

# SYNTHESIS, CHARL-. . LIC ACTIVITY OF Ni SUPPORTED ON BLEACHING CLAY

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#### **ABSTRACT**

Bleaching clay samples from Hudson WA Sdn Bhd were used to synthesis Ni(II) supported on clay. Metal supported on clay can serve as catalyst for hydrogenation reaction of crude palm oil. Acid analyses were employed using Ion Chromatography and titration technique to determine type and acid content. The nature of the bleaching clay examined by using X-ray Diffraction (XRD), Thermal Gravimetric Analysis (TGA), Fourier Transform Infrared (FTIR) and BET surface area. Nickel acetate with ethylene glycol as reducing agent was treated with bleaching clay to absorp metal: onto and into the interlayer of the clay. The adsorption of Ni(II) was investigated using BET surface area and x-ray diffraction. IC analysis and acid-base titration showed acid sulphuric as dominant acid at 0.0615 wt %. FTIR spectra and XRD pattern of this clay is identical with illite mineral It has a lattice spacing of 10.67 Å and has primitive bravais lattice properties. As much as  $\sim$  20 wt% of nickel can be intercalated and supported to bleaching clay. Metal supported clay have low load capacity compared with metal intercalated clay. However, particle diameter and surface area are increasing from 90 Å to 170 Å and 600  $m^2/g$  to 800  $m^2/g$  when load capacity is increases. Reactivity of Ni(II) supported bleaching clay is low where percentage of saturated fatty acid increases by 1 % to *2.5* % in hydrogenation reaction of crude palm oil at room temperature and pressure.

#### **ABSTRAK**

Sampel tanah liat peluntur dari Hudson MPA sdn bhd telah digunakan untuk menyediakan Ni(II) yang disokong atas tanah hat. Logam yang disokong atas tanah liat boleh digunakan sebagai pemangkin dalam tindak balas penghidrogenan minyak kelapa sawit mentah. Asid di dalam sampel dianalisa menggunakan kromatografi gas dan teknik titratan untuk menentukan jenis dan kandungan asid. Sifat semulajadi zarah tanah liat peluntur akan dianalisa dengan menggunakan pembelauan sinar x, analisis termal gravimetrik, fourier transformer infrared, dan luas permukaan BET. Nikel asetat dengan ethylene glycol sebagai ejen penurunan dirawat untuk menyokong nikel: di atas dan ke dalam lapisan dalam tanah hat peluntur. Penjerapan Ni(II) telah disiasat menggunakan luas permukaan BET dan pembelauan sinar x-ray. Keputusan menunjukkan bahawa lengkung IC dan titratan memperoleh asid sulfurik sebagai asid dominan pada 0.0615 % berat. Spektrum FTIR dan XRD tanah hat peluntur mi serupa dengan mineral ilhite. Sampel mi mempunyai jarak kekisi 10.67 A dan mempunyai sifat-sifat kekisi Bravais yang primitif. Sebanyak -20% berat nikel boleh masuk di atas dan dalam lapisan tanah hat peluntur. Logam yang menyokong tanah liat terluntur mempunyai kapasiti beban yang rendah berbanding dengan tanah liat yang berinterkalasi. Walaubagaimanapun, diameter zarah dan luas permukaannya meningkat daripada 90 Å hingga 170 Å dan 600 m<sup>2</sup>/g hingga 800 m<sup>2</sup>/g apabila kapasiti logam meningkat. Kereaktifan Ni(II) yang menyokong tanah liat terluntur adalah rendah di mana peratusan asid lemak tepu meningkat sebanyak 1 % hingga *2.5* % dalam tindak bahas penghidrogenan minyak sawit mentah pada keadaan bihik.

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# **LIST OF SYMBOLS**

 $\sim$ 

- $\mu$  Micro
- M Molar  $\sim$   $\sim$
- $\alpha$  Degree of Reduction
- $\theta$  Theta
- $\AA$  Armstrong (×10<sup>-10</sup>)
- d Spacing
- K Shape factor
- $\lambda$  X-ray Wavelength
- $\beta$  Line Broadening at Half Maximum of Peak in Radian
- I Intensity

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### **LIST OF ABBREVIATIONS**

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#### **CHAPTER 1**

#### **INTRODUCTION**

#### **1.1 INTRODUCTION**

Mineral can be described as an element or chemical compound that is normally crystalline and that has been formed as a result of geological processes. Mineral is a naturally occurring solid chemical substance with specific order of structure orientation and has its own chemical and physical characteristics. There are currently more than 4,000 known minerals in the world. However, it can be said that perhaps 100 of it can be called "common", 50 are "occasional", and the rest are "rare" to "extremely rare".

Clay is one of the common mineral. It can be classified primarily as ultrafmegrained mineral which are less than 2 micrometres in size on standard particle size classifications. So, it required special analytical techniques. Clay typically formed over long period of time by the gradual chemical weathering of rocks. The two main processes may involve slight physical and chemical alteration or decomposition and recrystallization. It is the result of the reaction between rocks, low concentration of carbonic acid and also some acidic solvents. These acidic solvents migrate through weathering rock after leaching through upper weathered layers. Some of clays formed are through hydrothermal activity. By comparison, a rock is an aggregate of minerals and/or mjneralojds and does not have a specific chemical composition. Minerals range in Composition from pure elements and simple salts to very complex silicates with thousands of known forms.

# **1.2 CLAY STRUCTURES**

Clay minerals are crystalline hydrated aluminosilicates  $(Al_2(SiO_3))$  and normally contain considerable amounts of transition metals, alkaline earth, alkaline metals and various type of cations. Generally speaking, two structural units are involved in the assembly of the crystalline lattice in clay minerals of the phyllosilicate group. The former is composed of oxygen atoms and hydroxyl groups in octahedral coordination, therefore alternating positions in relation to the corners of a geometric arrangement whose center is occupied by an aluminum atom, whereas the latter is arranged in tetrahedral coordination with a silicon atom at the center and oxygen atoms at the corners. In both cases, layered structures are formed in which every sheet is defined by the atomic plan that links the isolated elemental units, arranged either in octahedral or tetrahedral coordination. By superimposing several sheets, layers are formed that, separated by the interlayer space (space between the layers), constitute their structural repeating characteristic units.



**Figure 1.1:** A=1:1 clay and B=2:1 clay

Clay structure is commonly referred to 1:1 or 2:1 (Figure 1.1). These ratios referred to the layer of the clay. Those ratios are derived from the way in which these layers are built. Normally, 1:1 clay consists of one tetrahedral sheet and one octahedral sheet (A). While 2:1 clay consist of two tetrahedral sheets at the outer layer sandwiches with one octahedral sheet (B). Further explanation will be explained in the next subtopic by using the types of clay as example.

#### **1.3 TYPES OF CLAY**

Types of clay basically can be determined by the types of minerals and acidity of leaching water. There are several types of clay which are kaolinite, smectite, illite and chlorite.

**Table 1.1:** Types of clays

Group	<b>Chemical Composition</b>	<b>Layer Type</b>
Kaolinite	$[Si_4]A1_4O_{10}(OH)_8. nH_2O$ (n= 0 or 4)	1:1
<b>Illite</b>	$M_x[Si_{6.8}Al_{1.2}]Al_3Fe.025Mg_{0.7}5O_{20}(OH)_4$	2:1
<b>Smectite</b>	$M_x[S_{18}]Al_{3.2}Fe_{0.2}Mg_{0.6}O_{20}(OH)_4$	2:1
<b>Chlorite</b>	$(AI(OH)_{2.55})4[Si_{6.8}Al0_{1.2}\}Al_{3.4}Mg_{0.6})_{20}(OH)_{4}$	2:1:1

#### **1.3.1 Kaolinite**

Kaolinite basically have a chemical composition of  $Al_2Si_2O_5(OH)_4$ . Rocks that are rich in kaolinite are known as china clay, white clay, or kaolin. Kaolinite is a dioctahedral, two layered sheet of aluminosilicate of the 1:1 type in which the constituent layer is composed of two distinct layer surfaces. From one side, the layer face resembles the structure of Al(OH)<sub>3</sub>, with aluminum atoms coordinated Octahedrically with oxygen and hydroxyl groups. On the other hand, the opposite surface Of the layer resembles the structure of silica, where silicon atoms are coordinated tetrahedrically with oxygen atoms of the lattice. Therefore, hydroxyl groups are exposed at one side of the layer (Al side), whereas the other is decorated by oxygen atoms (Si side). Adjacent layers are linked to one another by hydrogen bonding involving aluminol (Al-OH) and siloxane (Si-0) groups. A very high cohesion results from this array of hydrogen bonding among kaolinite layers, and this definitely hinders both intercalation and functionalization reactions.



**Figure 1.2:** Structure of Kaolinite

#### **1.3.2 Smectite**

Smectite is a member of montmorillonite group. Most Smectite clays have Montmorillonite properties. It has the same chemical composition and structure as montmorillonite. The Montmorillonite name was actually given to Smectite clay that was found in the Montmorillon area of France. Clays are often named after their location. Smectite is a single layer consists of two sheets of silicon atoms linked tetrahedrically to oxygen atoms that belong to an intermediate aluminum sheet making it into three-layered sheets. This way, both layer surfaces are decorated with oxygen atoms in two-surface oxygen planes. As a result, there is a relatively weak interaction among layer, conferring anisotropic properties to the layered material and, as such, a relatively higher susceptibility to swelling and cleavage.

### 1.3.3 **Lllite**

Illite is a non-expanding mineral. It has 2:1 structure. The interlayer space is mainly occupied by poorly hydrated potassium cations responsible for the absence of swelling. Its structure is constituted by the repetition of tetrahedron - octahedron tetrahedron (TOT) layers. Structurally illite is quite similar to MMT with slightly more silicon, magnesium, iron, and water and slightly less tetrahedral aluminium and interlayer potassium. Chemical formula is given as  $(K,H_3O)(A1,Mg,Fe)_2(Si,Al)_4O_{10}[(OH)_2,(H_2O)].$ 



**Figure 1.3:** Structure of illite

#### **1.3.4 Chlorite**

Chlorite has a general formula of  $(Mg,Fe)_{3}(Si,Al)_{4}O_{10}(OH)_{2} \cdot (Mg,Fe)_{3}(OH)_{6}$ . Chlorites have a 2:1 sandwich structure (tetrahedral-octahedral-tetrahedral =  $t$ -o- $t$ ...). Unlike other 2:1 clay minerals, a chlorite's interlayer space in which the space between each 2:1 sandwich filled by a cation is composed of  $(Mg^{2+}, Fe^{3+})(OH)_6$ . This  $(Mg^{2+},$  $Fe^{3+}$ )(OH)<sub>6</sub> unit is more commonly referred to as the brucite-like layer, due to its closer resemblance to the mineral brucite  $(Mg(OH)_2)$ . Therefore, chlorite's structure appears as

[-t-o-t-brucite-t-o-t-brucite ...]. That is why chlorites are not always considered as clay but only classified as a separate group within the phyllosilicate.

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# **1.4 pIIYLLOSILICATE**

Phyllosilicate is a subgroup of silicate minerals. Silicate minerals can be defined as the components that make up the largest and most important class of rock-forming minerals including clay. They are classified based on the structure of their silicate group. Silicate minerals all contain silicon and oxygen. There are several types of silicate minerals which are:





 $\mathcal{F}_{\text{c},k}$ 

### **1.5 USES AND MODIFICATION OF CLAYS**

Clays has been used since prehistoric time. Usually as drinking vessel made of sun-dried clay. It is because clay exhibit plasticity when mixed with water in certain proportions. When dried, clay becomes firm and when fired, permanent physical and chemical changes occur. These reactions cause the clay to be converted into a ceramic material. Clay is also used for making pottery items, both utilitarian and decorative. Different types of clay, when used with different minerals and firing conditions, are used to produce earthenware, stoneware, and porcelain.

Modification of clay has been practiced globally especially in the chemical and oil industries. Organoclays, which have the metals in the clay replaced by large surfactant cations, such as long chain alkyl amine cations can be been used as liners in landfills to reduce transport of contaminants. Organoclays also could be used in wastewater treatment and spill control situations.

#### **1.6 BLEACHING** CLAY

Bleaching clay is clay which is highly adsorptive and consists mainly of hydrated aluminium silicates. It is used as bleaching agent, and as an adsorbent in decolorizing and refining oils and fats. Bleaching clay is a mineral substance characterized by the ability of absorbing basic colors and removing them from oils. It is composed mainly of alumina, silica, iron oxides, lime, magnesia, and water, in extremely variable proportions and is generally classified as sedimentary clay. In color it may be whitish, buff, brown, green, olive, or blue. It is semiplastic or nonplastiç and may or may not disintegrate easily in water. It was originally used in the fulling of wool to remove oil and grease but **IS** now used chiefly in bleaching and clarifying petroleum and secondarily in refining edible oils. Bleaching clay is mined in many parts of the United States. However Germany and India are the leading producers. Before it can be used, it has to be crushed and dried. In general Bleaching Earth is a decolourising agent which will change the tint of any coloured oil to a lighter shade by changing the basic colour units in oil, without

altering the chemical properties of the oil. Bleaching Earths are special clays activated by physical or chemical processes. Natural clays are found in special strata and are subjected to various processes such as desegregation in water, treatment by sulphuric acid solutions, filtration, baking, grinding, etc. Efficiency of bleaching earth depends on selecting the right grade and proper blends of the basic raw material, bentonite obtained from various mines. Hudson MPA Sdn Bhd is the leading supplier of various bleaching clay produced in India.

### **1.7 USES OF MODIFIED CLAY**

Intercalated bleaching clay was used as catalyst to drive certain reaction. Usually in hydrogenation reaction. Hydrogenation of aromatic arene such as benzene, naphthalene and anthracene also were found to be catalyzed under milder reaction condition to give monocyclic arenes. Other application is hydrogenation of palm oil into less unsaturated fatty acid.

# **1.8 PROBLEM STATEMENTS**

Modification of clay can give useful minerals mainly in chemical and oil processing. Much modification has been done by intercalating specific cationic particles into clay structure. By doing this, chemical and physical properties of clay can be , changed to suit the properties needed. However, research in this field is still in development stage and has not widely applied to industry. In order to intensify chemical processes, clay modification plays an essential role to achieve high yield, process efficiency, and cost reduction.

### **1.9 OBJECTIVES OF STUDY**

The objectives of this research are:

- 1. Prepare bleaching clay supported with nickel particles.
- 2. Characterize nickel particles supported on bleaching clay.
- 3. Study the use of nickel particles supported on bleaching clay as catalyst for hydrogenation of crude palm oil.

#### **CHAPTER 2**

#### **LITERATURE REVIEW**

#### **2.1 CLAYS AND ITS STRUCTURE**

The definition of "clay" was first formalized in 1546 by Agricola. It has been revised many times since, although the fundamentals involving plasticity, particle size, and hardening on firing were retained by most. The term "clay" refers 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. Although clay usually contains phyllosilicates, it may contain other materials that impart plasticity and hardeness when dried or fired. Associated phases in clay may include materials that do not impart plasticity and organic matter (Guggenheim and Martin, 1995).

Clays are primarily inorganic materials excluding peat, muck and some soils that contain large quantities of organic materials. Associated phases, such as organic phases, may be present. "Plasticity" refers to the ability of the material to be molded to any shape. The "fine-grained" aspect cannot be quantified, because a specific particle size is not a property that is universally accepted by all disciplines. For example, most geologists and soil scientists use particle size less than 2  $\mu$ m, sedimentologists use 4  $\mu$ m, and colloid chemists use  $1\mu$ m for clay-particle size. Sedimentologists may use the term "Clay" also to denote grain size only. It is more precise, however, to give the actual dimensions of the particles, e.g., particles less than 4  $\mu$ m (Guggenheim and Martin, 1995).

Recent research has shown that nanocomposites can be synthesized from a smectite mineral (Ishida et al., 2000), montmorillonite layered silicate, with a variety of polymeric host matrixes. Horch et al. (2002), said that montmorillonite has a 2:1 layered structure with a single layer of aluminum octahedral between two layers of silicon tetrahedral. Montmorillonites refer to hydrous aluminum silicates approximately represented by the formula (Na, Ca)(Al, Mg)<sub>6</sub>(Si4O10)<sub>3</sub>-(OH)<sub>6</sub>.nH2O. The Al<sup>3+</sup> or Si<sup>4+</sup> locations can be replaced by lower valent cations, which cause the montmori!lonite structure to have an excess of electrons. This negative charge is satisfied by loosely held cations from the associated water.



**Figure 2.1:** Cross-sectional model of MMT

Source: Horch et al. (2002)

Figure 2.1 above described about the cross-sectional model of the montmorillonite layered silicate platelet. This arrangement of atoms, called a smectite structure, consists Of two inward-facing layers of silicate tetrahedra (light blue) that flank a layer of alumina octahedra (dark blue). The entire platelet is between 7 and 10 Å (about 1 nm) thick, hence the name "nanoclay"(Horch et al., 2002).

# 2.2 **CLAY MODIFICATION**

Smectites are widely used in a range of applications because of their high cation exchange capacity, swelling capacity, high surface areas, and consequential strong adsorption/absorption capacities (Aither, 2000, 2003; Breen et al., 1997; Dentel et al., *1995* and He et al., 2001). According to Xi et al. (2005), among the swelling clays, the most common dioctahedral smectite is montmorillonite, which has two siloxane tetrahedral sheets sandwiching an aluminum octahedral sheet. Because of an isomorphic substitution within the layers (for example,  $Al^{3+}$  replaced by  $Mg^{2+}$  or  $Fe^{2+}$  in the octahedral sheet and  $Si^{4+}$  replaced by  $Al^{3+}$  in the tetrahedral sheet), the clay layer is negatively charged, which is counterbalanced by exchangeable cations such as  $Na<sup>+</sup>$  and  $Ca^{2+}$  in the interlayer.

The hydration of inorganic cations on the exchange sites causes the clay mineral surface to be hydrophilic. Thus, natural clays are ineffective sorbents for organic compounds (Kim et al., 2003 and He et al., 2004). However, such a difficulty can be overcome by ion exchange of the inorganic cations with organic cations.

Organomontmori!lonites are synthesized by intercalating cationic surfactants such as quaternary ammonium compounds into the interlayer space through ion exchange (He et al., 2004; Morfiand et al, 1986 and Soule et al. 2001).When using longchain alkylammonium cations, a hydrophobic partition medium within the clay interlayer can form and function analogously to a bulk organic phase. The interlayer height of clay before modification is relatively small, and the interlayer environment is then hydrophilic.

Xi et al. (2005) said, the intercalation of a cationic surfactant not only changes the surface properties from hydrophilic to hydrophobic but also greatly increases the basal spacing of the layers. Such surface-property changes effect the applications of the organoclay. In particular, the hydrophobic nature of the organoclay implies that the material can be used as a filter material for water purification.

On the other hand, Fusova et al. (2009) said, organic molecules not only adsorb on surficial exchangeable positions but also intercalate into interlayer space (between 2:1 layers) of the structure of smectite during intensive saturation of smectites. Intercalation into this guest structure can either induce a ion exchange reaction, when organic molecules are intercalated as cations, or are intercalated as neutral organic molecules and the original cations stay partly or completely in the interlayer of the smectite.

The clay minerals of smectite groups occur in the form of very small crystals with frequently defective structure. Theories about their structural constitution come from the hypothesis that their structure is compounded of 2:1 layers, between which are exchangeable hydrated ions. (The 2:1 layer is created when two tetrahedral sheets (with opposite polarity) are connected with one octahedral sheet in the middle. This latticed structure may lightly expand and bond molecules on the exterior and also interior) (Fusova et al., 2009).

Water molecules in the interlayer space of smectites and vermiculites can be displaced by many polar organic molecules. Neutral organic ligands can form complexes with the interlayer cations. The interlayer cations can be replaced by various types of organic cations. Alkylammonium ions are widely used in modifying bentonites. The other important group of organic compounds is cationic dyes and cationic complexes (Bergaya et al., 2006).

One of the most significant and widespread minerals of dioctahedral smectite is montmorillonite. It is characterized by no or very small substitution of  $Al^{3+}$  for  $Si^{4+}$  on the tetrahedron position. The charge of the 2:1 layers is produced primarily by octahedron substitution. Variability in chemical composition leads to their division into types: Wyoming, Chambers, Otay, Fe-montmorillonite. Monovalent (especially  $Na<sup>+</sup>, K<sup>+</sup>$ ) and divalent cations could appear in the interlayer of smectite. These compensate the negative charge of 2:1 layers (Fusova et al., 2009).

Bentonite is a, soft, very fine-grained heterogeneous rock of various colours, composed mostly of clay mineral montmorillonite, which originated mostly by submarine or atmospheric weathering of basic (to a smaller extent also of acid) volcanic rocks (mainly tuffs). Montmorillonite gives to bentonite its typical properties  $-$  high sorption capacity, characterized by a high value of cation exchange which is the ability to receive certain cations from solutions, and replace them with its own molecules  $-Mg$ , and in some cases also Ca and alkalis; internal swelling after contact with water (some bentonites do not swell, but have a high absorptive capacity as bleaching clays, especially when they are activated); high plasticity and binding ability. Bentonite also contains other clay minerals (kaolinite, illite, beidellite), Fe compounds, quartz, feldspars, volcanic glass, etc., which represent impurities and if possible they are removed during the mineral processing (Fusova et al., 2009).

Bentonites may be principally divided into:

- I. highly swelling Na-bentonites, the so-called Wyoming-type bentonites. Deposits of this raw material are found predominantly in the USA; in the Czech Republic, no Na-bentonite deposits have been found,
- 2. less swelling potassium, calcium and magnesium bentonites or their combinations. Such bentonites may be industrially enriched with sodium by the so-called activation, the swelling capacities of activated bentonites, however, still do not reach such values as those of Wyoming-type bentonites.

# 2.3 **INTERCALATION**

The adsorption of neutral molecules on smectites is driven by various chemical interactions: hydrogen bonds, ion-dipole interaction, co-ordination bonds, acid-base reactions, charge-transfer, and van der Waals forces. Polar molecules, such as alcohols, amines, amides, ketones, aldehydes, and nitriles, form intercalation complexes with smectites. Even acids are intercalated. Guest compounds can be intercalated from the vapour, liquid, and solid state. When intercalated from solutions, solvent molecules are generally co-adsorbed in the interlayer space.

Guest molecules may be intercalated in dried clay minerals or may displace the water molecules of hydrated smectites and vermiculites. The displacement of interlayer water molecules depends on the HSAB (hard and soft acids and bases) character of the interlayer cations and the interacting groups of the guest molecules. Water molecules around hard cations, like Na<sup>+</sup>, Mg<sup>2+</sup> and Ca<sup>2+</sup>, are only displaced by HO– or O= containing compounds but not by amines. In contrast, amines as soft bases displace water molecules from soft interlayer cations like  $Cu^{2+}$  and  $Zn^{2+}$  (Bergaya et al., 2006). Intercalation is achieved by three methods:

- 1. Reaction between smectite (solid phase) and a solution in which organic substance is included. This reaction is called "solid-liquid",
- 2. Reaction between smectite (solid phase) and steams of organic substance, which is called "solid-gas",
- 3. Reaction between smectite (solid phase) and organic substance, which is also in solid phase. This is called "solid-solid".

Sodium montmorillonite refers to the clay mineral in which the loosely held cation is the  $Na<sup>+</sup>$  ion (Burgoyne et al., 1986). The individual montmorillonite platelets exist as coordinated layers that measure 1 nm thick and several microns wide. The layers are Periodically spaced apart with a repeating distance in the nanometer range, known as the gallery spacing. Depending on the cation exchange capacity of the clay, the hydration of