



# UNIVERSITI TEKNIKAL MALAYSIA MELAKA

## BORANG PENGESAHAN STATUS TESIS\*

JUDUL: EFFECT OF NANOFILLER (PRISTINE MONTMORILLONITE AND HYDROTALCITE LAMELLAR CLAYS) OF CORROSION ACTIVITIES ON PAINT

SESI PENGAJIAN: 2008/09

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## APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering of UTeM as a partial fulfilment of the requirements for the degree of Bachelor of Manufacturing Engineering (Engineering Materials) with Honours. The member of the supervisory committed is as follow:

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## DECLARATION

I hereby, declared this report entitled effect of nanofiller (pristine montmorillonite and hydrotalcite lamellar clays) of corrosion activities on paint is the results of my own research except as cited in references.

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## ABSTRACT

This report represents a study on corrosion activities of nanofiller additives paint when compared to pristine epoxy paint. In this study, pristine montmorillonite and hydrotalcite lamellar is chosen as the nanofiller additives. The specimen is prepared by mixing of resin, curing agent, and nanofiller in ratio of 10 to one in term of weight. The shape of pellet form specimen is obtained by pouring the mixture into PVC pipe and left for curing for three (3) days under room temperature. The prepared samples are studied by using Tafel extrapolation plot under different solution (3.5 wt% of NaCl, 2 wt% HCl and 2 wt% NaOH) that provided different concentration of pH. Morphology studies are conducted by using Scanning Electron Microscope (SEM) and Energy-dispersive X-ray spectroscopy (EDX) under various magnifications. Result of study after calculation of corrosion penetration rate shows that the corrosion rate of epoxy – montmorillonite paint is lower than pristine epoxy paint. This shows that the additives of montmorillonite nanofiller will enhance the corrosion resistivity and service lifetime of paint. As for epoxy – hydrotalcite paint, the corrosion penetration rate shows a growing trend but this had been explained by the properties of hydrotalcite ion exchangeability to release of ions when contact with aggressive ions and suppress the attack of aggressive ions. EDX scanning indicates the participation of elements and proves the in-participation of foreign particles. The molecular vacancy observed in epoxy – hydrotalcite paint SEM micrograph after various testing conditions had been stated as no further references can be found.

## ABSTRAK

Laporan ini adalah berlandaskan kepada satu kajian berkaitan dengan kelakuan kakisan cat yang ditambah dengan pengisi apabila dibanding dengan cat epoxy tulin. Dalam kajian ini, “montmorillonite” dan “hydrotalcite” terpilih sebagai pengisi. Bahan salutan disediakan dengan campuran resin, bahan pelekat, and bahan pengisi dalam bahagian 10 kepada satu (1) keberatannya. Sampel ujian berbentuk palet didapati dengan tuangka campuran itu kepada paip PVC dan diletakkan sepanjang tiga (3) hari dalam suhu bilik untuk pengawetan. Sampel yang disediakan digunakan untuk kajian Tafel dalam keadaan kajian yang berlainan (3.5 wt% of NaCl, 2 wt% HCl and 2 wt% NaOH) yang akan memberikan kepekatan pH yang berlainan. Kajian morfologi akan dijalankan dengan menggunakan ‘Scanning Electron Microscope (SEM)’ dan juga ‘Energy-dispersive X-ray spectroscopy (EDX)’. Keputusan kajian menunjukkan kadar kakisan untuk sampel cat ‘epoxy – montmorillonite’ adalah lebih rendah dibanding dengan cat ‘epoxy’ tulin. Ini menunjukkan penambahan ‘montmorillonite’ akan meningkatkan ketahanan kakisan dan juga tempoh kegunaan cat. Untuk cat ‘epoxy – hydrotalcite’ pula, keputusan kadar kakisan menunjukkan peningkatan dibanding dengan cat ‘epoxy’ tulin. Perjumpaan ini boleh dijelaskan dengan sifat ‘hydrotalcite’ yang akan menyebarkan ion apabila bersentuhan dengan penyerangan ion dan akan tinda serangan ion. Keputusan EDS menunjukkan kewujudan elements dalam sampel dan tiada kehadiran zarah luaran. Kehilangan zarah yang dijumpai dari SEM permukaan sampel cat ‘epoxy – hydrotalcite’ dalam pelbagai keadaan kajian telah dicatatkan, tiada rujukan lain yang boleh menjelaskan perjumpaan itu.

## **DEDICATION**

*To my beloved father, mother, family and friends.*

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## LIST OF ABBREVIATIONS

AC	-	Alternating Current
ASTM	-	American Society for Testing and Materials
Ce	-	Cerium
$E_{\text{corr}}$	-	Corrosion Potential
EDX	-	Energy-dispersive X-ray spectroscopy
EIS	-	Electrochemical Impedance Spectroscopy
Emf	-	Electromotive force series
HCl	-	Hydrogen Chloride
$I_{\text{corr}}$	-	Corrosion Current
$i_{\text{corr}}$	-	Corrosion Current Density
K	-	Potassium
KHz	-	Kilo-Hertz
M	-	Moles
Mg	-	Magnesium
MMT	-	Montmorillonite
mV	-	Mili-volt
NaCl	-	Sodium Chloride
NaOH	-	Sodium Hydroxide
PPS	-	Polyphenylenesulfite
PVC	-	Polyvinyl Chloride
SEM	-	Scanning Electron Microscope
EDX	-	Energy-dispersive X-ray spectroscopy

# CHAPTER 1

## INTRODUCTION

### 1.1 Background

Corrosion may be defined as destructive phenomena, chemical or electrochemical, which affects the aesthetic appeal or an object, and in extreme cases may cause structural failure. According to Uhlig, “Corrosion is the destructive attack of a metal by chemical or electrochemical reaction with its environment” (Mario, 2008). It takes place when materials are converted to corrosion products, such as oxides, which are thermodynamically more stable, the process can occur very slowly or extremely rapidly, which is usually the critical parameter to be sought. Corrosion occurs not only on metallic materials but also non-metals like ceramics, plastics, and rubber. Corrosion processes are differentiated by the reaction mechanisms of metal-medium interaction, the corroding medium, the type of corrosion failure on the surface, and the character of additional effects the material is subjected to simultaneously with attack of corrosion medium. There are several types of corruptions including the uniform, galvanic, pitting, intergranular, selective leaching, erosion, and stress corrosion (Tushinsky et al., 2002). Corrosion has shown their detrimental effect like cause damages to appearance, contamination, liability or even loss of the valuable products, high maintenance and operating costs, waste annually, plant shutdown by corrosion failure and their dreadful effects on safety and reliability.

Corrosion was first drawn the attention from worldwide by Uhlig on year 1949, who estimated the total cost to the economy by summing materials and procedures related to corrosion control. Followed in the 1970s by a number of studies in various countries

such as United State, United Kingdom, and Japan, the corrosion prevention has become the objective of scientist for the ages.

To prevent the corrosion occurrence, corrosion scientists applied the coating technique on the product to dissolve in preference to it or as a physical barrier between the electrolytes and the electrodes. It is a cost effective corrosion protection method which act as a barrier to a corrosion solution. Coating prevents or retards the transfer of electrochemical charge from the solution to the metal underneath the organic coating (Light Truck Frame Project Team, 1999).

Clay nanofillers are a new generation of processed clays of interest in wide range of high performance composites, which is defined as clay having nanometre thick platelets. Natural smectite clays, particularly montmorillonite, have been the first choice for producing nanoclays, due to their availability, easy extraction, and relatively low cost. The heterogeneity of natural clay can be overcome by using the synthetic clays such as hydrotalcite (Hickman, 2002)

Montmorillonite  $\text{Na}_{0.2}\text{Ca}_{0.1}\text{Al}_2\text{Si}_4\text{O}_{10}(\text{OH})_2(\text{H}_2\text{O})_{10}$  was discovered in 1847 in Montmorillon. It is a very soft phyllosilicate mineral group that typically forms in microscopic crystals, forming 2:1 clay. The Montmorillonite minerals are composed of hydrous aluminium silicates in the form of extremely small particles. They take up water between their layers, causing swelling and change the interlayer spacing according to the mineral variety. In addition to being involved in organic exchange reactions, they react with and absorb some organic liquid.

Hydrotalcite or layered double hydrotalcite is a magnesium hydrate mineral with general formula  $(\text{Mg}_6\text{Al}_2(\text{CO}_3)(\text{OH})_{16} \cdot 4(\text{H}_2\text{O}))$ . Its name is derived from its resemblance with talc and its high water content. The structures of hydrotalcite are made up by hydroxide layers with water and anions sandwiched between them. Interest in hydrotalcites and derived materials arises from the wide applications like catalyst supports, processing of selective chemical nanoreactors, separation and membrane technology (Nalwa, 2004).

## **1.2 Problem Statement**

It is clear that material replacement is a better long-term choice than alternate coating technologies current available but the time and cost consumed for the invention are very high. The existence of nanofiller usage in coating had been highly significant for industry for many years. Yet, the understanding towards the formulated nanofiller coating is very limited (Anonymous 1, 2006). Previous researchs through montmorillonite and hydrotalcite nanofiller clays have generally shows improvement of corrosion resistance through the additives. Davood Zairei has conducted a research through the comparison between pure polymer and with nanofiller additives polymer coating and it shows that additives of filler will eventually increase the corrosion protection. Toshifumi Sugama has investigated the effect of montmorillonite filled polyphenylenesulfied nanocomposites coating on corrosion protection, from 0% nanofilled to 14% of montmorillonite nanoclay filled and the result indicates the proper amount of MMT nanofiller will upgrade the corrosion protection. As for Jian Long and his group, a comparison between Mg-Ce hydrotalcite film, Ce conventional film, and bare Mg alloy has been done. As the result shows the Mg-Ce hydrotalcite film has the better corrosion resistance. For all the research mentioned above, none of them shows the polarization of the material using Tafel extrapolation method and comparison between the performances of these selected nanofillers have never been done or the information are not published. Montmorillonite as one of the most commonly used nanofiller in the field can be used as a comparison to hydrotalcite nanofiller to show the rate of corrosion activities of the material.

## **1.3 Objectives**

1. To study the effect of different nanofiller additives on corrosion activities by Tafel extrapolation method.
2. To study the electrochemistry of nanofiller additives paint.
3. To compare the corrosion resistance of paint while applying different nanofillers.

## **1.4 Scope**

The research project will mainly focus on the study of corrosion activities of the nanofiller additives paint material. Two different nanofillers will be applied in the paint. The study of their performances toward corrosive environment will be the main concern in the report. Part of their electrochemistry will also be concerned as one of the study objectives. A few laboratory works such as Tafel corrosion test will be done as the need of the study. The research will be conducted in normal corrosive environment, and also in acidic and alkaline solution in different pH conditions.

## **CHAPTER 2**

### **LITERATURE REVIEW**

#### **2.1 Nanofillers**

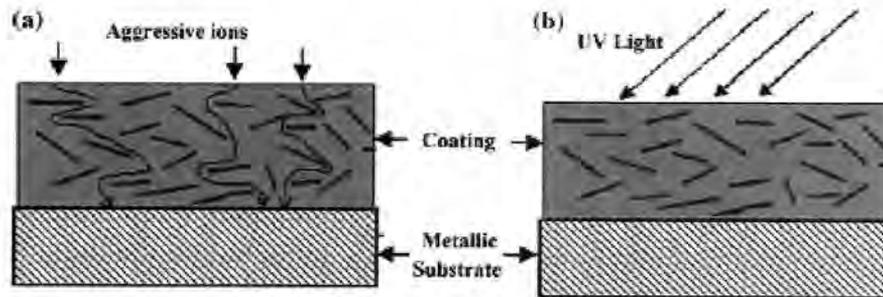
Nanofillers are nanosize particles that enhance polymer properties such as strength and tensile strength. There are different types of nanofillers such as inorganic particles, clay particles, graphite platelets, natural fibres, and carbon nanotubes. Nanoclays fillers offer less electrical and thermal conductivity but more economical cost. The most commonly used nanofillers are a nanoclays material, montmorillonite, which is layered smectite clay (Anonymous 2, 2008). Nanofillers have for many years had a high significance in the industry. Nanofillers are basically understood to be in solid form, which differ from polymer matrix in terms of their composition and structure. Inactive fillers or extenders raise the quantity and lower the price, while active fillers bring about targeted improvements in certain mechanical or physical properties. The activity of active fillers may have a variety of causes, such as the formation of a chemical bond or filling of a certain volume and disruption of the confirmation position of a polymer matrix, and also the immobilization of adjacent molecule groups and possible orientation of the polymer material (anonymous 1, 2006).

The presence of the clay in nanoscale dispersion lengthens the diffusion path of corrosive media and improves barrier properties. Consequently, the lifetime of the conductive polymer coatings can be significantly improved by incorporation of the clay. It is believed that, due to strong homogeneous impervious layers, the good corrosion protection properties of coating prevent corrosion media from seeping through it. As a

result, clay-based nanocomposite coatings may be considered energy-saving and environment-friendly materials (Davood et. al., 1998).

**- Corrosion resistivity of nanocomposite coatings based on conductive polymers and layered silicates**

It is believed that the presence of layered silicates improved the corrosion resistance of the ICP coatings, not only with decreasing the coating porosity and decreasing permeability of gases and liquids, but also with stabilizing their electronic structure (Zhu, 2003). Recently, researchers has found that incorporation of the inorganic nano-layers of MMT clay into the polymeric matrix effectively enhance the corrosion protection of pristine conducting polymers. As shown in figure 2.1, the presence of clay in nano-scale dispersion lengthens the diffusion path of corrosive media and improves the barrier properties. Clay also has a possible positive effect on protecting specimen from UV damage. In addition, clay platelets will improve the thermal stability.



**Figure 2.1:** Possible advantages of clay in coating.

**- Corrosion resistance properties of nanocomposites based on nonconductive polymer-layered silicates**

The corrosion resistance of polyphenylenesulfite (PPS)/MMT clay nanocomposite coating have also been studied. It was founded that MMT-PPS nanocomposite coating was effective in justifying corrosion compared with MMT-free coating. Recently, the effect of using the organic-philic MMT additives on the properties of epoxy coating has been evaluated. It was concluded that the additive results in improved coating properties such as adhesion, hardness, elasticity, and corrosion resistance (Garea et. al., 2006). In

nonconductive polymeric nanocomposites, the additional of a small amount of suitable grade of clay increases the corrosion resistance and better protection of substrates in different conditions.

## 2.2 Montmorillonite

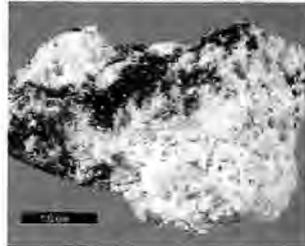


Figure 2.2: Montmorillonite image

Montmorillonite presented in figure 2.2 is a very soft phyllosilicate class silicates mineral that forms in microscopic crystals, forming clay. It is one of the members of the smectite family, 2:1 clay, meaning that it has 2 tetrahedral sheets sandwiching a central octahedral sheet as described in figure 2.3. The particles are plate-shaped with an average diameter of approximately 1 micrometer. Its water content is variable and it increases greatly in volume when it absorbs water. As presented in table 2.1, chemically it is hydrated sodium calcium aluminium magnesium silicate hydroxide  $(\text{Na,Ca})_{0.33}(\text{Al,Mg})_2(\text{Si}_4\text{O}_{10})(\text{OH})_2 \cdot n\text{H}_2\text{O}$ . Potassium, iron, and other cations are common substitutes; the exact ratio of cations varies with source.

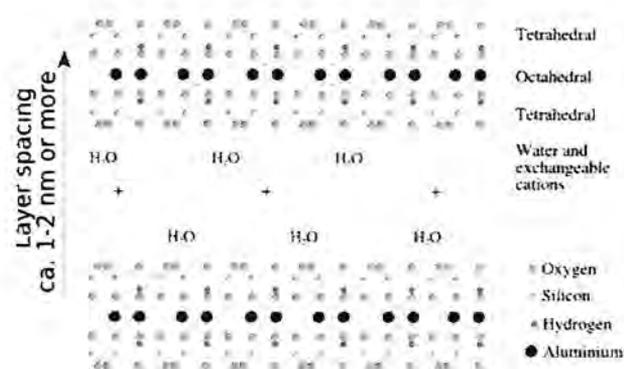


Figure 2.3: Structure of Montmorillonite.

Similar to other clays, montmorillonite swells with the addition of water. However, some montmorillonite expand considerably more than other clays due to water penetrating the interlayer molecular spaces and concomitant adsorption. The amount of expansion is due largely to the type of exchangeable cations contained in the sample (Anonymous 3, 2008). In montmorillonite clay, both Bronsted and lewis acidic catalytic sites are available; hence their natural occurrences as well as its ion exchange properties allow it to function efficiently as a catalyst. The interlayer cations are exchangeable, thus allowing alteration of the acidic nature of the material by simple ion-exchange procedure.

**Table 2.1:** Montmorillonite nominal physical constant (Anonymous 4, 2008)

<u>Molecular Weight</u> (g/mol.)	540.46
<u>Average Density</u> (g/cm <sup>3</sup> )	2.35
<u>Crystal System</u>	Monoclinic
<u>Member Of</u>	Smectite group
<u>Fracture</u>	Uneven to lamellar
<u>Luster</u>	Earthy (dull)
<u>Mohs Hardness @20°C</u>	1.5- 2.0
<u>Average Specific Gravity</u> (g/cc)	2.3- 3
<u>Color</u>	White, yellow
<u>Cleavage</u>	Perfect in one direction, basal
<u>Characteristic</u>	Crystals expand to many times their volume when added to water
<u>Field Indicators</u>	Softness, and soapy feel

As a mineral specimen, montmorillonite usually doesn't get much consideration. Usually, pure samples of montmorillonite are massive, dull and not very attractive. However, as with all minerals, there are exceptional specimens that defy the standard. Montmorillonite has been found as attractive pink inclusion in quartz crystals, and these make for interesting specimens (Anonymous 5, galleries 2008). As smectite clay, montmorillonite may be used in the absence of any surface modifier as a microfiller in various non-polar polymers or as a nanofiller in polar polymer. More often, montmorillonite is used with polymers of low intermediate polarity as reinforcing nanofiller after treatment with organic modifiers; these increase the distance between the

individual silicates layers and decrease inter gallery forces, thus, facilitating wetting and exfoliation to nanoplates within thickness (Pukanszky et. al., 2005)

### 2.2.1 Montmorillonite Clay:

In terms of chemistry, clay minerals are hydrous aluminium phyllosilicates, sometimes with variable amount of iron, magnesium, alkali metals, alkaline earths and other cations. In geology fine-grained sediments with particle sizes of less than 0.0039mm in size may be classified as clays. The small particles of clay helps fills in the empty space of mixture and it tend to stick to each other usually trap moisture in them. The additional of clay can slow down the drainage of the mixture material.

Clay mineral are divided into 4 groups, they are; kaolinite, smectite, illite, and chlorite group. Montmorillonite are categories under smectite group. This group is composed of several minerals including pyrophyllite, talc, vermiculite, sauconite, saponite, nontronite and montmorillonite. They differ mostly in chemical content. The general formula is  $(Ca, Na, H)(Al, Mg, Fe, Zn)_2(Si, Al)_4O_{10}(OH)_2 - xH_2O$ , where x represents the variable amount of water that members of this group could contain. The structure of this group is composed of silicate layers sandwiching a gibbsite (or brucite) layer in between, in an s-g-s stacking sequence. The variable amounts of water molecular would lie between the s-g-s sandwiches. Smectite group is named with effectiveness at absorbing toxins and expandable properties (Anonymous 6, 2008).

#### - Montmorillonite clay nanocomposite coating and their efficacy against corrosion

Alternating current (AC) electrochemical impedance spectroscopy (EIS) was used to evaluate the ability of the exposed coating films to protect the steel from corrosion. The specimen were mounted in the holder, and then inserted into an electrochemical cell. Computer programs were prepared to calculate theoretical impedance spectra and to analyze the experimental data. Specimens with a surface area of 13cm<sup>2</sup> were exposed to an aerated 0.5M sodium chlorite electrolyte at room temperature, and single-sine