PROCEEDING
BOOK I

CHEMMPRO 2014

MINERAL AND MATERIAL PROCESSING

International Seminar on Chemical Engineering
in conjunction with
Seminar Teknik Kimia Soehadi Reksowardojo (STKSR) 2014

"Minerals and Materials Processing Toward Sustainable Development"

Bandung, Indonesia
30 - 31 October 2014

Organized by :
Department of Chemical Engineering
Faculty of Industrial Technology
Institut Teknologi Bandung

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PREFACE

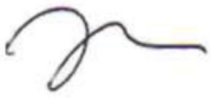
International Seminar on Chemical Engineering in conjuction with Seminar Teknik Kimia Soehadi Reksowardojo (STKSR) was held at East Hall, Institut Teknologi Bandung during 30 – 31 October 2014. This international conference had a theme of "Mineral and Material Processing" which was applicable with the enactment of Law No.4 of 2009 on Mineral and Coal Mining where the ban on the export of unprocessed minerals has been applied since January 2014. Chemical engineers hold a significant role in this area, especially to develop and implement appropriate processing technologies to the mineral resources, which also should considering the sustainable development.

There were five plenary lectures in this two-days conferences, with theme "Sustainable Mineral and Metal Processing" and "The Advancement of Chemical Engineering Technology" along with plenary discussion about "Overcome the Challenges in Indonesia's Mineral and Materials Processing Industry". This proceeding comprises the summary of these outstanding speech and the collected papers that has been presented in the parallel sessions. These papers are divided into several general themes: mineral processing, material processing, material refining and recovery, advance materials, nanotechnology, catalyst, polymers, and others.

The international conference provides an opportunity to publicize research works which done or in ongoing ones in many research institution and showcase their latest advancement and technologies. We have expectation in this occasion is not only a good place to exchange and discuss the progress of their research in chemical engineering that applicable to material and mineral processing, but also a venue to collect and to disseminate the most updated technologies and the researches of regional issue and public interest in order to contribute to the community and to draw support from the industrial and the governmental sectors.

We would like to grateful to all participants and sponsors who has contributed to the conference, to the organizing committee for their commitment in their busy days so that the conference is possible to be held and conducted successfully.

Thank you,



Dr. Dendy Adityawarman
Conference Chairman

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PROGRAM

Day 1: Thursday, 30 October 2014	
	Plenary Session 1
	Sustainable Mineral and Metal Processing
07.30-08.00	Registration
08.00-08.25	CheMMPRO Opening : Vice Rector of Communications, Partnerships, and Alumni Prof.Dr.Ir. Hasanuddin Z. Abidin, M.Sc.
08.25-08.50	Plenary Speaker: Prof. Geoffrey Brooks, Swinburne University of Technology
	Title: Development of Solar Thermal Processing of Minerals
08.50-09.15	Plenary Speaker: Assoc. Prof. M. Akbar Rhamdhani, Swinburne University of Technology.
	Title: Metals from Urban "Ores": Opportunities, Challenges and Technology
09.15-09.35	Discussion
09.35-10.00	Morning Tea / Coffee Break
	Plenary Session 2: Plenary Panel - Business Forum
	Overcome the Challenges in Indonesia's minerals and materials processing industry
10.05-10.25	R. Sukhyar (Directorate General of Mineral & Coal (Dirjen Minerba, Kementerian ESDM)
10.25-10.40	Ir. Hendra Santika, M.M. (PT. ANTAM)
10.40-10.55	Dr.Ir. Rozik B. Soetjipto (Freeport Indonesia)
10.55-11.10	Dinar Aryasena (PT Newmont Nusa Tenggara)
	Title: Throughput Prediction Model Development at Batu Hijau
11.10-11.25	Mr. Graham Brock (Direct Nickel)
	Title: Direct Nickel - Breakthrough Technology
11.25-12.30	Discussion
12.15-13.20	Lunch

Paralel Session 1: 30 October 2014 , 13.20-15.00

13.20-15.00	Room A Advanced material	Room B Industry	Room C Mineral & Material Processing	Room D Mineral Processing
	AM.02	AM.01	MAP.05	MIP.13
	AM.07	IN.02	MAP.06	MIP.05
	AM.04	IN.03	MAP.04	MIP.09
	AM.10	MRY.02	MIP.07	OT.01
	AM.06	MIP.10	MIP.08	MIP.06
15.00-15.30	Afternoon Tea / Coffee Break			

Parallel Session 2

15.30-17.10	Room A Advanced Material & Material Recovery	Room B Advanced Material & Material Processing	Room C Mineral & Material Processing	Room D Others
	AM.05	AM.14	MIP.01	AM.13
	AM.11	AM.12	MIP.03	OT.09
	MRY.07	AM.16	MRY.01	OT.11
	MRY.04	MAP.02	MRY.03	OT.06
	MRY.06	MAP.03	MAP.01	MRY.05
18.00-20.30	Gala Dinner. Venue: House - Sangkuriang			

Day 2: Friday, 31 October 2014				
Plenary Session 3				
The Advancement of Chemical Engineering Technology				
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08.25-08.50	Plenary Speaker: Assoc. Prof Wuled Lenggoro (TUAT, Japan)			
	Title: Assembly of Fine Particles Synthesized from the Gas-Phase			
08.50-09.15	Plenary Speaker: Assist Prof. Manabu Miyamoto (Gifu University)			
	Title: Shape Selectivity of MFI type Core-Shell Zeolite Catalysts			
09.15-09.30	Discussion			
09.35-10.00	Morning Tea / Coffee Break			
Parallel Session 3				
10.00-11.10	Room A Catalyst	Room B Process Modelling	Room C Others (Extraction)	Room D Polymer
	AM.03 CT.02 CT.05 OT.15	MRG.01 MIP.02 MIP.04 MRG.02	AM.08 AM.09 OT.02 OT.03	PL.01 PL.02 PL.03 AM.15
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Parallel Session 4				
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	CT.04 CT.03 OT.07 OT.08 OT.12	NP.01 NP.02 NT.01 NT.02 NT.03	OT.04 OT.05 OT.16 OT.10 OT.13	PL.05 PL.06 PL.09 PL.04 OT.14
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15.00-16.00	Closing Ceremony			

October 30th-31st 2014, Bandung Indonesia
NT.03

International Seminar Chemical Engineering in Minerals and Materials Processing
In conjunction with Seminar Teknik Kimia Soehadi Reksowardojo 2014

Catalytic Performance of Fe_3O_4 Nanoparticles Prepared By Coprecipitation in Oxidation of Methylene Blue and Rhodamine B by H_2O_2

Fauziatul Fajaroh and Sutrisno

Department of Chemistry UniversitasNegeri Malang
Jl Semarang 5 Malang 65119 East Java Indonesia
Email: fau_kim_um@yahoo.co.id

Abstract. Both Methylene Blue and Rhodamine B may cause water pollution. Remediation of water or waste water from these dyes is very important thing to do. This action can be conducted using H_2O_2 as oxidant. But interaction between a catalyst, especially magnetite (Fe_3O_4) nanoparticles, with H_2O_2 will generate more powerfull oxidant ·OH radical. The performancy of the particles as catalyst is a part of the properties of the particles. The aims of this study is to learn the catalytic performance of magnetite nanoparticles in oxidation of model solution of Methylene Blue and Rhodamine B with H_2O_2 as oxidant.This study was carried out in two main stages: (1) Preparation of catalyst, i.e synthesis of Fe_3O_4 nanoparticles by coprecipitation method using aqueous solution of $FeSO_4$ and $FeCl_3$ at room temperature, and characterization of the particles using XRD, BET, and VSM analysis; (2) evaluation of the performance of the particles as catalyst, i.e. oxidation of the dyes without and with catalyst. The result showed that magnetite nanoparticles weresuccesfully synthesized by coprecipitation methode. The particles were identified as magnetite due to JCPDS Card No.19-629 by XRD analysis. The particles show specific surface area of 120.750 m^2/g.The particlesexhibite ferromagnetism with saturation magnetization 62.824 emu/g. The particles was able to accelerate the degradation of Rhodamine B up to 56.24% and Methylene Blue up to 63.93% (within 1 hour). It was also shown that there is an optimum catalyst dosis for system that strengthens the conclusion about chain reaction mechanism of the catalytic reaction.

Keywords: *Fe_3O_4 nanoparticles, catalyst, Rhodamine B, MethyleneBlue.*

1 Introduction

The wide application of iron oxides nanoparticles, especially magnetite (Fe_3O_4) in many field such as technology, medical, and environment, promotes the development of their synthesis methods[1-4]. One simple methods for preparing the particles is coprecipitation, a method with promoting yield that can be conducted even at room temperature. Coprecipitation involves spontaneous reaction between Fe (II) and Fe (III) ions under basic condition attended

nucleation and growing to form ferric ferrous iron oxide (Fe_3O_4) particles after reach its saturated point. Formation of the particle which follow stoichiometric equation below:

$$Fe^{2+}{}_{(aq)} + 2Fe^{3+}{}_{(aq)} + 8OH^-{}_{(aq)} \rightleftharpoons Fe_3O_4{}_{(s)} + 4H_2O_{(l)} \qquad (1)$$

is more favor than the formation of $Fe(OH)_3$, due to fact that $Fe_3O_4$is less soluble in water than $Fe(OH)_3$.By coprecipitation, the desired particles.will be prepared by adjusting the parameters such as the kind and concentration of precursors and also the alkalinity of solution [5–7]. Material in nanoscale exhibite unique properties that different from its macroscale corresponding material, especially its surface area and reactivity. Due to these characters, there are many applications of this material, one of which is as a catalyst. In connection with the role of nanoparticles as catalysts, a new method in the treatment of organic waste has been developed. The method was called Advanced Oxidation Processes (AOPs) [8]. AOPs methods rely on the reactivity of free radicals, especially the hydroxyl radical in oxidizing organic wastes. These radicals are very reactive electrophylic that act quickly in oxidize organic substances through a chain reaction mechanism. Decomposition of H_2O_2 into •OH under acid condition with ions of Fe(II) in solution was called as homogeneous Fenton mechanism [9]. If the role of iron ions is replaced by iron oxide, the mechanism is called heterogeneous Fenton mechanisms or often called Fenton-Like Mechanism. Interaction between H_2O_2 with iron content in iron oxide will generate more •OH. Magnetite nanoparticles can be applied as a catalyst in this heterogeneous Fenton mechanism. Content of iron (II) and iron (III) on the surface of magnetite allowing the Fenton reaction.Its Surface area that much larger than the bulk magnetite increase its catalytic function. The catalyst will be isolated easily by an external magnet, thus allowing it to be reused [10]

Some Efforts to get an effective and efficient waste water treatment should always be done, including for the dyes wastes, especially Rhodamine B and Methylene Blue that we often find in textille industry. Both are cationic dyes which have a different structure complexity. Methylene blue has simpler structure. Both Methylene Blue and Rhodamine B may cause water pollution. Remediation of water or waste water from these dyes is very important thing to do.Various ways have been widely used to tackle the problem, such as coagulation, adsorption, and sonolysis [11-14].

In order to develop environmentally friendly remediation process of polluted water, an effort to examine the catalytic power of magnetite nanoparticles were synthesized by different conditions still needs to be done. Therefore, the

research aims are to study the performance of Fe_3O_4 (Magnetite) nanoparticles that was resulted in coprecipitation synthesis as a catalyst in the oxidation of Rhodamine B and Methylene Blue by H_2O_2. This activity is at once a part of the characterization of the synthesized nanoparticles. In this study, the influence of complexity structure of the dye on the performance of the catalyst will also investigated.

2 Experiment

This study was conducted with three main stages, namely: (1) the synthesis of magnetite nanoparticles by coprecipitation method (2) characterization of the nanoparticles by XRD, BET, and VSM, (3) test the potential of magnetite nanoparticles synthesized as oxidation catalysts of Rhodamine B and Methylene Blue. The tools used in this study were glass apparatus (glassware), a magnetic stirrer (Cimarec), a digital balance (Sartorius), SpectronicGenesys 20, XRD (PANalyticalXpert Pro), BET (Nova 1200 Quanta Chrome), and VSM (Oxford Type 1.2 H). Materials used include sodium hydroxide, iron (III) kloridaheksahidrat, iron (II) sulfate hepta hydrate, powder rhodamine B, methylene blue, hydrogen peroxide, aqua demineralization, and universal indicator.

Synthesis performed at room temperature by adding dropwise of a mixed solution of 0.6 M FeCl3 8 mL and 4 mL of 1.2 M solution FeSO4 into 25 mL of NaOH 1 M. The composition of this mixture is obtained through repeated trials to obtained magnetite in accordance with the standard XRD JCPDS Card No. 19-0629. Black Decomposit was then filtered, washed, and dried in an oven for 2 hours at a temperature of 65-70 ° C. Powder obtained will undergo further characterization by XRD, BET and VSM.

The performance of magnetite nanoparticles as catalysts was studied by determining the percent of oxidized dye (% oxidation) by measuring the absorbance of the reaction system at λ_{max} of each dyes. determination was from 540-560 nm wavelength for Rhodamine B and in the range of 560-670 nm for Methylene Blue. And then the calibration curve was made to determine the concentration of the dye in the wastewater models. Absorbance measurements performed every 15 minutes for 1 hour. Percent oxidation was calculated by the formula:

$$\% \text{ Oxidation} = (1 - A_t/A_0)\text{x } 100\% \quad (2)$$

With A_0 and $A_t$the absorbance in the initial conditions and after a time t. This step result the optimum volume of H_2O_2 30% required to oxidize the dyes. Degradation of Methylene Blue (without catalyst) is done at the same way, only the measurement of absorbance of the system on λ_{maks} 664 nm. The optimum catalyst mass was determined by the degradation of the dyes solution by H_2O_2 30% for 1 hour with a diverse mass of catalyst, which in the range 0.05-0.2 g. Percent oxidation was calculated with the same formula as in the determination of the optimum volume of 30% H_2O_2.Then, percent of the dyes oxidation compared with the percent oxidation without catalyst, in order to obtain information about the catalytic power of magnetite nanoparticles.

3 Result and Discussion

3.1 Synthesis and Characterization of Catalysts

Synthesis of magnetite nanoparticles by coprecipitation method under alkaline conditions resulted in a black precipitate that can be drawn in the form of powders by magnets. Fe_3O_4 black precipitate is formed according to equation (1). Diffractogram of black powder at 2θ angles of 25 to 65 was shown in Figure 1. There are some characteristic peaks at 30.5; 35.9; 37; 43.5; 53.6; 57.3; and 63.1 corresponding to typical peaks of Fe_3O_4 in accordance with JCPDS Card No. 19-0629. Spesific surface area of $Fe_3O_4$was determined by multi-point BET method of isothermal adsorption-desorption of N_2 gas. The results of the analysis using the multi-point BET method showed that the synthesized Fe_3O_4 powder has a specific surface area of 120.750 m2 / g. Assuming that the spherical particles and a non-porous, so the average particle diameter of Fe_3O_4 was 9.59 nm. This indicates that the magnetite nanoparticles has been succesfully synthesized.

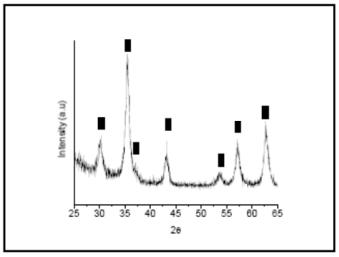

Figure 1 XRD Pattern of As-synthesized Magnetite Nanoparticle

Magnetization curve obtained from the analysis shown in Figure 2. The hysteresis of the magnetization curve shows after saturation is reached. When H is reduced then all domains are not returned in its original orientation. When H back to zero, the material shows the remanent magnetization (Mr) which can eliminated by the applying of coersivity field Hc which has opposite direction of the external field before. The emergence of hysteresis indicates that the synthesized magnetite behave as ferromagnetic material. Magnetite nanoparticles synthesized have a high saturation magnetization Ms of 62.824 emu / g that lower than bulk magnetite (Ms 92 emu / g) [10].

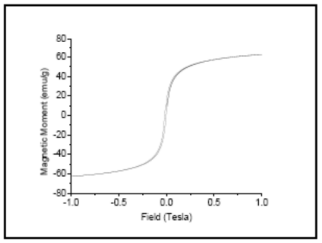

Figure 2 Magnetization Curve of As-synthesized Magnetite Nanoparticle

3.1 Application Magnetite Nanoparticles as Catalysts

3.1.1 Determination of λ_{maks} of Rhodamine B and Methylene blue solutionsand Preparation of calibration curve

The value of λ_{maks} of Rhodamine B and Methylene Blue was obtained from the calibration curve as shown in Figure 3 and 4. The λ_{maks} of Rhodamine B and Methylene Blue, respectively 553 and 664 nm. Determination of the concentration of dye that was used as the sample is based on a standard curve as shown in Figure 5 and 6. Based on these curves concentration of Rhodamine B and Methylene Blue 2.6 and 4 ppm respectively.

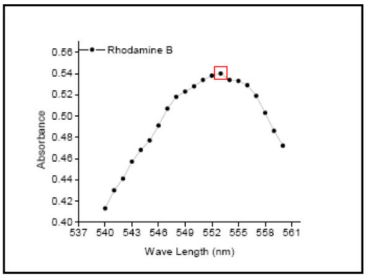

Figure 3 Calibration Curve of Rhodamine B

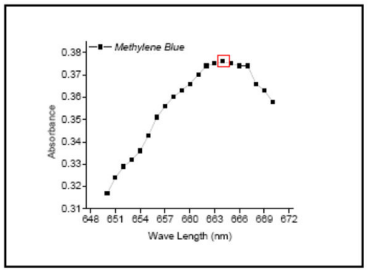

Figure 4 Calibration Curve of Methylen Blue

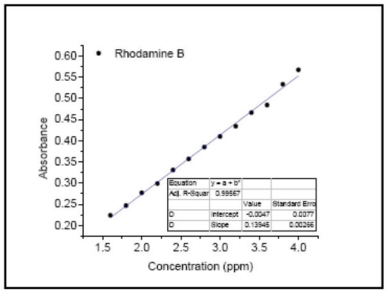

Figure 5 Standard Curve of Rhodamine B

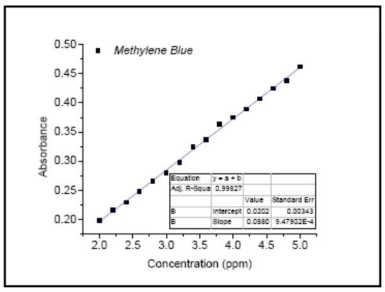

Figure 6 Standar Curve of Methylene Blue

3.1.2 Determination of Optimum Volume of H_2O_2 30% for oxidation Rhodamine B and Methylene Blue Without Catalyst

The oxidation process of both dyes by H_2O_2 takes place at pH system (pH 6), without addition of acid or base. Acidic conditions that are usually applied to homogeneous Fenton reaction will be able to dissolve the catalyst. On the otherhand, the system that is too alkaline will only make the oxidant decomposes into water and oxygen. The oxidation process is followed through color changes quantitatively observed by measuring the absorbance of the solution. The curves that showoxidation yield of Rhodamine B and Methylene Bluewere presented as Figure 7 and 8. As shown in Figure 7 and 8, there is an optimum volume of H_2O_2 (0.8 mL) which describes the optimum concentration of oxidizing agent was required in oxidation of both dyes. The addition of oxidant concentration after the optimum point is actually not profitable. This fact is in accordance with that obtained by Xue et al [9]. This fact strongly suggests that the reaction proceeds through the formation of hydroxyl radicals. The more hydroxyl radicals, the more possibilty of stabilization of these radicals. Equations (2) and (3) describesthis phenomenon.

$$H_2O_2 + \bullet OH \rightarrow \bullet O_2H + H_2O \quad (2)$$

$$\bullet O_2H + \bullet OH \rightarrow H_2O + O_2 \quad (3)$$

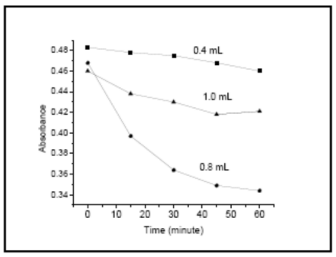

Figure 7 Determination of the optimum volume of 30% H_2O_2 Oxidation of Rhodamine B

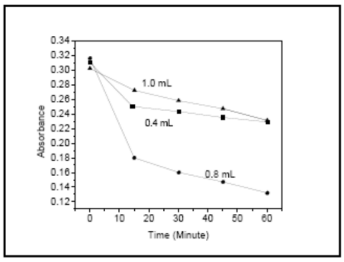

Figure 8 Determination of the optimum volume of 30% H2O2 Oxidation of Methylene Blue

3.1.3 Determination of Optimum Mass of Catalysts (Oxidation with Catalyst)

Percent of Rhodamine B and methylene blue that were oxidized in the presence of varying catalyst mass is given by Figure 9. For both Rhodamine B and Methylene Blue the greater mass of the catalyst initially make % oxidation to go down, but then the increase in mass of the catalyst make the oxidation faster and finally obtained the optimum catalyst mass (0,1 gram) which gives the largest degradation percent (56.24% and 63.93% for Rhodamine B and Methylene Blue). This fact is a proves that the magnetite nanoparticles are able to accelerate the oxidation of both dyes. Once the optimum point is reach, the addition of the mass of catalyst has lower the percent of oxidation. Supposedly percent oxidation is proportional to the amount of magnetite nanoparticles were used. Decreased activity of the catalyst occurs because the increased number of free radicals will provide a great opportunity on the possibility of collision

between free radicals to form a stable molecule[15]. The reaction that occurs in the formation of • OH and oxidation reactions of organic substances by • OH in general [8] alleged as follows:

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH^- + HO\bullet \quad (4)$$
$$Fe^{2+} + HO\bullet \rightarrow Fe^{3+} + OH^- \quad (5)$$
$$HO\bullet + RH \rightarrow H_2O + R\bullet \quad (6)$$
$$R\bullet + Fe^{3+} \rightarrow R^+ + Fe^{2+} \quad (7)$$
$$Fe^{3+} + H_2O \rightarrow Fe^{2+} + H^+ + HO\bullet \quad (8)$$

Based on the facts found and based on the equations above, it appears the role of Fe_3O_4 as a catalyst. In equation (4) and (5) Fe^{2+} is consumed, but in equation (7) and (8) Fe^{2+} is recovered.

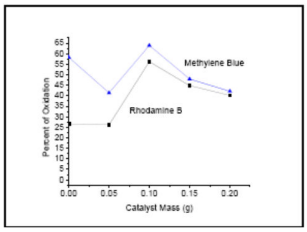

Figure 9 Oxidation Percent of Rhodamine B danMethylen Blue in the presence of catalyst

From Figure 9, it appears that percent of oxidation of Methylene Blue higher than Rhodamine B. It seems that complexity affects the performance of the catalyst structure. The simpler structurally Methylene Blue was more easily adsorbed on the surface of magnetite, and then undergo a process of oxidation.

4 Conclusions

1. Magnetite nanoparticles due to JCPDS Card No. 19-629 were synthesized using a coprecipitation method through a mixture of $FeCl_3$ and $FeSO_4$ at a ratio of 2: 1 under alkaline conditions. The particles have an average size of 9.59 nm, ferromagnetic, and have the saturation magnetization of 62.824 emu / g.

2. The magnetite nanoparticles was able to accelerate the oxidation of Rhodamine B and Methylene Blue. There was an optimum conditions in which Rhodamine B and Methylene Blue, respectively oxidized by 56.24 and 63.93% (within 1 hour). Methylene Blue is more easily oxidized than Rhodamine B. The complexity of the molecular structure factors also influence this catalityc oxidation process.
3. The existence of the optimum mass of catalyst on the oxidation process is a fact that reinforces the notion that this catalytic oxidation reaction follows the chain reaction mechanism.

5 Acknowledgements

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