

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

Judul Karya Ilmiah (Prosiding) : Synthesis and Characterization of Zeolite/Magnetite Composites from Iron Sand of Marina Beach

Nama/ Jumlah Penulis : Sriatun, Adi Darmawan, Sriyanti/3 orang

Status Pengusul : Penulis pertama

Identitas Prosiding :

- a. Judul Prosiding : Advance Science Letters
- b. ISBN/ISSN : 1936-6612. EISSN: 1936-7317
- c. Thn Terbit, Tempat Pelaks. : 2017/Vol. 23 No.7 6524-6526 (3)
- d. Penerbit/Organiser : American Scientific Publishers
- e. Alamat Repository/Web : https://www.ingentaconnect.com/content/asp/asp/2017/00000023/00000007/art00117.jsessionid_qb5q1ggvdm5o.x-ic-live-03

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

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Komponen Yang Dinilai	Nilai Maksimal Prosiding		Nilai Akhir Yang Diperoleh
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a. Kelengkapan unsur isi prosiding (10%)	3		3
b. Ruang lingkup dan kedalaman pembahasan (30%)	9		9
c. Kecukupan dan kemutakhiran data/informasi dan metodologi (30%)	9		8,5
d. Kelengkapan unsur dan kualitas terbitan/prosiding (30%)	9		8,5
Total = (100%)	30,00		29
Penulis Utama: 0,6 x 29 = 17,4			

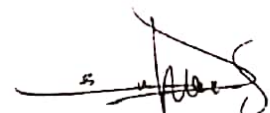
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- Kesesuaian dan kelengkapan unsur isi prosiding:**
Unsur isi prosiding sesuai dan lengkap sesuai dengan format penulisan karya ilmiah. Nilai 3
- Ruang lingkup dan kedalaman pembahasan:**
Ruang lingkup tentang sintesis dan karakterisasi komposit zeolit/magnetit dari pasir besi pantai Marina. Pembahasan cukup baik. Nilai 9
- Kecukupan dan kemutakhiran data/informasi dan metodologi:**
Data cukup, metodologi jelas, referensi mutakhir. Nilai 8,5
- Kelengkapan unsur dan kualitas terbitan/prosiding:**
Unsur terbitan lengkap, kualitas cukup baik. Nilai 8,5

Artikel tersebut masih terindeks Scopus sampai tahun 2017
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Nilai artikel diturunkan menjadi 26
Nilai pengusul : $60\% \times 26 = 15,6$


Dr. Dwi Haryanti, M.Sc
PAK UNDIP

Semarang, 2 April 2020
Reviewer I


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

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HASIL PENILAIAN SEJAWAT SEBIDANG ATAU PEER REVIEW
KARYA ILMIAH : PROSIDING

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b. Ruang lingkup dan kedalaman pembahasan (30%)	9		9
c. Kecukupan dan kemutakhiran data/informasi dan metodologi (30%)	9		8
d. Kelengkapan unsur dan kualitas terbitan /prosiding (30%)	9		8
Total = (100%)	30,00		28
Penulis Utama: $0,6 \times 28 = 16,8$			

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Ruang lingkup artikel ini adalah sintesis dan karakterisasi komposit zeolit/magnetit dari pasir besi pantai Marina dengan kebaruan yang tinggi. Pembahasan cukup dalam diulas disertai literatur pendukung. Nilai 9
- Kecukupan dan kemutakhiran data/informasi dan metodologi:**
Informasi cukup memadai, informasi literatur ada yang kurang mutakhir. Metodologi disajikan dengan detail sehingga mudah untuk peneliti lain mengulang. Nilai 8
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Artikel tersebut masih terindeks Scopus
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Nilai pengusul : $60\% \times 26 = 15,6$

Dr. Dwi Hudiyanto, M.Sc (PAK UNDIP).

Semarang, 23 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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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 kemutakhiran data/informasi dan metodologi (30%)	8,5	8	8,25
d. Kelengkapan unsur dan kualitas terbitan/prosiding(30%)	8,5	8	8,25
Total = (100%)	29	28	28,5
Penulis Utama (rata-rata): $0,6 \times 28,5 = 17,1$			

Semarang, 2 April 2020

Reviewer 2

Drs. Gunawan, M.Si, Ph.D
NIP. 196408251991031001

Unit Kerja : Departemen Kimia FSM UNDIP

Reviewer 1

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

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Nilai pengusul : $60\% \times 26 = 15,6$

Dr. Dwi Hidayat, M.Sc (PAK UNDIP).



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THE REPUBLIC OF INDONESIA
DIPONEGORO UNIVERSITY
FACULTY OF SCIENCES AND MATHEMATICS



CERTIFICATE

No : 603/UN7.3.8/HK/2016

This is to certify that
SRIATUN
as
Presenter

In the 6th International Seminar on New Paradigm and Innovation of Natural Sciences and its Application (ISNPINSA-6) held on 5 - 6 October 2016 at Grand Candi Hotel Semarang Indonesia with paper entitled as follows :

"Synthesis and Characterization of Zeolite/Magnetite Composites from Iron Sand of Marina Beach"

Dean of FSM UNDIP

Prof. Dr. Widowati, M.Si
NIP: 196902141994032002

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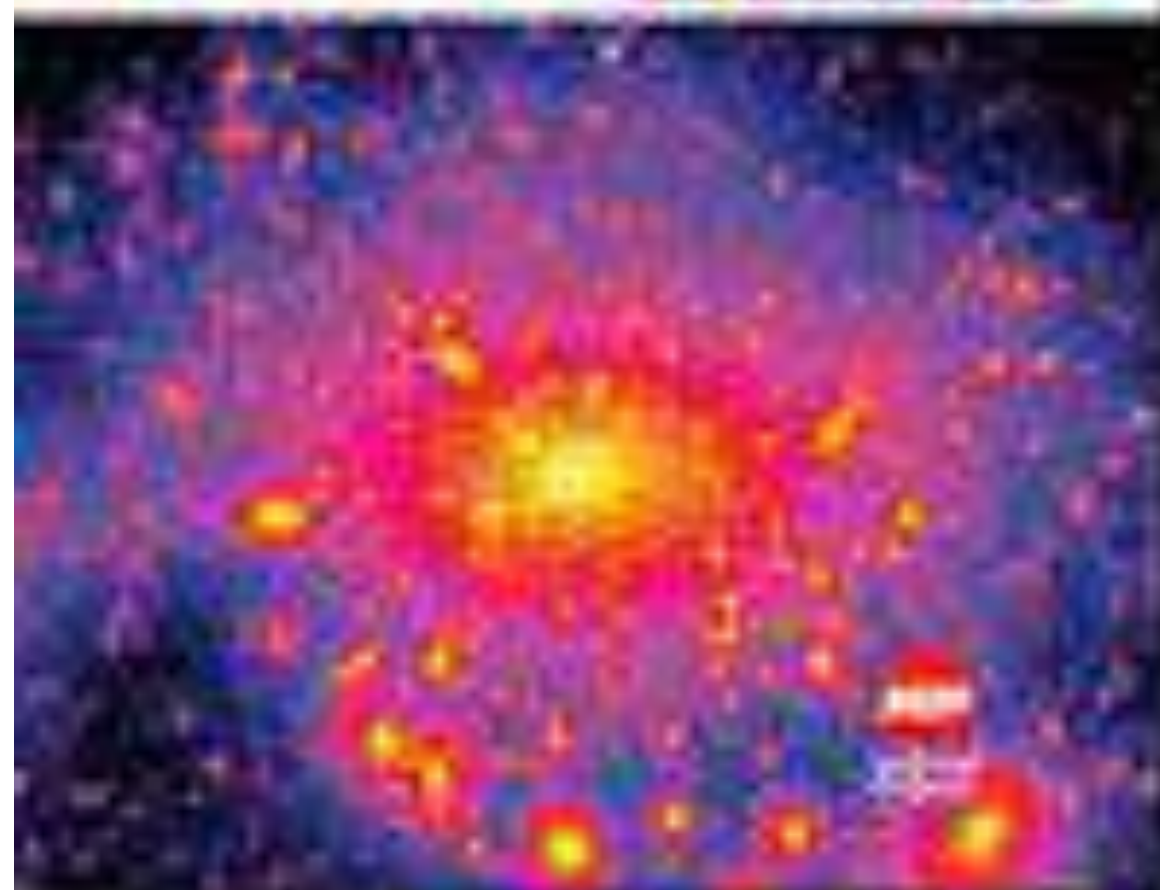
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Selected Peer-Reviewed Articles from the International Conference on Architecture and Built Environment 2016 (ICABE 2016), Kuala Lumpur, Malaysia, 5–6 October, 2016

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Inhalable and Respirable Dust Metal Concentration in Existing and Construction Stage of Airport Infrastructure

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Comparative Study of Thermal Surface Analysis on High-Rise Apartment Facades with Colonial and Neo-Minimalist Style Design in Penang, Malaysia

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Abstract

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Housing serves one of basic human needs. Hence housing quality positively relates to human well-being. This paper explores factors influencing housing demand in Perak. *Issue:* In Perak, the housing supply has yet to respond to the increasing housing demand with regards to the rising population. *Aim:* The aims of this research is to assess the current demand and supply of low-cost housing stock in Perak. *Method:* Content analysis was conducted based on secondary data gathered from government agencies such as National Property Information Centre (NAPIC), Department of Statistics Malaysia and Perak Land and Mineral Office. *Findings:* According to previous studies, nine factors were influencing the demand for low-cost housing. The factors include (i) population growth, (ii) birth rate, (iii) child mortality rate, (iv) income rate, (v) poverty rate, (vi) unemployment rate, (vii) housing stock (viii) inflation rate, and (ix) Gross Domestic Product (GDP). Recent studies discovered that out of the nine factors, only three of the factors significantly influence the housing demand in Perak. These factors were (i) demographic factors, (ii) monthly household income, and (iv) housing supply. The rate of population growth had drastically increased from the year 2000 to 2010. Additionally, more than 50% of the household in Perak had an average monthly income of less than RM 2,999. Despite these issues, the low-cost housing stock from the year 2010 to 2015 had significantly decreased even though the demand for these types of houses was high. The lack of low cost housing units leads to poor well-being as population face (i) burden of high rental, (ii) overcrowding and (iii) substandard housing. Therefore, constant monitoring by the related authorities on the construction of low cost houses is imperatively in need to ensure the continued supply of the low-cost housing targeted to the low-income population.

Keywords: Housing Demand; Housing Supply; Perak

Document Type: Research Article

Affiliations: Department of Urban and Regional Planning, International Islamic University Malaysia (IIUM), 53100 Kuala Lumpur, Malaysia

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Window design and the selection of glazing system have significant effect on daylight illuminance. Occupants' productivity is highly dependent on daylight, as it associates with numerous health advantages. Hence conducting a systematic investigation considering the performance of various window designs and glazing systems is highly important at the early stage of design process. For this purpose, this study attempts to develop a multivariate linear regression model for estimating the average daylight illuminance. To perform the simulations, an office room prototype was modelled by COMFEN 5Beta software. The prototype is a hypothetical office room, as its size, HVAC system and envelopes construction are based on the common practice in construction in Sweden. Because average daylight illuminance is sensitive to window size, orientation, glazing system, design model and position, 544 simulations were performed based on these variable to create an extensive dataset. A multivariate linear regression model was developed based on 90% dataset, which was chosen randomly. The obtained R^2 value was exceeded 96%, which shows an excellent fit for the developed model. The interaction between variables was also studied. The remaining 10% of dataset was utilized for validating the developed model. The validity of the model was further compared with another multivariate linear regression model, developed based on 50% of the dataset. The results show the effectiveness of multivariate linear regression models in supporting architects and predicting average daylight illuminance in early stage of design analysis.

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Source: Advanced Science Letters, Volume 23, Number 7, July 2017, pp. 6613-6617(5)

Publisher: American Scientific Publishers

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Laser treated polystyrene (PS) thin films were explored for simple, robust, and low-cost polymer based electronic applications. Polystyrene nanospheres of 500 nm were drop coated on silicon wafer before laser treatment was introduced to systematical investigated the structural transformation of treated PS. The relationship between the parameters used and the structural changes of PS, especially for its surface chemistry and the morphological, structural properties were characterized with Attenuated total-reflection Fourier transform infrared spectroscopy (ATR-FTIR), X-ray diffraction (XRD) and Field emission electron microscopy (FESEM). It was revealed that the morphological changes observed in the laser treated PS films were the dominant factor for the improvement of modified PS that can be used to tailor functional polymer such as organic light-emitting diodes (OLED), carbonaceous nanostructure, graphene, graphene oxide. Zwitter characteristic of the PS can be clearly observed during laser irradiation; over exposure of laser could be used to tailor different materials on the surface of the PS.

Keywords: Laser Irradiation; Polystyrene; Zwitter Characteristic

Document Type: Research Article

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Abstract



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The Medina of Tunis, or the vernacular heart of the Arabic-Islamic city, has been known for centuries as an important center of Muslim civilization and a crossroad of Muslim and non-Muslim cultures in North Africa. Its urban fabric, its built environment, and its architectural patterns are a manifestation of a creative and a sustainable process whereby the environment has had an important effect on the planning of the Medina. By analyzing the structure of this vernacular settlement, this paper aims to explore the concept of sustainability in this settlement while referring to the studies of 14th-century master builder Ibn Al-Rami. Its environmental qualities are explored through the gradual exploration of its urban components from the level of the street to the level of the courtyard to explain their participatory role in enhancing cultural and social values of Islam.

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Volume 23, Issue 7, July 2017, Pages 6524-6526

Synthesis and characterization of Zeolite /Magnetite composite from iron sand of marina beach [\(Article\)](#)

[Sriatun](#), Darmawan, A., Sriyanti

Chemistry Department, Sains and Mathematic Faculty, Diponegoro University, Indonesia

Abstract

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The synthesis and characterization of zeolite / magnetite composite was carried out. The iron sand from Marina Beach of Semarang Central Java was used as a source of magnetite and the natural zeolite from Bayat Klaten Central Java as a host material. The magnetite was impregnated into zeolite by a simple chemical route. The structural and crystallinity of the product were characterized by XRD. Whereas the content of the component and morphological properties were determined by SEM and EDX respectively. The XRD patterns show that composite of zeolite /magnetites has been formed and there were high intensity sharp peaks which means the samples are crystalline. The SEM EDX data explain that samples which prepared by zeolite : magnetite ratio 1:1 produced more homogenous size and shape of the particle. © 2017 American Scientific Publishers All rights reserved.

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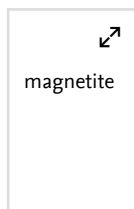
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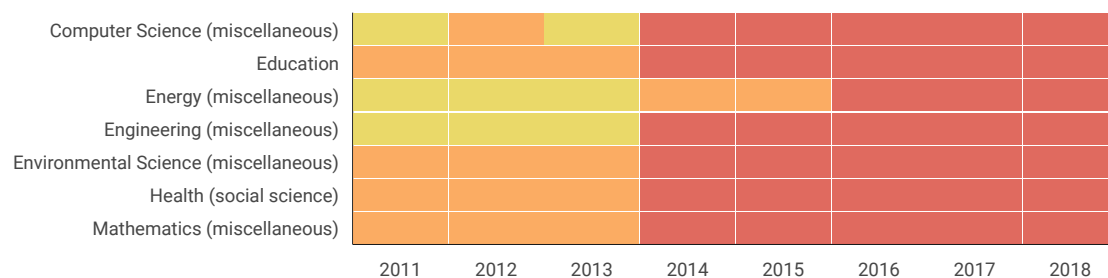
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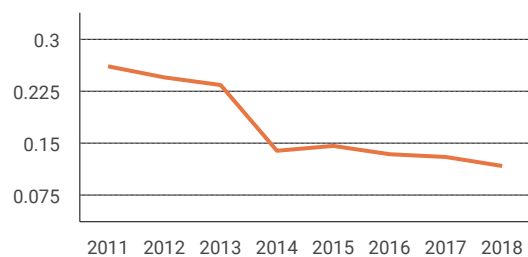
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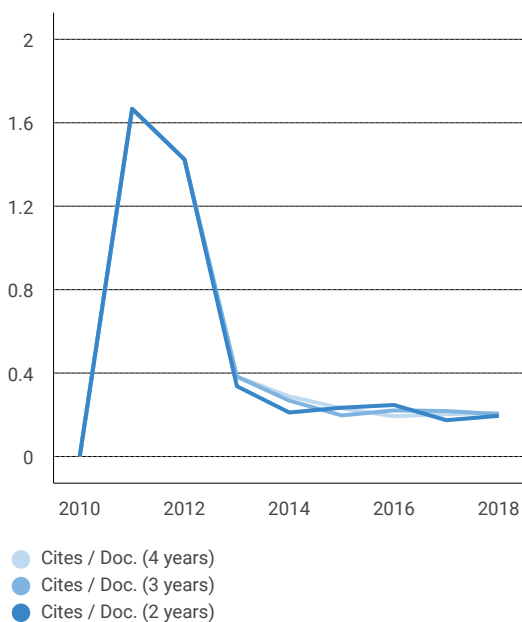
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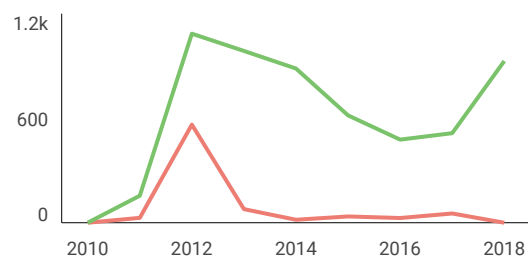
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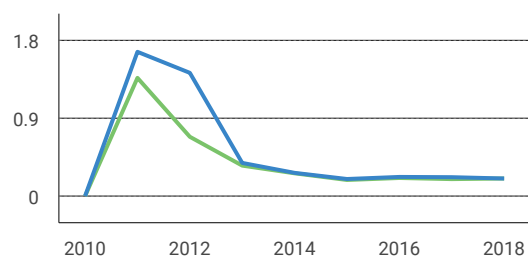
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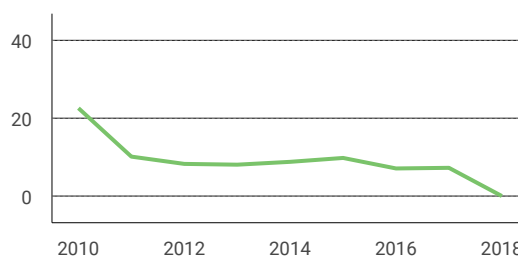
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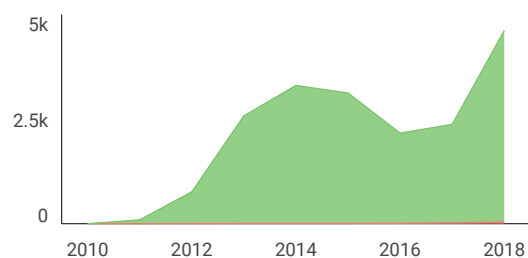
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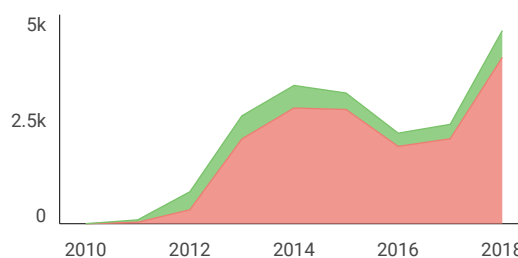
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Synthesis and Characterization of Zeolite/ Magnetite Composite from Iron Sand of Marina Beach

Sriatun*, Adi Darmawan, and Sriyanti

Chemistry Department, Sains and Mathematic Faculty, Diponegoro University, Indonesia

The synthesis and characterization of zeolite/magnetite composite was carried out. The iron sand from Marina Beach of Semarang Central Java was used as a source of magnetite and the natural zeolite from Bayat Klaten Central Java as a host material. The magnetite was impregnated into zeolite by a simple chemical route. The structural and crystallinity of the product were characterized by XRD. Whereas the content of the component and morphological properties were determined by SEM and EDX respectively. The XRD patterns show that composite of zeolite/magnetites has been formed and there were high intensity sharp peaks which means the samples are crystalline. The SEM EDX data explain that samples which prepared by zeolite: magnetite ratio 1:1 produced more homogenous size and shape of the particle.

Keywords: Synthesis, Characterization, Composite, Sand from Marina Beach.

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1. INTRODUCTION

Magnetite is one of natural inorganic mineral that has been widely in many application. It is main component in iron sand. Magnetite nanoparticles have been successfully synthesized from natural sand by coprecipitation method at room temperature.¹¹ Kartika et al. (2014) was synthesized magnetite from iron sand by dissolving acid. According Putra,⁸ his investigation showed that ferrous content in iron sand as mineral tetanomagnetic. Its composition is 60% Fe, 3.3% Al₂O₃, 0.26% SiO₂, 0.55% P₂O₅, 9.2% TiO₂, 0.6% MgO. In Indonesia, the spread or distribution of iron sand are in particular region. In Java island, there are three region that have large iron sand deposit, they are Southern coast in East Java, Northern coast in Central Java and Yogyakarta.⁹ Marina Beach is one part of Northern coast in Central Java, the location in Semarang. In this research iron sand from Marina Beach was used as magnetite sources.

In the other hand, zeolites are aluminosilicate compounds which are natural minerals or synthesized. Zeolites are microporous material which have many advantage, due to its high surface area and pore accessibility, thermal and chemical stability, good electrical conductivity they were widely used in industry such as waste water treatment, waste gas treatment, catalyst and molecular sieve. Besides that, the zeolite were also used in agriculture purposes and recently to produce the nanocomposites.¹⁰

Nanocomposite materials have various interesting properties, so the use of their materials have shown a high impact on society

and environment. Murphy et al.⁷ described that nanomaterial showed differences in shape, activity, conductivity and many other properties than bulk materials. Zeolites with their microporous and unique structure are good support and hosting materials. Whereas, magnetite in iron sand has high magnetic susceptibility properties.⁵ Incorporation of zeolites and magnetite to be a new material, that is zeolite/magnetite composites possess properties of both zeolite and magnetite.

Previously, many investigators extended about composites with zeolites as a support/hosting material. Oliviera et al. (2004) has synthesized zeolite/iron oxide composites to adsorb Cu²⁺, Zn²⁺ and Cr³⁺ ions. Barquist¹ used zeolite/iron oxide composites to adsorb of As and Cr ion and Fungaro et al.² used it to adsorb of water contaminant. Meanwhile, NaP1/nanomagnetite composite was synthesized by Aono et al. (2013) for Cs⁺ radioactive adsorption, carbon cryogel/zeolite was synthesized by Babic et al. (2011).

In this paper, we report the synthesis of zeolite doped magnetite that extracted from iron sand in Marina Beach of Semarang Central Java, whereas natural zeolite from Bayat, Klaten Central Java. New material zeolite/magnetite composites were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM) and energy dispersive X-ray (EDX).

2. EXPERIMENTAL SECTION

2.1. Preparation of Zeolite

For this study, natural zeolite from Bayat, Klaten Central Java was employed. This material was milled and sieved until the

*Author to whom correspondence should be addressed.

sized 100 mesh, then it was soaked in HF 1% solution for a period of 10 minutes. Furthermore, it was washed by aquadest until neutral condition, and dried at 100 °C for 4 hours.

2.2. Preparation of Magnetite

Magnetite was extracted from iron sands using quite strong permanent magnet in a simple and handmade magnetic separator. It was milled by VBM (vibration ball milling) at 1000 rpm for 1 hour, then dried at 100 °C during 2 hours. A number of the materials dissolved in chloride acid 37% to obtain $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ and $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$. They were mixed and homogenized at temperature 70 °C by stirrer magnetic at 600 rpm for 15 minutes. Solution was filtered, the filtrate was heated until slag formed, then it was powdered and calcined at 700 °C during 1 hour.

2.3. Synthesis of Zeolite/Magnetite

For the synthesis of zeolite/magnetite composite, 0,5 g of zeolite was added 25 ml of distilled water and slowly stir for few minutes until homogenate suspension was formed. After that a quantity of magnetite as extracted product at previous procedure was added into the solution with ratio 1:1 (w/w), then shaken for 1 hour. We used PEG 400 as a binder for both of the materials. While the mixture was being stirred vigorously a quantity of ammonium hydroxide 12,5% was slowly added. The synthesized product was filtered, washed with distilled water and finally, it was dried at 80 °C for 3 hours. The characterization product used SEM, EDX and XRD.

3. RESULTS AND DISCUSSION

Generally, Iron sand/natural sand contain magnetite and maghemite as main component that have magnetic susceptibility properties. Other component in iron sand are quartz, calcite, feldspar, amphibol pyroxene, biotite and tourmaline.⁹ In this research, we used magnetic separator as a separation technique to separate iron sands between magnetic component and non-magnetic component. Magnetite as a magnetic materials associated with non magnetic material so it can be separated by crushing. After this treatment, magnetic separator was used to separate magnetite again. This method has given high purity magnetite.

Nanoparticle size of magnetite to be a target in research, because materials with nanometer size can give very satisfying magnetic properties. Nanoparticle and homogenous magnetite can obtained by mechano processing in Vibration ball mill (VBM) of magnetite as a product extracted from iron sand. After this process, the colour of magnetite to be darker than ever. Magnetite which was gained from VBM was dried and calcined. The product was name PK.

The synthesis of magnetite nanoparticle was carried out by changing the crystal size of magnetite. The product magnetite from VBM process was dissolved in HCl 37% solution. So, there are mixture both of Fe^{3+} and Fe^{2+} in the solution. The reaction is shown in below.

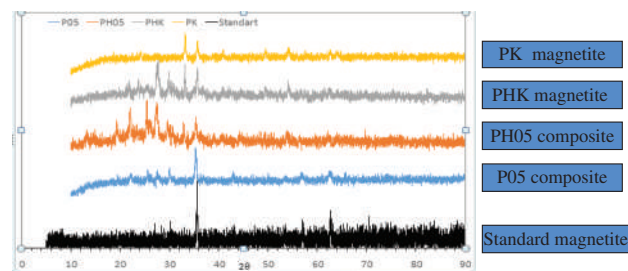
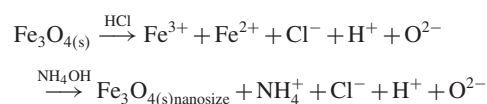


Fig. 1. X-ray diffraction patterns of magnetite and zeolite/magnetite composite.

The aim of adding NH_4OH into solution induced the coprecipitation process. Product of this process is thick black mud like precipitates falling down to the bottom of beaker glass. This may cause the nanoparticle magnetite was formed. The final product was name PHK.

Furthermore, the synthesized of zeolite/magnetite composite was employed to both of PK and PHK. One may observe the XRD spectra in Figure 1. Magnetite from VBM which has PK code sample shown congruity to standard magnetite, there are 3 high intensity peaks at $2\theta = 33.17$; 35.67 and 54.12 . They are a typical peak for magnetite. Magnetite as a product coprecipitation process was coded by PHK, given different pattern. Indeed, the pattern exhibited the mixture of magnetite at $2\theta = 35.62$ and 33.15 and maghemite at $2\theta = 27.60$; 29.82 and 23.67 . This result according to the Kalaori et al. (2014) that the adding of HCl caused Fe in Fe_3O_4 interacted and offered new phase of Fe. The composite of zeolite and magnetite was prepared by ratio 1:1 give P05 and PH05. The pattern of P05 shows highest peaks at $2\theta = 35.35$; 30.01 ; 25.59 and PH05 the main peaks at $2\theta = 25.43$; 27.39 ; 22.05 . This is indicate that formation of zeolite/magnetite composite has occurred. This result has similarity to the pattern XRD zeolite/ Fe_3O_4 which was synthesized by Fitriani et al.³ used microwave heating method. Intensities of the peaks sample are relatively high as indication of high crystallinity.

The XRD diffractogram can be used to quantify the proportion of crystallite size. The crystallite size is determined using Scherer equation.

$$D = \frac{57,3 \times k \times \lambda}{\beta \cos \theta}$$

Where, D is crystallite size average, k is an oxide constant, λ is the X ray wavelength, β is FWHM intensity in angular line,

Table I. Crystallite size by Scherer equation.

Kind of sample	Crystallite size (nm)	Composition (%)
PK	254,155	43,165
	229,382	34,262
	167,940	22,572
PHK	135,632	50,697
	210,020	28,920
	236,103	20,383
P05	226,999	61,237
	243,809	21,721
	269,283	17,043
PH05	312,193	27,451
	166,911	47,918
	272,722	24,631

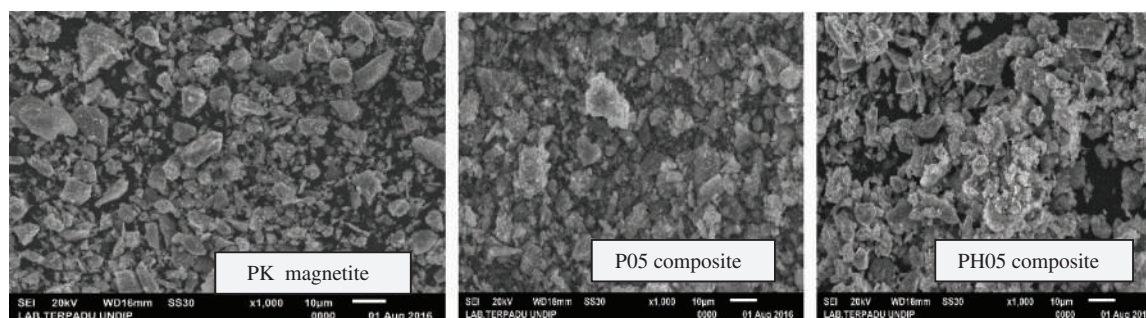


Fig. 2. SEM image of magnetite and zeolite/magnetite composite with ratio 1:1.

θ is the Bragg's angle in degree, 57,3 is correction factor from degree to radian. Table I shows the average crystallite size and their composition of synthesized magnetite from iron sand and zeolite/magnetite composite. There aren't specific trend for the result, treatment magnetite was dissolved in HCl before composite formed with zeolit (PHK) shows smaller crystallite size. But after composite formed both of P05 and PH05, the crystallite size were increased, it was caused magnetite entranced and merged into zeolite. Zeolite acted as supporting materials.

The result of scanning electron microscope (SEM) image of magnetite before and after combine to zeolite can observe on Figure 2. Typical image shows that the particle of zeolite/magnetite composite more distribute uniformly than magnetite only. In the EDX in Table II was found that main component in magnetite was Fe, but after composite was formed by ratio 1:1 used magnetite as product VBM (P05) was dominated by SiO_2 and Al_2O_3 . These are zeolite component, so it could predicted that the magnetite may entranced into zeolite pore. Meanwhile the composite which was synthesized by coprecipitation (PH05) the main component was Fe then Si. The content of Si in PH05 is higher than PK indicate that magnetite may dominate on surface of composite.

Table II. The components in magnetite and zeolite/magnetite composite.

Component	Composition (% weight)		
	PK	P05	PH05
C	34,78	27,32	15,79
Na_2O	0,66	0,68	—
MgO	1,78	2,66	1,67
Al_2O_3	4,28	9,37	5,60
SiO_2	6,74	42,37	18,74
K_2O	0,33	0,94	0,59
CaO	0,94	3,79	1,31
TiO_2	5,35	3,97	6,45
FeO	43,82	6,52	47,86
CuO	1,33	1,24	1,32
ZnO	—	1,13	—

4. CONCLUSION

Magnetite was synthesized from iron sand of Marina Beach Semarang. Treatment magnetite as product VBM with coprecipitation method gave different Fe phase. Zeolite/magnetite composite was obtained both of magnetite. The component of zeolite/magnetite composite was dominated by SiO_2 and Al_2O_3 . Meanwhile the composite which was synthesized by coprecipitation contained higher Fe than Si, but the content of Si in this product was higher than magnetite only that indicated that magnetite may dominate on surface of composite.

Acknowledgment: This research is financially supported by PNPB UNDIP 2016.

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by Sriatun Sriatun

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Synthesis and Characterization of Zeolite/ Magnetite Composite from Iron Sand of Marina Beach

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The synthesis and characterization of zeolite/magnetite composite was carried out. The iron sand from Marina Beach of Semarang Central Java was used as a source of magnetite and the natural zeolite from Bayat Klaten Central Java as a host material. The magnetite was impregnated into zeolite by a simple chemical route. The structural and crystallinity of the product were characterized by XRD. Whereas the content of the component and morphological properties were determined by SEM and EDX respectively. The XRD patterns show that composite of zeolite/magnetites has been formed and there were high intensity sharp peaks which means the samples are crystalline. The SEM EDX data explain that samples which prepared by zeolite: magnetite ratio 1:1 produced more homogenous size and shape of the particle.

Keywords: Synthesis, Characterization, Composite, Sand from Marina Beach.

1. INTRODUCTION

Magnetite is one of natural inorganic mineral that has been widely in many application. It is main component in iron sand. Magnetite nanoparticles have been successfully synthesized from natural sand by coprecipitation method at room temperature.¹¹ Kartika et al. (2014) was synthesized magnetite from iron sand by dissolving acid. According Putra,⁸ his investigation showed that ferrous content in iron sand as mineral tetramagnetite. Its composition is 60% Fe, 3.3% Al₂O₃, 0.26% SiO₂, 0.55% P₂O₅, 9.2% TiO₂, 0.6% MgO. In Indonesia, the spread or distribution of iron sand are in particular region. In Java island, there are three region that have large iron sand deposit, they are Southern coast in East Java, Northern coast in Central Java and Yogyakarta.⁹ Marina Beach is one part of Northern coast in Central Java, the location in Semarang. In this research iron sand from Marina Beach was used as magnetite sources.

In the other hand, zeolites are aluminosilicate compounds which are natural minerals or synthesized. Zeolites are microporous material which have many advantage, due to its high surface area and pore accessibility, thermal and chemical stability, good electrical conductivity they were widely used in industry such as waste water treatment, waste gas treatment, catalyst and molecular sieve. Besides that, the zeolite were also used in agriculture purposes and recently to produce the nanocomposites.¹⁰

Nanocomposite materials have various interesting properties, so the use of their materials have shown a high impact on society

environment. Murphy et al.⁷ described that nanomaterial showed differences in shape, activity, conductivity and many other properties than bulk materials. Zeolites with their microporous and unique structure are good support and hosting materials. Whereas, magnetite in iron sand has high magnetic susceptibility properties.⁵ Incorporation of zeolites and magnetite to be a new material, that is zeolite/magnetite composites possess properties of both zeolite and magnetite.

Previously, many investigators extended about composites with zeolites as a support/hosting material. Oliveira et al. (2004) has synthesized zeolite/iron oxide composites to adsorb Cu²⁺, Zn²⁺ and Cr³⁺ ions. Barquist¹ used zeolite/iron oxide composites to adsorb of As and Cr ion and Fungaro et al.² used it to adsorb of water contaminant. Meanwhile, NaPl/nanomagnetite composite was synthesized by Aono et al. (2013) for Cs⁺ radioactive adsorption, carbon cryogel/zeolite was synthesized by Babic et al. (2011).

In this paper, we report the synthesis of zeolite doped magnetite that extracted from iron sand in Marina Beach of Semarang Central Java, whereas natural zeolite from Bayat, Klaten Central Java. Natural material zeolite/magnetite composites were characterized by X-ray diffraction (XRD) and scanning electron microscopy (SEM) and energy dispersive X-ray (EDX).

2. EXPERIMENTAL SECTION

2.1. Preparation of Zeolite

For this study, natural zeolite from Bayat, Klaten Central Java was employed. This material was milled and sieved until the

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sized 100 mesh, then it was soaked in HF 1% solution for a period of 10 minutes. Furthermore, it was washed by aquadest until neutral condition, and dried at 100 °C for 4 hours.

2.2. Preparation of Magnetite

Magnetite was extracted from iron sands using quite strong permanent magnet in a simple and handmade magnetic separator. It was milled by VBM (vibration ball milling) at 1000 rpm for 1 hour, then dried at 100 °C during 2 hours. A number of the materials dissolved in chloride acid 37% to obtain $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ and $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$. They were mixed and homogenized at temperature 70 °C by stirrer magnetic at 600 rpm for 15 minutes. Solution was filtered, the filtrate was heated until slag formed, then it was powdered and calcined at 700 °C during 1 hour.

2.3. Synthesis of Zeolite/Magnetite

For the synthesis of zeolite/magnetite composite, 0.5 g of zeolite was added 25 ml of distilled water and slowly stir for few minutes until homogenate suspension was formed. After that a quantity of magnetite as extracted product at previous procedure was added into the solution with ratio 1:1 (w/w), then shaken for 1 hour. We used PEG 400 as a binder for both of the materials. While the mixture was being stirred vigorously a quantity of ammonium hydroxide 12.5% was slowly added. The synthesized product was filtered, washed with distilled water and finally, it was dried at 80 °C for 3 hours. The characterization product used SEM, EDX and XRD.

3. RESULTS AND DISCUSSION

Generally, Iron sand/natural sand contain magnetite and maghemite as main component that have magnetic susceptibility properties. Other component in iron sand are quartz, calcite, feldspar, amphibol pyroxene, biotite and tourmaline.⁹ In this research, we used magnetic separator as a separation technique to separate iron sands between magnetic component and non-magnetic component. Magnetite as a magnetic materials associated with non magnetic material so it can be separated by crushing. After this treatment, magnetic separator was used to separate magnetite again. This method has given high purity magnetite.

Nanoparticle size of magnetite to be a target in research, because materials with nanometer size can give very satisfying magnetic properties. Nanoparticle and homogenous magnetite can obtained by mechano processing in Vibration ball mill (VBM) of magnetite as a product extracted from iron sand. After this process, the colour of magnetite to be darker than ever. Magnetite which was gained from VBM was dried and calcined. The product was name PK.

The synthesis of magnetite nanoparticle was carried out by changing the crystal size of magnetite. The product magnetite from VBM process was dissolved in HCl 37% solution. So, there are mixture both of Fe^{3+} and Fe^{2+} in the solution. The reaction is shown in below.

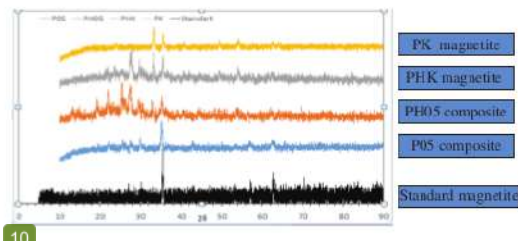
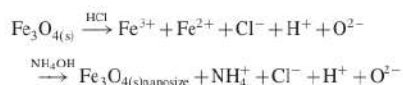


Fig. 1. X-ray diffraction patterns of magnetite and zeolite/magnetite composite.

The aim of adding NH_4OH into solution induced the coprecipitation process. Product of this process is thick black mud like precipitates falling down to the bottom of beaker glass. This may cause the nanoparticle magnetite was formed. The final product was name PHK.

Furthermore, the synthesized of zeolite/magnetite composite was employed to both of PK and PHK. One may observe the XRD spectra in Figure 1. Magnetite from VBM which has PK code sample shown congruity to standard magnetite, there are 3 high intensity peaks at $2\theta = 33.17$; 35.67 and 54.12 . They are a typical peak for magnetite. Magnetite as a product coprecipitation process was coded by PHK, given different pattern. Indeed, the pattern exhibited the mixture of magnetite at $2\theta = 35.62$ and 33.15 and maghemite at $2\theta = 27.60$; 29.82 and 23.67 . This result according to the Kalaori et al. (2014) that the adding of HCl caused Fe in Fe_3O_4 interacted and offered new phase of Fe. The composite of zeolite and magnetite was prepared by ratio 1:1 give P05 and PH05. The pattern of P05 shows highest peaks at $2\theta = 35.35$; 30.01 ; 25.59 and PH05 the main peaks at $2\theta = 25.43$; 27.39 ; 22.05 . This is indicate that formation of zeolite/magnetite composite has occurred. This result has similarity to the pattern XRD zeolite/ Fe_3O_4 which was synthesized by Fitriani et al.³ used microwave heating method. Intensities of the peaks sample are relatively high as indication of high crystallinity.

The XRD diffractogram can be used to quantify the proportion of crystallite size. The crystallite size is determined using Scherer equation.

$$D = \frac{57,3 \times k \times \lambda}{\beta \cos \theta}$$

Where, D is crystallite size average, k is an oxide constant, λ is the X ray wavelength, β is FWHM intensity in angular line,

Table 1. Crystallite size by Scherer equation.

Kind of sample	Crystallite size (nm)	Composition (%)
PK	254,155	43,165
	229,382	34,262
	167,940	22,572
PHK	135,632	50,697
	210,020	28,920
	236,103	20,383
P05	226,999	61,237
	243,809	21,721
	269,283	17,043
PH05	312,193	27,451
	166,911	47,918
	272,722	24,631

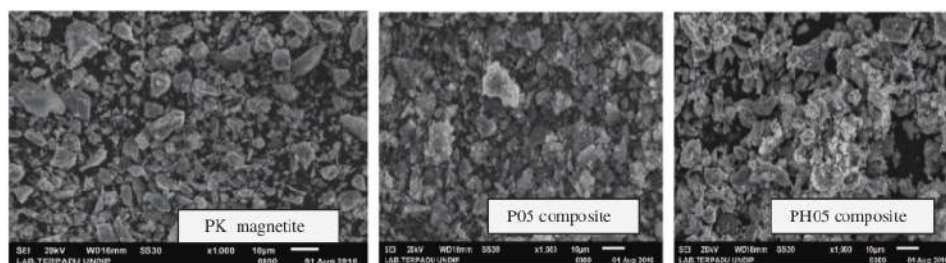


Fig. 2. SEM image of magnetite and zeolite/magnetite composite with ratio 1:1.

θ is the Bragg's angle in degree, 57,3 is correction factor from degree to radian. Table I shows the average crystallite size and their composition of synthesized magnetite from iron sand and zeolite/magnetite composite. There aren't specific trend for the result, treatment magnetite was dissolved in HCl before composite formed with zeolite (PHK) shows smaller crystallite size. But after composite formed both of P05 and PH05, the crystallite size were increased, it was caused magnetite entranced and merged into zeolite. Zeolite acted as supporting materials.

The result of scanning electron microscope (SEM) image of magnetite before and after combine to zeolite can observe on Figure 2. Typical image shows that the particle of zeolite/magnetite composite more distribute uniformly than magnetite only. In the EDX in Table II was found that main component in magnetite was Fe, but after composite was formed by ratio 1:1 used magnetite as product VBM (P05) was dominated by SiO_2 and Al_2O_3 . These are zeolite component, so it could predicted that the magnetite may entranced into zeolite pore. Meanwhile the composite which was synthesized by coprecipitation (PH05) the main component was Fe then Si. The content of Si in PH05 is higher than PK indicate that magnetite may dominate on surface of composite.

Table II. The components in magnetite and zeolite/magnetite composite.

Component	Composition (% weight)		
	PK	P05	PH05
C	34,78	27,32	15,79
Na_2O	0,66	0,68	—
MgO	1,78	2,66	1,67
Al_2O_3	4,28	9,37	5,60
SiO_2	6,74	42,37	18,74
K_2O	0,33	0,94	0,59
CaO	0,94	3,79	1,31
TiO_2	5,35	3,97	6,45
FeO	43,82	6,52	47,86
CuO	1,33	1,24	1,32
ZnO	—	1,13	—

CONCLUSION

Magnetite was synthesized from iron sand of Marina Beach Semarang. Treatment magnetite as product VBM with coprecipitation method gave different Fe phase. Zeolite/magnetite composite was obtained both of magnetite. The component of zeolite/magnetite composite was dominated by SiO_2 and Al_2O_3 . Meanwhile the composite which was synthesized by coprecipitation contained higher Fe than Si, but the content of Si in this product was higher than magnetite only that indicated that magnetite may dominate on surface of composite.

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