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Characterization of clays in Trichinopoly group Tamilnadu, India

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Abstract

A combination of analytical techniques such as X-ray fluorescence(XRF), X-ray diffraction(XRD) and Fourier transform Infra red (FTIR) spectral analysis were employed to characterize clays from Trichinopoly group, Tamilnadu India. The results obtained show that enriched with high amount of silica(SiO_2) 53.20-58.40% followed by alumina(Al_2O_3) 18.33-25.51%, Iron oxide(Fe_2O_3) 2.32-5.72%, calcium(CaCO_3) 2.35-4.10% and magnesium(MgO) 2.04-3.76% and trace amounts of other elements such as Ti, K, Na and Mn (0.07-1.13%,0.99-2.84%, 1.52-3.93% and 0.01-0.05% respectively). The XRD studies showed that the clay deposits consist predominantly of kaolinite, dickite and quartz with traceable amounts of illite, muscovite and microcline minerals. The infra red spectral analysis between $400\text{-}4000\text{cm}^{-1}$ revealed interesting wave numbers and absorption bands. The presence of above minerals were further confirmed by FTIR analysis.

Keywords: Characterization, Clay, XRD, XRF and FTIR, Trichinopoly Group

1. Introduction

Clays are raw materials abundantly found and widely used by the ancient civilizations to make figures and artifacts (Norton, 1974, Haber and Smith, 1991) ^[1, 2]. Clays are used in the manufacture of ceramic products such as bricks, roofing tiles, porcelain, sanitary wares, wall tiles, floor tiles and are also used in different industrial and chemical processes. The clay minerals are hydrated aluminosilicates with layered structures that are responsible for the characteristic properties of the clays. There are a variety of clay minerals including kaolinite, halloysite, illite, montmorillonite, chlorite, among others (Lamia Bouchhima *et al.* 2013) ^[3]. Because of their low cost, abundance in most areas, high sorption properties and potential for ion exchange, clay materials are strong candidates as adsorbents. The adsorption capabilities result from a net negative charge on the structure of minerals. This negative charge gives clay the capability to adsorb positively charged species. Their sorption properties also come from their high surface area and high porosity (Alkan *et al.* 2004, Sagar Nayak and Singh, 2007) ^[4, 5]. The clays are composed mainly of alumina and water, frequently with appreciable quantities of iron, alkalis and alkali earths (Grim, 1968) ^[8]. Two structural units are involved in the atomic lattices of most clay minerals. One unit consists of closely packed oxygen and hydroxyls in which magnesium atoms are embedded in an octahedral combination so that they are equidistant from six oxygen or hydroxyls. The second unit is built of silica tetrahedrons. The silica tetrahedrons are arranged to form a hexagonal network that is repeated indefinitely to form a sheet of composition, $\text{Si}_4\text{O}_6(\text{OH})_4$ (Grim, 1968) ^[8]. In view of this natural architectural design of clay, it is imperative to always characterize them before putting it to use. There is no report in the literature to our knowledge that the clay deposit of Trichinopoly group has been characterized. Hence, this paper deals with the characterization of clay with the help of XRF, XRD and IR analytical techniques.

2. Materials and Methods

Representative clay samples were collected from Trichinopoly group of formation, Tamil Nadu. This sample was fractionated into different fractions of varying particle sizes using standard sieves of mesh sizes having geometrical mean particle diameters 140, 100 and 50 μm . The chemical composition of clay was determined by XRF.

XRD pattern of clay were obtained on a powder X-ray diffraction model Philips with $\text{CuK}\alpha$ radiation having a scanning speed of $0.04^\circ/\text{s}$. The clay was analysed for its vibrational spectra with the aid of FTIR using Perkin Elmer 1800 model instrument in the range of $400\text{-}4000\text{ cm}^{-1}$ as KBr pellet technique.

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3. Results and Discussion

3.1 Chemical characterization

The result of the oxide analysis is shown in table.1. The clays of Trichinopoly group enriched with high amount of silica (53.20-58.40%), alumina (18.33-25.51%), Iron (2.32-5.72%), calcium (2.35-4.10%) and magnesium (2.04-3.76%). The traceable (less than 1%) amounts of other elements such as Ti,

K, Na and Mn (0.07-1.13%, 0.99-2.84%, 1.52-3.93% and 0.01-0.05% respectively). When silica content of clay is high, their source of silica can be used for the production of floor tiles. According to Onukwuli *et al.* (1996)^[9], the high content SiO₂ and Al₂O₃ and low content of CaO present in the clay suggested that the clay is dominated by kaolinite in nature.

Table 1: Chemical composition of the Clay

| Sample No. | SiO ₂ | Al ₂ O ₃ | Fe ₂ O ₃ | CaO | MgO | Na ₂ O | K ₂ O | MnO | TiO ₂ | P ₂ O ₅ |
|------------|------------------|--------------------------------|--------------------------------|------|------|-------------------|------------------|------|------------------|-------------------------------|
| TG1 | 53.20 | 20.93 | 3.71 | 2.79 | 2.76 | 1.52 | 0.99 | 0.01 | 0.79 | 0.01 |
| TG2 | 58.4 | 19.81 | 3.84 | 4.1 | 2.79 | 2.89 | 1.33 | 0.02 | 0.07 | 0.0078 |
| TG3 | 57.53 | 18.33 | 4.21 | 2.59 | 2.82 | 2.36 | 2.84 | 0.02 | 1.13 | 0.02 |
| TG4 | 55.65 | 25.51 | 2.32 | 2.35 | 2.38 | 1.97 | 1.23 | 0.01 | 0.95 | 0.009 |
| TG5 | 56.29 | 19.07 | 5.72 | 4.05 | 2.04 | 2.86 | 1.47 | 0.05 | 0.67 | 0.07 |
| TG6 | 54.09 | 18.88 | 3.95 | 2.42 | 3.76 | 3.93 | 1.91 | 0.02 | 0.93 | 0.02 |

TG1-Peruvalappur, TG2-Kulakkanatham, TG3-Saradamangalam, TG4-Mugilpadi, TG5-Adanur, TG6-Kuttur

3.2 XRD Characterization

XRD is used to determine the mineralogical composition of clay deposits. The occurrence of minerals in clay were identified by comparing 'd' values (selected powder diffraction data for minerals, 1974). The possible minerals with their 'd' values present in the analysed samples are given in the Table 2. The characterization of XRD patterns indicates the presence of kaolinite, illite, Dickite are found as major clay mineral phases. The non clay mineral phases are identified as quartz and microcline. Further, the occurrence of above minerals were confirmed by FTIR study.

Table 2: X-Ray diffraction 'd' values of the clay fraction

| d (Å°) | Possible mineral |
|--|------------------|
| 4.49, 3.76, 3.73, 3.66, 3.14, 3.18, 3.21, 3.28, 2.57, 1.91, 1.81 | Kaolinite |
| 4.25, 4.04, 3.74, 3.64, 3.23, | Dickite |
| 3.47, 3.24, 2.93, 2.65, 2.11 | illite |
| 2.84, 3.77 | Muscovite |
| 4.26, 3.34, 2.99, 1.77 | Quartz |
| 15.66, 15.58, 15.51, 15.20 | Microcline |

Reference: JCPD file, Carroll, (1970)

3.3 FTIR Characterization

The observed adsorption wave numbers from the peaks were tabulated with their corresponding minerals are shown in the table 3. The FTIR spectra were recorded over a range of 4000 - 400 cm⁻¹.

Table 3: The observed adsorption wave numbers and corresponding minerals from FTIR spectra

| S. No | Assignment | Wavelength cm ⁻¹ | Minerals |
|-------|---|------------------------------------|------------|
| 1 | Inner surface OH stretching vibration | 3700 | Kaolinite |
| 2 | OH stretching of inner surface hydroxyl group | 3924, 3884, 3776, 3619, 3611, 3391 | Kaolinite |
| 3 | Si - O stretching of clay minerals | 1035 | Kaolinite |
| 4 | Si - O of Quartz | 695 | Quartz |
| 5 | Si - O stretching | 786 | Quartz |
| 6 | Si - O deformation | 426 | Orthoclase |
| 7 | Si - O - Si bending of silicates | 781 | Muscovite |
| 8 | Si - O - Si deformation | 472 | Illite |

Reference: Russell (1987), Summer (1995), Marel and Bentelspacher (1976)

In sample Si-O stretching and bending as well as the OH bending absorption tells us more about the kaolinite minerals

present in the clay (Nwosu *et al.* 2013)^[10]. Most of the band such as 3700 cm⁻¹, 1035 cm⁻¹, 693 cm⁻¹, 468 cm⁻¹ show the presence of kaolinite (Nayak and Singh, 2007)^[5]. In the deformation site of the wave length at the region 472 - 466.83 cm-1 indicate the presence of illite.

The presence of quartz can also be observed in the region of 786 cm⁻¹, 694 cm⁻¹ and 467 cm⁻¹. The appearance of ν(Si-O-Si) and δ (Si-O) bands also support the presence of quartz (Marel and Bentelspacher, 1976, Nwosu *et al.* 2013)^[6, 10]. The vibrations observed at 426 cm⁻¹ indicate the possibility of presence of orthoclase.

4. Conclusion

The chemical compositional analysis (XRF) exhibits that the clay deposits of the study area are composed of silica and alumina in major quantities. The minor and trace oxide composition are Iron, calcium, magnesium and other elements. The XRD study suggests the presence of kaolinite, illite, muscovite are found as major clay mineral phases. The presence of above mineral were further confirmed by FTIR analysis.

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