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**SYNTHESIS, CHARACTERIZATION AND CATALYTIC ACTIVITY OF PALLADIUM
SUPPORTED ON BLEACHING CLAY**

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ABSTRACT

Bleaching clay (BC) is one example of phyllosilicate clay that consists of two tetrahedral sheets sandwiching a central octahedral sheet. Palladium (Pd) is proposed to adsorb on the layers of BC by adsorption process to form the palladium supported in bleaching clay (Pd-BC) structure. The layer structure of BC with the ability of exchangeable cation allowed the adsorption process of Pd. In this research, pretreatment of BC involved the acid content analysis using ion chromatography (IC) and acid-base titration, fourier transform infrared (FTIR) spectroscopy and thermogravimetric analysis (TGA) to determine its physical and chemical characteristics. The adsorption process is done by adding PdCl₂ solution of variable Pd contents (1, 2, 3 % w/w) into BC-ethanol suspension and stirred for 4 hours, before the mixture is filtered, washed three times with distilled water and dried at room temperature. The characterization with X-ray diffraction (XRD) is meant to study layer spacings, while N₂ sorption is to obtain surface areas and pore structure. The hydrogenation process of palm oil was performed using Pd-BC as catalyst in order to test the catalytic activity of Pd-BC. The catalyst is dissolved in isopropanol before the mixture is bubbled with H₂ gas for 60 minutes, then palm oil is added at room temperature and the hydrogenation process is continued for another 90 minutes. After that the hydrogenated palm oil is extracted with hexane and esterified with methanol in NaOH before the product is analyzed by gas chromatography (GC) to determine the percentage of fatty acid methyl esters (FAME). The acid-base titration and IC analysis of BC revealed that BC sample has about 1.26 to 1.8213 % w/w of sulphuric acid, while FTIR and TGA confirmed the presence of clay mineral in BC and its thermal process. The adsorption process with different Pd contents varied the distribution of Pd cations between the layers of BC and its adsorption capacity. Pd-BC samples have smaller surface area, but larger layer spacing than BC. Pd-BC samples are achieved for catalytic hydrogenation of palm oil and cause lower percentage of C18:2 FAME through higher percentage of C18:0 and C18:1 FAMES.

ABSTRAK

Tanah liat pelunturan (BC) adalah salah satu contoh tanah liat phyllosilikat yang terdiri daripada dua lapisan tetrahedral yang mengapit lapisan oktahedral. Palladium (Pd) dicadangkan untuk dijerap di atas lapisan BC melalui proses penjerapan untuk membentuk struktur palladium dijerap tanah liat pelunturan (Pd-BC). Struktur lapisan BC dengan kemampuan pertukaran kation membolehkan proses jerapan Pd berlaku. Kajian ini melibatkan pengolahan BC dan pencirian sifat-sifat fizikal dan kimia BC. Kajian awal terhadap BC melibatkan analisis kandungan acid menggunakan kromatografi ion (IC) dan pentitratan asid-bes, spektroskopi inframerah transformasi fourier (FTIR) dan analisis thermogravimetrik (TGA). Proses jerapan dilakukan dengan menambah larutan PdCl₂ dengan variasi kandungan Pd (1, 2, 3 % w/w) ke dalam suspensi BC-etanol dan dikacau selama 4 jam, sebelum campuran ditapis, dicuci tiga kali dengan air suling dan dikeringkan pada suhu bilik. Proses pencirian BC dan Pd-BC dimulakan dengan difraksi sinar-X (XRD) untuk menganalisis ruang dan jarak lapisan sampel, dan menggunakan jerapan N₂ untuk menentukan luas permukaan. Proses hidrogenasi minyak kelapa sawit dilakukan menggunakan Pd-BC sebagai mangkin untuk menguji aktiviti pemangkinan Pd-BC. Mangkin dilarutkan dalam isopropanol sebelum campuran digelembungkan dengan gas H₂ selama 60 minit, kemudian minyak sawit ditambahkan pada suhu bilik dan proses hidrogenasi diteruskan untuk 90 minit. Setelah itu minyak kelapa terhidrogenasi diekstrak dengan heksana dan diesterifikasikan dengan metanol dalam NaOH sebelum produk dianalisis dengan kromatografi gas (GC) untuk menentukan peratusan asid lemak metil ester (FAME). Pentitratan asid-bes dan analisis IC mendedahkan kandungan asid sulfurik dalam BC sekitar 1.26 hingga 1.8213 % w/w, manakala analisis FTIR dan TGA mengesahkan kehadiran mineral tanah liat dalam BC serta proses habanya. Proses jerapan dengan variasi kandungan Pd dapat mengubah sebaran kation Pd antara lapisan BC dan mengubah kapasiti jerapannya. Analisis pencirian Pd-BC memperoleh keputusan luas permukaan yang lebih rendah, namun jarak lapisan yang lebih besar berbanding BC. Aktiviti pemangkinan Pd-BC mengesahkan hidrogenasi minyak sawit dengan pengurangan peratusan FAME C18:2 menerusi peningkatan peratusan FAME C18:0 dan C18:1.

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LIST OF ABBREVIATIONS

BC	Bleaching clay
BET	Brunauer-Emmett-Teller
BJH	Barrett-Joyner-Halenda
C16:0	Palmitic acid
C18:0	Stearic acid
C18:1	Oleic acid
C18:2	Linoleic acid
CPO	Crude palm oil
FAME	Fatty acid methyl ester
FTIR	Fourier transform infrared spectroscopy
GC	Gas chromatography
IC	Ion chromatography
N	Normality
Pd	Palladium
Pd-BC	Palladium supported on bleaching clay
TGA	Thermogravimetric analysis
XRD	X-ray diffraction
w/w	Weight per weight

CHAPTER 1

INTRODUCTION

1.1 BACKGROUND OF STUDY

Clay is a natural inorganic substance occurring in great abundance in nature, which is one of the significant component of soil. Soil texture separates clay from other types of soil; silt and sand, based on the fractions of each soil separately (sand, silt, and clay) present in a soil. Clay can be classified based on its smallest particle size compared with silt and sand, with the diameter of less than 0.002 mm.

Clay consists of fine-grained minerals of aluminium phyllosilicate sheets with the certain percentage of water and exchangeable cations trapped between these sheets. These aluminium phyllosilicate sheets are composed of crystal layers of silicate (SiO_4) tetrahedral and alumina (AlO_4) octahedral, that combine together by the ratio of 1:1 or 2:1 depending on the types of clay minerals. These sheets are attracted together by polar attraction due to the uncharged or negatively charged sheets with positively charged layers of exchangeable cations. This type of attraction provides the layer structure of clay that formed large surface area between that layer spacings for the purpose of molecular adsorption and interaction. In the micrograph view, the layer spacings are picturized as the porous.

The structure and composition of clay generally influence its physical properties. The ability of layer structure of clay to trap an amount of water molecules made the clay has high waterholding capacity. When mixed with certain amount of water, clay exhibits plasticity property which is enable it to be sculpted and molded into different shapes. When dried or fired, clay turn hard and firm because of its

shrinkage. Clay shows high cohesion property due to its intermolecular polar attraction, which explained its impermeability of water. The existence of exchangeable cations between its layer structures enables clay to have cation exchange capacity due to adsorption process.

Based on its composition of minerals, clay can be classified into four main groups; kaolinite, smectite, illite and chlorite groups. Kaolinite is a clay mineral with the chemical composition $\text{Al}_2\text{Si}_2\text{O}_5(\text{OH})_4$. It is a layered silicate mineral, with the silicate tetrahedral sheet linked through oxygen atoms to one octahedral sheet of alumina, in the 1:1 ratio. This type of clay mineral includes the minerals kaolinite, dickite, halloysite and nacrite. Smectite group of clay is composed of several minerals including pyrophyllite, talc, vermiculite, sauconite, saponite, nontronite and montmorillonite, and they are differ mostly in chemical content. The general formula for smectite is $(\text{Ca}, \text{Na}, \text{H})(\text{Al}, \text{Mg}, \text{Fe}, \text{Zn})_2(\text{Si}, \text{Al})_4\text{O}_{10}(\text{OH})_2 \cdot n\text{H}_2\text{O}$. This group is composed of silicate layers sandwiching an alumina octahedral layer in between that formed the 2:1 ratio. Variable amounts of water molecules would lie between these sandwich layers.

Illite is a non-expanding, clay-sized, micaceous mineral. The chemical formula is given as $(\text{K}, \text{H}_3\text{O})(\text{Al}, \text{Mg}, \text{Fe})_2(\text{Si}, \text{Al})_4\text{O}_{10}[(\text{OH})_2, (\text{H}_2\text{O})]$, but there is considerable ion substitution potential. Its structure is constituted by the repetition of tetrahedron-octahedron-tetrahedron layers of 2:1 ratio. The layer spacing is mainly occupied by poorly hydrated potassium cations responsible for the absence of swelling. Chlorite group of clay also has a 2:1 sandwich structure of tetrahedral-octahedral-tetrahedral with layer spacing composed of $(\text{Mg}^{2+}, \text{Fe}^{3+})(\text{OH})_6$ that formed the typical general formula of $(\text{Mg}, \text{Fe})_3(\text{Si}, \text{Al})_4\text{O}_{10}(\text{OH})_2 \cdot (\text{Mg}, \text{Fe})_3(\text{OH})_6$.

Clay has been used widely in ceramic industry to make pottery and porcelain items. In the construction industry, clay is the main component in cement production. As the component of soil, the clay soil is originally used in agricultural sector for wetland cultivation such as paddy, jute, kenaf and hemp, since clay can trap huge amount of water. Recent interest in used of clay considers its absorption, surface and colloidal properties for catalytic application.

1.2 PROBLEM STATEMENT

The cation exchange capacity of clay may increase its adsorption and catalytic properties due to modification obtained by its structures. The exchangeable cations between layer structures of clay can be replaced by other cations, usually from transition metal groups by adsorption process. This process modified the microstructure of clay by four main effects; distribution of supported cations, layer spacing, adsorption capacity and specific surface area.

As the new cations are supported on the clay layer structures, the distribution of that supported cations may differ from the previous exchangeable cations. The distribution of supported cations affected clay's physical properties such as its shear stress, plasticity, cohesion and viscosity. This distribution is related with the amount of supported cations used for the adsorption process.

The supported cations can also influence the layer sizes of modified clay, hence its porosity. These porous sites are important as catalytic sites for adsorption and interaction of reactants. The modified layer can be penetrated by molecule of certain molecular sizes, which make it having specific permeability. This modified properties affect the catalytic properties of clay.

The modified clay has modified adsorption properties, which is described through isotherm, the amount of adsorbate on the adsorbent. The adsorption properties of clay are highly related with the distribution of cations between its layer spacings, which is determined the availability of specific active sites for the adsorption process. This active sites are described as specific surface area, total surface area per unit of mass, solid or bulk volume, or cross-sectional area of the material.

The specific surface area is related with the particle size distribution, layer sizes, supported particles and the structure of material. Larger specific surface area will provide more active sites for adsorption activity which increased the adsorption capacity of that material.

1.3 OBJECTIVES

The three main objectives of this study are to characterize the physical and chemical properties of bleaching clay (BC) sample, to synthesize the nanoparticles of Pd supported on BC (Pd-BC), and characterize the physical and chemical properties of Pd-BC, and to conduct the hydrogenation reaction process using Pd-BC as catalyst.

1.4 SCOPES OF STUDY

Based on the objectives, the scopes of this study have been identified to consist of four main parts. The first scope is to characterize the physical and chemical properties of bleaching clay sample. The determination of chemical properties of bleaching clay sample includes the acid content analysis using acid-base titration and ion chromatography (IC). The physical characterization of bleaching clay sample involved the thermogravimetric analysis (TGA) to test its mass stability over different temperature, fourier transform infrared (FTIR) spectroscopy to determine the chemical bonds and related compounds exist in bleaching clay sample, N₂ sorption of surface area analyzer to construct adsorption/desorption isotherm and surface area determination, and X-ray diffractometer (XRD) to analyze the layer spacing and possible structures of bleaching clay.

The second scope is about the synthesis of Pd-BC with variable Pd contents chosen to test the effect on physical and chemical properties of Pd-BC. Pd contents for adsorption process will be varied with three mass ratios of Pd contents to clay chosen being 1, 2 and 3 % w/w.

The third scope of this study is about the characterization analysis of Pd-BC. This scope is important in order to determine the physical and chemical properties of modified clay. The characteristics of modified clay are compared with that of normal clay to analyze the significance of adsorption process. In this research, three main characteristics of Pd-BC are analyzed; surface area determination and

adsorption/desorption isotherm using N_2 sorption, and sizes of layer spacing using X-ray diffractometer (XRD).

The main interest on modified clay is its catalytic activity. The third scope of this research is the catalytic activity of Pd-BC for the hydrogenation of palm oil into hydrogenated palm oil. Fresh palm oil is hydrogenated with H_2 gas, extracted using hexane and esterified with methanol in NaOH to form glycerols and fatty acid methyl ester (FAME), then the products are analyzed using gas chromatography (GC) to determine the percentage of FAME to be compared with FAME of fresh palm oil. The analysis results of percentage of conversion of palm oil into hydrogenated oil reflected the successful of hydrogenation process using Pd-BC as catalyst.

CHAPTER 2

LITERATURE REVIEW

2.1 CLAY

2.1.1 General View of Clay

The definition of "clay" was first formalized in 1546 and has been revised many times since, although the fundamentals involving plasticity, particle size, and hardening on firing were retained by most (Guggenheim et al., 1995). Clays and clay minerals have been mined since the Stone Age; today they are among the most important minerals used by manufacturing and environmental industries, and are mainly found on or near the surface of the Earth (Foley, 1999), as in Figure 2.1. Clay minerals form part of a subclass of the silicate class of minerals known as the phyllosilicates, the largest and most complex groups of minerals with approximately 30 % of all minerals are silicates, while geologists estimate that 90 % of the Earth's crust is made up of silicates (Woodward et al., 2002).

Clay principally is a fine-grained inorganic material having the ability to demonstrate marked plasticity when wet, and properties such as adsorption, hydration, ion exchange, and hardening when exposed to different environmental conditions (Kassim, 2009). Clays may be composed of mixtures of finer grained clay minerals and clay-sized crystals of other minerals such as quartz, carbonate, and metal oxides (Foley, 1999). Guggenheim et al. (1995) extended the term "clay" to a naturally occurring material composed primarily of fine-grained minerals, which is generally plastic at appropriate water contents and will harden when dried or fired. They also defined the term "clay mineral" as phyllosilicate minerals and to minerals

which impart plasticity to clay and which harden upon drying or firing (Guggenheim et al., 1995).

In mineralogical terms, clay particles are typically less than 2 μm but may be 10 μm or more in length (Woodward et al., 2002). Foley (1999) applied the term “clay” both to materials having a particle size of less than 2 μm and to the family of minerals that has similar chemical compositions and common crystal structural characteristics. According to Guggenheim et al. (1995), most geologists and soil scientists use particle size less than 2 μm , sedimentologists use 4 μm , and colloid chemists use 1 μm for clay-particle size.



Figure 2.1: Massive kaolinite deposits at the Hilltop pit, Lancaster County, South Carolina; the clays formed by the hydrothermal alteration and weathering of crystal tuff

Source: Foley (1999)

2.1.2 Microscopic Structure of Clay

Clay minerals share a basic set of structure and chemical characteristic (e.g. they are largely aluminosilicates with layer structures) and yet each clay mineral has its own unique set of properties that determine how it will interact with other chemical species (Viruthagiri et al., 2010). Piner et al. (2003) refer the term “clay” to a class of materials generally made up of layered silicates that are similar to mica, with these clay particles spanning centimeters in lateral dimensions, the in-plane dimensions of the individual clay layers are on the order of a micron, and the thickness of a single clay nanoplatelet is on the order of a nanometer. The crystalline clay minerals belong to the mineral family termed phyllosilicates (Greek phyllon, leaf) which is because of their leaf-like or planar structure, comprised of two basic building blocks, a sheet of silicate tetrahedral and the other an octahedral sheet, which are the fundamental structural units of these minerals, as explained by Kassim (2009) in Figure 2.2. Although clay usually contains phyllosilicates, it may contain other materials that impart plasticity and harden when dried or fired (Guggenheim et al., 1995).

The layers of clays are characterized by strong intralayer covalent bonds within the individual sheets comprising the clay, with only weak van der Waals interactions between adjacent clay sheets that generated strong in-plane covalent bonds (Piner et al., 2003). The sheets of tetrahedrons are connected to each other by weakly bonded cations and often have water molecules trapped between the sheets (Woodward et al., 2002). The bonds that link the unit layers of clay together to form particles are the reason for the variable response of the clay minerals in terms of plasticity and consistency (Kassim, 2009).

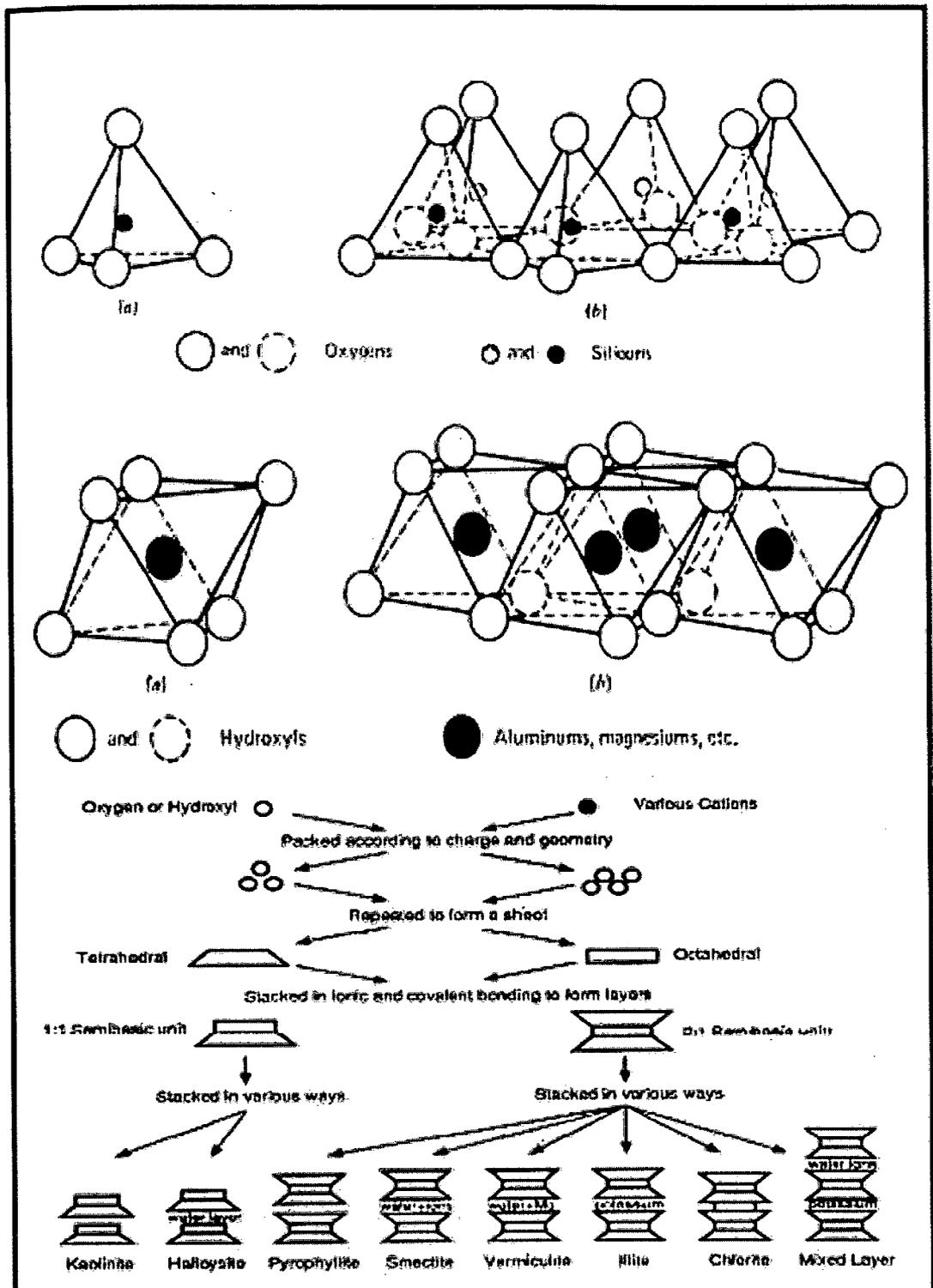


Figure 2.2: Basic sheet arrangements of silica tetrahedral and aluminum octahedral and synthesis pattern of clay minerals

Source: Kassim (2009)

2.1.3 Classification of Clay

The clay minerals are classified into two different groups; 1:1 type minerals or the kaolin group and the 2:1 type minerals or the illites and smectite groups (Kassim, 2009). Woodward et al. (2002) categorized clay into four main groups; kaolinite, montmorillonite/smectite, illite and chlorite groups.

According to Kassim (2009), kaolinite mineral is the most prominent member of kaolin group. As shown schematically in Figure 2.3, kaolinite formation is favored when alumina is abundant and silica is scarce because of the 1:1 silica to alumina structure. Conditions leading to kaolinite formation usually include areas where precipitation is relatively high, and there is good drainage to ensure leaching of cations and iron. As can be seen each kaolinite particle is comprised of a series of hexagonal shape layers much like the pages of a book. These layers are bound to other adjacent layers by hydrogen bonding. Consequently, cations and water do not enter between the structural layers of kaolinite. Therefore, in contrast with other clay minerals, kaolinite exhibits less plasticity, cohesion, and swelling. Woodward et al. (2002) explained the general structure of the kaolinite group is composed of silicate sheets bonded to aluminium oxide/hydroxide layers referred to as gibbsite layers, then the silicate and gibbsite layers are tightly bonded together with only weak bonding existing between the silicate-gibbsite paired layers.

In addition, Kassim (2009) explained that montmorillonite is the most common mineral of smectite group. Conditions favoring formation of montmorillonite type minerals are high pH, high electrolyte content, and the abundance of silica. Also climatic conditions where there is poor leaching and drainage favor the formation of smectite. This mineral has small, very thin, and filmy particles. The layers are loosely held together by very weak bonding. Hence exchangeable cations and associated water molecules are easily attracted between the layer spacings, causing expansion of the crystal lattice, as showed in Figure 2.4. It should be noted that, the type of adsorbed cations exerts a controlling influence on the behavior of these minerals. The structure of this group is composed of silicate layers sandwiching a gibbsite layer in a silicate-gibbsite-silicate stacking sequence

and the varying amounts of water molecules lie between the silicate-gibbsite-silicate sandwiches (Woodward et al., 2002).

In the same paper, Kassim (2009) described that illite is one of the most common clay minerals found in soil deposits encountered in engineering practice. They form under conditions similar to those leading to the formation of smectites. In addition, the presence of potassium is essential. The high stability of illite is responsible for its abundance and persistence in soils and sediments. The crystal layers of these mineral are characterized by an octahedral sheet sandwiched between two tetrahedral sheets. As can be seen in Figure 2.5, non-exchangeable potassium ions are strongly attracted in the layer spacing, acting as a binding agent, preventing expansion of the crystal. Therefore, illite minerals are quite non-expansive. The morphology of these particles is often flaky and thin at the edges. This group is basically hydrated microscopic muscovite mica, which the structure is composed of silicate layers sandwiching a gibbsite layer in a silicate-gibbsite-silicate stacking sequence and the variable amounts of water molecules would lay between the silicate-gibbsite-silicate sandwiches (Woodward et al., 2002).

As mentioned by Woodward et al. (2002) before, another type of clay is chlorite group, which the structure of this group is composed of silicate layers sandwiching a brucite layer in a silicate-brucite-silicate stacking sequence. However, there is an extra weakly bonded brucite layer in between the silicate-brucite-silicate sandwiches, which gives the clay a silicate-brucite-silicate, brucite, silicate-brucite-silicate sequence. Variable amounts of water lie between the silicate-brucite-silicate and brucite layers.

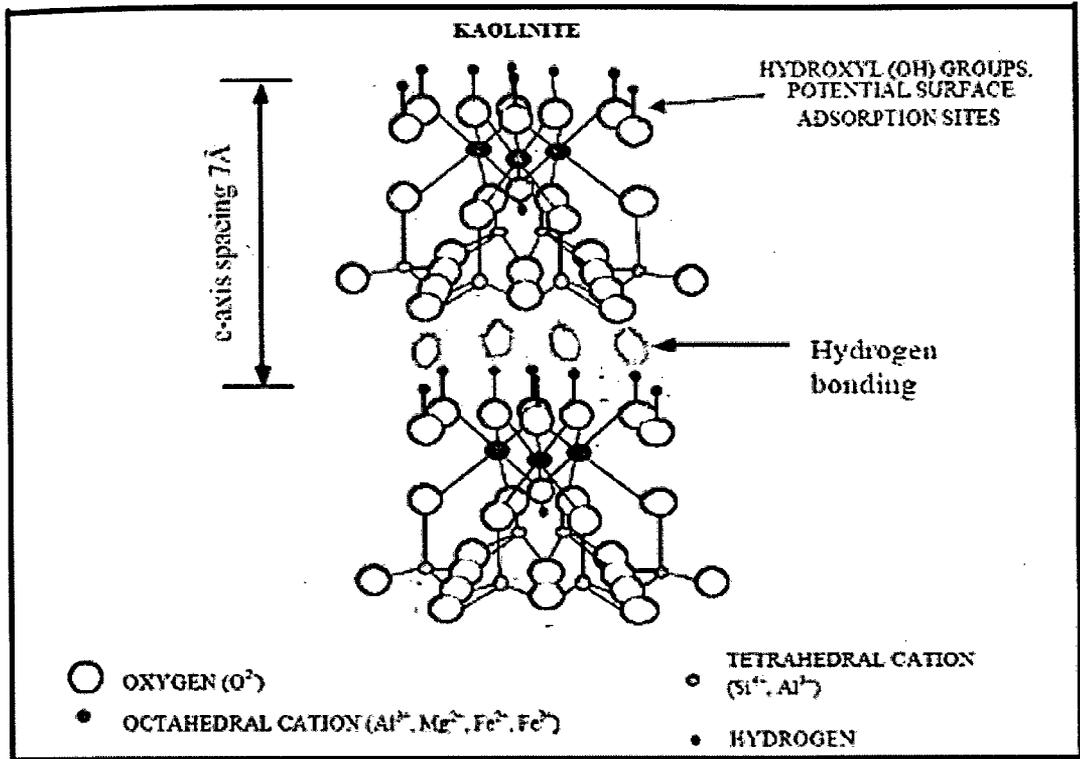


Figure 2.3: Structure of kaolinite

Source: Kassim (2009)

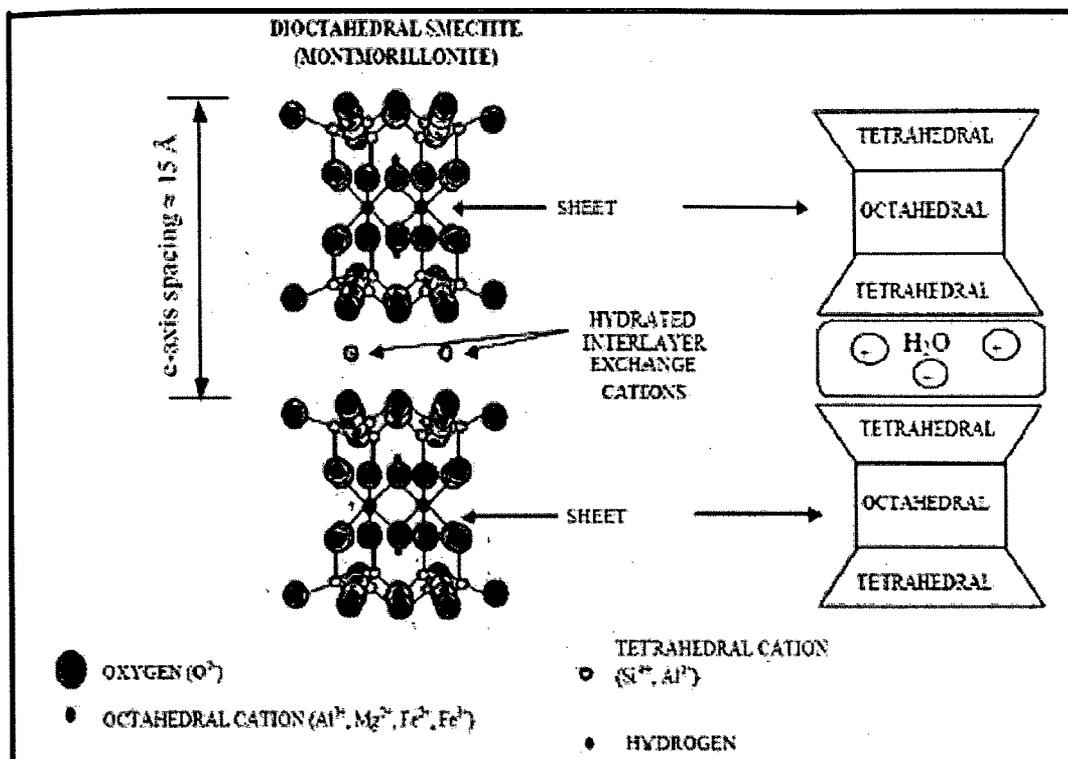


Figure 2.4: Structure and morphology of montmorillonite

Source: Kassim (2009)