Characterization of technical kaolin using XRF, SEM, XRD, FTIR and its potentials as industrial raw materials

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Abstract. Technical Kaolin is a commercially sold kaolin with a relatively low level of purity and is often used as a filler of paints, paper, and ceramics. In this study, we tested the technical properties of kaolin physically and chemically, which include XRF, SEM, XRD, and FTIR. Based on the XRF test, the main composition of kaolin, SiO$_2$, Al$_2$O$_3$, K$_2$O, Fe$_2$O$_3$, and TiO$_2$ were 46.66%, 39.63%, 0.84%, 0.55% and 0.274% respectively, while the rest were impurities. The FTIR spectra showed the functional groups of Al-OH, Al-O, and Si-O. While the XRD diffractogram identified kaolinite as the main mineral phase in the presence of quartz, chlorite, halloysite, and cristobalite tested in small quantities in the sample.

1. Introduction

The term of kaolin and kaolinite comes from the word Kau-ling, Jiangxi province, China where locals use this white clay for ceramics/porcelain. Mineralogy is used in this way against hydrated aluminum silicate mineral groups such as kaolinite, halloysite, metahalloysite and fire clay. Kaolin including non-metallic minerals clay type of soil that has fine grains, it is plastic when moist, harden when dry and petrified when heated. Kaolin or commonly referred as china clay is one type of mineral containing a crystal compound with the main content of kaolinite (Al$_2$O$_3$.2SiO$_2$.2H$_2$O). Kaolin can be distinguished from other clay minerals due to its softness, white degree (whiteness) and easily dispersed in water or other solutions. Because of these physical properties, kaolin is widely used in the paper industry as a filler or coating material. According to the theory, pure kaolin contains 46% silicate, 40% alumina and 14% water [1]. Moreover, it is hardly found that kaolin that meets the composition, since there are other impurities such as titan (TiO$_2$), iron (Fe$_2$O$_3$), lime (CaO) or potassium (K$_2$O).

Kaolin is formed from strong weathering results of crystalline rocks, especially acidic ones, such as granite, diorite, dacite, etc. This weathering process is usually chemical weathering or hydrothermal alteration and can also be called the soil formation process commonly caused by acidic hot water. The process of weathering frozen rocks into kaolin usually occurs on the surface or very close to the ground surface. In Indonesia, kaolin deposits are mostly the result of granite rock alteration. The kaolin mineral belongs to a 1:1 clay wherein the crystal consists of octahedral sheets of aluminum piled on a silica tetrahedral sheet. The sheets extend continuously and accumulate on top of the other. The unit cell is not symmetrical with the silica tetrahedral sheet on one side and the octahedral...
sheet of aluminum on the other side. As a result, the basic plane of oxygen atoms in a crystal unit is confronted with the basal plane consisting of OH ions in the next layer. The negative charge on kaolin minerals is caused by the dissociation of the open hydroxyl group. This hydroxyl group usually attaches to the silica contained in the broken tetrahedral (T) plate. At high pH, the hydrogen of the hydroxyl group will decompose slightly and the clay surface becomes negatively charged, which is derived from the oxygen charge.

The negative charge due to this phenomenon is different from that caused by the isomorphic substitution process, which then changes due to pH change. Kaolin has a Cation Exchange Capacity (CEC) ranging from 1-10 meq/100 gr and a small surface area of 7-30 m²/g. This small surface area limits the ability of kaolin in cation absorption.

The use of kaolin for the first time in the manufacture of ceramics, since the nature of plasticity, has easily shaped and smoothed, no shrinkage time dries and produces good color after burning. In addition to the ceramics industry, kaolin is used in the rubber industry as a filler, developer, and amplifier. While in the paper industry, other than as filler and kaolin developer also serves as a coating or wrapping of cellulose fibers. Kaolin is also widely used in the paint industry, ink, medicine, cosmetics, wine purifier, carrier material for insecticide, crayon filler, oil adsorbent and also the catalyst.

Kaolin is commercially sold on the market generally in different purity. The higher the kaolin purity level, the higher the selling price. Technical Kaolin or kaolin with relatively high levels of impurities are often found in chemicals stores at low prices and their use is generally the main ingredient of ceramic making. This technical kaolin must be purified before use as in the medical and cosmetic industries.

In this study, we examined the technical kaolin characteristics using XRF, SEM, XRD, FTIR and compared the results obtained with pure kaolin characteristics based on the literature.

2. Research Method
2.1. Apparatus and Materials
The used apparatuses are FTIR spectrophotometer (Shimadzu FTIR - 4200 type A), XRD (Shimadzu XRD-6000), JEOL JSM-6360, XRF, a set of glass tools (Pyrex), a sieve of 130 mesh Retsch), and technical kaolin.

2.2. Technical kaolin Preparation
The technical kaolin purchased from one of the largest chemicals stores in Medan, North Sumatra, Indonesia, was sieved to obtain a uniform size by using a mesh size of 130 mesh. Kaolin is then further characterized by using XRF, SEM, XRD, and FTIR.

3. Results and Discussion
The kaolin technical composition was analyzed using XRF instrument and obtained the results as shown in Table 1. From Table 1, it is identified that the technical kaolin containing SiO₂ and Al₂O₃ are
46.66% and 39.63. The data obtained is not much different from the data pure kaolin according to the theory [2] ie the composition of SiO$_2$ and Al$_2$O$_3$ respectively by 46% and 40%. Based on the proximity of silicate and alumina compositions obtained between technical kaolin and pure kaolin showed that technical kaolin has silicate and aluminate levels near pure kaolin although there are still many impurities it contains such as K$_2$O, Fe$_2$O$_3$, TiO$_2$, MgO, SO$_3$ (Table 1).

| Table 1. Technical Kaolin Composition Based on XRF Analysis |
|------------------------------------------------------------|
| Compound         | Value | Unit |
|------------------|-------|------|
| Loss on ignition | 14.32 | %    |
| SiO$_2$          | 46.66 | %    |
| Al$_2$O$_3$      | 39.63 | %    |
| Fe$_2$O$_3$      | 0.55  | %    |
| MgO              | 0.03  | %    |
| SO$_3$           | 0.08  | %    |
| K$_2$O           | 0.84  | %    |
| CaO              | 0.00  | %    |
| Na$_2$O          | 0.02  | %    |
| P$_2$O$_5$       | 0.021 | %    |
| TiO$_2$          | 0.274 | %    |
| Mn$_2$O$_3$      | 0.008 | %    |
| Cr$_2$O$_3$      | 0.002 | %    |

The XRF analysis results (Table 1) were compared with kaolin standards for the ceramic industry (Table 2). The data shows the proximity of the composition value obtained.

| Table 2. Composition of Ceramic Grade Kaolins [8] |
|------------------------------------------------------|
| Compound         | a    | b    | c    | Unit |
|------------------|------|------|------|------|
| Loss on ignition | 13.0 | 12.22| 12.1 | %    |
| SiO$_2$          | 47   | 48   | 48   | %    |
| Al$_2$O$_3$      | 38   | 37   | 37   | %    |
| Fe$_2$O$_3$      | 0.39 | 0.7  | 1.0  | %    |
| MgO              | 0.22 | 0.30 | 0.30 | %    |
| K$_2$O           | 0.8  | 1.85 | 2.0  | %    |
| CaO              | 0.1  | 0.06 | 0.07 | %    |
| Na$_2$O          | 0.15 | 0.10 | 0.10 | %    |
| TiO$_2$          | 0.03 | 0.02 | 0.05 | %    |

(a) ECC Super standard porcelain  
(b) ECC grolleg; earware  
(c) ECC Remblend; sanitaryware

Based on the absorption of FTIR spectra from kaolin samples in Figure 2, a sharp peak uptake of 1639, 1820, and 1928 cm$^{-1}$ are observed in the absorption of the -OH buckling vibrations trapped in the crystal lattice. The absorption bands appearing at wave numbers 1010 and 1022 cm$^{-1}$ show vibration Si-O- which is a typical uptake of kaolinite minerals [3, 4]. While at the peak of uptake area 3660 cm$^{-1}$ indicates the existence of vibration -OH which has the environmental difference is -OH bound to Al octahedral atoms on the surface or on the inter-layer silicate. The functional group wave numbers are almost identical to kaolin spectra data presented by previous researchers [5] in the wave
numbers 1010 and 1032 cm\(^{-1}\) (presence of Si-O vibration), 1633 cm\(^{-1}\) (buckling vibration -OH) and 3696 cm\(^{-1}\) (vibration -OH, octahedral).

Figure 2. Infrared Spectra of Technical Kaolin

The identification of the kaolin mineral compositing component is done by comparing the peak intensity position (2θ) in the sample X-ray diffraction-gram with the peak position value of the diffraction intensity(2θ) standards contained in the Joint Committee for Powder Diffraction Standards (JCPDS). The price of the peak intensity of the technical kaolin is shown in Figure 3. The absorption peaks appear at 2θ (°) = 11.68°; 12.26°; 19.84°; 21.22°; 22.96°; 24.81°; 26.57°; 34.94°; 38.35°.

Figure 3. X-ray Diffractogram of Technical Kaolin
The price of 2θ (°) for kaolinite minerals is 12.26°; 24.81°, chlorite mineral is 19.84°, mineral quartz is 21.22°; 26.57°, cristobalite was 22.96° [4] and the halloysite mineral was 11.68°, mineral clay was 34.94°; 38.35° [6]. Table 3 shows the value of pure d (Å) kaolin and the technical kaolin has no different, which from the data shows the SiO₂ content of both kaolin and a special characteristic of kaolinite.

| Table 3. Data of technical XRD Kaolin |
|---------------------------------------|
| d standard (Å) | d sample (Å) |
| 7.14          | 7.2086       |
| 3.57          | 3.5849       |
| 2.34          | 2.3451       |

The morphological analysis of the technical kaolin in the form of photographs using SEM is presented in Figure 4 which shows that the dominant content of typical kaolin morphology in the form of heterogeneous layered sheets with heterogeneous sizes.

The kaolin structure has a size of 1-10 μm with the number of sheets per layer of about 10-50 pieces [7]. In technical kaolin morphology, it is clear that there is a large quantity of quartz mineral which is the impurity of kaolin mineral.

Figure 4. Photo of SEM Technical Kaolin

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4. Conclusion
The results of the kaolin technical analysis using XRF, FTIR, SEM and XRD instruments have clearly shown that the commercially sold kaolin is a kaolinite mineral compound. The XRF results show the composition of SiO₂ and Al₂O₃ in the technical kaolin of 46.66% and 39.63%, respectively, wherein the silicate and alumina compositions are very close to the pure kaolin composition. The results of the FTIR, XRD and SEM analyses also support the XRF analysis. From FTIR spectra and XRD diffractogram obtained special peaks of kaolinite minerals.
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