₄ and is often called China clay.¹⁻² Natural kaolinite is a long-term chemical weathering product of aluminosilicate rocks. The process of forming kaolinite occurs due to weathering and alteration of hydrothermal processes in many igneous rocks containing feldspar, where aluminum silicate and feldspar potassium minerals are converted into kaolinite.^{3,4} Kaolinite has a 1:1 layer structure where each layer consists of tetrahedral sheets (SiO₄) and octahedral sheets containing Al³⁺ ions.⁵⁻⁶ The kaolinite particles have about 50 silicate layers, where the layers are bound together by the Van der Waals force and hydrogen bonds.⁷ As with other clay minerals, kaolinite having a particle size of the colloid group is < 2 μm.⁸ The chemical-physical properties of kaolin depend on the geological and climatic conditions in which kaolin deposits are formed.⁹⁻¹⁰

Jaboi kaolin as the name suggests originates from the Jaboi area of Sukajaya District, Sabang, Weh Island, Aceh Province. The kaolin deposit in this Aceh province is very large reaching 450 million tons and has not been utilized¹¹. The location of kaolin is located at the foot of Jaboi mountain which is ± 100 m from geothermal sources (Fig.-2). Research on the use of Jaboi kaolin has been done by several

researchers^{11,12}, but the studies were not studied the physical and chemical properties of Jaboi kaolin overall and utilization of natural kaolin is limited only as a source of alumina and as adsorbent. Researchers¹¹ only mentioned the composition of Jaboi kaolin from XRF analysis at one sampling point and did not include the results of other characterizations. The same thing was also reported by other researchers¹², where both researchers only reported the use of Jaboi kaolin with the results of chemical and physical characterization is very limited. Optimizing the use of Jaboi kaolin in the industry needs to be carried out research on the quality and characteristics of Jaboi kaolin to be suitable and can be used in certain industries.

Fig.-1: Stages of Kaolinite Formation from Feldspar ⁴

Fig.-2: Geological map of the Weh Island.¹³

This study aims to examine the physical-chemical properties and determine the Jaboi kaolin mineralogy in Suka Jaya sub-district in an effort to see the compatibility of this natural kaolin as a raw material in the industry.

EXPERIMENTAL

Material and Methods

In this study the sample to be tested was natural kaolin from the Jaboi area and as a comparison used commercial kaolin purchased from one of the chemical distributors in the city of Medan. The characterization test for kaolin was carried out using several instruments: XRF (PanAlytical Serie Axios,

Philips), XRD (Shimadzu XRD-6000), FTIR (Shidmazu IR Prestige-21), SEM-EDX (Carl Zeiss-Bruker, Type EVO MA10) and DSC / TGA (Shidmazu, DSC/TGA- 60).

General Procedure

The natural kaolin used came from the Jaboi area, Sukajaya sub-district, Sabang city, Aceh Province, Indonesia. This area is located on 95°12'00"E - 95°23'00"E and 05°46'00"N- 05°55'00"N, about 15 km south of Sabang city. Kaolin is taken at three sampling points with a depth of 25 cm below the soil surface. Kaolin is crushed and sieved to obtain a uniform size using a 100 mesh sieve and dried in an oven at 105°C for about 2 hours. Commercial kaolin that is used as a comparison is also carried out the same treatment. Furthermore, the characterization of Jaboi kaolin is done using XRF, XRD, FTIR, SEM EDX and DSC / TGA.

Detection Method

Jaboi kaolin and commercial kaolin compositions were determined using X-ray fluorescence (XRF) machines (PanAlytical Serie Axios, Philips). X-ray diffraction (XRD (Shimadzu XRD-6000) was conducted in ambient conditions with (Cu K α) diffractometer to determine the constituent minerals of Jaboi kaolin, the scan speed of 2 degree min⁻¹, step size of 0.02°, and 2 θ from 7° to 70°. The infrared spectrum of absorption for kaolin was measured using Fourier transform infrared spectroscopy (FTIR Shidmazu IR Prestige-21) at wavelengths range of 4000-500 cm⁻¹. The morphology and elemental composition of the Jaboi kaolin were observed from SEM EDX (Carl Zeiss-Bruker, EVO MA10 Type) at an acceleration voltage of 15 kV. Magnification of 1,000 images was chosen to reveal the kaolinite layer. The samples will be checked initially coated surface using gold plating by sputtering to improve conductivity. Thermal analysis was carried out using DSC / TGA (Shidmazu, DSC/TGA- 60). Samples used as much as 5 mg with a flow rate of 20 ml/min at a temperature range of 20°C - 600°C.

RESULTS AND DISCUSSION

X-ray Fluorescence Analysis of Jaboi Kaolin

Jaboi kaolin content analyzed using XRF tool and the results obtained are shown in Table-1. Commercial kaolin is used as a comparison. XRF analysis results showed that Jaboi kaolin contained SiO₂ and Al₂O₃ of 84.8% and 2.96%, while other impurities composition such as K₂O, Fe₂O₃, TiO₂, MgO, SO₃ were under 1%. Based on the data obtained shows a very high silica content while the alumina content is very low. This is caused by the presence of Jaboi kaolin in the volcano area which affects the silica content to be so large. When compared with the commercial kaolin composition, there were differences in the levels of silica and alumina where the composition of commercial kaolin SiO₂ and Al₂O₃ were 45.98% and 40.26% respectively.

Table-1: Composition of Jaboi Kaolin vs Commercial Kaolin Based on XRF Analysis.

Compound	Unit	Three Sampling Points			Average	Commercial Kaolin
		Point 1	Point 2	Point 3		
Loss on ignition	%	10.68	9.72	12.6	11.000	14.12
SiO ₂	%	82.75	87.75	83.9	84.800	45.98
Al ₂ O ₃	%	3.46	2.35	3.07	2.960	40.26
Fe ₂ O ₃	%	1.39	0.26	0.26	0.637	0.78
MgO	%	0.34	0.18	0.16	0.227	0.04
SO ₃	%	0.04	0.04	0.00	0.027	0.09
K ₂ O	%	1.78	0.13	0.22	0.710	0.75
CaO	%	0.11	0.13	0.14	0.127	0.00
Na ₂ O	%	0.13	0.05	0.05	0.077	0.03
P ₂ O ₅	%	0.03	0.02	0.03	0.027	0.033
TiO ₂	%	0.55	1.029	0.978	0.852	0.321
Mn ₂ O ₃	%	0.02	0.006	0.007	0.011	0.009
Cr ₂ O ₃	%	0.01	0.002	0.005	0.006	0.003

Natural kaolin research was also carried out by several researchers^{14,15}, such as natural kaolin in Balikesir region¹⁴, Turkey having silica levels of 63.11% and alumina levels of 25.43%. While kaolin originating from Tabarka, Tunisia is reported to have silica and alumina levels of 53.2% and 27.3%, respectively.¹⁵ Other research¹⁶ reported the results of the analysis obtained kaolin containing silica and alumina amounted to 60.54% and 22.55%. The difference in silica and alumina levels (mineralogical composition) of natural kaolin is strongly influenced by the geology of the kaolin deposit. Kaolin deposits can be sedimentary, residual, or hydrothermal and in almost every example kaolin has different properties and must, therefore, be fully tested and evaluated to determine their utilization.¹⁷

Fourier Transform Infrared Analysis of Jaboi Kaolin

The spectra of Jaboi kaolin and commercial kaolin are shown in Fig.-3. The spectral analysis will help in the identification of various forms of minerals present in the clay. The presence of kaolinite minerals can be detected by differences in position and relative intensity of OH stretching and bending band in the IR spectrum.⁶ Jaboi kaolin shows absorption bands at wavelengths of 3600-3700 cm^{-1} , uptake at 3620 and 3606 cm^{-1} due to stretching vibrations of hydroxyl groups.¹⁸ The bands at 3620 and 3606 cm^{-1} are hydroxyl groups on the surface of the octahedral alumina layer which are connected to oxygen atoms from the adjacent silica tetrahedral layer.^{6,24,19}

The presence of kaolinite is also clearly visible at 3620 cm^{-1} . Jaboi kaolin spectra show sharp absorption in the regions of 1606, 1822 and 1938 cm^{-1} which is the absorption of the bend-OH vibration trapped in the crystal lattice. Si-O stretching vibration which is a typical absorption of kaolinite minerals is shown in 1118, 1002, 898 and 746 cm^{-1} .¹⁹

Spectra in the range of 779 to 797 cm^{-1} indicate the presence of cristobalite minerals while quartz is predicted at spectra of 628 cm^{-1} .²⁰⁻²¹ The FTIR spectra of Jaboi kaolin when compared to commercial kaolin there are close absorption peaks, both for the hydroxyl group, the kaolinite, quarsa and cristobalite minerals.

Fig.-3: Jaboi Kaolin and Commercial Kaolin Infrared Spectra

X-ray Diffraction Analysis of Jaboi Kaolin

The XRD patterns of the Jaboi kaolin and commercial kaolin are given in Fig.-4. The diffraction peaks of each clay mineral are typical, the diffraction angle 2θ is related to the crystal lattice plane of the analyzed mineral.

Identification of the constituent components of kaolin minerals is done by comparing the peak position of diffraction intensity (2θ) of the sample with the standard peak position of diffraction intensity (2θ) on the Joint Committee for Powder Diffraction Standards (JCPDS).²² Based on XRD analysis results, it can be seen that Jaboi kaolin consists of kaolinite minerals shown at 2θ ($^{\circ}$) = 21.52 with d spacing 4.125²³, quartz at 2θ ($^{\circ}$) = 21.82 with d spacing 4.068 and cristobalite at 2θ ($^{\circ}$) = 35.9 with d spacing 2.49.²⁴ From the diffraction intensity of the three minerals, quartz is the mineral with the largest composition, followed by the minerals kaolinite and cristobalite. This is according to the results of FTIR spectra which indicate the

presence of the three minerals. Commercial kaolin contains kaolinite as the main constituent mineral at 2θ ($^\circ$) = 12.26 and 38.35 with d spacing 7.21 and 2.345.²⁴ While the quartz minerals as impurities are found at 2θ ($^\circ$) = 24.81 with d spacing 3.585.

Fig.-4: Diffraction Pattern of Jaboi Kaolin and Commercial Kaolin

Surface Morphology of Jaboi Kaolin

SEM image (Fig.-5a and Fig.-5b) shows the morphology of Jaboi kaolin and commercial kaolin. Based on the results of the micrographs show that hexagonal platelets of the mineral kaolinite with a non-uniform size, in addition to the presence of quartz and cristobalite impurities.^{19,22,25} Jaboi kaolin has a pore size in the range of 2.145-6.701 μm . Impurity minerals, both quartz and cristobalite, are seen in the form of chunks that cover the mineral kaolinite. Commercial kaolin micrograph results show hexagonal platelet with a smaller and uniform size. This indicates that impurities in commercial kaolin are relatively small. This result is supported by the measurement of the pore diameter obtained smaller than the pore diameter of Jaboi kaolin, which is in the range of 1.117 – 4.020 μm . The EDX results showed that the composition of the Jaboi kaolin element consisted of 31.94% silicon, 1.33% aluminum, 14.61% carbon and 50.78% oxygen. While from the results of EDX commercial kaolin obtained 16.27% silicon, 16.2% aluminum, 2.44% carbon and 46.46% oxygen. The EDX results of the two types of kaolin are in accordance with XRF analysis as shown in Table-1.

Fig.-5a: SEM EDX Jaboi Kaolin

DSC/TGA Analysis

Kaolin phase transformation in the temperature range of 20⁰C - 600⁰C was studied using differential thermal analysis. Jaboi kaolin derivatogram is shown in Fig.-6. The DSC curve shows an endothermic effect at 20⁰C - 40⁰C, a sharp peak (endothermic peak) that occurs due to the removal of hygroscopic water or evaporation of water.

Fig.-5b: SEM EDX Commercial Kaolin

The endothermic curve is quite sharp and wide there is also at a temperature of 90⁰C - 200⁰C caused by the release of bonds of water molecules. This evaporation process requires heat $\Delta H = 169.74$ J/g or $\Delta H = 40.55$ cal/g. Based on the derivatogram, there is no visible crystallization of the Jaboi kaolin. From the results of the thermal gravimetric analysis (TGA) analysis, kaolin heated from a temperature of 20⁰C - 600⁰C changes in mass by 6-8%.²⁶

Fig.-6: DSC/TGA Curves of Jaboi Kaolin

CONCLUSION

This study studied the physical and chemical properties of Jaboi kaolin through characteristic tests using XRF, FTIR, XRD, SEM-EDX and DSC/TGA. Based on the XRF test, the main composition of Jaboi kaolin was obtained as follows: SiO₂ (silica) and Al₂O₃ (alumina) respectively at 84.8% and 2.96%, while the remaining impurities with concentrations below 1%. The results of FTIR spectra and diffraction intensity using XRD can be seen that the mineral constituents of Jaboi kaolin are quartz, kaolinite and cristobalite. Quartz is the main mineral, followed by kaolinite and cristobalite. The SEM test obtained the typical morphology of Jaboi kaolin, in the form of a heterogeneous group of layered hexagonal sheets with pore sizes in the range 2.145 - 6.701 μm. EDX results showing the composition of Jaboi kaolin elements consisting of 31.94% silicon, 1.33% aluminum, 14.61% carbon and 50.78% oxygen. The results of EDX Jaboi kaolin are supported by XRF, FTIR and XRD analysis results. Based on the derivatogram in thermal analysis, there is no visible crystallization of the Jaboi kaolin. Jaboi kaolin characterization results indicate that kaolin is very suitable for use as a raw material in the refractory and ceramics industries.

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