

CERTIFICATION OF APPROVAL

**CATIONIC EFFECTS ON THE MORPHOLOGY OF HYDROTALCITE**

By

VIVNESH KUMAR SANTHA MOORTHY

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Approved by,

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( Dr. Suriati Sufian )

UNIVERSITI TEKNOLOGI PETRONAS

TRONOH, PERAK

May 2013

## **CERTIFICATION OF ORIGINALITY**

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and the original work contained herein have not been undertaken or done by unspecified sources or persons.

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**VIVNESH KUMAR SANTHA MOORTHY**

## ABSTRACT

Hydrotalcites have been attracting a lot of interest from the researchers because of their wide possible applications such as CO<sub>2</sub> absorbent, ion exchangers, fire retardants, base catalysts and precursors for well-mixed oxides for various catalytic applications. When hydrotalcites are synthesized in different conditions, they can present different morphologies. The purpose of the research is to study the morphology differences of hydrotalcite which associate them with their optimum potential applications.

The first chapter will give a brief introduction on the background of the project. Besides, the problem statement of this project will be stated to address the main purpose and significance of conducting this research project. Apart from that, the objective of this research project and also the scope of study will also be highlighted in the following chapter.

A detailed literature review will be discussed in the following chapter. In this chapter, the concept and basic understanding of the project is shown. This chapter will highlight what hydrotalcite is and how it is formed.

Finally in the last chapter, the research methodology, the experimental approach and procedures as well as the key milestones will be presented.

## ACKNOWLEDGEMENT

In completion of this Final Year Project, I would like to express my deepest gratitude to all parties that involved in making this project as a meaningful and great achievement for me. Throughout the project timeframe, I have learned a lot of new things and gained so much experience that will be a huge value for me in the future.

First and foremost, I would like to thank UTP's Faculty of Chemical Engineering for providing me guidance and tools to complete the project. With their strong support and advice, I was able to plan and conduct my project very well.

Special thanks should go to my Final Year Project supervisors, Dr.Suriati Sufian and Phd student Mr. Mohammad Abdus Salam, the key peoples to teach and guide me in conducting this project. Their strong support and willingness to share their knowledge and wide expertise has given me a very important experience in this project study.

Besides that, I would like to thank the lab assistants who had helped me on workstation. I truly appreciate their help and their sharing of information in order for me to execute the project well.

Finally, my deepest appreciation goes to my family and my friends in UTP for their continuous support and encouragement which have enabled me to do my best for this project. I hope that after this project completion, all the findings and knowledge that I have shared through this dissertation would be useful for everyone in the study field.

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## CHAPTER 1

### PROJECT BACKGROUND

#### 1.1 Alternative Energy Sources

The world currently relies heavily on coal, oil, and natural gas for its energy. Fossil fuels are non-renewable, that is, they draw on finite resources that will eventually dwindle, becoming too expensive or too environmentally damaging to retrieve. In contrast, the many types of renewable energy resources-such as wind and solar energy-are constantly replenished and will never run out.

Most renewable energy comes either directly or indirectly from the sun. Sunlight, or solar energy, can be used directly for heating and lighting homes and other buildings, for generating electricity, and for hot water heating, solar cooling, and a variety of commercial and industrial uses. The sun's heat also drives the winds, whose energy, is captured with wind turbines. Then, the winds and the sun's heat cause water to evaporate. When this water vapor turns into rain or snow and flows downhill into rivers or streams, its energy can be captured using hydroelectric power.

Biomass can be used to produce electricity, transportation fuels, or chemicals. The use of biomass for any of these purposes is called bioenergy. Geothermal energy taps the Earth's internal heat for a variety of uses, including electric power production, and the heating and cooling of buildings. And the energy of the ocean's tides come from the gravitational pull of the moon and the sun upon the Earth. There is also energy from the ocean that we can harness to generate electricity. The tidal energy and temperature difference in ocean are some of the ways to create energy.

Hydrogen also can be found in many organic compounds, as well as water. It's the most abundant element on the Earth. But it doesn't occur naturally as a gas. It's always combined with other elements, such as with oxygen to make water. Once separated from another element, hydrogen can be burned as a fuel or converted into electricity.



## 1.2 Hydrogen Storage

Hydrogen gas is an ideal alternative fuel for the next generation. Hydrogen is considered an alternative fuel for two reasons: It is renewable, and it is the most abundant element on the earth. Hydrogen comprises more than 75 percent of the environment; so if it became a primary fuel, dependence on foreign sources of fuel would be eliminated. However, hydrogen in nature exists primarily in combination with other elements. For hydrogen to be useful as a fuel, it must exist as free hydrogen ( $H_2$ ). One common source of hydrogen is water, which is 11.2 percent hydrogen by weight (Kushnir, 2000).

Hydrogen storage describes the methods for storing hydrogen gas for subsequent use. The methods span many approaches, including high pressures, cryogenics, and chemical compounds that reversibly release hydrogen gas upon heating [5].

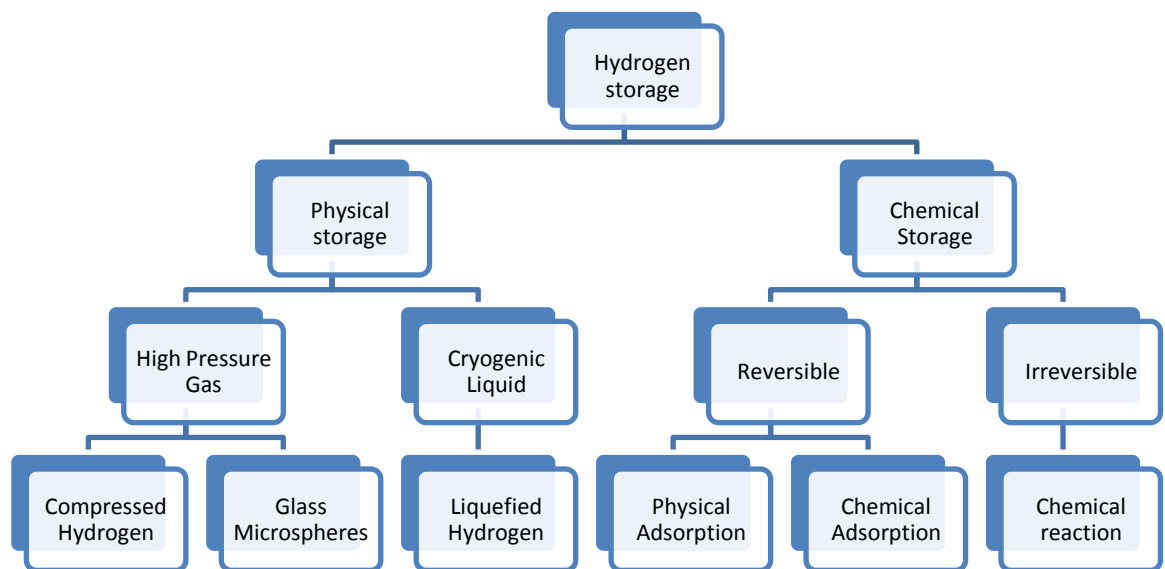


Figure 1: Types of Hydrogen Storage

### **1.2.1 Compressed Hydrogen**

Compressed hydrogen (CGH<sub>2</sub> or CGH<sub>2</sub>) is the gaseous state of the element hydrogen kept under pressure. Compressed hydrogen in hydrogen tanks at 350 bar (5,000 psi) and 700 bar (10,000 psi) is used for mobile hydrogen storage in hydrogen vehicles. It is used as a fuel gas commonly nowadays [10].

### **1.2.2 Glass Microspheres**

The basic concept for how glass micro spheres can be used to store hydrogen gas onboard a vehicle can be described by a three step process (charging, filling, and discharging)[11]. First, the hollow glass spheres are filled with H<sub>2</sub> at high pressure (350 - 700 bars) and high temperature (ca. 300°C) by permeation in a high pressure vessel. Next, the micro spheres are cooled down to room temperature and transferred to the low-pressure vehicle tank. Finally, the micro spheres are heated to (ca. 200-300°C) for controlled release of H<sub>2</sub> to run the vehicle. The main problem with glass micro spheres is the inherently low volumetric densities that can be achieved and the high pressures required for filling. The glass micro spheres slowly leak hydrogen at ambient temperatures.

### **1.2.3 Liquefied Nitrogen**

Liquid hydrogen (LH<sub>2</sub>) is the liquid state of the element hydrogen. Hydrogen is found naturally in the molecular H<sub>2</sub> form [12]. To exist as a liquid, H<sub>2</sub> must be pressurized above and cooled below hydrogen's Critical point. However, for hydrogen to be in a full liquid state without boiling off, it needs to be cooled to 20.28 K (-423.17 °F/-252.87°C) while still pressurized. One common method of obtaining liquid hydrogen involves a compressor resembling a jet engine in both appearance and principle. Liquid hydrogen is typically used as a concentrated form of hydrogen storage. As in any gas, storing it as liquid takes less space than storing it as a gas at

normal temperature and pressure. Once liquefied, it can be maintained as a liquid in pressurized and thermally insulated containers.

#### **1.2.4 Physical Adsorption**

Physical adsorption is a method or also known as physisorption is a method to require a very porous material that can maximize the contact surface area for the sorption of hydrogen [13]. Examples are materials such as polyethylene, Teflon, nanotubes and etc.

#### **1.2.5 Chemical Adsorption**

Chemical adsorption or chemisorption is a kind of adsorption which involves a chemical reaction between the surface and the adsorbate [14]. This type of method allows the hydrogen to be adsorbed directly into the material itself. For example, metal hydrides are used as a source for hydrogen storage.

#### **1.2.6 Chemical Reaction**

The chemical reaction route for hydrogen storage involves displacing chemical reactions for both hydrogen generation and hydrogen storage. For reactions that may be reversible on-board a vehicle, hydrogen generation and hydrogen storage take place by a simple reversal of the chemical reaction as a result of modest changes in the temperature and pressure. Sodium alanate-based complex metal hydrides are an example. In many cases, the hydrogen generation reaction is not reversible under modest temperature/pressure changes. Therefore, although hydrogen can be generated on-board the vehicle, getting hydrogen back into the starting material must be done off-board. Sodium borohydride is an example.

### 1.3 Hydrogen Storage Advantages and Disadvantages

The major advantage is that hydrogen stores approximately 2.8 times the energy per unit mass as gasoline. Hydrogen is the lightest element occurring in nature and contains a large amount of energy in its chemical bond. Because of its low density, liquid hydrogen weighs less than petroleum-based fuels. The density of gaseous hydrogen is 0.0899 grams per liter (g/l). Liquid hydrogen boils at -252.77 degrees Celsius, and it has a density of 70.99 g/l. With these properties, hydrogen has the highest energy-to-weight ratio of all fuels: 1 kilogram (kg) of hydrogen has the same amount of energy as 2.1 kg of natural gas or 2.8 kg of gasoline (Kushnir, 2000).

Besides that, hydrogen is the cleanest fuel available. Hydrogen-fueled vehicles and gas turbine engines have negligible emissions of air pollutants. Hydrogen-powered-fuel-cell vehicles have zero emissions. On the other hand, platforms powered by petroleum-based fuels emit significant amounts of air pollutants (hydrocarbons, carbon monoxide, nitrogen oxides, sulfur oxides, and particulate matter), air toxics (either confirmed or suspected human carcinogens, including benzene, formaldehyde, 1,3-butadiene, and acetaldehyde), and carbon dioxide.

One of the disadvantages is at normal atmospheric pressure, a gram of hydrogen takes up the same amount of space as about 3 gallons of gas. In order to store enough hydrogen to make it a viable option for fuel, hydrogen must be pressurized and stored in a container that can handle the extreme pressure of the hydrogen inside. Hydrogen can also be stored in a pressurized liquid form. However, in liquid form, hydrogen must be kept at cryogenic temperatures to maintain the hydrogen's stability. Neither pressurized hydrogen gas nor pressurized hydrogen liquid is easy to implement in regular daily use. Hydrogen is extremely reactive. Hydrogen has the potential to spontaneously combust and can be very flammable. Hydrogen stored in high pressure can be susceptible to explosion and may be fatal [8].

Containers to reliably hold hydrogen in daily use in pressurized form are still under development. Developing this technology is costly and time-consuming. Till then, the process of hydrogen storage will be highly costly compared to conventional fossil fuels.

## **1.4 Background Study**

Human dependence towards fossil fuel nowadays is very high. Petroleum production is expected to decrease significantly by 2025, the year that AAN concepts and force structures are scheduled to be operational. Current oil production is 25 billion barrels of oil per year; by 2025, annual oil production most likely will be between 18 and 19 billion barrels, less than the annual production during the oil shortages of the 1970's. Many have switched their attention to alternative energy sources as possible solution for the fore seen situation. Hydrogen fuel cell has been mentioned as one the main solution for the problem.

This study intends to research on the potential of hydrotalcite like minerals as hydrogen storage media. The revolutionary method used to synthesis the hydrotalcite is called sol-gel related co precipitation (SRGCP) method. This study will focus on the effects of cationic substitution and pH on the synthesis. Through this approach, the intended objective is to find the real capability of hydrotalcites hydrogen storage capability.

## **1.5 Problem Statement**

As global depletion of the fossil fuel arrives in future, energy demands will be looked upon other sources. Alternative sources are highly in demand in research to provide continuity. Hydrogen has been introduced as one of alternative for fossil fuels. There have been many researched regarding hydrogen storage as alternative, but neither can reach heights set by fossil fuels.

As another alternative for hydrogen storage, hydrotalcites are used to store hydrogen in this project. Hydrotalcite is not a well known mineral that can be used in many purposes. Currently hydrotalcite is generally used in pharmaceutical industry as antacid mainly consumed for ulcer like diseases. Recent researches also have showed that hydrotalcite can be also used as hydrogen storage cell.

Hydrotalcites will be synthesized using sol-gel related precipitation technique in this research. There will be two parameters that may vary the morphology of the hydrotalcite; cationic substitution and preparation pH. Finally, the characteristics of the synthesized hydrotalcite will be investigated using several applicable characterization methods.

## **1.6 Objective**

The main objectives of the project are:

- 1) The effects of trivalent cation substitution on the morphology of hydrotalcite
- 2) To characterize the hydrotalcite using FTIR, XRD, FESEM and BET techniques.

Once we are able to study the morphology, we will be adapting in identifying a purpose for each separate synthesized hydrotalcite.

## **1.7 Scope Of Study**

- 1) The hydrotalcite compound will be synthesized using sol-gel related coprecipitation method (SRGCP) with different cationic substitution.
- 2) The characterization study will be done using BET and FESEM Analysis.

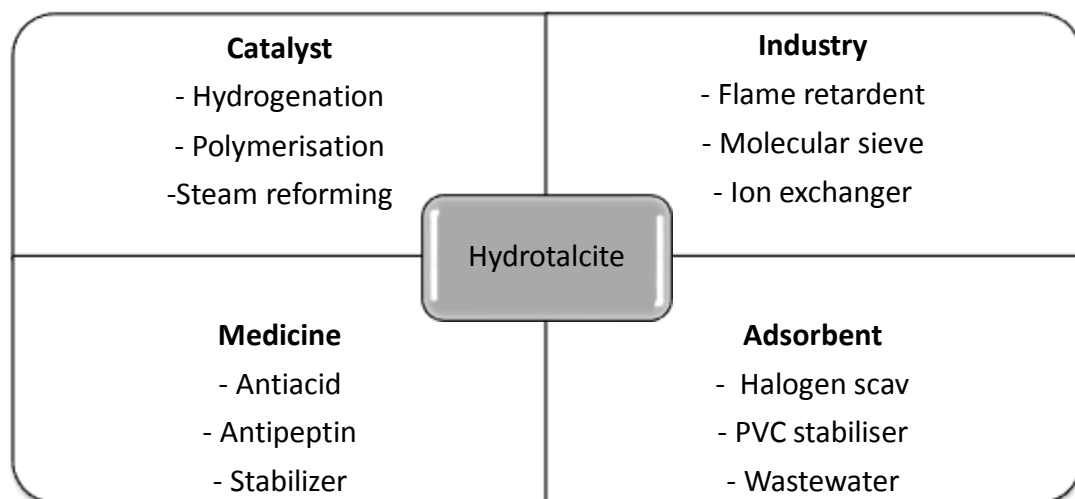
## CHAPTER 2

### LITERATURE REVIEW

#### 2.1 Hydrotalcite

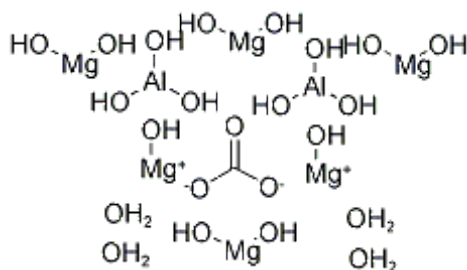
The layered double hydroxides (LDHs) are a large class of natural and synthetic compounds whose layered structure is derived from that of brucite,  $\text{Mg}(\text{OH})_2$  [9]. In LDHs, two cations of different charge substitute on the sites corresponding to the Mg of brucite to give an overall positive charge to the hydroxide layer. The positive charge is balanced by monatomic or small complex anions which are intercalated between the layers. The interlayer species are loosely bound and can be exchanged readily. This ability to exchange anions between the positively charged structural layers contrasts with the exchange of interlayer cations in silicate clays and channel cations in zeolites and many other classes of micro porous materials with hetero polyhedral frameworks which have negatively charged structural frameworks (Mills *et al*, 2012).

To date, 44 minerals have been described as natural examples of LDH phases; they are commonly known to mineralogists as the ‘hydrotalcites’ or ‘hydrotalcite group’ of minerals. Hydrotalcites are anionic clays which are not commonly found and rarer in nature compared to cationic clays such as smectites. The first hydrotalcite was discovered in Sweden around 1842 [1]. The anionic clays based on hydrotalcite-like compounds have found many practical applications.



**Figure 2: Schematic table of possible functions for hydrotalcite material**

The exact formula for hydrotalcite,  $[Mg_6Al_2(OH)_{16}CO_3 \cdot 4H_2O]$ , and of the other isomorphous minerals was presented by E. Manasse, professor of Mineralogy at the University of Florence (Italy), who was also the first to recognize that carbonate ions were essential for this type of structure [2].



**Figure 2: Hydrotalcite structure.**

Hydrotalcite structure is basically derived from brucite structure,  $Mg(OH)_2$ , where the  $Mg^{2+}$  ion are octahedrally surrounded by six  $OH^-$  ions. The hydrotalcite like structure can be obtained when  $Mg^{2+}$  ions or other divalent cations are replaced by trivalent cations of similar radius. A multiple array of compositions are possible for synthetic hydrotalcites, based upon the general formula  $[M_{1-x}^{2+}M_x^{3+}(OH)_2]b^+[A^{n-}]_{b/n} \cdot mH_2O$ , where  $M^{2+}$  and  $M^{3+}$  are the divalent and trivalent cations in the octahedral positions within the hydroxide layers.  $A^{n-}$  is an interlayer anion with a negative charge  $n$ ,  $b$  is the layer and  $m$  is the number of water molecules.



## **2.2 SOL-GEL Related Coprecipitation Method (SGRCP)**

The sol-gel process is a versatile solution process for making advanced materials, including ceramics and organic-inorganic hybrids. In general, the sol-gel process involves the transition of a solution system from a liquid "sol" (mostly colloidal) into a solid "gel" phase. Utilizing the sol-gel process, it is possible to fabricate advanced materials in a wide variety of forms: ultrafine or spherical shaped powders, thin film coatings, fibers, porous or dense materials, and extremely porous aerogel materials. An overview of various sol-gel processes is illustrated above in a graphical form.

The starting materials used in the preparation of the "sol" are usually inorganic metal salts or metal organic compounds such as metal alkoxides. In a typical sol-gel process, the precursor is subjected to a series of hydrolysis and polymerization reactions to form a colloidal suspension, or a "sol". Further processing of the "sol" makes it possible to make materials in different forms.

## **2.3 Hydrotalcite Characterization Methods**

### **2.3.1 XRD Characterization**

X-ray diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a crystalline material and can provide information on unit cell dimensions. The analyzed material is finely ground, homogenized, and average bulk composition is determined. The functions of XRD characterization are [7]:

- To measure the average spacing's between layers or rows of atoms
- To determine the orientation of a single crystal or grain
- To find the crystal structure of an unknown material
- To measure the size, shape and internal stress of small crystalline regions

### **2.3.2 BET and FESEM Analysis**

BET analysis provides precise specific surface area evaluation of materials by nitrogen multilayer adsorption measured as a function of relative pressure using a fully automated analyzer. The technique encompasses external area and pore area

evaluations to determine the total specific surface area in  $\text{m}^2/\text{g}$  yielding important information in studying the effects of surface porosity and particle size in many applications. This technique characterizes pore size distribution independent of external area due to particle size of the sample.

Field Emission Scanning Electron Microscope (FESEM) is a microscope that uses electron to form an image. This process analysis generates high resolution images.

## CHAPTER 3

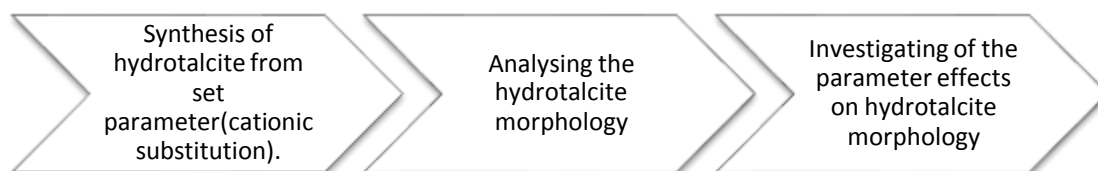
### METHODOLOGY

#### 3.1 Research Methodologies And Project Activities

The chosen methodology for this research is by exploration and discovery because the effects of the parameters applied during the synthesis is not apparent and has to be investigated. Once the results are obtained, the results shall be compared with other research results to show a correlation or difference. The result will be mainly the effect of the factors on the morphology of the hydrotalcite. The results also can be further used to find appropriate uses for hydrotalcite depending on its morphology behavior.

#### 3.2 Experimental Procedures

The figure below shows the general experimental procedures that will be implemented in this research project.



**Figure 3: The schematic diagram depicting the general approach in this project**

##### 3.2.1 Raw Materials And Chemicals Needed

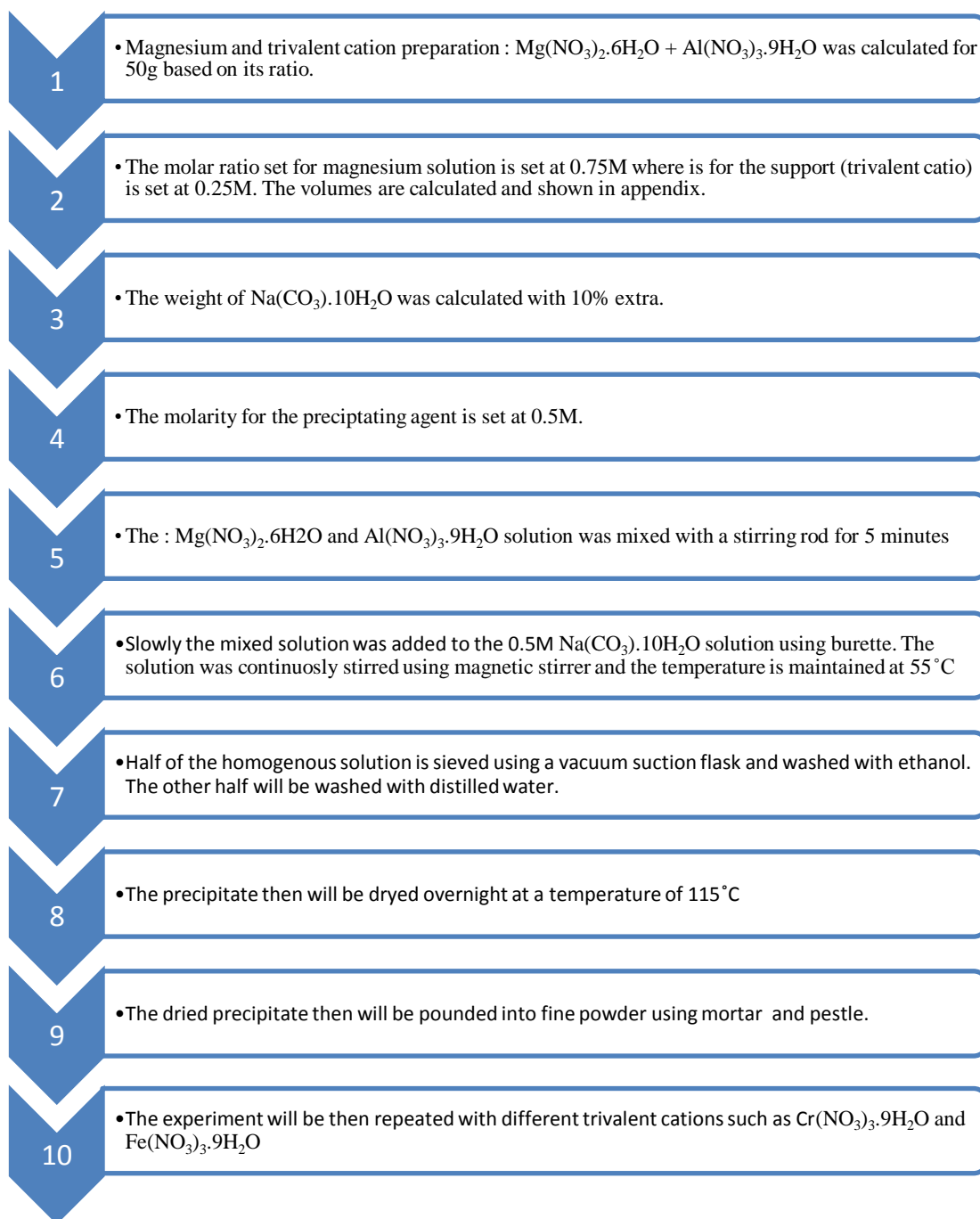
In the experiments that are going to be conducted, several raw materials and chemicals are needed. There are:

- i. Aluminium nitrate nonahydrate
- ii. Iron nitrate nonahydrate
- iii. Chromium nitrate nonahydrate
- iv. Magnesium nitrate hexahydrate
- v. Sodium carbonate hexahydrate
- vi. Ethanol

### 3.2.2 Experimental Method

In this project, the magnesium based will be prepared using the sol-gel related coprecipitation method with three different trivalent cations. The molar ratio set for magnesium and trivalent cation is 3:1.

#### 1. Coprecipitation Method to Test Cationic Effect:



**Figure 4: Preparation for synthesis process for hydrotalcite for Cationic Substitution**

### 3.3 Taguchi Method

The experimental is based on the orthogonal array series. The array consists of two levels, which are cationic substitution and wetting solution for precipitation. All factors were arranged in L<sub>4</sub> series orthogonal array. Below are Taguchi design method in Table 1 and Table 2.

**Table 1 : Factor and Level for Taguchi Method**

<b>Factors</b>	<b>Level</b>
Cation Substitution	Mg-Fe
	Mg-Al
	Mg-Cr
Wetting Solution	Ethanol
	Distilled Water

**Table 2: Designed Experiment with Taguchi Method**

Experiment	Cation Substitution	Wetting Solution
A	Mg-Fe	Ethanol
B	Mg-Fe	Distilled Water
C	Mg-Cr	Ethanol
D	Mg-Cr	Distilled Water
E	Mg-Al	Ethanol
F	Mg-Al	Distilled Water

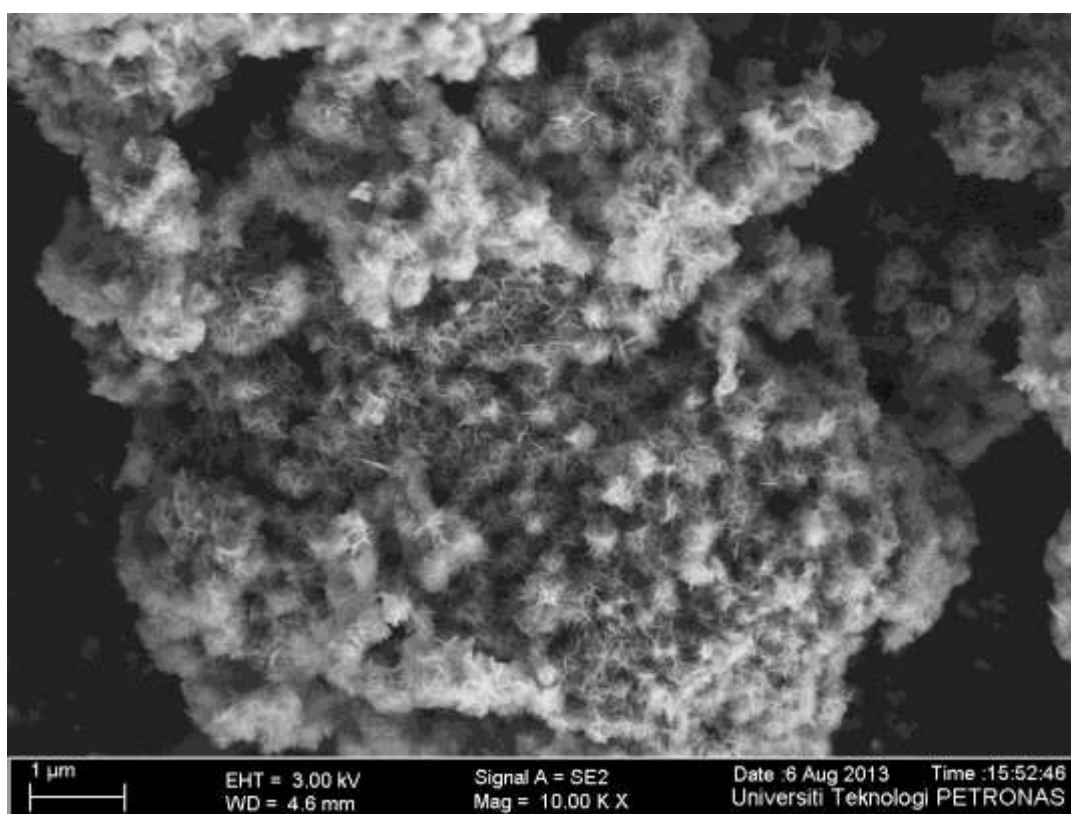
## CHAPTER 4

### RESULT AND DISCUSSION

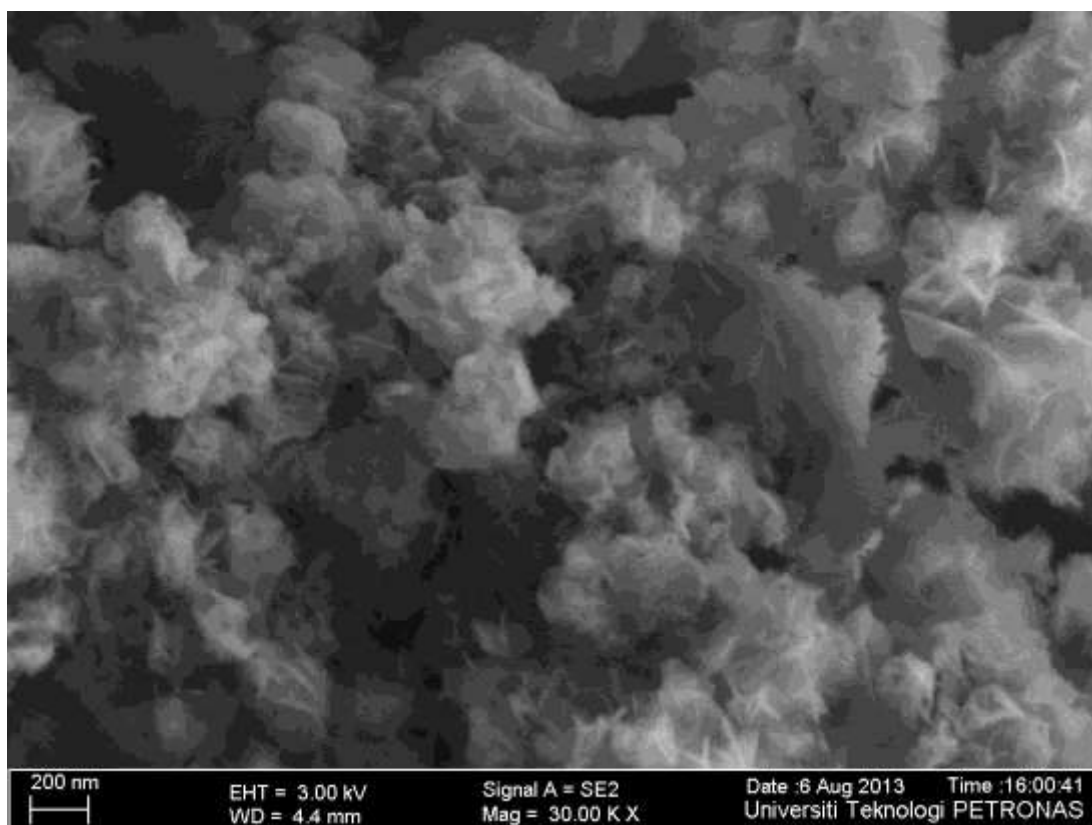
#### 4.1 Field Emission Scanning Electron Microscope (FESEM) Analysis

The analyses of hydrotalcites were conducted using the FESEM analysis. This type of method enables the morphology of the HTlcs to be observed carefully. Figures here some of the results obtained for the FESEM analysis:

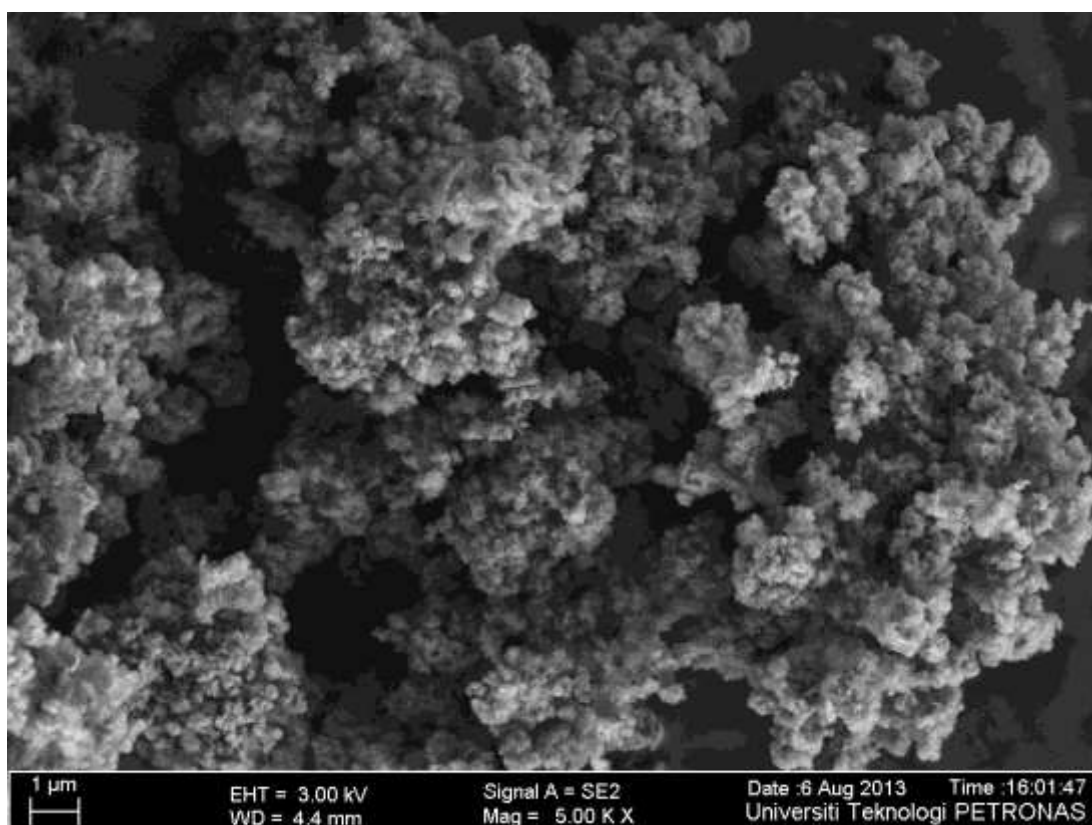
Mg-Fe:



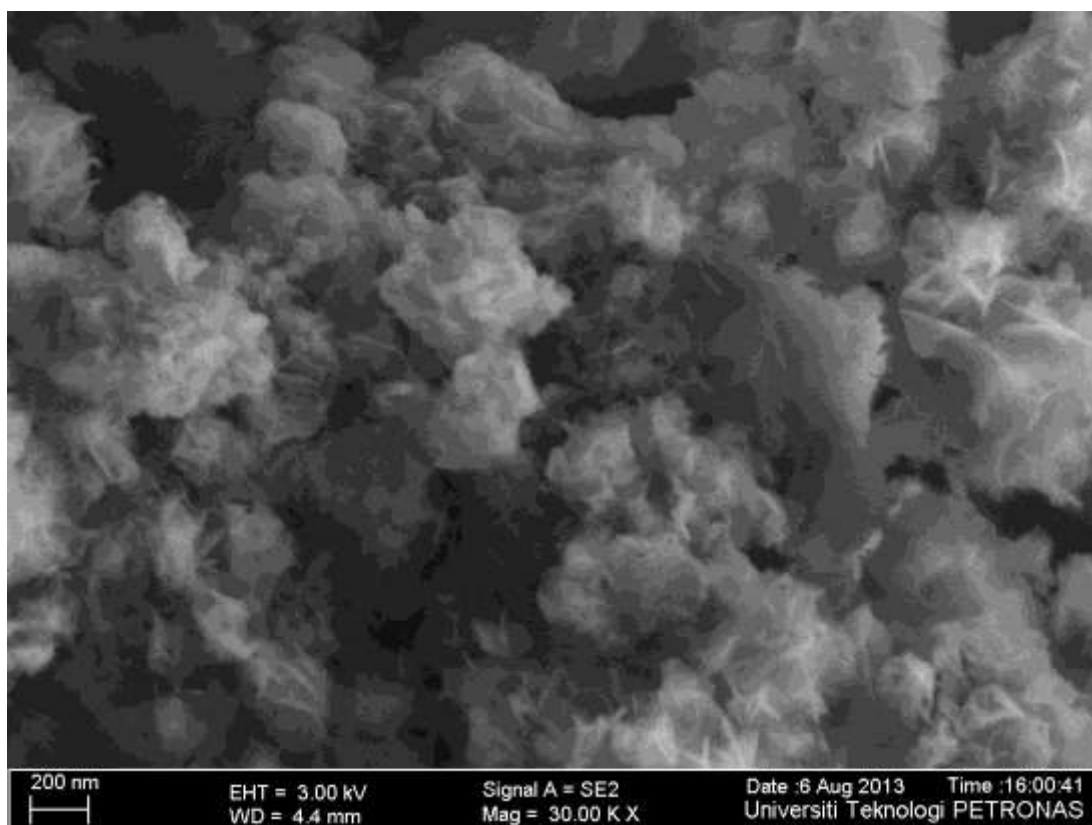
**Figure 5: FESEM image of HTlcs Mg-Fe, wetted with ethanol**



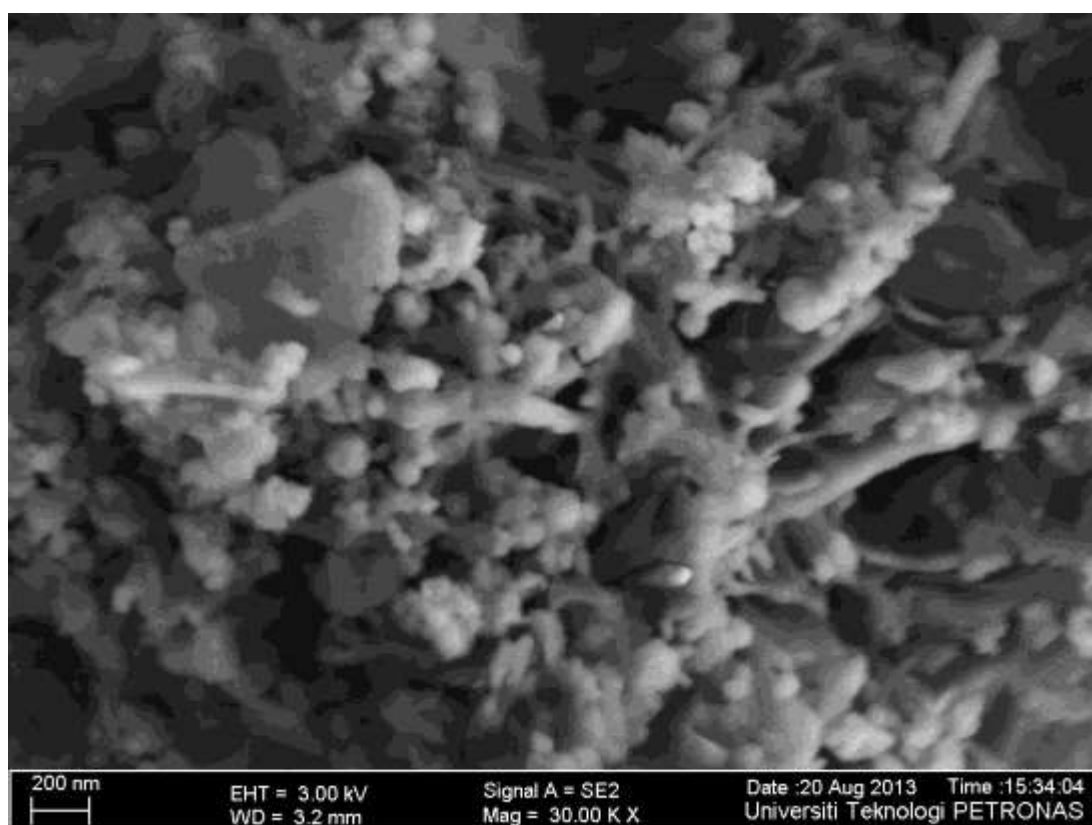
**Figure 6: FESEM image of HTlcs Mg-Fe, wetted with ethanol**



**Figure 7: FESEM image of HTlcs Mg-Fe, wetted with distilled water**

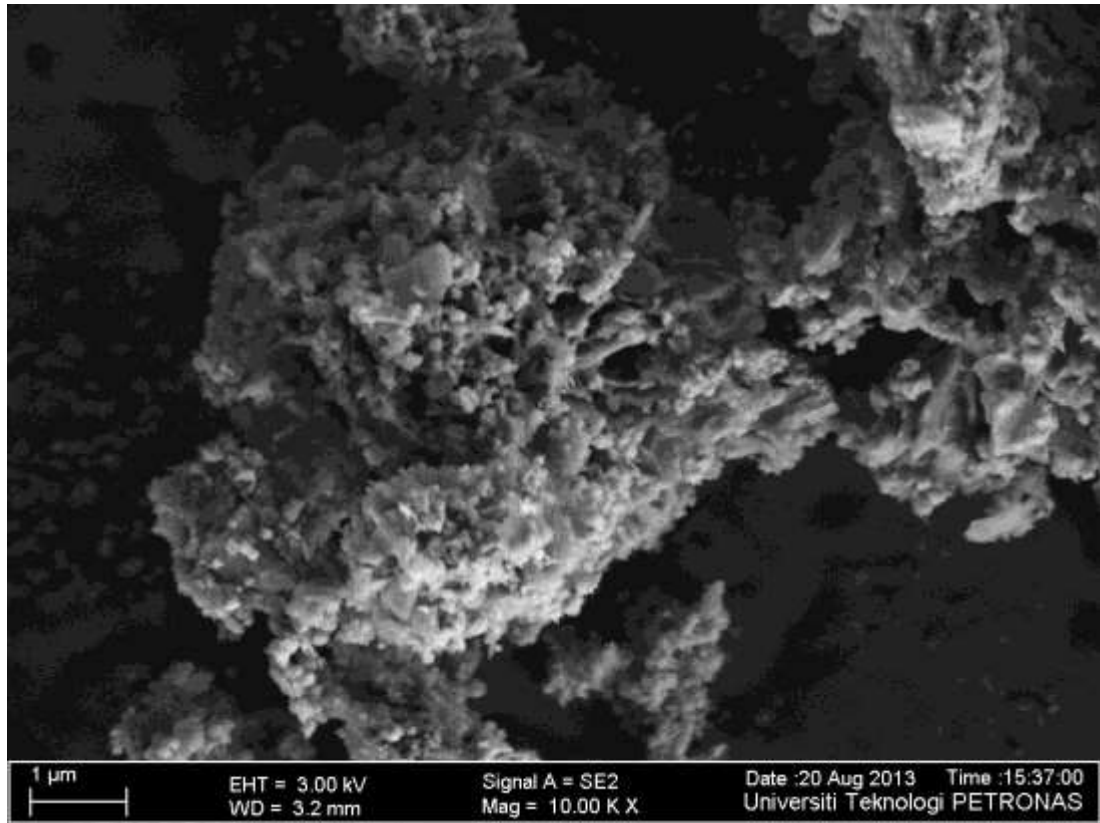


**Figure 8: FESEM image of HTlcs Mg-Fe, wetted with distilled water**

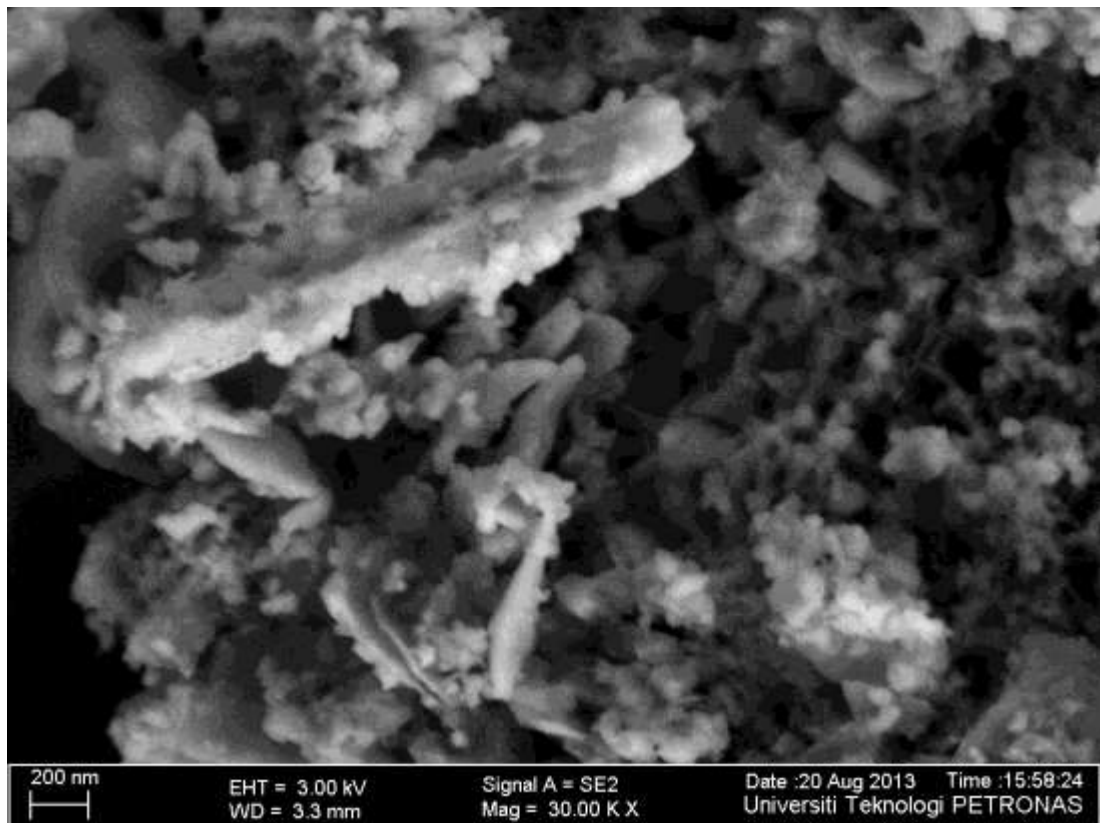


**Figure 9: FESEM image of HTlcs Mg-Cr, wetted with ethanol**

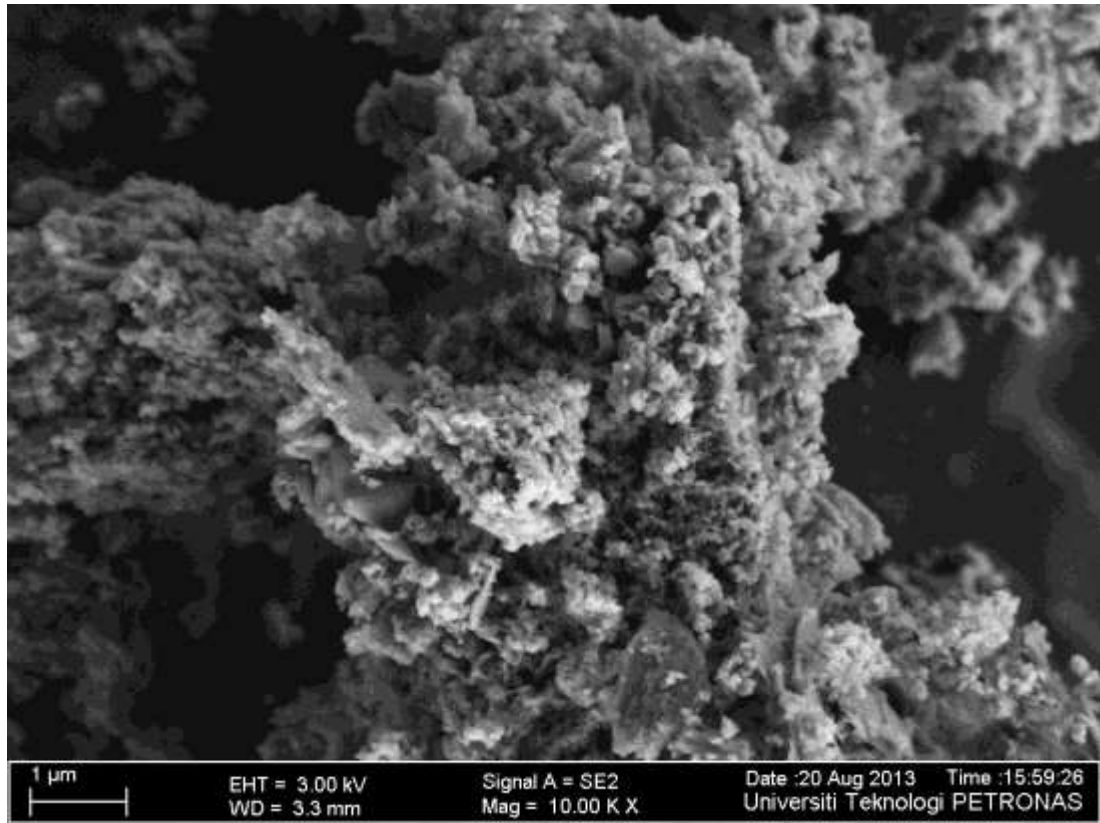




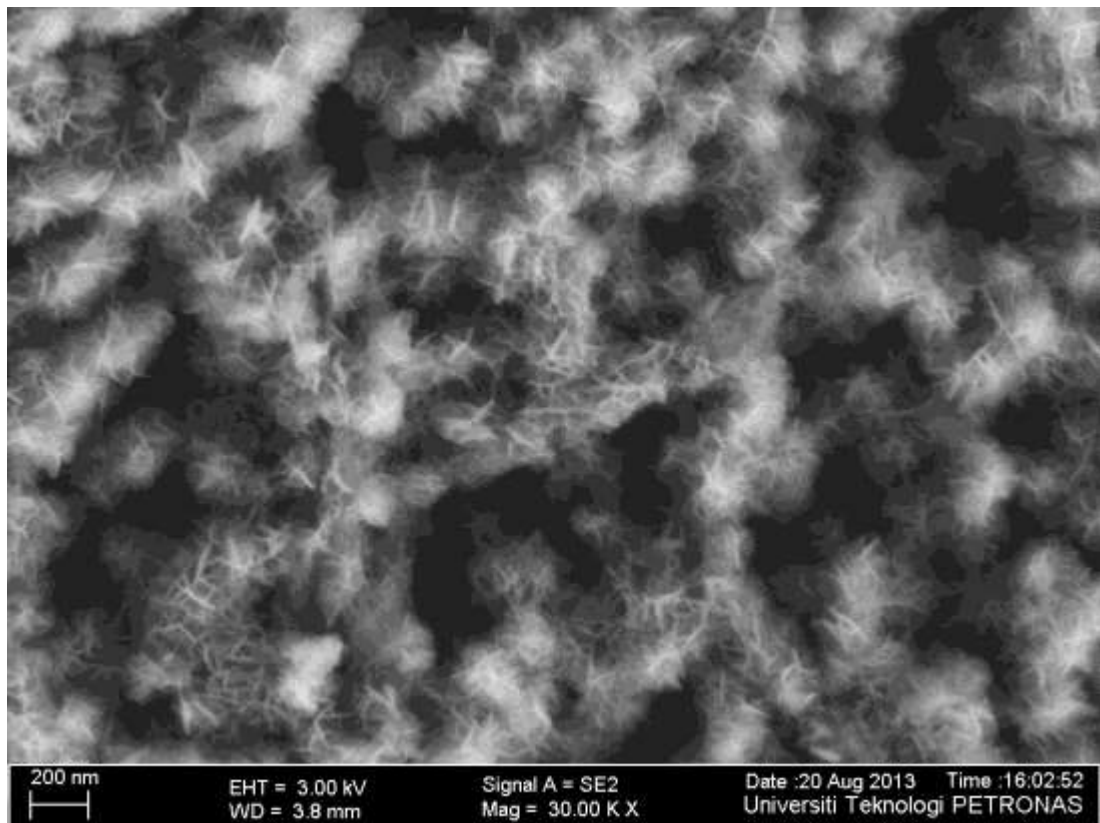
**Figure 10: FESEM image of HTlcs Mg-Cr, wetted with ethanol**



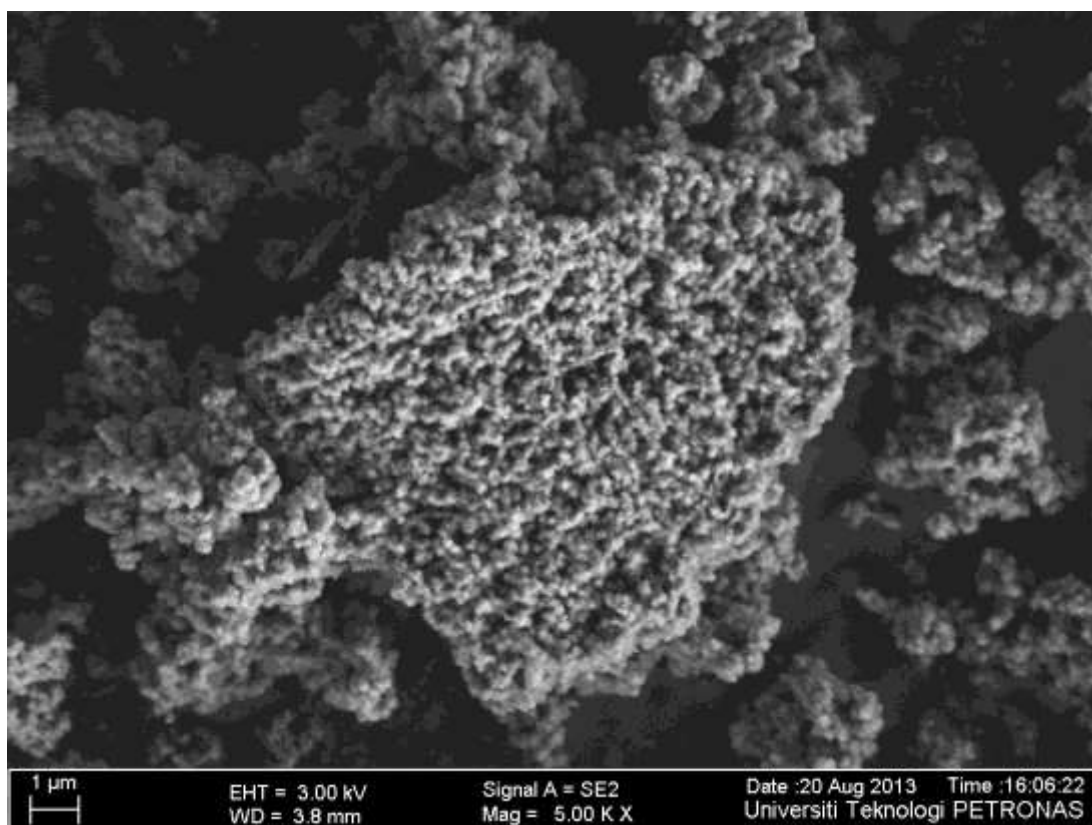
**Figure 11: FESEM image of HTlcs Mg-Cr, wetted with distilled water**



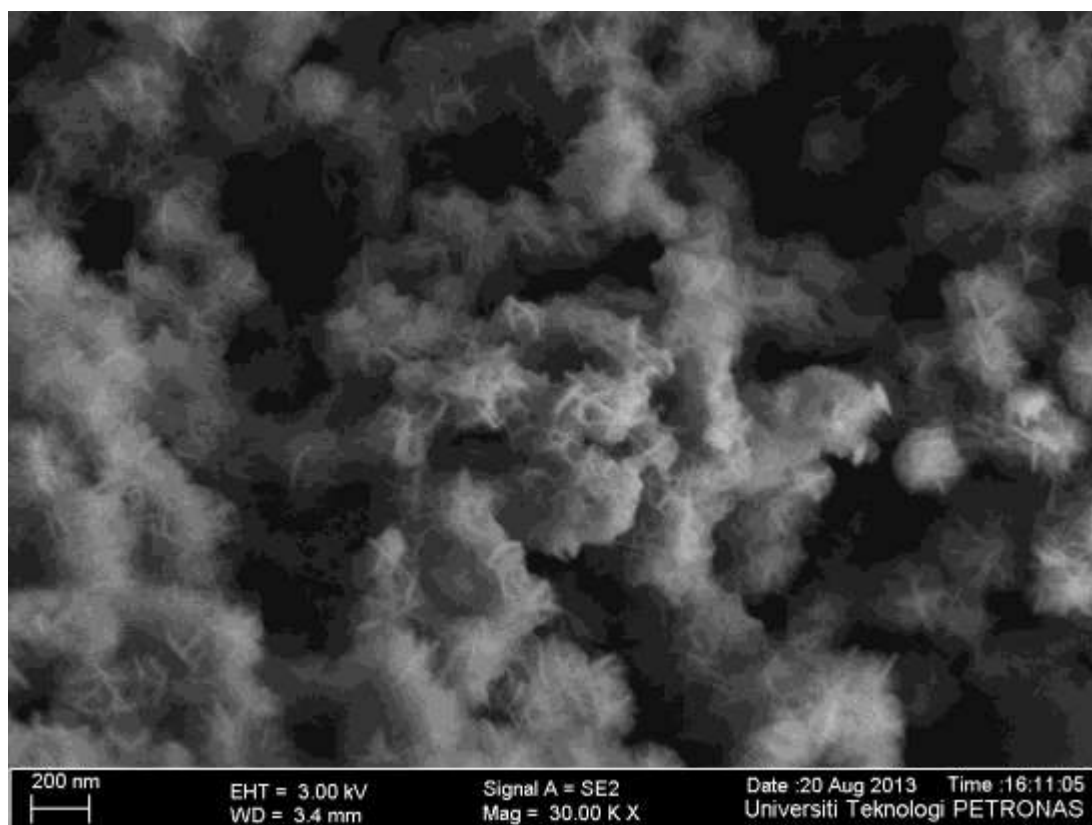
**Figure 12: FESEM image of HTlcs Mg-Cr, wetted with distilled water**



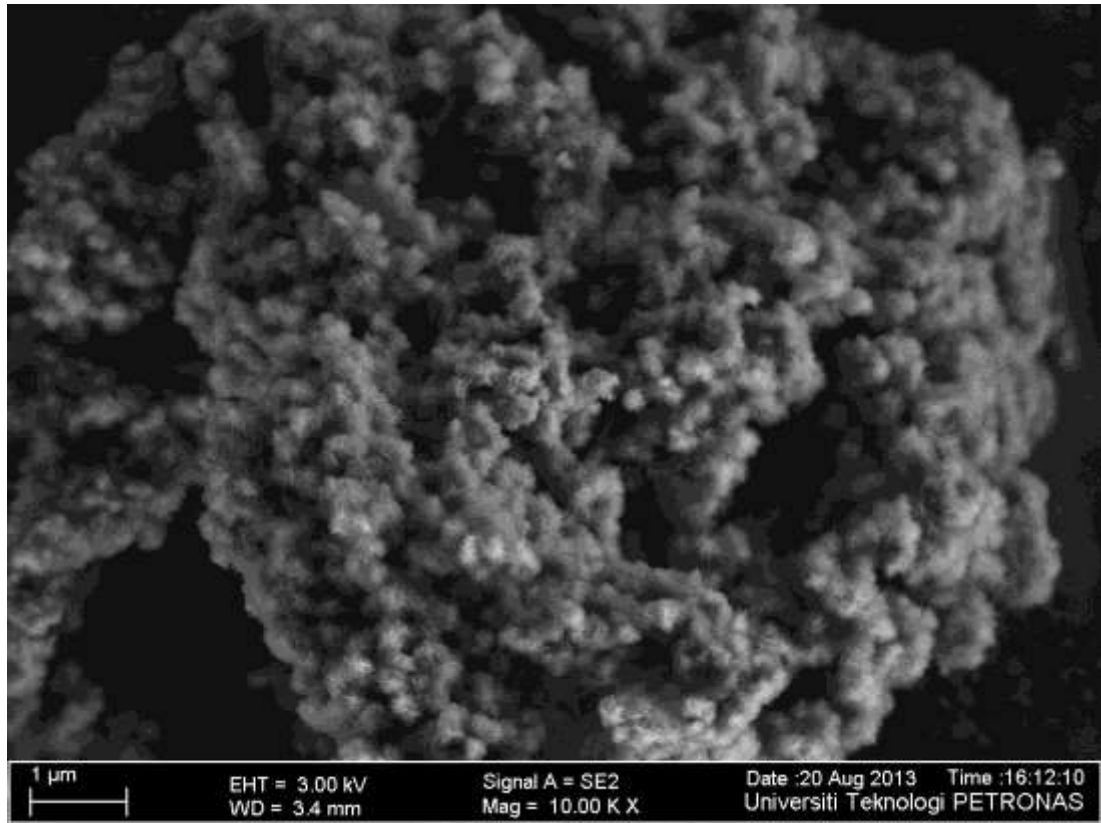
**Figure 13: FESEM image of HTlcs Mg-Al, wetted with ethanol**



**Figure 14: FESEM image of HTlcs Mg-Al, wetted with ethanol**



**Figure 15: FESEM image of HTlcs Mg-Al, wetted with distilled water**



**Figure 16: FESEM image of HTlcs Mg-Al, wetted with distilled water**

These are the obtained FESEM images of the synthesized hydrotalcite with being wetted with ethanol and distilled water. Figure 7 and 8 are the FESEM images of Mg-Fe hydrotalcite wetted with distilled water. In figure 5, hexagonal and rhombohedral lattice structure of the hydrotalcite can be observed. However, based on the visual, there is around only some hydrotalcite was formed as there is the cotton like surface that may present to be unreacted nitrate precursors.

In figure 7, the coral-like morphology which indicated a porous material [16]. The hydrotalcite formed from chromium are more plate like as can be observed figure 9, 10, 11 and 12. From all the images, it can be said that hydrotalcites consist of agglomerates of thin-plate like crystals which represent the layered structure [17].

## 4.2 BET Surface Area Analysis

Specific surface area of the sample was determined by the adsorption of nitrogen gas at 200°C for 2 hours. The textural properties of the materials are presented in Figure 17 and 18, and Table 3.

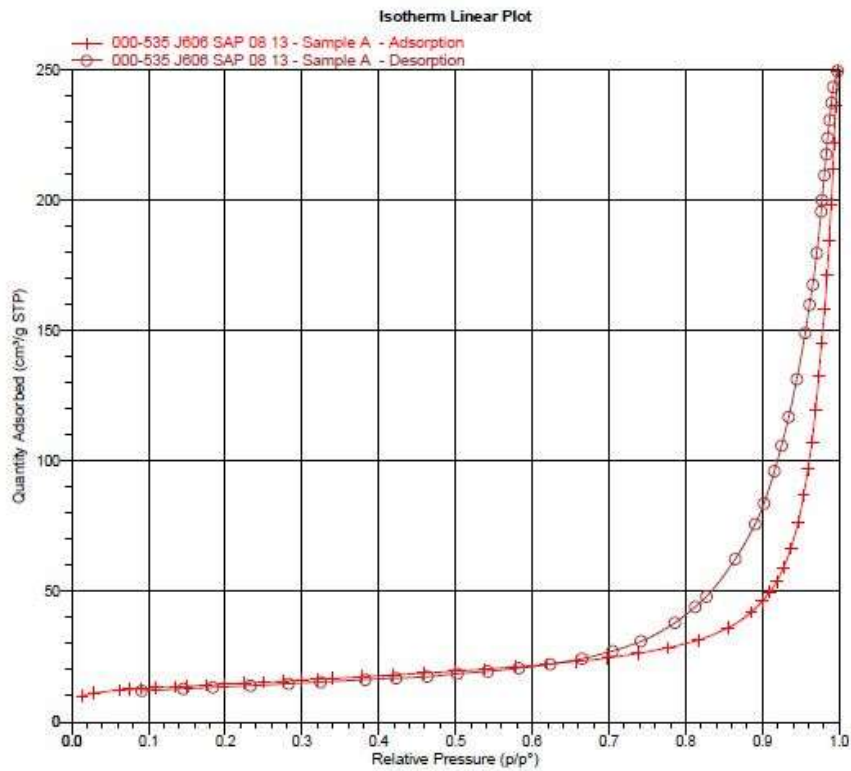


Figure 17: Isotherm Curve of Nitrogen Gas adsorption onto Mg-Fe, wetted with ethanol

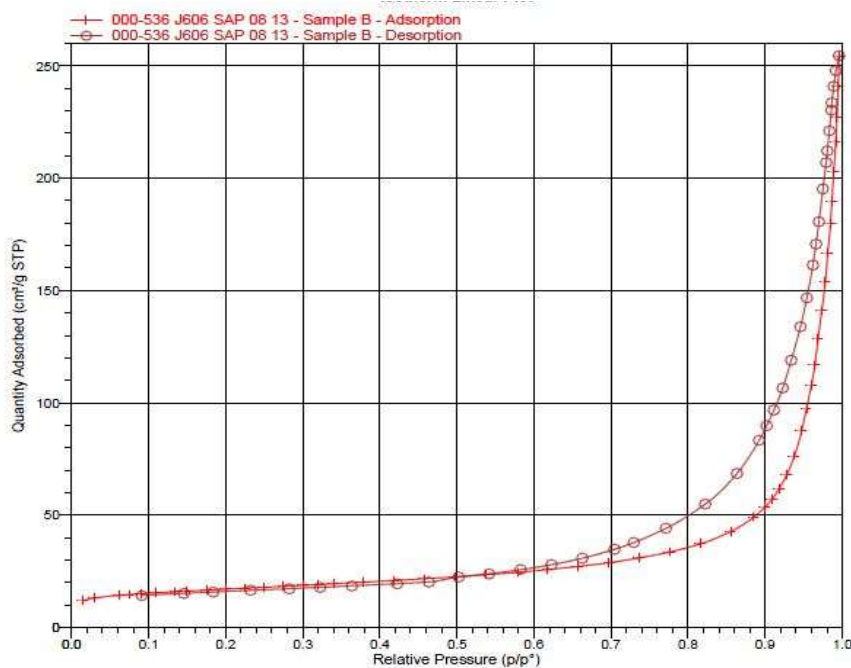


Figure 18: Isotherm Curve of Nitrogen Gas adsorption onto Mg-Fe, wetted with distilled water

Material (Mg-Fe)	BET Surface Area (m <sup>2</sup> /g)	Pore volume (cm <sup>3</sup> /g)	Pore Size	
			Adsorption	Desorption
Ethanol	50.4529	0.343004	319.552	255.550
Distilled Water	59.3531	0.351670	285.172	224.691

**Table 3: Textural Properties of the Synthesized Hydrotalcites**

As shown in above table, the specific surface area of the sample increases as the distilled water is used as washing liquid for the sample.

## CHAPTER 5


### CONCLUSION AND RECOMMENDATIONS

Hydrotalcites were successfully synthesized and characterizations of the hydrotalcites are still in characterization processes. From the FESEM analysis, we can observe the materials coral-like morphology which means proves the material is porous and corresponds to other findings.

The recommendations on this study are including the study of future hydrogen storage capability of the material. Thermogravimetric analysis (TGA) and Magnetic Suspension Balance (MSB) study will further improve the research on hydrogen storage capability. Thermogravimetric analysis or thermal gravimetric analysis (TGA) is a method of thermal analysis in which changes in physical and chemical properties of materials are measured as a function of increasing temperature (with constant heating rate), or as a function of time (with constant temperature and/or constant mass loss). TGA can provide information about chemical phenomena including chemisorptions, desolvation (especially dehydration), decomposition, and solid-gas reactions [17]. This would allow to study on the hydrogen adsorption on hydrotalcite.

Further parameters of the experiment can be researched as well to improve the HTlcs such as the pH of the synthesis of HTlcs. A higher constant pH that has to be maintained during the experiment can facilitate higher incorporation of Mg material. Other parameter such as adding another metal nitrate precursor to create a variation and study the differences.

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## APPENDICES

### APPENDIX A : Calculation for Hydrotalcite Synthesis

Chemicals	Symbol	Formula	MW (g/mol)
Magnesium Nitrate Hexahydrate	U	$\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	256.41
Iron (III) Nitrate Nonahydrate	W	$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	404.00
Chromium Nitrate Nonahydrate	X	$\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	400.15
Aluminium Nitrate Nonahydrate	Y	$\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	375.13
Sodium Carbonate Decahydrate	Z	$\text{Na}(\text{CO}_3) \cdot 10\text{H}_2\text{O}$	286.12

Molar ratio: Mg-Fe = 3:1

$$\text{Total weight} = (3 \times 256.41 + 1 \times 404.00) = 1173.23 \text{ g}$$

$$\text{Weight of U} = \frac{3 \times 256.41}{1173.23} \times 50 \text{ g} = 32.78 \text{ g}$$

$$\text{Weight of W} = \frac{1 \times 404.00}{1173.23} \times 50 \text{ g} = 17.22 \text{ g}$$

Molar ratio: Mg-Cr = 3:1

$$\text{Total weight} = (3 \times 256.41 + 1 \times 400.15) = 1169.38 \text{ g}$$

$$\text{Weight of U} = \frac{3 \times 256.41}{1169.38} \times 50 \text{ g} = 32.89 \text{ g}$$

$$\text{Weight of X} = \frac{1 \times 400.15}{1169.38} \times 50 \text{ g} = 17.11 \text{ g}$$

Molar ratio: Mg-Al = 3:1

$$\text{Total weight} = (3 \times 256.41 + 1 \times 375.13) = 1144.36 \text{ g}$$

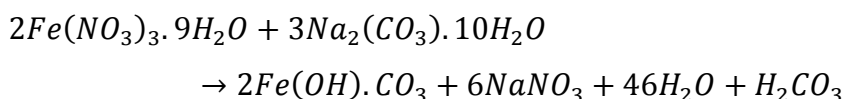
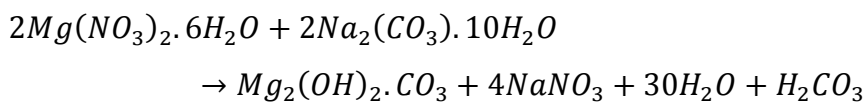
$$\text{Weight of U} = \frac{3 \times 256.41}{1144.36} \times 50 \text{ g} = 33.61 \text{ g}$$

$$\text{Weight of Y} = \frac{1 \times 375.13}{1144.36} \times 50 \text{ g} = 16.39 \text{ g}$$

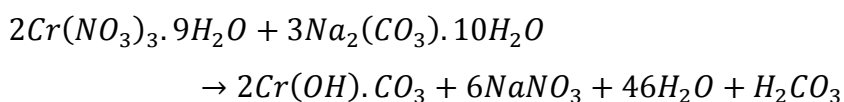
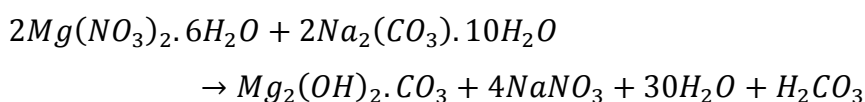
**APPENDIX B: Calculation For Amount of Sodium Carbonate Decahydrate For Each Synthesis.**

Reaction Equations:

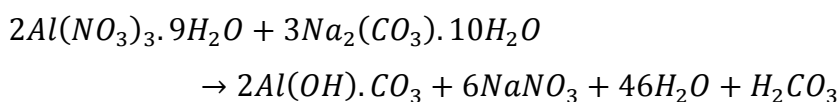
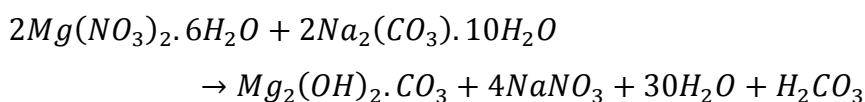
**Mg-Fe:**



**Mg-Cr:**



**Mg-Al:**



Molar ratios:

$$\frac{Mg}{Na} = \frac{2}{2} = 1$$

$$\frac{Fe}{Na} = \frac{2}{3} = 1$$

$$\frac{U/256.41}{Z/286.14} = 1$$

$$\frac{W/256.41}{Z/404.00} = \frac{2}{3}$$

$$Z_1 = 1.116 U$$

$$Z_2 = 1.144 W$$

Amount of  $\text{Na}(\text{CO}_3) \cdot 10\text{H}_2\text{O}$ :

$$Z = 1.116 U + 1.144 W$$

Amount of  $\text{Na}(\text{CO}_3) \cdot 10\text{H}_2\text{O}$  used in the synthesis is 10% more, so:

$$Z_{actual} = 1.1 \times Z$$

Material required for the synthesis of hydrotalcite:

Material Combination	Material (g)				
	$\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	$\text{Cr}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	$\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	$\text{Na}(\text{CO}_3) \cdot 10\text{H}_2\text{O}$
Mg-Fe	32.78	17.21	0	0	49.18
Mg-Cr	32.89	0	17.11	0	49.34
Mg-Al	33.61	0	0	16.39	50.42

## APPENDIX C : Calculation for Volume of Distilled Water for Each Material

The molar ratio for each component of sol-gel related coprecipitation method:

$$\text{Mg} = 0.75\text{M}$$

$$\text{Support (Trivalent Cation)} = 0.25\text{M}$$

$$\text{Precipitating Agent } [\text{Na}(\text{CO}_3) \cdot 10\text{H}_2\text{O}] = 0.5\text{M}$$

$$\text{Number of moles} = \frac{\text{Mass}}{\text{Molecular weight}}$$

$$\text{Molarity} = \frac{\text{Number of moles}}{\text{Volume}}$$

Therefore,

$$\text{Volume} = \frac{\text{Mass} / \text{Molecular weight}}{\text{Molarity}}$$

### **Mg-Fe:**

Magnesium Nitrate Hexahydrate = 170.50 mL

Iron (III) Nitrate Nonahydrate = 170.50 mL

Sodium Carbonate Decahydrate = 343.78 mL

### **Mg-Cr:**

Magnesium Nitrate Hexahydrate = 171.03 mL

Chromium Nitrate Nonahydrate = 171.03 mL

Sodium Carbonate Decahydrate = 345.00 mL

### **Mg-Al:**

Magnesium Nitrate Hexahydrate = 174.80 mL

Aluminium Nitrate Nonahydrate = 174.80 mL

Sodium Carbonate Decahydrate = 352.50 mL

## APPENDIX D: EDX Reports

C

Spectrum processing :

No peaks omitted

Processing option : All elements analyzed (Normalised)

Number of iterations = 5

Standard :

C CaCO<sub>3</sub> 1-Jun-1999 12:00 AM

O SiO<sub>2</sub> 1-Jun-1999 12:00 AM

Na Albite 1-Jun-1999 12:00 AM

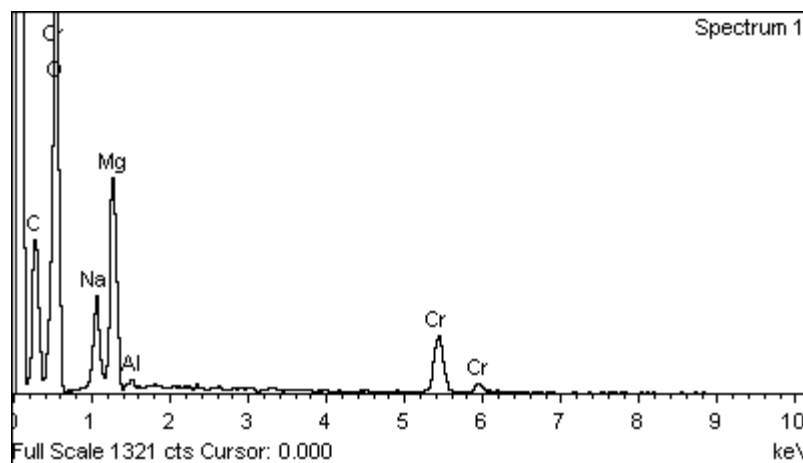
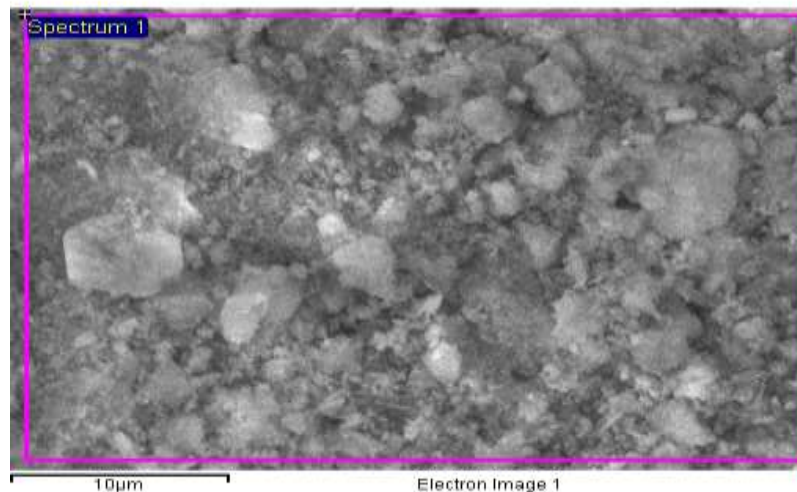
Mg MgO 1-Jun-1999 12:00 AM

Al Al<sub>2</sub>O<sub>3</sub> 1-Jun-1999 12:00 AM

Cr Cr 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%

Comment:



---

C K	31.88	41.19
O K	50.89	49.36
Na K	4.41	2.97
Mg K	7.58	4.84
Al K	0.27	0.16
Cr K	4.97	1.48
Totals	100.00	

---

Spectrum processing :

No peaks omitted

Processing option : All elements analyzed (Normalised)

Number of iterations = 5

Standard :

C CaCO<sub>3</sub> 1-Jun-1999 12:00 AM

O SiO<sub>2</sub> 1-Jun-1999 12:00 AM

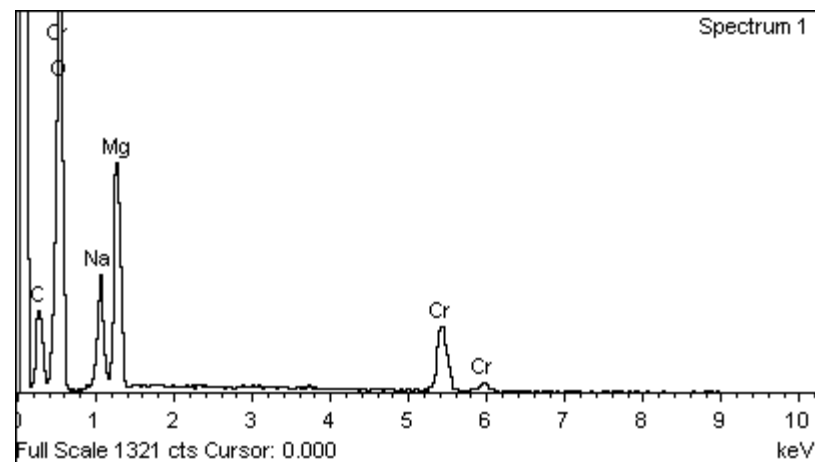
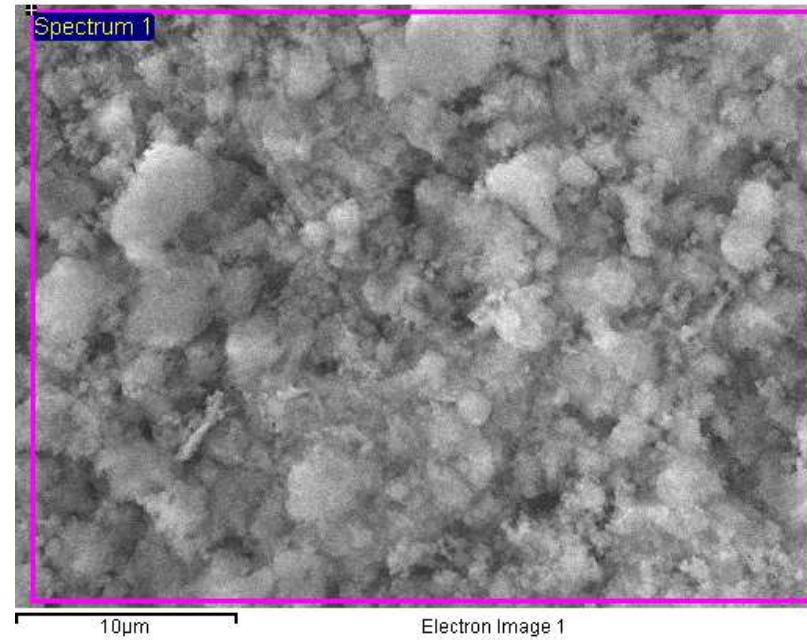
Na Albite 1-Jun-1999 12:00 AM

Mg MgO 1-Jun-1999 12:00 AM

Cr Cr 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	24.44	33.09
O K	53.24	54.11
Na K	5.97	4.23
Mg K	9.72	6.50

Comment:





---

Cr K	6.63	2.07
Totals	100.00	

---

Spectrum processing :

No peaks omitted

Processing option : All elements analyzed (Normalised)

Number of iterations = 5

Standard :

C CaCO<sub>3</sub> 1-Jun-1999 12:00 AM

O SiO<sub>2</sub> 1-Jun-1999 12:00 AM

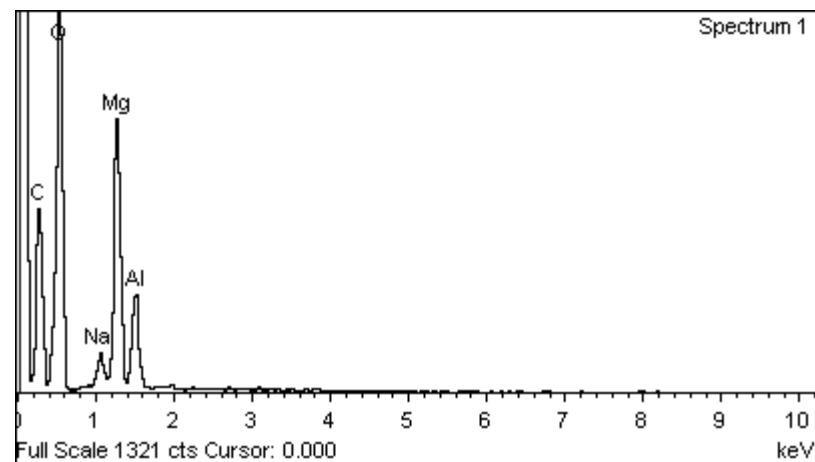
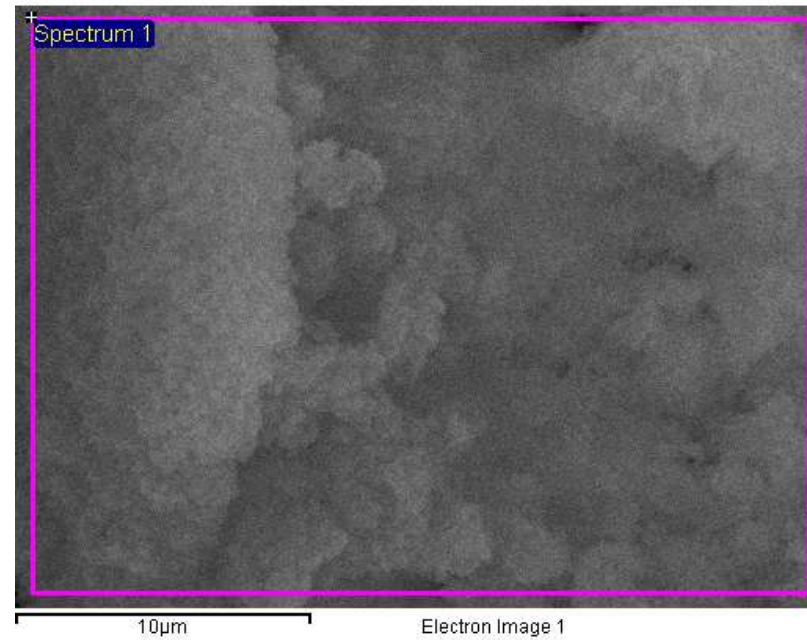
Na Albite 1-Jun-1999 12:00 AM

Mg MgO 1-Jun-1999 12:00 AM

Al Al<sub>2</sub>O<sub>3</sub> 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	35.28	43.75
O K	52.50	48.88
Na K	1.45	0.94
Mg K	8.03	4.92

Comment:



---

AI K	2.73	1.51
Totals	100.00	

---

Spectrum processing :

No peaks omitted

Processing option : All elements analyzed (Normalised)

Number of iterations = 5

Standard :

C CaCO<sub>3</sub> 1-Jun-1999 12:00 AM

O SiO<sub>2</sub> 1-Jun-1999 12:00 AM

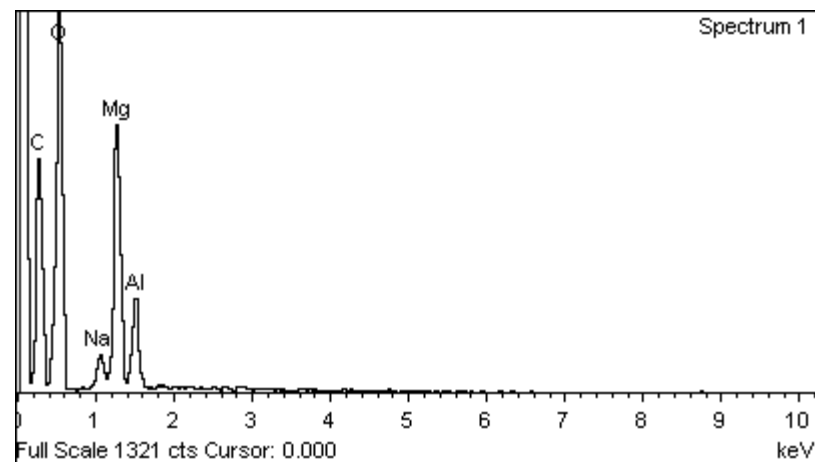
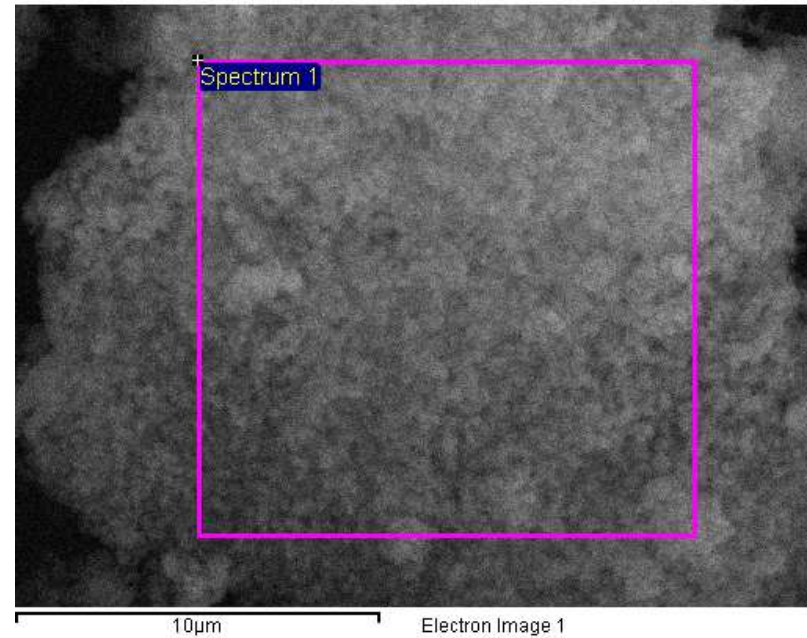
Na Albite 1-Jun-1999 12:00 AM

Mg MgO 1-Jun-1999 12:00 AM

Al Al<sub>2</sub>O<sub>3</sub> 1-Jun-1999 12:00 AM

Element	Weight%	Atomic%
C K	38.16	46.73
O K	50.81	46.70
Na K	1.30	0.83
Mg K	7.33	4.43

Comment:



AI K	2.40	1.31
Totals	100.00	