LEMBAR
HASIL PENILAIAN SEJAWAT SEBIDANG ATAU *PEER REVIEW*
KARYA ILMIAH : PROSIDING

Judul Karya Ilmiah (Prosiding)	:	Zeolite/Magnetite Composites as Catalyst on the Synthesis of Methyl Ester`(MES) from Cooking Oil		
Nama/ Jumlah Penulis	:	Sriatun, Adi Darmawan, Sriyanti, Wuri Cahyani, Hendri Widyandari		
Status Pengusul	:	Penulis pertama		
Identitas Prosiding	:	a. Judul Prosiding	:	IOP Conference Series: Journal of Physics
		b. ISBN/ISSN	:	1742-6588, E-ISSN:1742-6596
		c. Thn Terbit, Tempat Pelaks.	:	2018/1025/012135
		d. Penerbit/Organiser	:	IOP Publishing
		e. Alamat Repository/Web	:	https://iopscience.iop.org/journal/1742-6596
		Alamat Artikel	:	https://iopscience.iop.org/article/10.1088/1742-6596/1025/1/012135/pdf Url Turnitin (9%) http://eprints.undip.ac.id/80669/26/C26_Zeolitemagnetite_composites_as_catalysts_on_the_Synthesis_of_Methyl_Esters_(MES)_from_cooking_oil.pdf
		f. Terindeks di (jika ada)	:	**SCOPUS (SJR 0,221)**

Kategori Publikasi Makalah (beri ✓ pada kategori yang tepat) : ☑ *Prosiding* Forum Ilmiah Internasional
☐ *Prosiding* Forum Ilmiah Nasional

Hasil Penilaian *Peer Review*:

Komponen Yang Dinilai	Nilai Maksimal Prosiding		Nilai Akhir Yang Diperoleh
	Internasional ☑	Nasional ☐	
a. Kelengkapan unsur isi prosiding (10%)	3		3
b. Ruang lingkup dan kedalaman pembahasan (30%)	9		9
c. Kecukupan dan kemutahiran data/informasi dan metodologi (30%)	9		8,6
d. Kelengkapan unsur dan kualitas terbitan /prosiding (30%)	9		8,6
Total = (100%)	**30,00**		**29,2**
Penulis Utama: 0,6 x 29,2 = 17,52			

Catatan Penilaian Paper oleh Reviewer :

1. **Kesesuaian dan kelengkapan unsur isi prosiding:**
   Paper ini telah berisi unsur-unsur penting dalam karya ilmiah, hubungan antara judul, abstak, pendahuluan sampai pembahasan telah sesuai. Nilai 3
2. **Ruang lingkup dan kedalaman pembahasan:**
   Ruang lingkup tentang komposit zeolit/magnetit sebagai katalis dalam sintesis MES dari minyak goreng bekas. Pembahasan dibahas dengan baik dan detil. Nilai 9
3. **Kecukupan dan kemutakhiran data/informasi dan metodologi:**
   Data disampaikan dengan baik dan lengkap dan cukup mutakhir, metodologi cukup detil. Nilai 8,6
4. **Kelengkapan unsur dan kualitas terbitan/prosiding:**
   Unsur terbitan lengkap, kualitas terbitan prosiding cukup baik. Nilai 8,6

Semarang, 3 April 2020
Reviewer 1

Dr. Bambang Cahyono, MS
NIP. 196303161988101001
Unit Kerja : Departemen Kimia FSM UNDIP

LEMBAR
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		f. Terindeks di (jika ada)	:	**SCOPUS (SJR 0,221)**

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Hasil Penilaian *Peer Review*:

Komponen Yang Dinilai	Nilai Maksimal Prosiding		Nilai Akhir Yang Diperoleh
	Internasional ☑	Nasional ☐	
a. Kelengkapan unsur isi prosiding (10%)	3		3
b. Ruang lingkup dan kedalaman pembahasan (30%)	9		9
c. Kecukupan dan kemutahiran data/informasi dan metodologi (30%)	9		8
d. Kelengkapan unsur dan kualitas terbitan/prosiding (30%)	9		8
Total = (100%)	**30,00**		**28**
Penulis Utama: 0,6 x 28= 16,8			

Catatan Penilaian Paper oleh Reviewer :

1. **Kesesuaian dan kelengkapan unsur isi prosiding:**
   Unsur isi prosiding lengkap dan sesuai dengan standart IOP Publishing. Nilai 3
2. **Ruang lingkup dan kedalaman pembahasan:**
   Ruang lingkup artikel ini adalah katalis komposit zeolit/magnetit untuk sintesis ME dari minyak goreng bekas dengan keterbaruan yang tinggi. Pembahasan diulas dengan baik (dalam) disertai literartur pendukung. Nilai 9
3. **Kecukupan dan kemutakhiran data/informasi dan metodologi:**
   Data yang disajikan cukup memadai sebagai artikel dan informasi yang disampaikan ada yang tahun kemautakhirannya kurang, metodologi diulas dengan detil sehingga peneliti lain bisa mengulang dengan baik. Nilai 8
4. **Kelengkapan unsur dan kualitas terbitan/ prosiding:**
   Unsur terbitan lengkap, kualitas terbitan ada yang kurang terbaca (gambar) da salah penulisan (typo). Nilai 8

Semarang, 14 Maret 2020
Reviewer 2

Drs. Gunawan, M.Si, Ph.D
NIP.196408251991031001
Unit Kerja : Departemen Kimia FSM UNDIP

LEMBAR
HASIL PENILAIAN SEJAWAT SEBIDANG ATAU *PEER REVIEW*
KARYA ILMIAH : PROSIDING

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		f. Terindeks di (jika ada)	:	**SCOPUS (SJR 0,221)**

Kategori Publikasi Makalah (beri ✓pada kategori yang tepat) :
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☐ *Prosiding* Forum Ilmiah Nasional

Hasil Penilaian *Peer Review* :

Komponen Yang Dinilai	Nilai Reviewer		Nilai Rata-rata
	Reviewer I	Reviewer II	
a. Kelengkapan unsur isi prosiding (10%)	3	3	3
b. Ruang lingkup dan kedalaman pembahasan (30%)	9	9	9
c. Kecukupan dan kemutahiran data/informasi dan metodologi (30%)	8,6	8	8,3
d. Kelengkapan unsur dan kualitas terbitan/prosiding(30%)	8,6	8	8,3
Total = (100%)	**29,2**	**28**	**28,6**
Penulis Utama(rata-rata): 0,6 x 28,6 = 17,16			

Semarang, 3 April 2020

Reviewer 2

Drs. Gunawan, M.Si, Ph.D
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Unit Kerja : Departemen Kimia FSM UNDIP

Reviewer 1

Dr. Bambang Cahyono, MS
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The 7th International Seminar on New Paradigm and Innovation on Natural Science and Its Application

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Journal of Physics: Conference Series, Volume 1025, The 7th International Seminar on New Paradigm and Innovation on Natural Science and Its Application 17 October 2017, Semarang, Indonesia

2018 *J. Phys.: Conf. Ser.* **1025** 011001

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Abstract

PREFACE

The 7th International Seminar on New Paradigm and Innovation on Natural Sciences and Its Application (ISNPINSA-7) is annual conferences organized by Faculty of Sciences and Mathematics (FSM) Diponegoro University and has been successfully conducted since 2011. The aims of ISNPINSA are to facilitate brain storming and state of the art information in field of sciences and mathematics; to increase innovation of technology that can be applied in industries; to contribute in formulating strategy to increase the role of science for community; and to stimulate collaboration between industries, researchers and government to increase community welfare. The theme of 7th ISNPINSA in 2017 is "*Science and Data Science for Sustainable Development Goals*".

The scope of the field of participants comes from various fields including biology, physics, chemistry, statistics, mathematics, informatics, environment, public health, and relevant fields that contribute to sustainable development. The conference was held in Semarang, Indonesia on October, 17th, 2017. There were three keynote speakers and three invited speaker who came from Japan, Italy, Malaysia, Philipines and Indonesia. The number of participants of this seminar were more than 200

consist of researchers, lecturers, postgraduate and undergraduate students from various universities and after the selection process there are 132 articles selected to be published in the present conference proceeding.

The Editors

Dr. Budi Warsito

Sapto Purnomo Putro, Ph.D.

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Lists of Conference Photographs, Referees, Speakers and Committees are available in this pdf.

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PREFACE

The 7th International Seminar on New Paradigm and Innovation on Natural Sciences and Its Application (ISNPINSA-7) is annual conferences organized by Faculty of Sciences and Mathematics (FSM) Diponegoro University and has been successfully conducted since 2011. The aims of ISNPINSA are to facilitate brain storming and state of the art information in field of sciences and mathematics; to increase innovation of technology that can be applied in industries; to contribute in formulating strategy to increase the role of science for community; and to stimulate collaboration between industries, researchers and government to increase community welfare. The theme of 7th ISNPINSA in 2017 is "*Science and Data Science for Sustainable Development Goals*".

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Subject Area and Category	Physics and Astronomy Physics and Astronomy (miscellaneous)
Publisher	Institute of Physics
Publication type	Journals
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Scope	The open access Journal of Physics: Conference Series (JPCS) provides a fast, versatile and cost-effective proceedings publication service.

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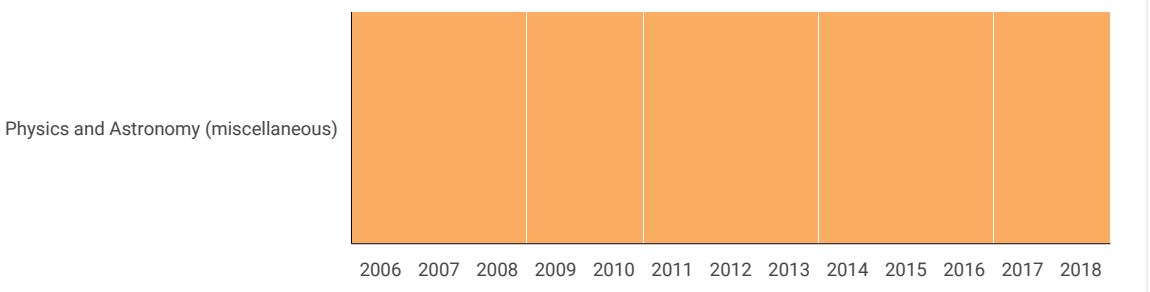

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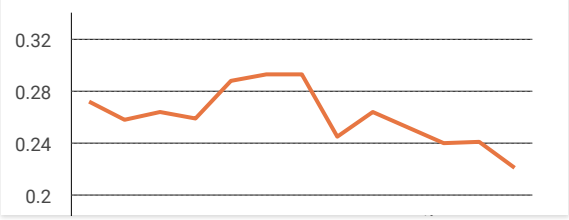

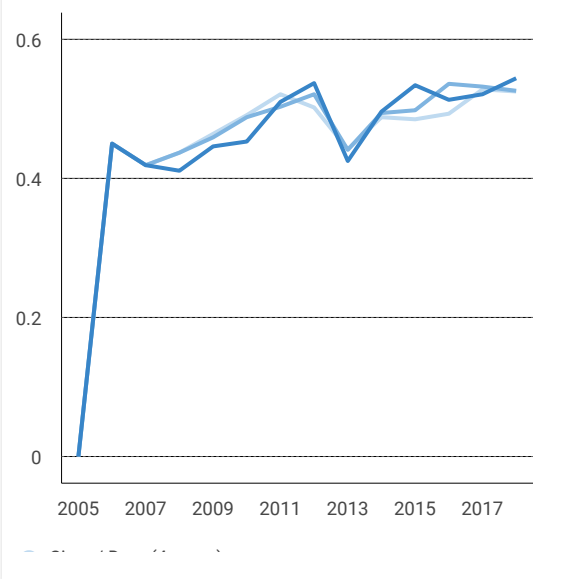

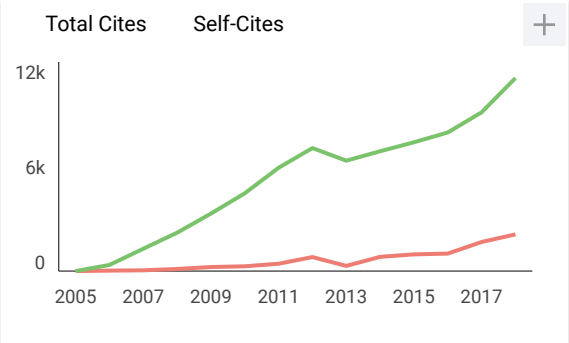

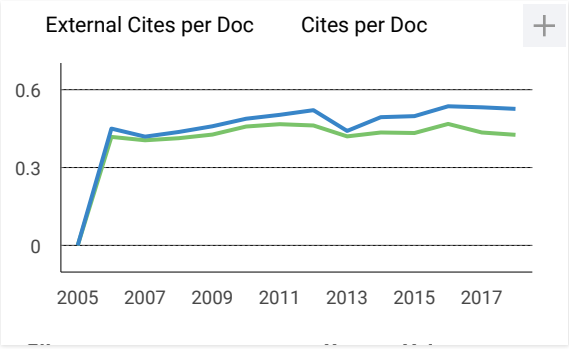

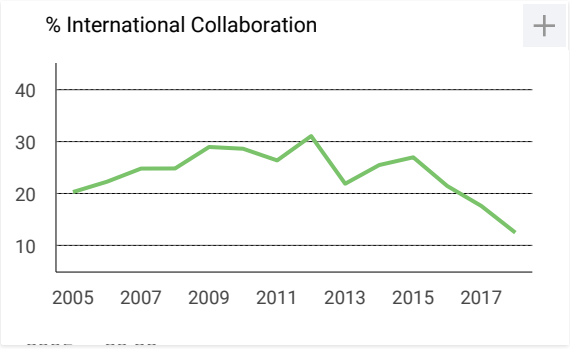

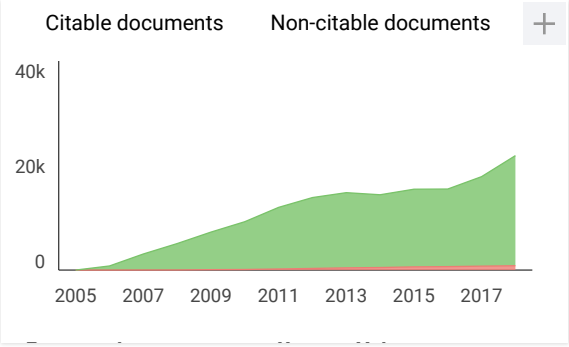

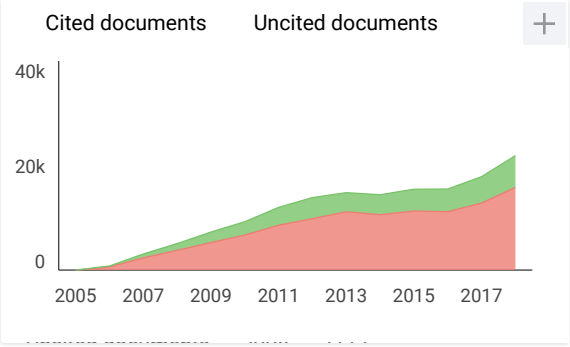

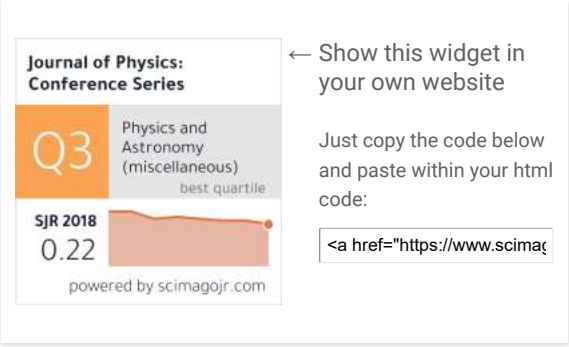

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Lipid production from tapioca wastewater by culture of *Scenedesmus sp.* with simultaneous BOD, COD and nitrogen removal

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Abstract. The use of microalgae to produce biodiesel or possibly remove nutrients from industrial wastewater has gained important attention during recent years due to their photosynthetic rate and its versatile nature to grow in various wastewater systems. In this study, a microalgae, *Scenedesmus sp.*, was cultured to enhance the lipid production and nutrients removal from tapioca wastewater sample. To assess lipid production, *Scenedesmus sp.* was cultured in different concentration of tapioca wastewater sample (from 0 to 100 %), and nutrient removal including BOD, COD, NH_4, NO_2, NO_3 level by *Scenedesmus sp.* was assessed in 100% of tapioca wastewater culture. After 8 days of culture, it was found out that 50% of tapioca wastewater sample resulted in highest concentration of lipid content than that of the other concentrations. The level of environment indicator as nutrient removal such as BOD, COD, NH_4, NO_2, NO_3 were also decreased up to 74%, 72%, 95%, 91%, and 91%, respectively. The pH condition changed from initial condition acidic (pH: 4) to neutral or basic condition (pH: 7-8) as recommended in wastewater treatment system. This research provided a novel approach and achieved efficient simultaneous lipid production and nutrients removal from tapioca wastewater sample by Scenedesmus's culture system.

Keyword: *Scenedesmus sp., tapioca wastewater, lipid production*

1. Introduction

The energy crisis is one of the most important problems faced by all people over the world in the 21st century. The highest consumption of fossil fuels has result in greenhouse effect and causes global climate change [1,2]. One of a type of renewable energy is microalgae biomass-based biofuel, which is considered as one of the most potent substitutes for fossil fuel [2]. However, to increase the production of microalgae biomass, several strategies should be developed, such as modification of culture medium and environmental factors. Hence, one of promising strategies is using wastewater sample as

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Zeolite / magnetite composites as catalysts on the synthesis of methyl esters (MES) from cooking oil

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Zeolite/magnetite composites as catalysts on the Synthesis of Methyl Esters (MES) from cooking oil

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Abstract. The using of zeolite/magnetite composite as a catalyst for the synthesis of methyl esters (MES) of cooking oil has been performed. In this study the natural magnetite was extracted from the iron sand of Semarang marina beach and milled by high energy Milling (HEM) with ball: magnetite ratio: 1:1. The composites prepared from natural zeolite and natural magnetite with zeolite: magnetite ratio 1:1; 2:1; 3:1 and 4:1. Preparation of methyl ester was catalyzed by composite of zeolite/magnetite through transeserification reaction, it was studied on variation of catalyst concentration (w/v) 1%, 3%, 5% and 10% to feed volume. The reaction product are mixture of methyl Oleic (MES), methyl Palmitic (MES) and methyl Stearic (MES). Character product of this research include density, viscosity, acid number and iodine number has fulfilled to SNI standard 7182: 2015.

Keywords: composite, zeolite/magnetite, catalyst, MES

1. Introduction

Increased transportation demand leads to an increase in energy demand globally. Transportation sector is a sector that consumes the most energy, so it can increase environmental emission. Transportation using fuel oil is mainly processed petroleum products, while the world's oil reserves and Indonesia began to thin out. In an effort to find solutions to these problems, various studies reveal the existence of a variety of raw materials from renewable resources capable of being processed into a more environmentally friendly fuel as it reduces CO_2 emissions.

Methyl esters are a major component of biodiesel which is the most appropriate solution to replace the fuel fossils as the world's major source of energy transport, because biodiesel is a material fuel that can replace petrol diesel on a machine. Biodiesel is consisting of a mixture of mono-alkyl esters of long chain fatty acids, which are used as an alternative to fuel from diesel engines. Biodiesel or methyl esters are obtained from the transesterification of animal fats, vegetable oils, and even cooking oil waste, with catalytic process [1].

Actually Vegetable oil has a calorific value that is almost the same with conventional fuel, but usage directly as fuel is still encountered obstacles. When compared to biodiesel, vegetable oils have a much higher viscosity value at temperature 40^{o}C. This may cause problems in the pumping and spraying process when they used as fuel [2]. The incandescent point (flash point) also much larger than diesel oil, this is it inhibit the injection process and result incomplete combustion. The use of oil

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vegetable as a fuel also leaves residue carbon in the engine injector. Efforts to reduce viscosity and flash point vegetable oils, among others, by dilution of oil with solvents, emulsification, pyrolysis, and transesterification. The transesterification process is the most common way done because it does not require energy and temperature tall one. This reaction will produce methyl or ethyl esters, depending on the type of alcohol reacted. Methanol is used more as a source of alcohol because the chain is shorter, more polar and the price cheaper than other alcohols [3].

Both homogeneous and heterogeneous catalysts can be used in transeterification reactions. Basic homogeneous catalysts such as KOH as well as sodium and potassium alkoxide [3], NaOH [4], while acidic ones such as sulfuric acid, sulfonic acid and hydrochloric acid are usually used as catalysts in transesterification reactions. The using of homogeneous catalysts has a disadvantage that is difficulty in the separation of methyl ester products. The lack of homogeneous catalysts can be overcome by using a heterogeneous catalyst that is a solid catalyst, becaused many advantages are obtained when transesterification reactions apply the use of heterogeneous catalysts such as catalysts easily separated from the product, not corrosive and safer and more environmentally friendly [5-7]. Researches on heterogeneous/solid catalysts has been performed such as alkaline or alkaline earth oxides, alkali metals, zeolites, hydrotalcites [8-12], CaO [13], natural bentonite-supported CaO [14].

Zeolite is a porous material widely used as a catalyst based on its properties that have Bronsted acid sites and Lewis acid sites contained in pore zeolite. However, zeolites have less optimum activity and selectivity to convert reactants into desired products. Therefore we need an active phase that can minimize the less expected conditions. The widely used active phases are generally metals or metal oxides. The utilization of natural zeolite as catalyst support for transesterification of palm oil has reported. In the research, KOH was supported into natural zeolit [15]. In this work used magnetite (Fe_3O_4). Magnetite is in great demand as a catalyst because of its biocompatibility, stability, large surface area, and super paramagnetic properties [16]. Magnetite (Fe_3O_4) and γ-Fe_2O_3 which had spiral structure are used to improve biodiesel production [17]. However the magnetite embedded in zeolite as a catalyst in the transesterification reaction of used cooking oil has not been reported. As we know that in the Central Java region has great natural resource potential that is natural zeolite that found in Bayat Klaten area and iron sand/magnetite which is contained in sand of Marina Semarang beach. Therefore, in this research the utilization of both materials as raw material to preparation composite zeolite/magnetite. Furthermore the composite was applied as a catalyst for the transesterification reaction of used cooking oil to produce methyl esters.

2. Experimental Method

2.1. Materials

Sand from Marina beach Semarang, natural zeolite from Bayat Klaten. Both areas are located in Central Java, used cooking oil. Fluoric acid p.a, methanol absolute, polyethylenglycol (PEG) 4000 p.a reagents were supplied by Merck.

2.2. Instruments

X-ray diffraction (XRD) Rigaku Multiplex with Cu Kα radiation (λ = 1.54184 A^o) at generator voltage 40 kV and current 40 mA. Scanning Electron Microscopy (SEM) JEOL, JED 2300. GCMS-TQ8030 Shimadzu Serial O207051.

2.3. Preparation zeolite/magnetite composite catalyst

Zeolite as catalyst support was prepared by activation of natural zeolite from Bayat Klaten using HF 1% solution. The activated zeolite washing until it reaches a neutral pH, then dried to a constant weight. Characterization using XRD is used to determine the constituent components and the structure of material. Magnetite was prepared by extraction methodof sand from marina beach. Treatment by using HEM-3D (High energy milling) method with planetary ball milling is used to purify and reduce the size of magnetite particles. The mass ratio of magnetite:ball was 1:1, speed 400 rpm for 5 h. The milled magnetite characterized by X-ray Diffraction (XRD) and Scanning Electron Microscopy

(SEM). The catalyst of zeolite/magnetite composite was prepared in a way activated natural zeolite was mixed with milled magnetite (Fe_3O_4). The various of zeolite:magnetiteratio mass were 1:1, 2:1, 3:1 and 4:1.The next step, addition of aquadest to the mixture and stirred in a magnetic stirrer at room temperature at 1200 rpm. An amount of PEG 4000 solution as binder was slowly added suspension. The product was dried and characterized by X-ray Diffraction (XRD.

2.4. Application zeolite/magnetite composite as transesterification catalyst

Catalytic activity tests of catalyst to transesterification reaction with used cooking oil as feed. Previously step the used cooking oil are filtered to eliminate impurities and heated to remove the water. Transesterification of used cooking oil in excess methanol (v/v) (1:12) by composite catalyst was carried out at 60^{o}C for 4 hours. The concentration variations of catalyst were 1%, 3%, 5% and 10% (w/v). The resulting product is separated in a separating funnel for 8 hours to form 2 layers. The top layer was methyl ester and the bottom layer was glycerol. The top layer is separated and evaporated to remove the excess methanol which not participate in reaction. Furthermore, the components of the product transesterification were determined by gas chromatography mass spectrometry (GC-MS). Besides that the physical characters such as density, viscosity, acid number, iodine number and content of water also determined. Product methyl ester is weighed.

3. Results and Discussions

3.1. Characterization of zeolite/magnetite composite catalyst

Zeolite activation is done by using fluoric acid. HF solution is selected for zeolite activation because HF only slightly lowers the radius of the zeolite pores, so the radius of zeolite is still large enough and easy to do further modification. The aim of activation by fluoric acid to form decationation that influenced the zeolite surface area to increase due to the decrease of impurity covering the pores of zeolite. The increase of surface area are needed, therefore the catalytic performance of zeolite as catalyst support can be more optimal. The activation process causing the skeleton of zeolite easy to substitute with metal.

The X-ray diffraction of activated natural zeolite shown in table 1. The highest intensity peak is at 2θ = 25.56 °. Identification of peaks mordenite minerals in activated natural zeolites was matched with JCPDS 6-239 data while clinoptilonite minerals were matched with JCPDS data 39-1383 as shown in the Table 1.

Table 1. XRD data of activated natural

Component	2θ Standart (deg)	2θ observed (deg)
Mordenite JCPDS 6-239	13.2	13.36
	19.7	19.52
	25.7	25.56
	27.7	27.62
Clinoptilolite JCPDS 39-1383	22.1	22.18
	26.6	26.54
	29.9	29.98

The effect of the separation using permanent magnet can be seen in figure 1. It is clearly difference in color and size of the iron sand.

Sand before extraction | Extracted material (magnetite) | Milled material (milled magnetite)

Figure 1. The difference of magnetite appearance

Extracted materials from sand is magnetite/iron sand has more dark color. This is due to the reduction of impurities from the iron sand. After milling treatment using HEM-3D, it appears that magnetite has a finer size than before the HEM-3D process. The diminished magnetic particle size results in increased surface area, where it is necessary to enlarge sites that react with feeds. The evidence of magnetite in milled material can be seen in Table 2.

Table 2. The 2Θ XRD data of milled material

Component	2θ Standart (deg)	2θ observed (deg)
Magnetite JCPDS 89-4319	30.083	30.093
	35.434	35.462
	43.064	43.099
	56.949	56.981
	62.536	62.592

The catalytic performance of the magnetite will be affected by the high magnetite surface area. However, due to the small particle size, agglomeration often occurs if the catalyst applied at high temperature. Therefore, magnetite particles supported in the zeolite matrix to prepare composite zeolite/magnetite as a heterogeneous catalyst. This step is expected can be improve both catalytic properties at the transesterification stage in the preparation of methyl esters from used cooking oil. The presence of magnetite in composite that the magnetic properties is expected to facilitate the separation so as to prevent the formation of hydrogen bonds so as to reduce the purity of glycerol. The evidence showing the composite has been formed on Table 3.

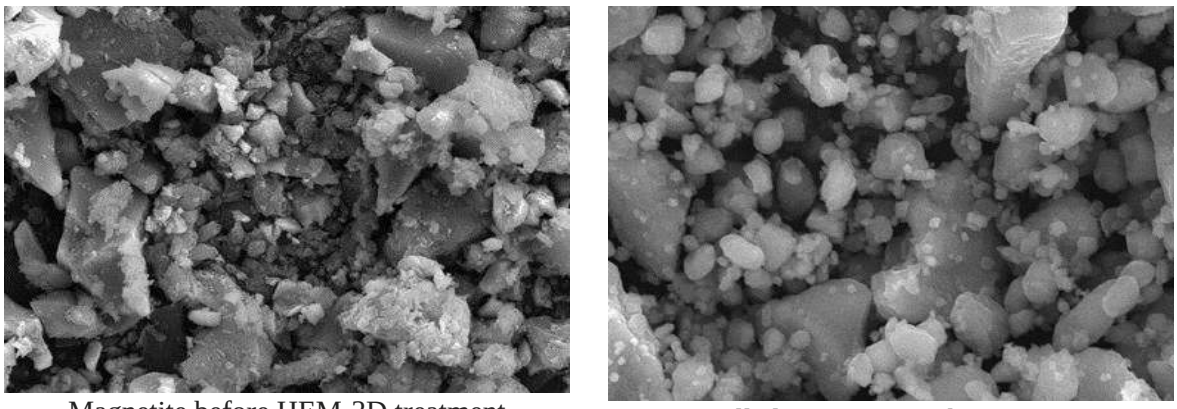
Table 3. The 2Θ XRD data of composite

Component	2θ Standart (deg)	2θ observed (deg)
Mordenite JCPDS 6-239	13.2	-
	19.7	-
	25.7	25.6
	27.7	27.54
Clinoptilolite	22.1	22.14
JCPDS 39-1383	26.6	26.58

	29.9	28.22
Magnetite	30.083	30.1
JCPDS 89-4319	35.434	35.46
	43.064	43.12
	56.949	56.92
	62.536	62.56

3.2. Structure morphology of magnetite

A slightly difference was observed in surface structure of the appearance of the magnetite and after treatment by HEM-3D. The magnetite extracted from the sand shows the area of crystals or magnetite particles appear less clear and regular. In addition, the images in Figure 2 also reveal that the material still contains impurities. While on the magnetite of treatment with HEM-3D has a crystal or particles with the edge specified. From the figure 2 also investigated that the particle size of milled magnetit seems more smooth and homogeneous. It indicates that the milling treatment using a planetary ball mill (high energy milling/HEM-3D) causing a very strong collision between the magnetite particles and the spheres/balls on the planetary ball mill, therefore reducing on magnetite particles size.



Magnetite before HEM-3D treatment Milled magnetite with HEM-3D

Figure 2. SEM images of magnetite

3.3. Zeolite/magnetite composite as catalyst in transesterification reaction

In this transesterification reaction, triglycerides in used cooking oil react with methanol to produce methyl esters and glycerol. This reaction will more quickly achieve equilibrium with the addition of a catalyst. In biodiesel (methyl ester) synthesis, methanol is mostly used as alcohol because of its low cost, availability and production of alkyl esters with lower viscosity [18]. In this research, transesterification reaction of used cooking oil with methanol was catalyzed by zeolite/magnetite composite as heterogeneous catalysts. This composite were formed from zeolite superseded by magnetite. The use of this zeolite/magnetite composite as heterogenous catalyst will be more effectively used in transesterification reactions because it has two active sites of zeolite and magnetite that work simultaneously and for faster triglyceride splitting. Furthermore, this is because the zeolite/magnetite catalyst has two catalytically active sites namely the active site of zeolite in the form of Bronsted acid and the active site of the magnetite in the form of oxide. The Bronsted acid in this zeolite initiates triglycerides to convert the C = O bond to a C-OH bond because the Brønsted acid is able to attack the C = O bond to the C-OH bond. Whereas in this magnetite the iron oxide FeO will bind to methanol.

Based on GC-MS analysis, its known that in this transesterification reaction produce methyl ester compounds.

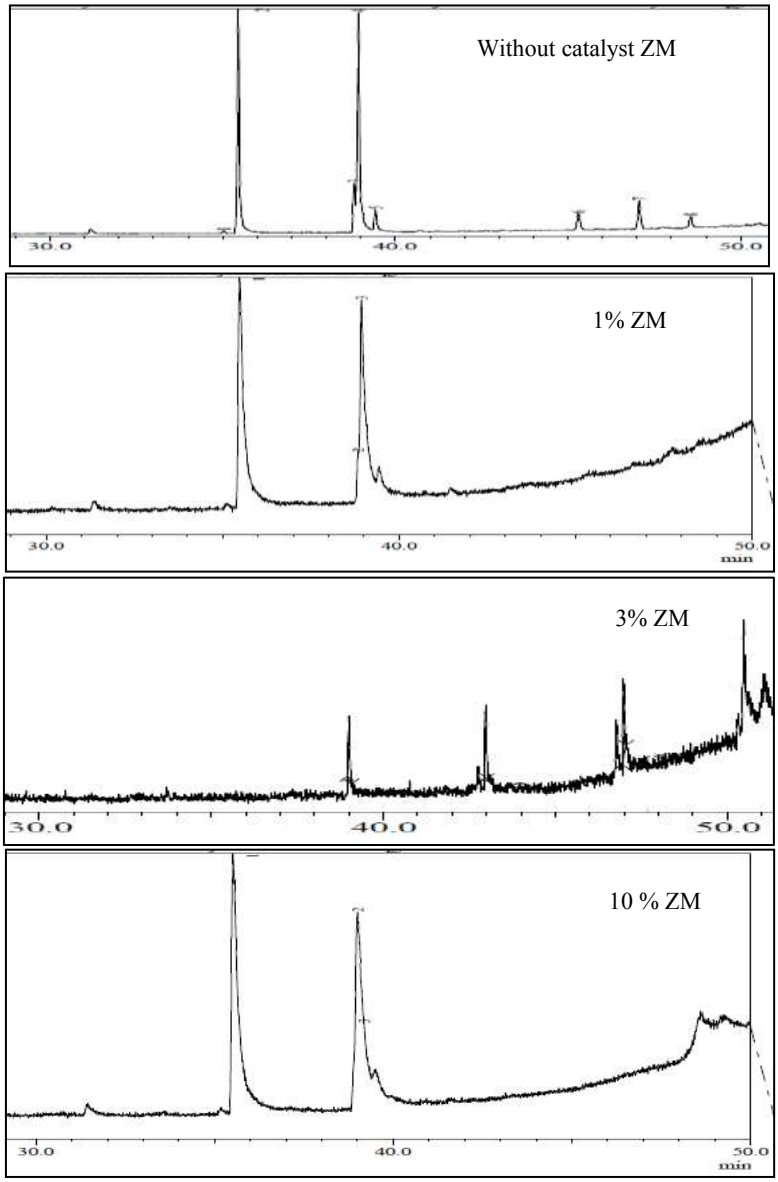


Figure 3. Chromatogram GC of transesterification product

Figure 3 shows the chromatogram GC of transesterification product. Moreover, fragmentation using mass spectroscopy (MS) indicates the presence of methyl esters in the reaction product. Methyl ester of transesterification product without catalyst produces methyl oleic/9-octadecanoic acid (m/z = 296) compound which is the highest peak fragmentation compound followed by methyl palmitic/hexadecanoic acid (m/z = 270) and Methyl stearic/octadecanoic acid (m/z = 270), meanwhile the using of concentration 1% ZM (zeolite/magnetite composite) catalyst resulted the methyl Palmitat (m/z = 270) as the highest peak fragmentation compound followed by methyl Oleic (m/z = 296). For concentration 10% ZM catalyst produces the methyl oleic (m/z = 296) compound which is the highest peak fragmentation followed by Methyl palmitic (m/z = 270).

The influence of the catalyst concentration ranging 1-10 % (w/v) on the transesterification of the used cooking oil is presented in Figure 4. The Fig. 4 shows transesterification reaction efficiency to yielded methyl ester strongly depends upon the catalyst amount, therefore increasing of the catalyst amount would increases the yield percentage. However, utilization of zeolite/magnetite composite catalyst in large amount less effective precisely because the content of water in methyl ester product is

too great. It beyond which no considerable change in oil conversion is observed due to increasing the viscosity of the mixture. Mansir [19] reveals that catalytic conversion of triglycerides to biodiesel/methyl ester involves the use of catalyst which lowers the activation energy and speeds up the reaction. In this process, the amount of catalyst utilized affects the biodiesel content. Usually biodiesel production reaction involves the use of catalyst. Lower catalyst loading is applied in the initial stage of the reaction and the conversion yield of biodiesel is measured. The subsequent catalyst loading will be increased periodically at constant reaction parameters until optimum biodiesel content from a particular feedstock is observed.

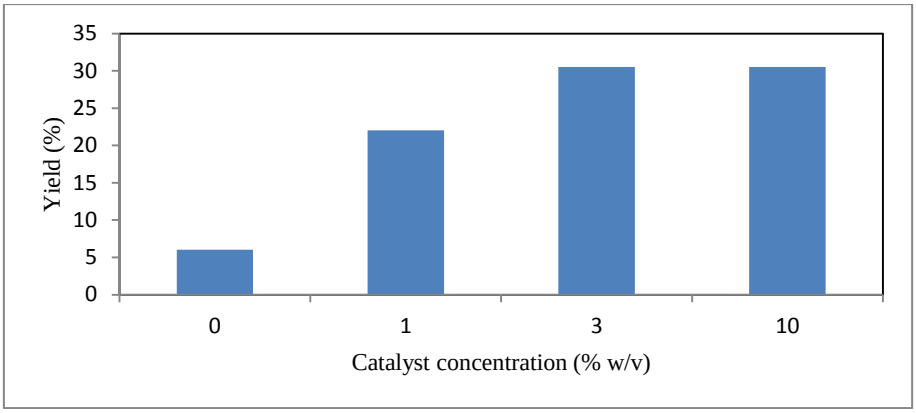


Figure 4. Correlation between Yield of methyl ester and catalyst concentration

3.4. The properties of methyl ester as transesterification product
Investigation of the product of transesterification addressed to its physical properties. The physical properties include density, viscosity, acid number, iodine number and water content. The properties should be determined because reflecting the quality of the methyl ester. Hence it can be decided whether the methyl ester is feasible as a fuel substitute or not.

3.4.1. Density. Density methyl ester as a product from transesterification reaction using various catalyst presents in Figure 5.

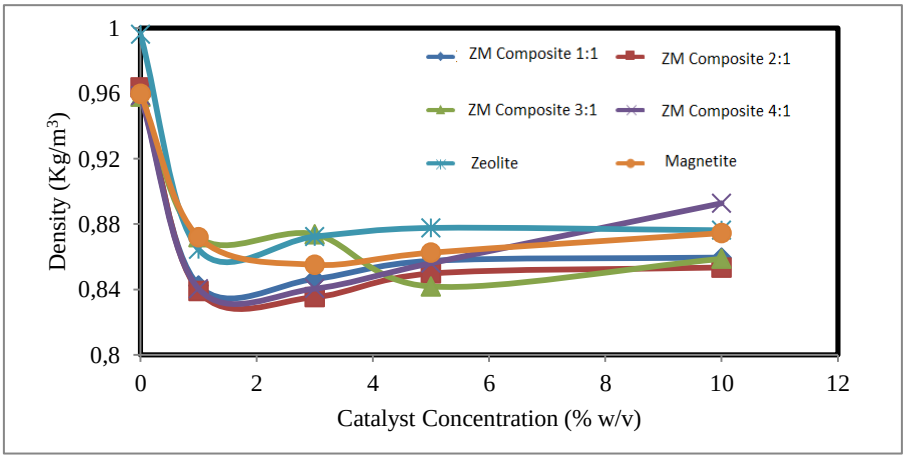


Figure 5. Density of methyl ester using various of catalyst

In Fig. 5 can observe the effect of zeolite/magnetite catalyst concentration to the density of methyl ester. The using of zeolite only, magnetite or composite zeolite/magnetite catalyst causes the density decreased sharply. At various concentrations of catalysts the use of composite catalysts showed less

density than zeolite or magnetite only as catalyst. This suggests that composite catalysts are more effective because of the role of multiple active sites derived in magnetite (Fe_3O_4) as acid sites that initiate reactions with methanol and acid-active sites in zeolite. In all variations of concentration catalyst the range 1-10%, composite which was synthesized by zeolite/magnetite ratio 2:1 shows the lowest density.

3.4.2. Viscosity. The viscosity of methyl ester often called kinematic viscosity is the value of methyl ester treacly as fuel at 40°C. Viscosity of methyl ester as a product from transesterification using various catalyst presents in Figure 6.

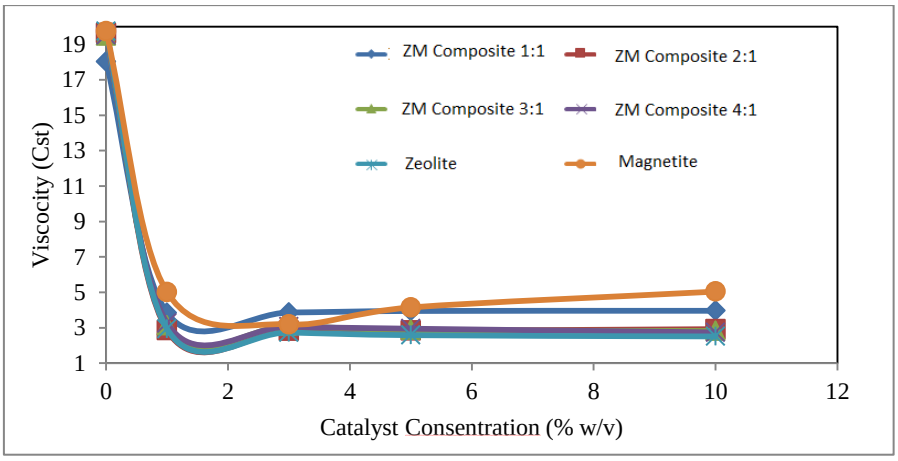


Figure 6. Viscosity of methyl ester using various of catalyst

Viscosity is one of the important properties that must be known if the methyl esters are used instead of fuels. This is because these properties correlate with their performance on the machine. When overly high viscosity will make the fuel become larger droplets that will result in a deposit on the machine, however if the viscosity is too low it will produce a spray that is too smooth to form a rich zone that causes the formation of soot [20]. The role of the catalyst in influencing the viscosity properties has a tendency similar to that of the methyl ester density. Hence in all variations of concentration catalyst the range 1-10%, composite which was synthesized by zeolite/magnetite ratio 2:1 shows the lowest viscosity.

3.4.3. Acid number. The acid number is milligram amount of KOH needed for neutralized 1 gram of sample. The acid number is calculated from the percentage value of fatty acids free. The acid number of methyl ester as a product from transesterification using various catalyst presents in Figure 7.

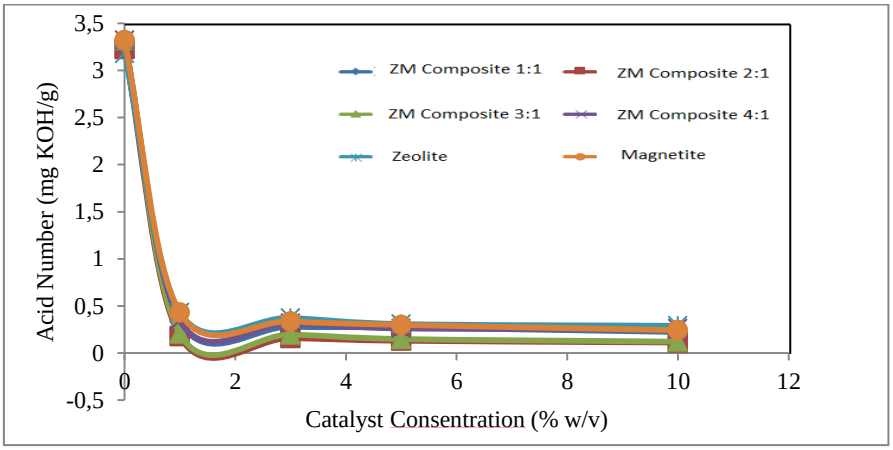


Figure 7. Viscosity of methyl ester using various of catalyst

Large acid numbers show large free fatty acids derived from oil hydrolysis or due to poor processing. The higher the acid number the lower the quality. In this work, the catalyst plays a very significant role in decreasing the acid number. In all variations of concentration catalyst the range 1-10%, composite which was synthesized by zeolite/magnetite ratio 2:1 shows the lowest acid number. When the reaction using zeolite/magnetite ratio 2:1 composite as catalyst cause the acid number increasing slightly. This data reveals that the need for magnetite in assisting the process of initiation of reactions between feeds and methanol.

3.4.4. Iodine number. The iodine number called iodine absorption number is the mass of iodine in grams absorbed at 100 grams of a chemical under the test conditions used. Iodine numbers are often used to determine the amount of unsaturation in fatty acids. The unsaturation is in the form of a double bond, which will react with the iodine compound. The higher the iodine number, the more the C = C bonds present in the fat [21]. Iodine number of methyl ester as a product from transesterification using various catalyst presents in Figure 8.

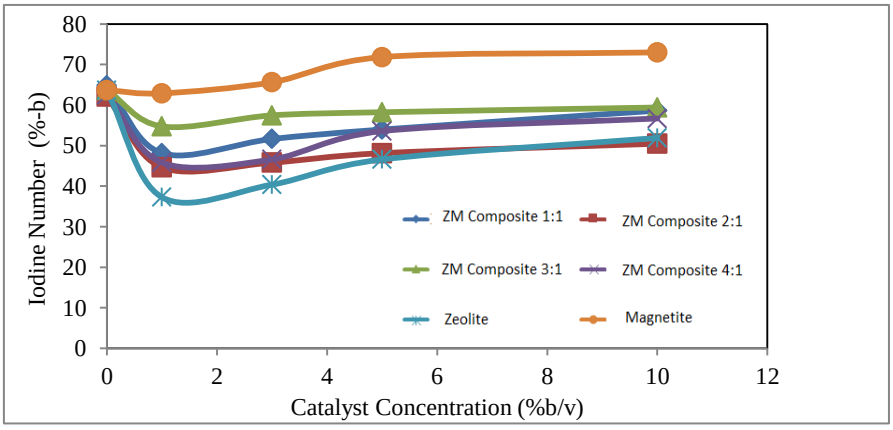


Figure 8. Iodine number of methyl ester using various of catalyst

The higher the iodine value, the higher the number of double-bonded fatty acids contained in the methyl ester. The more double bonds held in the sample have greater potential to experience the polymerization process due to lower stability. The influence of the rise of methyl ester unsaturation can cause CO_2 gas to increase the degree of unsaturation associated with the iodine number. The longer the carbon chain the lower the CO_2 emissions and the higher the iodine number the lower the CO_2 emissions produced [20]. If the methyl ester would be use as biofuel so the number of iodine must

be low. Therefore based on the data in Fig. 8, the low iodine number is belonged by methyl esters of transesterified results catalyzed by zeolites. The explanation argument for this fact is that iodine is most likely absorbed by the pores of the zeolite, where it cannot occur in magnetite because magnetite is not a porous material. This is also cause the highest iodine number on magnetite catalyst. The ability adsorption of zeolite is reduced when zeolite compose with magnetite.

3.4.5. Water content. Water content is the difference of weight methyl ester before and after evaporating. In this work data of water content in methyl ester presents in Figure 9. In this data there is a tendency on catalyst has similarity of role with the viscosity data. This means that the lowest water content in the use of catalysts only zeolite, and the highest water content in magnetite. The role of the pores present in the zeolite structure absorb the water, therefore the catalyst having relatively high porosity as in the zeolite/magnetite 3: 1 composite also shows a low water content.

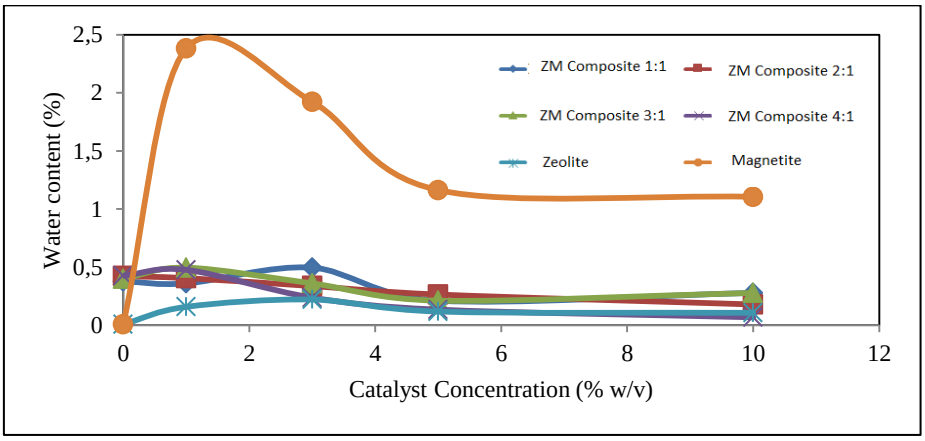


Figure 9. The water content of methyl ester using various of catalyst

4. Conclusion

Preparation of methyl ester was catalyzed by composite of zeolite/magnetite through transeserification reaction using variation of catalyst concentration 1%, 3%, 5% and 10% (w/v) produce a mixture of methyl Oleic (MES) Palmitic Acid (MES) and Stearic Acid (MES). Character product such as density, viscosity, acid number and iodine number has fulfilled to SNI standard 7182: 2015.

Acknowlegment

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Zeolitemagnetite composites as catalysts on the Synthesis of Methyl Esters (MES) from cooking oil

by Sriatun Sriatun

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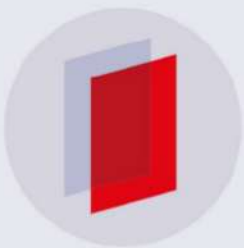

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Zeolite/magnetite composites as catalysts on the Synthesis of Methyl Esters (MES) from cooking oil

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Abstract. The using of zeolite/magnetite composite as a catalyst for the synthesis of methyl esters (MES) of cooking oil has been performed. In this study the natural magnetite was extracted from the iron sand of Semarang marina beach and milled by high energy Milling (HEM) with ball: magnetite ratio: 1:1. The composites prepared from natural zeolite and natural magnetite with zeolite: magnetite ratio 1:1; 2:1; 3:1 and 4:1. Preparation of methyl ester was catalyzed by composite of zeolite/magnetite through transeserification reaction, it was studied on variation of catalyst concentration (w/v) 1%, 3%, 5% and 10% to feed volume. The reaction product are mixture of methyl Oleic (MES), methyl Palmitic (MES) and methyl Stearic (MES). Character product of this research include density, viscosity, acid number and iodine number has fulfilled to SNI standard 7182: 2015.

Keywords: composite, zeolite/magnetite, catalyst, MES

1. Introduction

Increased transportation demand leads to an increase in energy demand globally. Transportation sector is a sector that consumes the most energy, so it can increase environmental emission. Transportation using fuel oil is mainly processed petroleum products, while the world's oil reserves and Indonesia began to thin out. In an effort to find solutions to these problems, various studies reveal the existence of a variety of raw materials from renewable resources capable of being processed into a more environmentally friendly fuel as it reduces CO_2 emissions.

Methyl esters are a major component of biodiesel which is the most appropriate solution to replace the fuel fossils as the world's major source of energy transport, because biodiesel is a material fuel that can replace petrol diesel on a machine. Biodiesel is consisting of a mixture of mono-alkyl esters of long chain fatty acids, which are used as an alternative to fuel from diesel engines. Biodiesel or methyl esters are obtained from the transesterification of animal fats, vegetable oils, and even cooking oil waste, with catalytic process [1].

Actually Vegetable oil has a calorific value that is almost the same with conventional fuel, but usage directly as fuel is still encountered obstacles. When compared to biodiesel, vegetable oils have a much higher viscosity value at temperature 40°C. This may cause problems in the pumping and spraying process when they used as fuel [2]. The incandescent point (flash point) also much larger than diesel oil, this is it inhibit the injection process and result incomplete combustion. The use of oil

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vegetable as a fuel also leaves residue carbon in the engine injector. Efforts to reduce viscosity and flash point vegetable oils, among others, by dilution of oil with solvents, emulsification, pyrolysis, and transesterification. The transesterification process is the most common way done because it does not require energy and temperature tall one. This reaction will produce methyl or ethyl esters, depending on the type of alcohol reacted. Methanol is used more as a source of alcohol because the chain is shorter, more polar and the price cheaper than other alcohols [3].

Both homogeneous and heterogeneous catalysts can be used in transeterification reactions. Basic homogeneous catalysts such as KOH as well as sodium and potassium alkoxide [3], NaOH [4], while acidic ones such as sulfuric acid, sulfonic acid and hydrochloric acid are usually used as catalysts in transesterification reactions. The using of homogeneous catalysts has a disadvantage that is difficulty in the separation of methyl ester products. The lack of homogeneous catalysts can be overcome by using a heterogeneous catalyst that is a solid catalyst, becaused many advantages are obtained when transesterification reactions apply the use of heterogeneous catalysts such as catalysts easily separated from the product, not corrosive and safer and more environmentally friendly [5-7]. Researches on heterogeneous/solid catalysts has been performed such as alkaline or alkaline earth oxides, alkali metals, zeolites, hydrotalcites [8-12], CaO [13], natural bentonite-supported CaO [14].

Zeolite is a porous material widely used as a catalyst based on its properties that have Bronsted acid sites and Lewis acid sites contained in pore zeolite. However, zeolites have less optimum activity and selectivity to convert reactants into desired products. Therefore we need an active phase that can minimize the less expected conditions. The widely used active phases are generally metals or metal oxides. The utilization of natural zeolite as catalyst support for transesterification of palm oil has reported. In the research, KOH was supported into natural zeolit [15]. In this work used magnetite (Fe_3O_4). Magnetite is in great demand as a catalyst because of its biocompatibility, stability, large surface area, and super paramagnetic properties [16]. Magnetite (Fe_3O_4) and γ-Fe_2O_3 which had spiral structure are used to improve biodiesel production [17]. However the magnetite embedded in zeolite as a catalyst in the transesterification reaction of used cooking oil has not been reported. As we know that in the Central Java region has great natural resource potential that is natural zeolite that found in Bayat Klaten area and iron sand/magnetite which is contained in sand of Marina Semarang beach. Therefore, in this research the utilization of both materials as raw material to preparation composite zeolite/magnetite. Furthermore the composite was applied as a catalyst for the transesterification reaction of used cooking oil to produce methyl esters.

2. Experimental Method

2.1. Materials

Sand from Marina beach Semarang, natural zeolite from Bayat Klaten. Both areas are located in Central Java, used cooking oil. Fluoric acid p.a, methanol absolute, polyethylenglycol (PEG) 4000 p.a reagents were supplied by Merck.

2.2. Instruments

X-ray diffraction (XRD) Rigaku Multiplex with Cu Kα radiation ($\lambda = 1.54184$ A^o) at generator voltage 40 kV and current 40 mA. Scanning Electron Microscopy (SEM) JEOL, JED 2300. GCMS-TQ8030 Shimadzu Serial O207051.

2.3. Preparation zeolite/magnetite composite catalyst

Zeolite as catalyst support was prepared by activation of natural zeolite from Bayat Klaten using HF 1% solution. The activated zeolite washing until it reaches a neutral pH, then dried to a constant weight. Characterization using XRD is used to determine the constituent components and the structure of material. Magnetite was prepared by extraction methodof sand from marina beach. Treatment by using HEM-3D (High energy milling) method with planetary ball milling is used to purify and reduce the size of magnetite particles. The mass ratio of magnetite:ball was 1:1, speed 400 rpm for 5 h. The milled magnetite characterized by X-ray Diffraction (XRD) and Scanning Electron Microscopy

(SEM). The catalyst of zeolite/magnetite composite was prepared in a way activated natural zeolite was mixed with milled magnetite (Fe_3O_4). The various of zeolite:magnetiteratio mass were 1:1, 2:1, 3:1 and 4:1.The next step, addition of aquadest to the mixture and stirred in a magnetic stirrer at room temperature at 1200 rpm. An amount of PEG 4000 solution as binder was slowly added suspension. The product was dried and characterized by X-ray Diffraction (XRD.

2.4. Application zeolite/magnetite composite as transesterification catalyst

Catalytic activity tests of catalyst to transesterification reaction with used cooking oil as feed. Previously step the used cooking oil are filtered to eliminate impurities and heated to remove the water. Transesterification of used cooking oil in excess methanol (v/v) (1:12) by composite catalyst was carried out at 60°C for 4 hours. The concentration variations of catalyst were 1%, 3%, 5% and 10% (w/v). The resulting product is separated in a separating funnel for 8 hours to form 2 layers. The top layer was methyl ester and the bottom layer was glycerol. The top layer is separated and evaporated to remove the excess methanol which not participate in reaction. Furthermore, the components of the product transesterification were determined by gas chromatography mass spectrometry (GC-MS). Besides that the physical characters such as density, viscosity, acid number, iodine number and content of water also determined. Product methyl ester is weighed.

3. Results and Discussions

3.1. Characterization of zeolite/magnetite composite catalyst

Zeolite activation is done by using fluoric acid. HF solution is selected for zeolite activation because HF only slightly lowers the radius of the zeolite pores, so the radius of zeolite is still large enough and easy to do further modification. The aim of activation by fluoric acid to form decationation that influenced the zeolite surface area to increase due to the decrease of impurity covering the pores of zeolite. The increase of surface area are needed, therefore the catalytic performance of zeolite as catalyst support can be more optimal. The activation process causing the skeleton of zeolite easy to substitute with metal.

The X-ray diffraction of activated natural zeolite shown in table 1. The highest intensity peak is at 2θ = 25.56 °. Identification of peaks mordenite minerals in activated natural zeolites was matched with JCPDS 6-239 data while clinoptilonite minerals were matched with JCPDS data 39-1383 as shown in the Table 1.

Table 1. XRD data of activated natural

Component	2θ Standart (deg)	2θ observed (deg)
Mordenite JCPDS 6-239	13.2	13.36
	19.7	19.52
	25.7	25.56
	27.7	27.62
Clinoptilolite JCPDS 39-1383	22.1	22.18
	26.6	26.54
	29.9	29.98

The effect of the separation using permanent magnet can be seen in figure 1. It is clearly difference in color and size of the iron sand.

Sand before extraction | Extracted material (magnetite) | Milled material (milled magnetite)

Figure 1. The difference of magnetite appearance

Extracted materials from sand is magnetite/iron sand has more dark color. This is due to the reduction of impurities from the iron sand. After milling treatment using HEM-3D, it appears that magnetite has a finer size than before the HEM-3D process. The diminished magnetic particle size results in increased surface area, where it is necessary to enlarge sites that react with feeds. The evidence of magnetite in milled material can be seen in Table 2.

Table 2. The 2Θ XRD data of milled material

Component	2θ Standart (deg)	2θ observed (deg)
Magnetite JCPDS 89-4319	30.083	30.093
	35.434	35.462
	43.064	43.099
	56.949	56.981
	62.536	62.592

The catalytic performance of the magnetite will be affected by the high magnetite surface area. However, due to the small particle size, agglomeration often occurs if the catalyst applied at high temperature. Therefore, magnetite particles supported in the zeolite matrix to prepare composite zeolite/magnetite as a heterogeneous catalyst. This step is expected can be improve both catalytic properties at the transesterification stage in the preparation of methyl esters from used cooking oil. The presence of magnetite in composite that the magnetic properties is expected to facilitate the separation so as to prevent the formation of hydrogen bonds so as to reduce the purity of glycerol. The evidence showing the composite has been formed on Table 3.

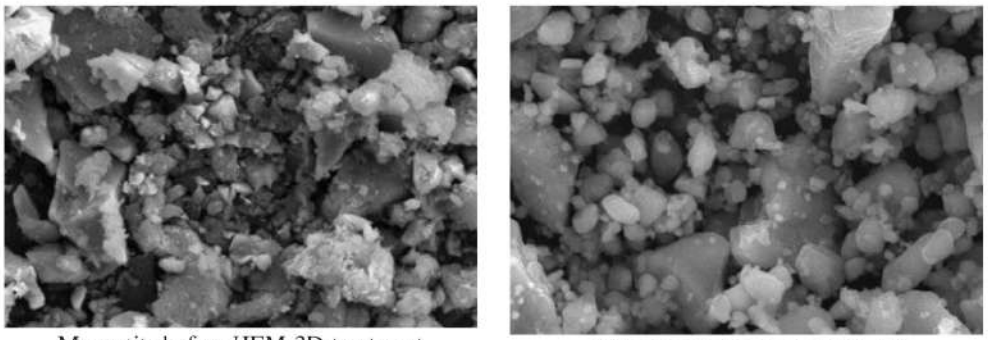
Table 3. The 2Θ XRD data of composite

Component	2θ Standart (deg)	2θ observed (deg)
Mordenite JCPDS 6-239	13.2	-
	19.7	-
	25.7	25.6
	27.7	27.54
Clinoptilolite JCPDS 39-1383	22.1	22.14
	26.6	26.58

	29.9	28.22
Magnetite	30.083	30.1
JCPDS 89-4319	35.434	35.46
	43.064	43.12
	56.949	56.92
	62.536	62.56

3.2. Structure morphology of magnetite

A slightly difference was observed in surface structure of the appearance of the magnetite and after treatment by HEM-3D. The magnetite extracted from the sand shows the area of crystals or magnetite particles appear less clear and regular. In addition, the images in Figure 2 also reveal that the material still contains impurities. While on the magnetite of treatment with HEM-3D has a crystal or particles with the edge specified. From the figure 2 also investigated that the particle size of milled magnetit seems more smooth and homogeneous. It indicates that the milling treatment using a planetary ball mill (high energy milling/HEM-3D) causing a very strong collision between the magnetite particles and the spheres/balls on the planetary ball mill, therefore reducing on magnetite particles size.



Magnetite before HEM-3D treatment Milled magnetite with HEM-3D

Figure 2. SEM images of magnetite

3.3. Zeolite/magnetite composite as catalyst in transesterification reaction

In this transesterification reaction, triglycerides in used cooking oil react with methanol to produce methyl esters and glycerol. This reaction will more quickly achieve equilibrium with the addition of a catalyst. In biodiesel (methyl ester) synthesis, methanol is mostly used as alcohol because of its low cost, availability and production of alkyl esters with lower viscosity [18]. In this research, transesterification reaction of used cooking oil with methanol was catalyzed by zeolite/magnetite composite as heterogeneous catalysts. This composite were formed from zeolite superseded by magnetite. The use of this zeolite/magnetite composite as heterogenous catalyst will be more effectively used in transesterification reactions because it has two active sites of zeolite and magnetite that work simultaneously and for faster triglyceride splitting. Furthermore, this is because the zeolite/magnetite catalyst has two catalytically active sites namely the active site of zeolite in the form of Bronsted acid and the active site of the magnetite in the form of oxide. The Bronsted acid in this zeolite initiates triglycerides to convert the C = O bond to a C-OH bond because the Brønsted acid is able to attack the C = O bond to the C-OH bond. Whereas in this magnetite the iron oxide FeO will bind to methanol.

Based on GC-MS analysis, its known that in this transesterification reaction produce methyl ester compounds.

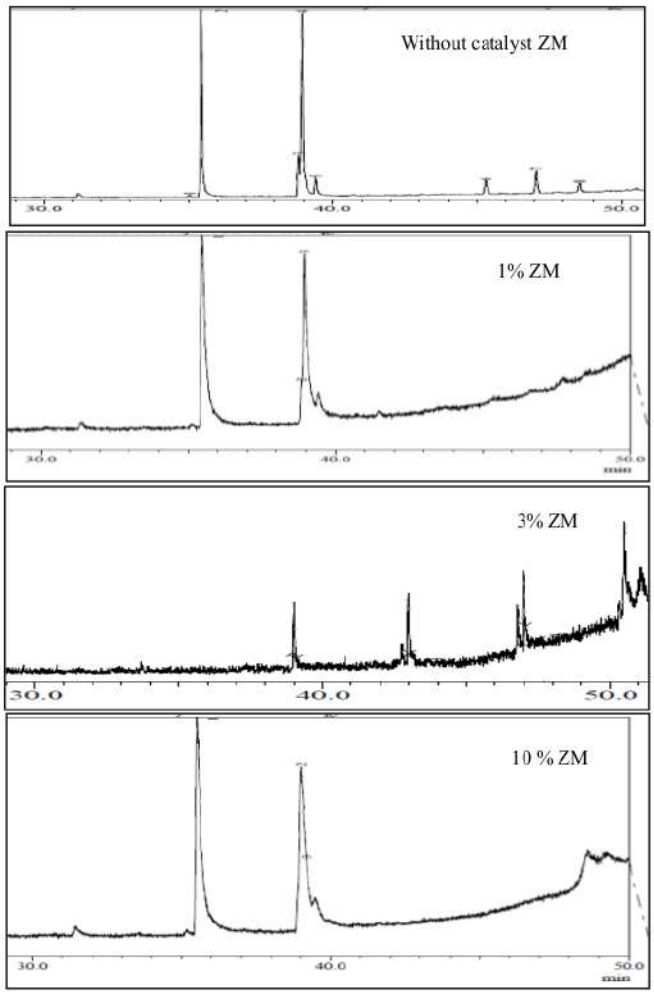


Figure 3. Chromatogram GC of transesterification product

Figure 3 shows the chromatogram GC of transesterification product. Moreover, fragmentation using mass spectroscopy (MS) indicates the presence of methyl esters in the reaction product. Methyl ester of transesterification product without catalyst produces methyl oleic/9-octadecanoic acid (m/z = 296) compound which is the highest peak fragmentation compound followed by methyl palmitic/hexadecanoic acid (m/z = 270) and Methyl stearic/octadecanoic acid (m/z = 270), meanwhile the using of concentration 1% ZM (zeolite/magnetite composite) catalyst resulted the methyl Palmitat (m/z = 270) as the highest peak fragmentation compound followed by methyl Oleic (m/z = 296). For concentration 10% ZM catalyst produces the methyl oleic (m/z = 296) compound which is the highest peak fragmentation followed by Methyl palmitic (m/z = 270).

The influence of the catalyst concentration ranging 1-10 % (w/v) on the transesterification of the used cooking oil is presented in Figure 4. The Fig. 4 shows transesterification reaction efficiency to yielded methyl ester strongly depends upon the catalyst amount, therefore increasing of the catalyst amount would increases the yield percentage. However, utilization of zeolite/magnetite composite catalyst in large amount less effective precisely because the content of water in methyl ester product is

too great. It beyond which no considerable change in oil conversion is observed due to increasing the viscosity of the mixture. Mansir [19] reveals that catalytic conversion of triglycerides to biodiesel/methyl ester involves the use of catalyst which lowers the activation energy and speeds up the reaction. In this process, the amount of catalyst utilized affects the biodiesel content. Usually biodiesel production reaction involves the use of catalyst. Lower catalyst loading is applied in the initial stage of the reaction and the conversion yield of biodiesel is measured. The subsequent catalyst loading will be increased periodically at constant reaction parameters until optimum biodiesel content from a particular feedstock is observed.

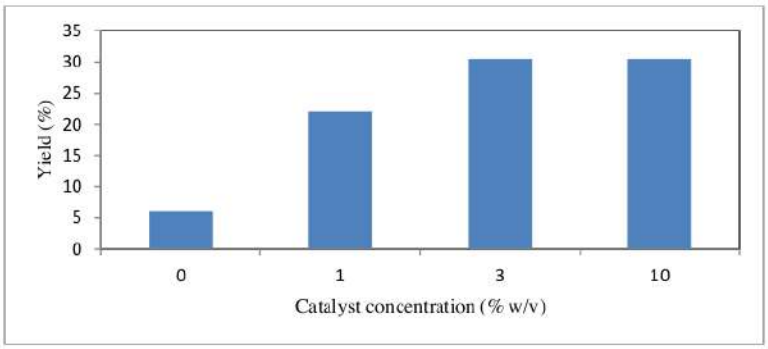


Figure 4. Correlation between Yield of methyl ester and catalyst concentration

3.4. The properties of methyl ester as transesterification product

Investigation of the product of transesterification addressed to its physical properties. The physical properties include density, viscosity, acid number, iodine number and water content. The properties should be determined because reflecting the quality of the methyl ester. Hence it can be decided whether the methyl ester is feasible as a fuel substitute or not.

3.4.1. Density. Density methyl ester as a product from transesterification reaction using various catalyst presents in Figure 5.

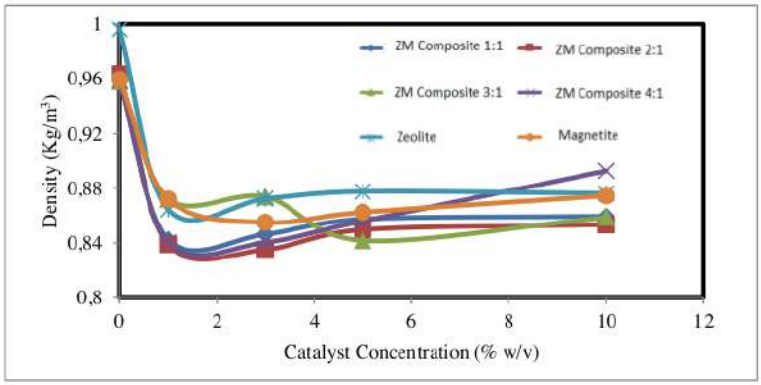


Figure 5. Density of methyl ester using various of catalyst

In Fig. 5 can observe the effect of zeolite/magnetite catalyst concentration to the density of methyl ester. The using of zeolite only, magnetite or composite zeolite/magnetite catalyst causes the density decreased sharply. At various concentrations of catalysts the use of composite catalysts showed less

density than zeolite or magnetite only as catalyst. This suggests that composite catalysts are more effective because of the role of multiple active sites derived in magnetite (Fe_3O_4) as acid sites that initiate reactions with methanol and acid-active sites in zeolite. In all variations of concentration catalyst the range 1-10%, composite which was synthesized by zeolite/magnetite ratio 2:1 shows the lowest density.

3.4.2. Viscosity. The viscosity of methyl ester often called kinematic viscosity is the value of methyl ester treacly as fuel at 40°C. Viscosity of methyl ester as a product from transesterification using various catalyst presents in Figure 6.

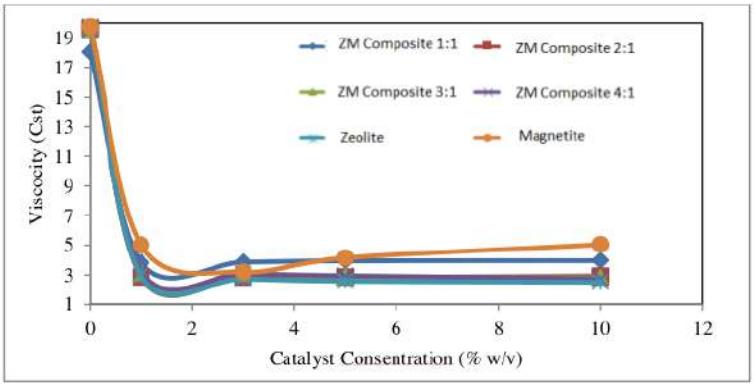


Figure 6. Viscosity of methyl ester using various of catalyst

Viscosity is one of the important properties that must be known if the methyl esters are used instead of fuels. This is because these properties correlate with their performance on the machine. When overly high viscosity will make the fuel become larger droplets that will result in a deposit on the machine, however if the viscosity is too low it will produce a spray that is too smooth to form a rich zone that causes the formation of soot [20]. The role of the catalyst in influencing the viscosity properties has a tendency similar to that of the methyl ester density. Hence in all variations of concentration catalyst the range 1-10%, composite which was synthesized by zeolite/magnetite ratio 2:1 shows the lowest viscosity.

3.4.3. Acid number. The acid number is milligram amount of KOH needed for neutralized 1 gram of sample. The acid number is calculated from the percentage value of fatty acids free. The acid number of methyl ester as a product from transesterification using various catalyst presents in Figure 7.

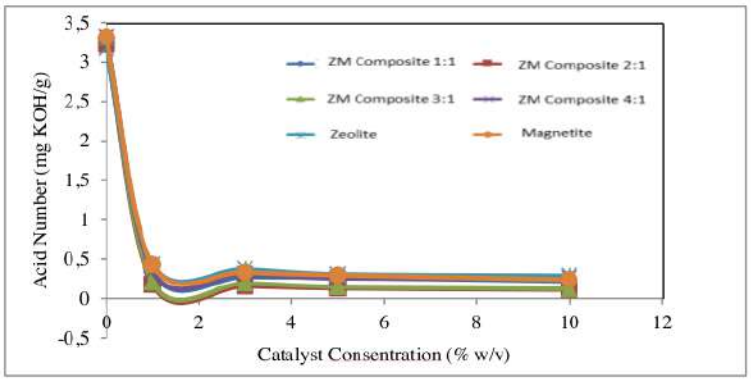


Figure 7. Viscosity of methyl ester using various of catalyst

Large acid numbers show large free fatty acids derived from oil hydrolysis or due to poor processing. The higher the acid number the lower the quality. In this work, the catalyst plays a very significant role in decreasing the acid number. In all variations of concentration catalyst the range 1-10%, composite which was synthesized by zeolite/magnetite ratio 2:1 shows the lowest acid number. When the reaction using zeolite/magnetite ratio 2:1 composite as catalyst cause the acid number increasing slightly. This data reveals that the need for magnetite in assisting the process of initiation of reactions between feeds and methanol.

3.4.4. Iodine number. The iodine number called iodine absorption number is the mass of iodine in grams absorbed at 100 grams of a chemical under the test conditions used. Iodine numbers are often used to determine the amount of unsaturation in fatty acids. The unsaturation is in the form of a double bond, which will react with the iodine compound. The higher the iodine number, the more the C = C bonds present in the fat [21]. Iodine number of methyl ester as a product from transesterification using various catalyst presents in Figure 8.

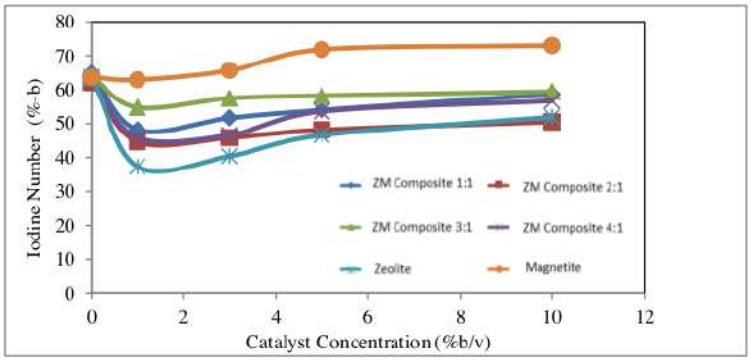


Figure 8. Iodine number of methyl ester using various of catalyst

The higher the iodine value, the higher the number of double-bonded fatty acids contained in the methyl ester. The more double bonds held in the sample have greater potential to experience the polymerization process due to lower stability. The influence of the rise of methyl ester unsaturation can cause CO_2 gas to increase the degree of unsaturation associated with the iodine number. The longer the carbon chain the lower the CO_2 emissions and the higher the iodine number the lower the CO_2 emissions produced [20]. If the methyl ester would be use as biofuel so the number of iodine must

be low. Therefore based on the data in Fig. 8, the low iodine number is belonged by methyl esters of transesterified results catalyzed by zeolites. The explanation argument for this fact is that iodine is most likely absorbed by the pores of the zeolite, where it cannot occur in magnetite because magnetite is not a porous material. This is also cause the highest iodine number on magnetite catalyst. The ability adsorption of zeolite is reduced when zeolite compose with magnetite.

3.4.5. Water content. Water content is the difference of weight methyl ester before and after evaporating. In this work data of water content in methyl ester presents in Figure 9. In this data there is a tendency on catalyst has similarity of role with the viscosity data. This means that the lowest water content in the use of catalysts only zeolite, and the highest water content in magnetite. The role of the pores present in the zeolite structure absorb the water, therefore the catalyst having relatively high porosity as in the zeolite/magnetite 3: 1 composite also shows a low water content.

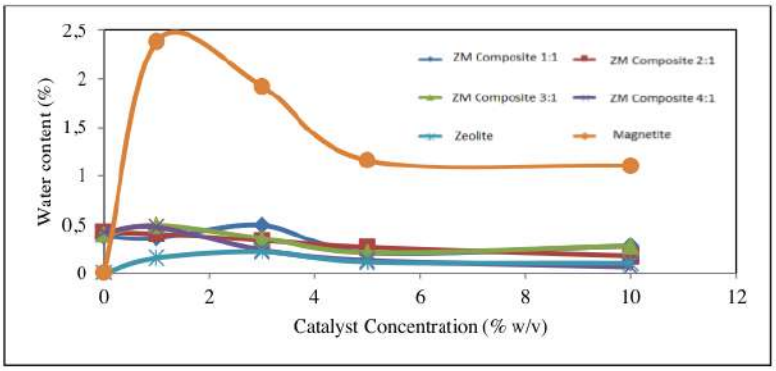


Figure 9. The water content of methyl ester using various of catalyst

4. Conclusion

Preparation of methyl ester was catalyzed by composite of zeolite/magnetite through transesterification reaction using variation of catalyst concentration 1%, 3%, 5% and 10% (w/v) produce a mixture of methyl Oleic (MES) Palmitic Acid (MES) and Stearic Acid (MES). Character product such as density, viscosity, acid number and iodine number has fulfilled to SNI standard 7182: 2015.

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