

**Optimization of Iron (III) Oxide Catalyst Using Taguchi Method for
Development of Carbon Nanofiber**

by

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CERTIFICATION OF APPROVAL

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A project dissertation submitted to the
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Universiti Teknologi PETRONAS
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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 that the original work contained herein have not been undertaken or done by unspecified sources or persons.

NORAZLINA BINTI MD YAACOB

ABSTRACT

Carbon nanofiber (CNF) has widely developed due to its potential application in hydrogen storage material in solid form. CNF is synthesized from the chemical vapor deposition (CVD) of carbon containing gasses over transition metal of the catalyst. The size of catalyst will control the type of CNF produced. This research work is to find the most optimum condition for Iron (III) oxide in developed catalyst using Taguchi Method as the design of experiment.

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CHAPTER 1

INTRODUCTION

1.1 Problem statement

Environmental concerns regarding the use of fossil fuels and their predicted exhaustion are globally important issues. Hydrogen is a promising substitute for fossil fuels in mobile applications, with the additional benefit of potentially allowing the production of zero emission vehicles. However, to facilitate the commercialisation of this technology suitable hydrogen storage systems need to be developed. In recent years, activated carbons, CNF and carbon nanotubes (CNT) have all attracted considerable attention as potential hydrogen storage materials [1-6]. CNF is synthesized from the CVD of carbon containing gasses over transition metal of the catalyst such as iron, nickel, copper, cobalt and many others. Before synthesized CNF, preparation of metal catalyst is essential as it determines the size and structure that would be obtained in the CNF. During the growth of CNF, hydrocarbon gas or vapour must undergo dissociate absorption onto the surface of catalyst particle before the carbon begin to diffuse through the particle. The interaction of the hydrocarbon molecules and the catalyst surface will crate carbon pore and structure. Thus the crystallographic orientation and chemical composition of the catalyst particle are important as it will control the diameter of CNF produced. There is higher possibility to produced CNF when Iron (Fe) is used as the catalyst in CVD. Therefore, to find the optimum condition for Iron Oxide catalyst production, Taguchi Method has been used in this paper to design the experiment as also used in previous studies in finding the most optimum condition.

1.2 Objectives

To find the most optimum condition for develop catalyst using Taguchi Method as design of experiment (DoE) and to characterize the catalyst using XRD, FESEM, and Raman Spectroscopy.

1.3 Scope of study

The scope for this project has selected the DoE using Taguchi Method. Three factors with three levels of variations are arranged in matrix form according to Orthogonal Array L₉. The factor involves are temperature, calcinations time and weight of salt used. The calcination temperature is chosen within the range 300°C to 500°C, calcinations is within 2 hours to 6 hours and last parameter is weight of salt 3g, 5g and 7g respectively.

CHAPTER 2

LITERATURE REVIEW

2.1 What is Design of Experiment?

2.1.1 Systematic Approach to Data Collection Design of experiments (DoE) is a systematic, rigorous approach to engineering problem-solving that applies principles and techniques at the data collection stage so as to ensure the generation of valid, defensible, and supportable engineering conclusions. In addition, all of this is carried out under the constraint of a minimal expenditure of engineering runs, time, and money. DoE Problem Areas There is 4 general engineering problem areas in which DoE may be applied:

1. Comparative
2. Screening/Characterizing
3. Modeling
4. Optimizing

Design of Experiments (DoE) is widely used in research and development, where a large proportion of the resources go towards solving optimization problems. The key to minimizing optimization costs is to conduct as few experiments as possible. DoE requires only a small set of experiments and thus helps to reduce costs.

2.1.1.1 **Comparative** In the first case, the engineer is interested in assessing whether a change in a single factor has in fact resulted in a change/improvement to the process as a whole.

2.1.1.2 **Screening Characterization** In the second case, the engineer is interested in "understanding" the process as a whole in the sense that he/she wishes (after design and analysis) to have in hand a ranked list of important through unimportant factors (most important to least important) that affect the process.

2.1.1.3 **Modeling** In the third case, the engineer is interested in functionally modeling the process with the output being a good-fitting (= high predictive power) mathematical function, and to have good (= maximal accuracy) estimates of the coefficients in that function.

2.1.1.4 **Optimizing** In the fourth case, the engineer is interested in determining optimal settings of the process factors; that is, to determine for each factor the level of the factor that optimizes the process response.

The ultimate goal in catalyst design is maximization of activity and selectivity of the catalyst. The processes and parameters used in catalyst design affect activity and selectivity via various surface properties such as total surface area, active surface area, metal content, pore structure and so on. Therefore the surface properties should be optimized to obtain desired activity and selectivity.

However there are two major difficulties in this task. First the desired combination of surface properties that lead the maximum activity and selectivity are usually unknown and can not be found easily. Second these values of surface properties may not be obtained within the same range of catalyst preparation parameters. To overcome these difficulties a two step process may be employed. In the first step, the approximate range of catalyst preparation parameters can be found by optimizing various surface properties. Then a more detailed study can be performed within these ranges to maximize activity and selectivity. On the other hand, catalyst preparation is an expensive and tedious process. Therefore, optimizing surface properties should be considered as "preliminary" and achieved using as small a number of experiments as possible. Taguchi method widely used in industrial experiments may be used for this purpose.

2.2 Taguchi Method

Taguchi's approach is totally based on statistical design of experiments [8], and this can economically satisfy the needs of problem solving and product or process design optimization [9]. By applying this technique one can significantly reduce the time required for experimental investigation, as it is effective in investigating the effects of multiple factors on performance as well as to study the influence of individual factors to determine which factor has more influence, which has less influence [8, 9].

The most important stage in the design of an experiment lies in the selection of control factors. As many as possible factors should be included, so that it would be possible to identify non-significant variables at the earliest opportunity. Taguchi creates a standard orthogonal array to accommodate this requirement. Depending on the number of factors, interactions and levels needed, the choice is left to the user to select either the standard or column-merging method or idle-column method, or etc. Two of the applications in which the concept of S/N ratio is useful are the improvement of quality through variability reduction and the improvement of measurement. The S/N ratio characteristics can be divided into three categories when the characteristic is continuous [8]:

Goal	S/N Ratio Formula
nominal is best	$\frac{S}{N} = 10 \log \left(\frac{\bar{Y}^2}{s^2} \right)$
larger-is-better (maximize)	$\frac{S}{N} = -10 \log \left(\frac{1}{n} \sum_i \frac{1}{Y_i^2} \right)$
smaller-is-better (minimize)	$\frac{S}{N} = -10 \log \left(\frac{1}{n} \sum_i Y_i^2 \right)$

where, \bar{y} is the average of observed data, s^2 is variance of y , n is number of observations, and y_i is the observed data. For each type of the characteristics, with the above S/N ratio transformation, the higher the S/N ratio the better is the result. To simplified the analysis, Taguchi suggests the analyzing the means and S/N ratio using a conceptual approach that involves graphing the effect and identifying the factors visually that appear to be significant without using ANOVA [7]. Conceptual S/N ratio is useful to study the effect of catalyst metals and temperature on the

diameter of CNF effects of the factors involves in the process by looking at the slope of the plotted graph and the mean of the S/N ratio values. The factor optimum condition is determined with this value catalyst according to Taguchi method design of experiment using a standard orthogonal array L₉.

2.3 Size of catalyst desired in CNF development

Based on literature review, most of the size of Fe₂O₃ catalyst will be between 16 – 25 nm. Therefore, the author target on this paper work will be this range. The size of catalyst is important since it will determine the size of CNF in industry usage like lithium-ion batteries.

2.4 Techniques in synthesizing Fe₂O₃ catalyst

2.4.1 Precursor dispersion

A $\mu^3/4$ Fe₂O₃ powder of nanosized particles has been successfully prepared by effectively dispersing the precipitated hydroxide precursor in a sodium chloride matrix. In particular, the hydroxide precursor was converted into crystalline $\mu^3/4$ Fe₂O₃ particles approximately 10 nm in size when it was mechanically activated in the sodium chloride matrix for 20 h. The subsequent calcination at 600 °C for 20 h resulted in a limited degree of coarsening in particle size while the crystallinity of $\mu^3/4$ Fe₂O₃ was further established. The effectiveness of obtaining ultrafine $\mu^3/4$ Fe₂O₃ powders by mechanical activation in the sodium chloride matrix was demonstrated by comparing the powder with those obtained via other routes, such as mechanical activation without sodium chloride as the matrix, calcination at 600 °C, and then mechanical activation in sodium chloride matrix, respectively. None of these processing routes led to a powder comparable in particle characteristics to that derived by the precursor dispersion [11].

2.4.2 Fe₂O₃ films grown by the spin-on gel deposition

The sol preparation method starts with an aqueous solution of an iron salt. In this experiment, we use both Ferric Nitrate and Ferrous Sulfide as the precursor salts to prepare the first aqueous solution. Then, an iron hydroxide powder was precipitated

by adding an aqueous ammonium hydroxide solution. After the powder was separated, it was peptized in acetic acid to constitute the sol required. From this sol, films can be deposited by either the spin-on or dipping techniques. In this work, spin-on at about 4500 rpm in ~40% humid air was selected to produce the films onto glass slide substrates. Films with six spin-on steps and intermediate drying thermal processes at about 100°C during 15 minutes were fabricated. Some samples were thermally treated at 150, 300, 400 and 500°C to find the temperature at which the desired phase is obtained. Usually, a final thermal annealing process during 2 hours at 500°C ensures that we get the hematite phase. Also, sample with 10, 30 and 50 weight percentage of tin in the solution were made by adding the appropriate amount of tin chloride to the starting aqueous solution.

By using the sol-gel spin-on method outlined with the specific sol prepared, nanocrystalline hematite films were obtained with grain size larger than ~31nm. It was found that the hematite structure is achieved by using temperature between 400°C and 500°C [12].

2.4.3 Precipitation method (different procedures)

In this work, we report the synthesis of magnetite (Fe₃O₄) nanoparticles in aqueous solutions without any surfactants. The Fe₃O₄ particles have an average diameter less than 10 nm and a narrow size distribution. This particle size is smaller than heretofore reported. The colloidal suspensions of the magnetite can be then directly oxidized by aeration to form colloidal suspensions of γ -Fe₂O₃. The procedure that we report for the synthesis of Fe₃O₄ and γ -Fe₂O₃ nanoparticles in aqueous solutions contains significant new synthesis modifications. The reaction steps in our process are as follows:



To prepare Fe₃O₄ nanoparticles, homogeneous in size and composition, it is important to perform the above reaction in an aqueous solution with a molar ratio of Fe(II)/Fe(III)) 0.5 and a pH) 11-12. Volumes of 0.85mL of 12.1 N HCl and 25 mL of purified, deoxygenated water (resistance of 17.8 M, by nitrogen gas bubbling for

30 min) were combined, and 5.2 g of FeCl₃ and 2.0 g of FeCl₂ were successively dissolved in the solution with stirring. The resulting solution was added dropwise into 250 mL of 1.5 M NaOH solution under vigorous stirring. The last step generated an instant black precipitate. The paramagnetism was checked in situ by placing a magnet near the black precipitate of Fe₃O₄.

The precipitate was isolated in the magnetic field, and the supernatant was removed from the precipitate by decantation. Purified deoxygenated water was added to the precipitate and the solution decanted after centrifugation at 4000 rpm. After repeating the last procedure three times, 500mL of 0.01M HCl solution was added to the precipitate (with stirring) to neutralize the anionic charges on the nanoparticles. The cationic colloidal nanoparticles were again separated by centrifugation (4000 rpm) and peptized by adding water. The result was a clear, transparent cationic colloid (hydrosol). Samples for transmission electron microscopy (TEM) samples were prepared on 400 mesh copper grids coated with Formvar film. A drop of Fe₃O₄ nanoparticle sol was carefully placed on the copper grid surface and dried. TEM experiments were carried out on a Hitachi- 600 transmission electron microscope. Figure 1 shows a TEM image of Fe₃O₄ nanoparticles. The nanoparticles are nearly spherical and have an average diameter of 8.5 ± 1.3 nm [13].

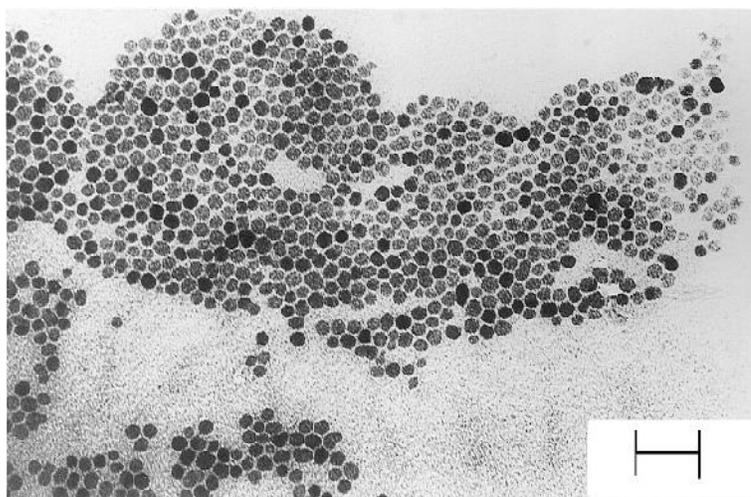


Figure 1: Scanning electron micrograph of Fe₃O₄ particles. Bar length is 30 nm. [13]

CHAPTER 3

METHODOLOGY

The experiment trial to produce iron (III) oxide catalyst is using Taguchi Method. Three factors with three levels of variation are arranged in matrix form according to Orthogonal Array L₉. Temperature, calcinations time and weight of salt used are the factors that involve. Detail on Taguchi Design method shown in Table 1 and Table 2 respectively.

Table 1: Factor and Level for Taguchi Method.

Factors	Level
Temperature	300°C 400°C 500°C
Calcinations time	2 hours 4 hours 6 hours
Salt weight	3g 5g 7g

Table 2: Designed experiment using Taguchi Method.

Experiment	Temperature (°C)	Calcinations Time (hour)	Weight of Salt (g)
1	300	2	3
2	300	4	5
3	300	6	7
4	400	2	5
5	400	4	7
6	400	6	3
7	500	2	7
8	500	4	3
9	500	6	5

300°C – 500°C was chosen as the calcinations temperature. It was reported that at this range of temperature, the crystal structure of Iron (III) Oxide can be produced as the temperature for calcinations greater than 250°C [20]. As the temperature for calcinations increase the crystalline phase of Iron (III) Oxide emerges more clearly however, its crystal size will grow bigger [21]. Therefore 500°C been chosen as the maximum temperature in order to produce as small as possible nano-size catalyst.

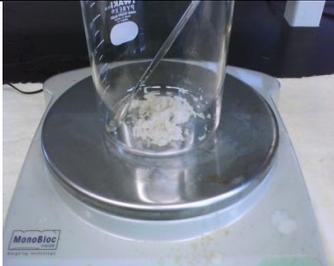
From literature review, mostly experiment was conducted at 4 hours [22,23] and 6 hours [21,24]. 2 hours calcinations time was included to see weather possible smaller size of catalyst can be produce as minimum calcinations time.

Previously, it is reported that the concentration or weight of salt will also affect the size of catalyst produced. As the concentration increase, the bigger size will be produced [25]. 5g for salt was used to produce Iron (III) Oxide using precipitation method was reported [24]. As one of control factor, 3g and 7g were selected to study the behavior of salt concentration in this experiment.

3.1 Catalyst Preparation

For this paper work, precipitation method is chose to synthesize the Iron (III) Oxide (Fe_2O_3). Iron Nitrate Nanohydrate salt is used as the salt precursor. 3g, 5g, and 7g of the salt precursor are weighted and dissolve in 100ml dionized water. The aqueous solution was then mixed with 2M ammonia solution. The precipitate produced then will filtered out and wash several times with dionized water until it is nitrate free. The precipitate is dried over night at 80°C and calcine in the furnace at certain temperature as hours that design using Taguchi Method earlier. The solid catalyst then will grinded into fine powder.

The samples are analyzed using x-ray diffraction (XRD), field emission scanning electron microscopy (FESEM) and Raman Spectroscopy to determine the crystallinity, particle size and to observe the morphology of the carbon structure.

NO	PROCEDURE	
1.	Weighted 3g, 5g, and 7g of the salt precursor respectively.	

2.	100ml deionized water was added to dissolve the salt.		
3.	2M excess ammonia was mixed with aqueous solution.		
4.	The precipitate produced was filtered out and wash several times with deionized water until it was nitrate free.		
5.	Precipitate that left over was dried over night at 80°C.		
6.	The flakes were then ground.		
7.	The catalyst was calcined at design temperature for the selected parameters.		

8. Precipitate was again ground back and sent for testing.

- XRD
- FESEM
- Raman Spectroscopy



CHAPTER 4

SAMPLE CHARACTERIZATION

4.1 Signal to Noise Ratio

The most optimum condition to produce the catalyst is this project will be chosen based on the condition which has the highest S/N ratio.

$$S/N = -10 \text{ Log}_{10} (\text{MSD})$$

- For smaller: $\text{MSD} = (y_1^2 + y_2^2 + y_3^2 + \dots)/n$
- For bigger: $\text{MSD} = (1/y_1^2 + 1/y_2^2 + 1/y_3^2 + \dots)/n$

4.2 XRD

Each sample will scanned with 2θ of 0° to 80° and the data is collected in step mode with interval of 0.02° . The peaks in x-ray diffraction pattern are directly related to the atomic distance (d). The crystal structure also can be determine as well the size of crystal using Scherer equation.

$$D_{\text{size}} = k\lambda/(\beta - \beta_0) \cos \theta$$

where:

k is constant = 0.9

λ is X-ray wave length = 1.5406nm

$\beta - \beta_0$ is FWHM from XRD

θ is angle of diffraction in radian

4.3 FESEM

The morphology and the structure of each sample is studies with Field Emission Scanning Electron Microscopy (FESEM) at accelerating voltage of 20kV. Energy

dispersion X-ray (EDXS) will utilize to determine element present in the samples. This is done by using ZAF quantitative method at accelerating voltage 20kV and spot size of 3.

4.4 Raman Spectroscopy

Raman spectroscopy is the measurement of the wavelength and intensity of inelastically scattered light from molecules. The Raman scattered light occurs at wavelengths that are shifted from the incident light by the energies of molecular vibrations [16]. Typical is use to identify the molecular structure of chemical substance.

CHAPTER 5

RESULT AND DISCUSSIONS

5.1 Sample Observation

A mixing of iron (III) nitrate nanohydrate with 2M ammonia solution had led to a dark brown precipitation that was iron (III) hydroxide. The precipitate was collected after filtration and dried overnight in the oven at 80°C. The precipitate became flakes and turned to dark silver porous solid. The flakes were then ground into powder and calcined at design temperature to convert the precursor to Fe₂O₃. After calcinations the catalyst turns back into flakes and again ground into powder. Once its ground, the powder was in dark red color which is Fe₂O₃.

5.2 Yield of Catalyst

All samples synthesized at 300°C, 400°C and 500°C results a yield around 70 to 99%. The yield of Fe₂O₃ is affected during the filtration. The particles might drain out during filtration process. Hence, the yield for all samples shown in Table 3.

Table 3: Percentage yield for first and second run.

Sample	Temperature (°C)	Calcinations Time (hour)	Weight of Salt (g)	Yield (%) 1 st Run	Yield (%) 2 nd Run
1	300	2	3	74.03	76.43
2	300	4	5	92.00	94.53
3	300	6	7	91.79	89.45
4	400	2	5	99.00	97.02
5	400	4	7	92.53	97.23
6	400	6	3	98.70	94.11
7	500	2	7	98.51	93.72
8	500	4	3	97.22	81.34
9	500	6	5	99.00	98.13

5.3 Main Effect if Iron Oxide Catalyst

Using Taguchi method is it effective in investigating the effects of multiple factors on performance as well as to study the influence of individual factors to determine which factor has more influence, which has less influence [8][9]. Taguchi also provided some key procedures to analyze the result obtain from experiment. By computing the average value is been chose to evaluate this experiment result. The influence factors are being observed. Figure 2 showed the behavior each factor based on average yield of catalyst.

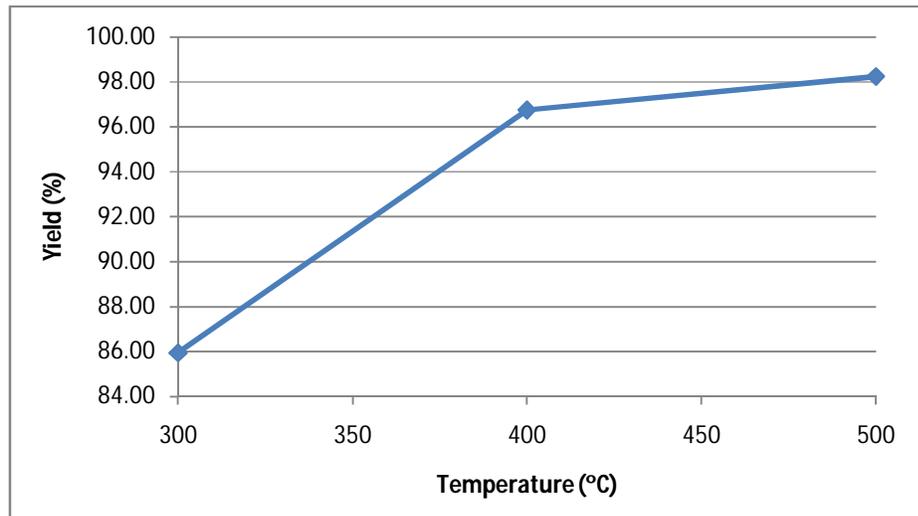


Figure 2: (a) Yield versus Temperature for Fe₂O₃

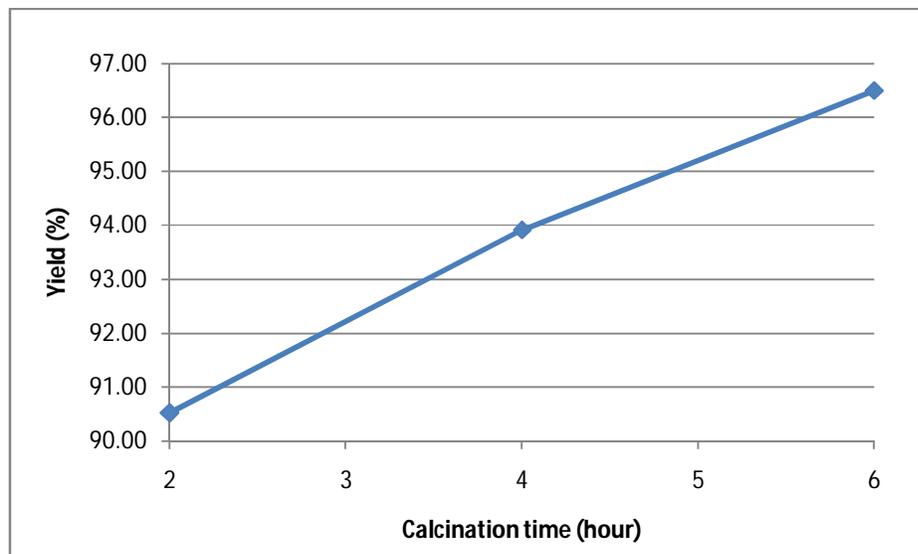


Figure 2: (b) Yield versus Calcination time for Fe₂O₃

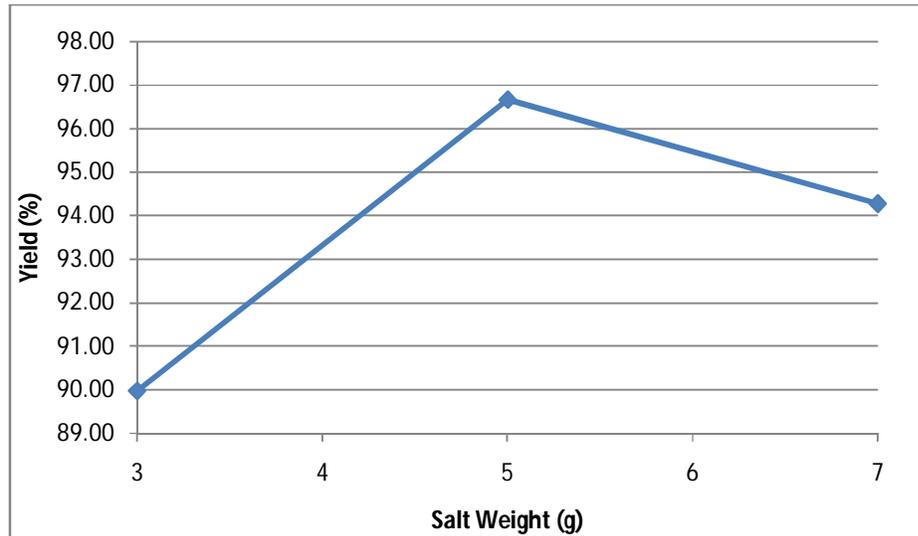


Figure 2: (c) Yield versus Salt Weight for Fe_2O_3

It is observed that the yield of Iron Oxide catalyst is directly proportional with the calcinations time. This result showed that the longer the calcinations time the more time taken for Iron Oxide to fully develop. Meanwhile the yield is increase together with increasing in temperature. Higher the temperature might give more energy for Iron Hydroxide react to form Iron Oxide catalyst. Salt weight behavior showed that weak effect on Iron Oxide catalyst. However at 5g of salt precursor the yield to form Iron Oxide is higher. This showed that at 5g the amount of salt precursor given enough quantity to react with 2M ammonia solution during precipitation process.

5.4 Signal to Noise Ratio

Taguchi suggests the analyzing the S/N ratio using a conceptual approach that involves graphing the effect. The higher the S/N ratio the better is the result [7]. Table 4 showed the S/N ratio of Iron Oxide catalyst. Therefore the optimum condition to produce Iron Oxide catalyst is at 500°C , 6 hours calcinations time with 5g of salt precursor.

Table 4: Signal to Noise ratio of Iron Oxide catalyst

Temperature (°C)	Calcinations Time (hour)	Weight of Salt (g)	S/N
300	2	3	37.53
300	4	5	39.40
300	6	7	39.15
400	2	5	39.83
400	4	7	39.55
400	6	3	39.68
500	2	7	39.66
500	4	3	39.05
500	6	5	39.87

5.5 Field Emission Scanning Electron Microscopic (FESEM) of Iron Oxide

The FESEM images of the catalyst are shown in Figure 3. At 200nm with EHT 5kV the surface morphology of Iron Oxide clearly sees. The shape is found to be in nanorice shape called hematite [17]. Energy dispersion X-ray (EDXS) was utilized to determine the element present in the sample. The result showed only Iron and Oxygen element exist in the sample. Therefore, it confirmed that all the nitrate has been remove during filtration by several wash with dionized water.

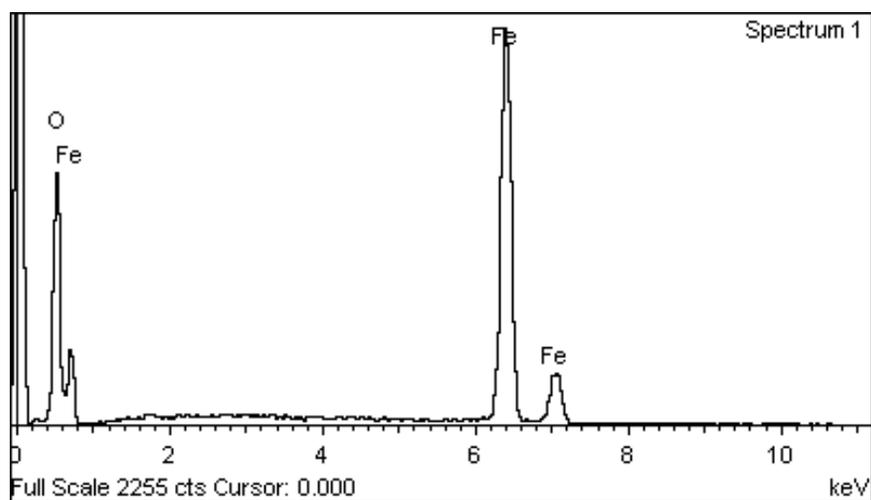


Figure 3: Energy dispersion X-ray (XRDS) Iron (III) Oxide

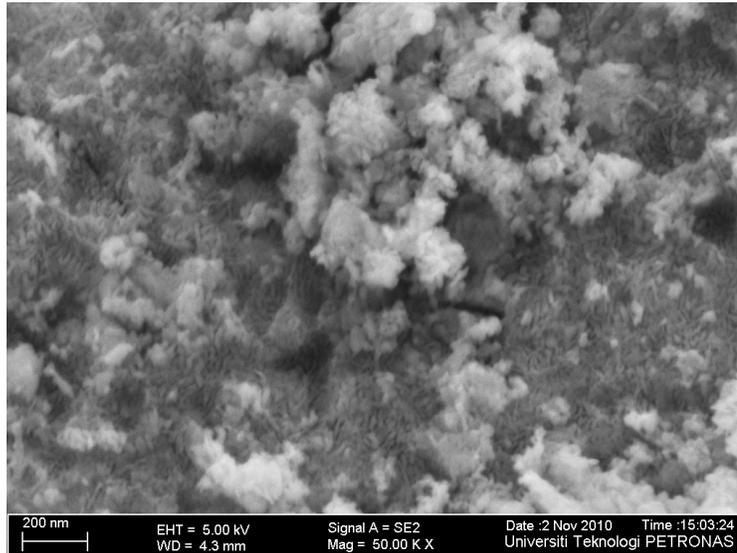


Figure 4: (a) FESEM image of 7g Iron Oxide at 300°C for 6 hours calcination time.

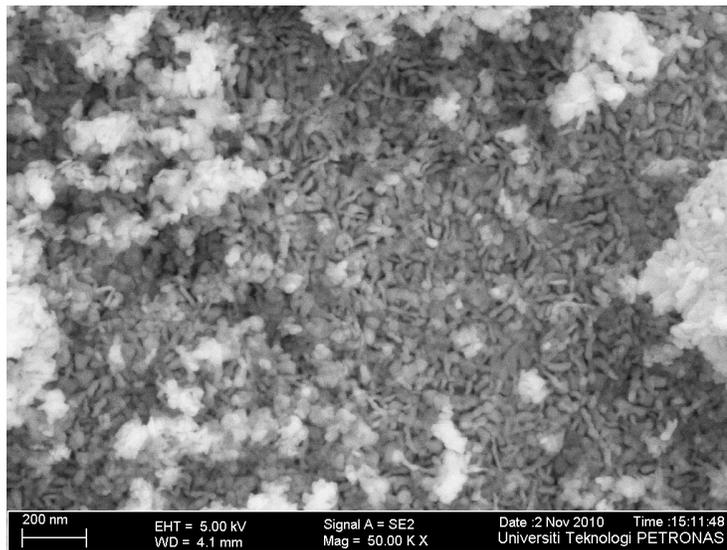


Figure 4: (b) FESEM image of 3g Iron Oxide at 400°C for 6 hours calcination time.

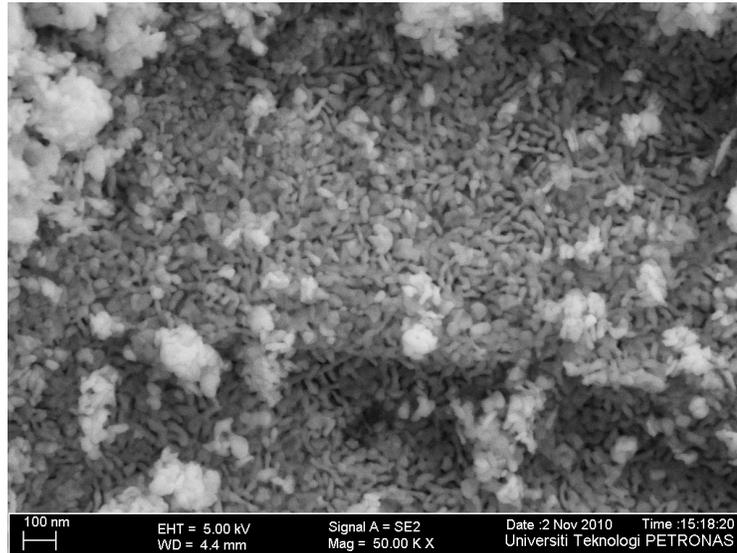


Figure 4: (c) FESEM image of 5g Iron Oxide at 500°C for 6 hours calcination time.

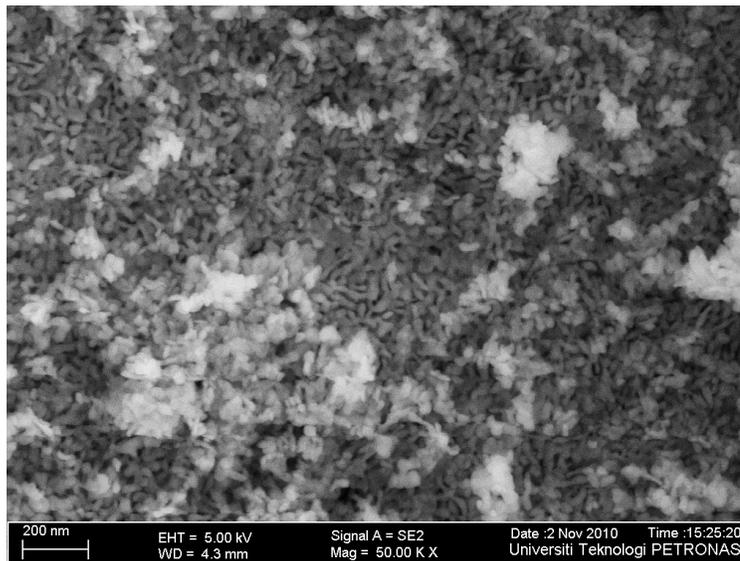


Figure 4: (d) FESEM image of 7g Iron Oxide at 500°C for 2 hours calcination time.

5.6 Raman Spectroscopy of Iron Oxide

The molecular structure of chemical substance in catalyst can be determined using Raman Scattering technique. The single frequency will be used to irradiate the sample. The radiation scattered from molecule which is the vibrational unit of energy different from the incident beam will be detected and recorded. Graph below shown Raman Scattering graph is plot by Raman Scattering versus the wave number or called Raman Shift.

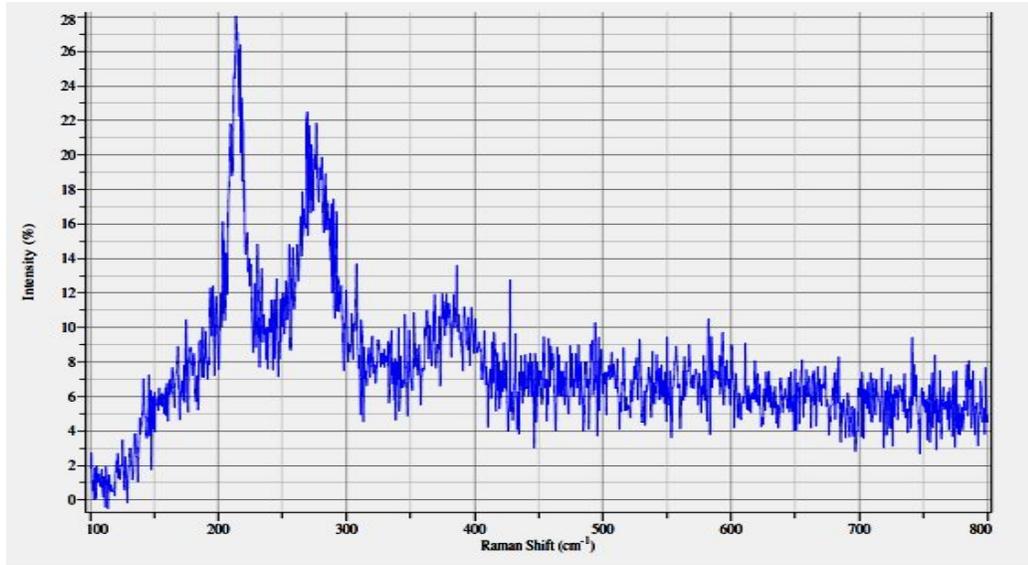


Figure 5: (a) Raman Spectroscopy image of 7g Iron Oxide at 300°C for 6 hours calcination time.

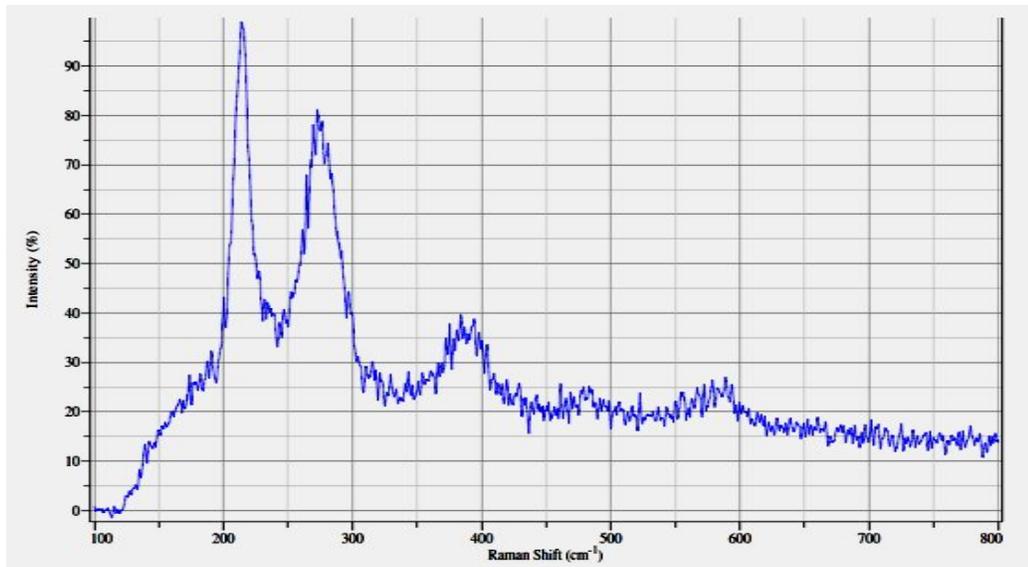


Figure 5: (b) Raman Spectroscopy image of 3g Iron Oxide at 400°C for 6 hours calcination time.

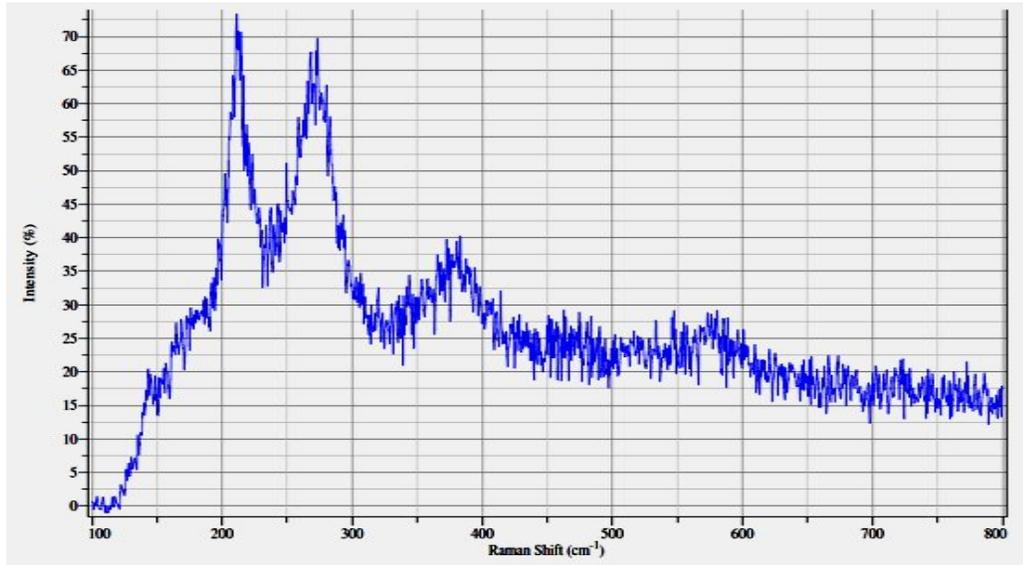


Figure 5: (c) Raman Spectroscopy image of 5g Iron Oxide at 500°C for 6 hours calcination time.

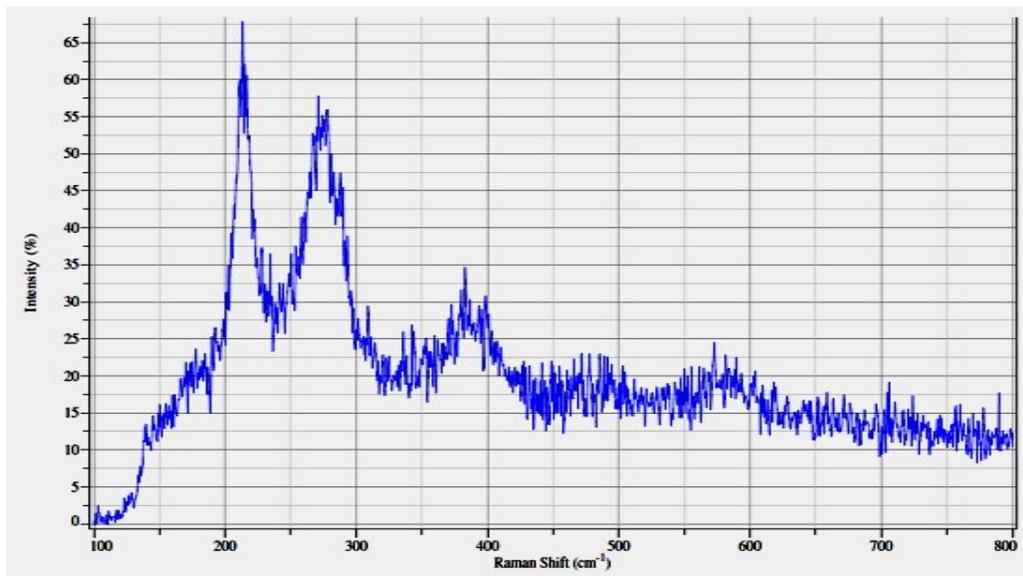


Figure 4: (d) Raman Spectroscopy image of 7g Iron Oxide at 500°C for 2 hours calcination time.

The band for Iron Oxide is broad and the strongest peaks are observed at Raman Shift 212cm^{-1} , 267cm^{-1} , 388cm^{-1} , 492cm^{-1} and 583cm^{-1} . The comparison with literature review showed that this value were found to have lower wave number [18,19]. This due to high degree of disorder because Iron Oxide have smaller partiel size and different specific area. The peak at 212cm^{-1} until 388cm^{-1} are belongs to Fe-O vibrational group. Meanwhile, peak at 492cm^{-1} is due to the vibration energy of

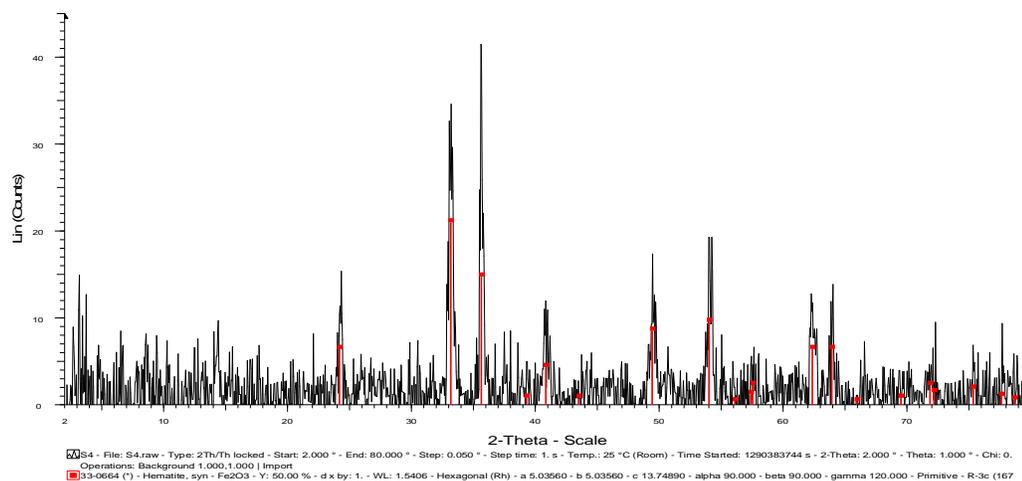


Figure 7 (b): Detail XRD pattern of Fe_2O_3

The result on X-ray diffraction (XRD) analysis can be referred to Figure 7. XRD pattern at different temperature can be seen in Figure 7 (a). At 300°C which was the lowest temperature the peaks were short and broad. This indicates that at this temperature the catalyst were incomplete and not fully developed. Meanwhile, at 400°C and 500°C the peaks clearly can be seen. The stronger peaks shown that at higher temperature better crystallization occurred to produce Fe_2O_3 .

Figure 7 (b) shown the detail peaks of Fe_2O_3 were summerized. Its showed that a pure face phase of hexagonal (Rh) Fe_2O_3 with space group of Primitive R-3c was synthesized. The lattice parameters were $a = b = 5.0356$, $c = 13.7489$, $\alpha = \beta = 90^\circ\text{C}$ and $\gamma = 120^\circ\text{C}$. The peaks that been detected known as crystallographic planes at (012), (104), (110), (006), (113), (202), (024), (116), (211), (018), (214), (300), (208), (1010), (220), (036), and (223). These had been compared with literature review and found out to be matched [22, 26]. Using Scherer equation to calculate the average crystal size of Fe_2O_3 . The result was 24.34nm [APPENDIX I].

CHAPTER 6

CONCLUSION

The catalyst Iron (III) Oxide is successfully synthesized by precipitation method with the purity confirmed with FESEM and Raman Spectroscopy and later with XRD. The most optimum condition for Iron Oxide is at 500°C, 6 hours calcinations time with 5g salt precursor. The average particle size Iron Oxide catalyst is 68.57nm with yield of 98.56%. The average catalyst size of Fe₂O₃ was 24.34nm.

CHAPTER 7

RECOMMENDATION

However, some action may be taken for future improvement to get more accurate result or eliminate disturbance during experiment been carry out.

1. To achieve 100% yield, careful action must be taken during filtration to avoid the precipitate drain out together with nitrate.
2. To eliminate temperature drop during calcinations time while transfer the sample inside the furnace, fast action or shorten time must be taken in order to do that.
3. Add more control factor could bring more accurate result. Example; the concentration of precipitation agent which is ammonia solution or by using different kind of precipitation agent.

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APPENDIX I

Calculation for crystal size using Scherer equation.

$$D_{\text{size}} = k\lambda/(\beta - \beta_0) \cos \theta$$

where:

k is constant = 0.9

λ is X-ray wave length = 1.5406nm

$\beta - \beta_0$ is FWHM from XRD (0.00599)

θ is angle of diffraction in radian (0.312746)

$$\begin{aligned} D_{\text{size}} &= (0.9 \times 1.5406\text{nm})/(0.00599) \cos 0.312746 \\ &= \underline{\underline{24.34\text{nm}}} \end{aligned}$$