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KARYA ILMIAH : JURNAL ILMIAH

Judul Jurnal Ilmiah (Artikel)	:	Microstructure Characterization of Natural Magnetite from Sand Marina Beach By High Energy Milling		
Jumlah Penulis	:	4 orang		
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Identitas Jurnal Ilmiah	:	a. Nama Jurnal	:	ORIENTAL JOURNAL OF CHEMISTRY An International Open Free Access, Peer Reviewed Research Journal
		b. Nomor ISSN	:	0970-020 X
		c. Vol, No., Bln Thn	:	Vol. 34, No.(2): Page. 868-874, 2018
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Semarang, 30 Maret 2020
Reviewer 1

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

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Semarang, 20 Maret 2020
Reviewer 2

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

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HASIL PENILAIAN SEJAWAT SEBIDANG ATAU *PEER REVIEW*
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d. Kelengkapan unsur dan kualitas terbitan/jurnal (30%)	10	11	10,5
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Penulis Utama (rata-rata): 0,6 x 37,5 = 22,5			

Semarang, 30 Maret 2020

Reviewer 2

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

Reviewer 1

Dr. Bambang Cahyono
NIP. 196303161988101001
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TOC Volume 34 Number 2

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Sequential Injection at Valve Mixing (SI-VM) for Determination of Albumin-Creatinine Ratio in Urine

Akhmad Sabarudin

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Structural Analysis of Powdered Manganese(II) of 1,10-Phenanthroline (phen) as Ligand and Trifluoroacetate (TFA) as Counter Anion

Kristian Handoyo Sugiyarto , Cahyorini Kusumawardani, Hari Sutrisno and Muhammad Wahyu Arif Wibowo

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Polyimide Composite Materials Containing Modified Nanostructured Boron Carbide

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Synthesis and Characterization of Natural $Ca(OH)_2$/KF Superbase Catalyst for Biodiesel Production from Palm Oil

I. F. Nurcahyo[1,2] , Karna Wijaya[1], Triyono[1], Arief Budiman[3] and Yun Hin Taufiq-Yap[4,5]

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Electrochemical Preparation of the Ti/Ni-Sb-SnO_2 for Phenol Removal by *In-situ* Generated Ozone

Ali Reza Rahmani, Mohammad Taghi Samadi, Mohammad Reza Samarghandi and Ghasem Azarian

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Novel Organotin(IV) Complexes Derived from Chiral Benzimidazoles: Synthesis, Molecular Structure and Spectral Properties

A. Akremi[1] , A. Noubigh[2] and M. J. A. Abualreish[3]

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Study on the Effect of Deposition Parameters on Characteristics of Electrochemically Synthesized Polyaniline and Poly O-Toluidine

Monimul Huque[1], Golam Haider[2] , Shah Rokonuzzaman[2] and Aksaruzzaman Nuri[2]

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An Efficient Synthesis and In Vitro Antimicrobial Screening of 2-Cyanoimino -4-aryl-6-(1,1'-biphenyl-4-yl)-3,4-dihydro-1H-Pyrimidines

Synthesis of High-Sulfur Polymers by Redox Copolymerization of Elemental Sulfur with Pyrrole

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Anticancer Activity of Cu(II), Pd(II) and Zn(II) Complexes of Phosphonate with Glutamine Amino Acid

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Physicochemical Characteristics and Photocatalytic Activity of Silver Nanoparticles-decorated on Natural Halloysite (An aluminosilicate clay)

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Synthesis and Antibacterial Activity 1-Monolaurin

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Microstructure Characterization of Natural Magnetite from Sand Marina Beach By High Energy Milling

Sriatun, A. Darmawan, Sriyanti and W. Cahyani

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Phytosterol Screening of Skin and Seed Extracts of Wild Grape Ampelocissus Martinii Planch. Fruits

Wilaiwan Simchuer and Prasong Srihanam

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Carbon–Carbon Bond Formation Reaction with Pd/reduced Graphene Oxide Composite

Padmakar Anant Kulkarni, Suresh Shamrao Shendage and Ashok Ganuji Awale

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Utilization of Natural Zeolite Clipnotilolit-Ca as a Support of ZnO Catalyst for Congo-Red Degradation and Congo-Red Waste Applications with Photolysis

Zilfa, Rahmayeni, Yeni Stiadi and Adril

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Phytosterol Screening of Skin and Seed Extracts of Wild Grape *Ampelocissus martinii* Planch

WILAIWAN SIMCHUER and PRASONG SRIHANAM*

Creative Chemistry and Innovation Research Unit, the Center of Excellence in Chemistry, Department of Chemistry, Faculty of Science, Mahasarakham University, Kantarawichai, Maha Sarakham 44150, Thailand.

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(Received: October 29, 2017; Accepted: January 05, 2018)

ABSTRACT

Reports on phytosterol in wild grape, *Ampelocissus martinii* Planch fruits are rarely available world wide. We have investigated the total triterpenoid content (TTC) and total sterol content (TSC) in skin and seed extracts from different growth stages of wild grape fruits using a spectrophotometric assay. In addition, we analyzed some free sterols using high performance liquid chromatography (HPLC). The mature fruit showed the highest content of TTC in both the skin and seed extracts. The TSC content was found the highest from skin extract in the ripe stage. The two most common phytosterols found were stig masterol and β-sitosterol. The stig masterol content was found the highest in the skin extract of the immature, while β-sitosterol was found in the seed extract of the ripe fruits. This work indicated that the skin and seed of wild grape fruits might be new sources of phytosterol.

Keywords: Wild grape, Phytochemical, Phytosterol, Spectrophotometric assay.

INTRODUCTION

Phytochemicals are secondary metabolites that are non-nutritive, naturally occurring, and biologically active compounds and found in the plant kingdom[1]. Phytochemicals enrich the biological activity potential, including anti-inflammation, anti-cancer, antimicrobial, and antioxidant. There are more than a thousand known phytochemicals[2]. Phytosterols are one group of triterpenoid and pervasive in plants. Generally, their structures look like to cholesterol, but side chain has differed in the alkyl position. Among more than 200 different compounds of phytosterol, β-sitosterol, stigmasterol, and campesterol are predominate types in higher plants and human diets. The several of vegetable oil, nuts, fruit, berries, grapes, and cereals are important sources of the plant sterols. Wild grape is a potent source of antioxidants and anti-bacterials from their

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Carbon–Carbon Bond Formation Reaction with Pd/reduced Graphene Oxide Composite

PADMAKAR ANANT KULKARNI, SURESH SHAMRAO SHENDAGE* and ASHOK GANUJI AWALE

Department of Chemistry, KET'S Vinayak Ganesh Vaze College of Arts, Science and Commerce Mithagar Road, Mulund (E) Mumbai-400081, India.
*Corresponding author E-mail: sureshsshendage@gmail.com

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(Received: December 03, 2017; Accepted: February 01, 2018)

ABSTRACT

Palladium nanoparticles supported on reduced graphene oxide (Pd/rGO) catalyst was prepared by using alovera extract in sunlight. The catalyst (Pd/rGO) was employed for the Suzuki cross coupling reactions. The characterization of Pd/rGO was done by scanning electron microscopy (SEM), transmission electron microscopy (TEM) and X-ray diffraction analysis (XRD). The average particle size of Pd was found to be 7±2 nm. The compound showed excellent catalytic activity with good recyclability for C-C cross coupling reactions.

Keywords: Solar energy, Alo vera, Heterogeneous catalysis, Greener synthesis, Palladium nanoparticles, Reduced graphene oxide, Cross coupling reactions.

INTRODUCTION

In last decades Suzuki–Miyaura cross-coupling reaction has gained considerable attention because of its use in making active pharmaceutical ingredients, agrochemicals, polymers, bioinorganic materials and natural products on a laboratory as well as industrial scale[1]. In 1981 the first method was reported for the preparation of biaryls via C-C cross-coupling reaction[2]. In general, homogeneous palladium complexes are used as catalysts in the Suzuki reaction[3].

The reported homogeneous catalysts are found to have greater catalytic properties than heterogeneous catalysts in the formation of C-C coupling reactions[4-6]. The several researchers focused on the development of palladium complexes which efficiently catalyze the reaction[7].

The palladium complexes usually show greater catalytic activity. The main drawbacks of these catalysts are availability, stability, recovery, and high cost in addition to their sensitivity to air and moisture. So reactions catalyzed by

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Volume 34, Issue 2, 2018, Pages 868-874

Microstructure characterization of natural magnetite from sand marina beach by high energy milling (Article) (Open Access)

Sriatun, Darmawan, A., Sriyanti, Cahyani, W.

Department of Chemistry, Diponegoro University, Semarang, Central of Java, 50275, Indonesia

Abstract

View references (16)

In this work, we performed an experimental investigation the change of microstructure of magnetite by high energy milling -3D (HEM-3D) method using planetary ball milling at 400 rpm speed. The present studies mainly focusses on the effect of milling on crystallinity and phase of magnetite by XRD, particle size by PSA and the morphology by SEM. The increasing of the ball mass in the milling process, mass ratio magnetite : ball (P/B) 1: 1, 1: 3 and 1: 5 give the magnetite particles smaller (< 1μm), the crystallinity decreases but the peaks at (2 2 0), (3 1 1), (4 0 0), (5 1 1), and (4 4 0) were keep appearing. This shows that the phase of cubic spinel does not change. Rising the milling time for 1 h, 3 h and 5 h can lead to decreasing of size and crystallinity. Even milling time for 5 h on mass ratio of magnetite : ball (P/B) 1: 5 causes the magnetite phase to change to amorphous.

SciVal Topic Prominence

Topic: Titanomagnetite | Ilmenite | Carbothermal Reduction

Prominence percentile: 88.130

Chemistry database information

Substances

magnetite

Author keywords

High energy milling -3D. | Microstructure | Natural magnetite | Sand marina beach

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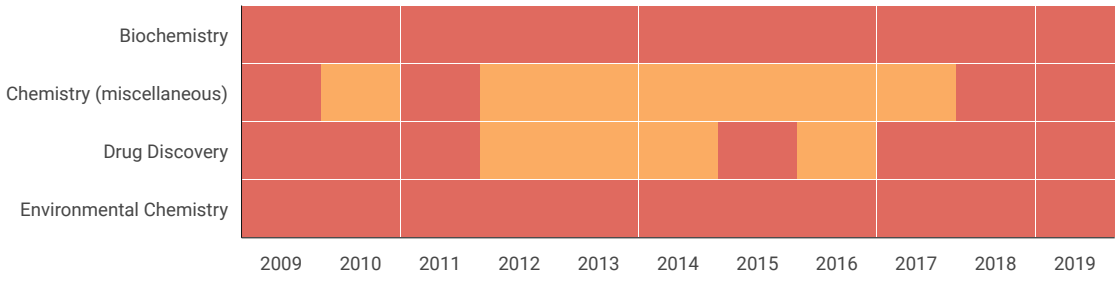

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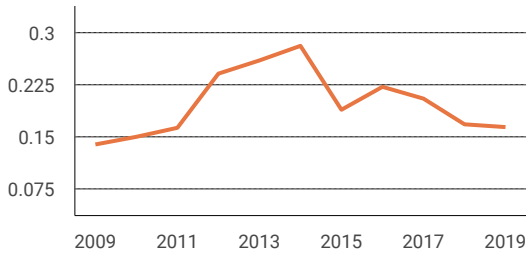

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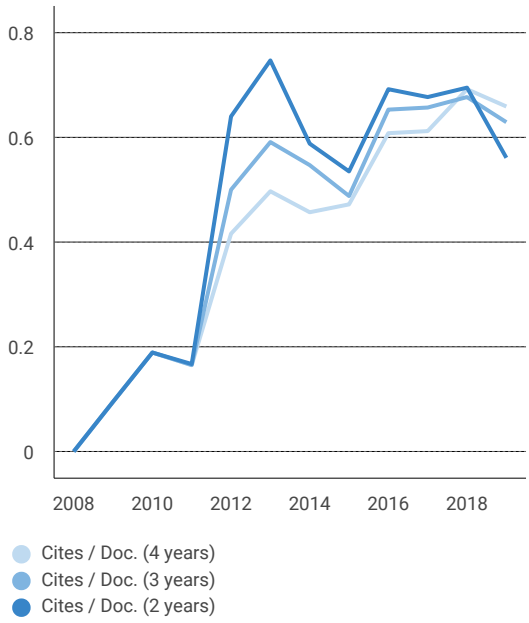

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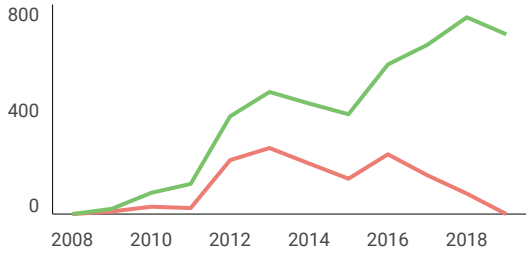

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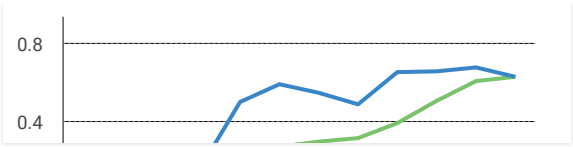

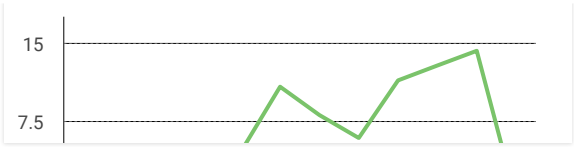

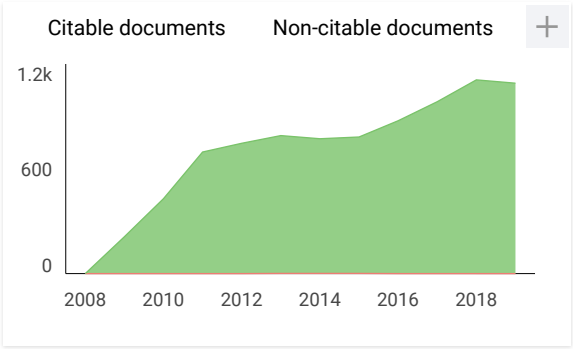

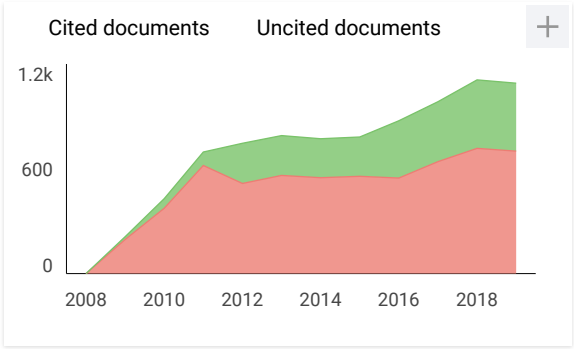

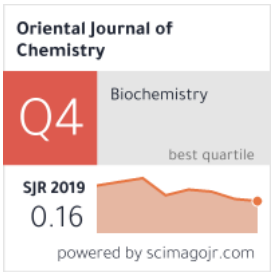

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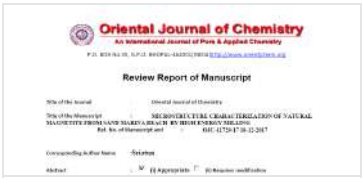

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MICROSTRUCTURE CHARACTERIZATION OF NATURAL MAGNETITE FROM SAND MARINA BEACH BY HIGH ENERGY MILLING

ABSTRACT


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Microstructure Characterization of Natural Magnetite from Sand Marina Beach by High Energy Milling

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ABSTRACT

In this work, we performed an experimental investigation the change of microstructure of magnetite by high energy milling-3D (HEM-3D) method using planetary ball milling at 400 rpm speed. The present studies mainly focusses on the effect of milling on crystallinity and phase of magnetite by XRD, particle size by PSA and the morphology by SEM. The increasing of the ball mass in the milling process, mass ratio magnetite: ball (P/B) 1: 1, 1: 3 and 1: 5 give the magnetite particles smaller (< 1μm), the crystallinity decreases but the peaks at (2 2 0), (3 1 1), (4 0 0), (5 1 1), and (4 4 0) were keep appearing. This shows that the phase of cubic spinel does not change. Rising the milling time for 1 h, 3 h and 5 h can lead to decreasing of size and crystallinity. Even milling time for 5 h on mass ratio of magnetite: ball (P/B) 1: 5 causes the magnetite phase to change to amorphous.

Keywords: Microstructure, Natural magnetite, Sand marina beach, High energy milling-3D.

INTRODUCTION

Iron sand occurs naturally in several regions throughout the world. Iron sand is one of Indonesia's natural mineral resources, which is spread over the islands along the coast of Java Island, Kalimantan and Sumatra. Iron sand is a special type of sand that's rich in the metal iron, the color is dark gray or black, consisting of (Fe) iron as a major element and a small amount of Ti, Si, Ca, Mn and Vanadium. They provide a raw material of relatively low grade, whereas in the southern coast of Yogyakarta containing 5.85 % to 95.11% of iron. In addition to magnetite in iron sand also contains other minerals such as rutile, ilmenite and hematite[1]. While most sand contains at least some trace of iron, therefore it has a distinct dark-gray or black color, which is in stark contrast to the white-yellow color of regular sand.

Iron sand is a magnetic material that is widely used in various fields such as electronics, energy, chemistry, ferrofluidics, catalysts, and medical diagnostics[2]. The application of iron sand was inseparable from the development of studies of nanomaterials demanding that they be in the order of nanometers. Magnetite or Fe_3O_4 is one of the iron oxide phases which has the greatest magnetic or ferromagnetic properties among the other phases. Iron oxide has four phases, namely magnetite (Fe_3O_4), maghemite (γ-Fe_2O_3), hematite (α-Fe_2O_3), and geotite (FeO(OH)). Only magnetite and maghemite have magnetic properties[3].

Magnetite (Fe_3O_4) is known as a class of iron oxide compound with a cubic inverse spinel structure and has face centered cubic close packed oxygen anions and Fe cations occupying interstitial tetrahedral and octahedral sites[4,5]. Nano-sized magnetite particles provide many advantages such as for the separation of magnetic contaminants in water, large of surface area and the ability to bind through electro-chemical interactions to form sludge. It is also applied to drug delivery and magnetic resonance technology and others.

For the synthesis of nanosized magnetite particles can be synthesized through various methods such as mechanical milling[6], sol-gels, direct decomposition[7], co-precipitation[8], microwave-heating[9] and solvothermal[10,11]. Mechanical milling method is one way to reduce the magnetite size is the cheapest and easy. Mechanical milling is defined as the mechanical breakdown of magnetite into smaller without changing their state of aggregation. The method was used to increase the surface area and induce defects which is needed for subsequent operations such as chemical reactions, sorption. Milling also to increase the proportion of regions of high activity in the surface[12].

Furthermore, this research the small size of magnetite from iron sand was prepared by mechanical milling method using high energy planetary ball mill. Kinetic energy of the balls depends not only on its velocity, but also on its mass and how long the collision occurred, due to in this work investigated the ratio of magnetite and ball mass in the planetary ball mill and the time of impact during collision.

MATERIALS AND METHODS

Materials

Iron sand was taken from Marina Beach in Semarang.

Instrumentations

Magnet permanent, High energy planetary ball mill-3D, X-ray diffraction (XRD) Rigaku Multiplex with Cu Ká radiation (λ = 1.54184 A^o) at generator voltage 40 kV and current 40 mA, Particle Size Analyzer (PSA) Horiba SZ-100, Scanning electron microscope (SEM) JEOL JED 2300.

Procedure

Magnetite preparation

The natural iron sand from Marina Beach Semarang cleaned and washed using aquadest, dried in oven at 80 ^{o}C for 24 hours. Natural magnetite was extracted from natural iron sand using permanent magnet until 12 times. This treatment produces powder material dark gray-black color. Refinement of magnetite particles carried out by mechanical milling method using high energy planetary ball mill (HEM-E3D) instrument. The milling was done on mass ratio of magnetite: ball (P/B) 1:1, 1:3 and 1:5, speed 400 rpm. Milling of magnetite carried out for 1, 3 and 5 hours. Milled magnetite dried at 150 ^{o}C for 1.5 hours. Finally, the microstructure characterization of product was done by X-ray diffraction (XRD) to find out the structure of magnetite crystals, PSA to determine the size of magnetite particle, SEM to know the surface morphology.

RESULTS AND DISCUSSIONS

In this work the change of crystal structure, particle size and morphology of magnetite was investigated. The method is high energy milling (HEM) used planetary ball mill. The choice of this method due to it can reduce the material up to the nano order (nano particle) inside a relatively short time under conditions atmosphere at room temperature during process milling. This method using energy collision between the crushing balls and chamber walls are rotated and driven in a certain way. The change of crystal structure, particle size and morphology of magnetite was studied on variation the mass ratio magnetite:ball (P/B 1:1, 1:3 and 1:5) and milling time (1, 3 and 5 hours).

Physical changes of magnetite

The process of separation of magnetite compounds from iron sand is done repeatedly, it is intended that the compound to be obtained has a high purity. The separation process with magnets also uses a certain distance, the farther the magnet is closer to the iron sands, the less iron oxide attaches. This makes the sample (magnetite) higher purity and less impurities, although there is still the possibility of the other oxide compounds sticked to a permanent magnet. The Fig. 1 following is the embodiment of magnetite extracted from iron sand.

(a) (b)

Fig. 1. The original iron sand from marina beach before extraction treatment with permanent magnet (A) Magnetite after extraction treatment

The extracted iron sand powder then performed mechanical milling with several variations of the mass ratio of magnetite:ball (P/B) 1:1, 1:3 and 1:5 for 1, 3 and 5 h at speed 400 rpm. Magnetite obtained from the milling results has a softer texture and dark black as shown in Figure 2.

Fig. 2. Milled magnetite

It is clearly from Fig. 1A and 1B and Fig. 2, the difference in color and size of iron sand. In iron sand that has been separated with permanent magnet looks blacker than iron sand that has not been separated. This is due to the reduction of impurities from the iron sand so that the iron sand look blacker after extraction using permanent magnet as much 12 times. This shows that the separation of iron sand from impurity elements by this method more effectively. The size of iron sand after mechanical milling becomes smaller and softer than the separated iron sand. This is the advantages of mechanical milling method that ball mill is not sensitive to metal. The superiority of high energy milling is able to produce smaller particles in shorter milling time[12].

Crystal structure of magnetite

Based on the results of the analysis using X-ray diffraction on magnetite powder before milling treatment with HEM-3D obtained X-ray diffraction pattern as shown in Fig. 3. There are five highest peaks at 2è angle of 30.09°; 35.46°; 43.09°; 56.98°; and 62.59°. Furthermore the highest peaks were compared with Joint Committee of Powder Diffraction Standard (JCPDS) number 89-4319 with the highest peaks at 2θ angle of 30.083°; 35.434°; 43.064°; 56.949°; and 62.536°. Based on data obtained from XRD, the compound is magnetite.

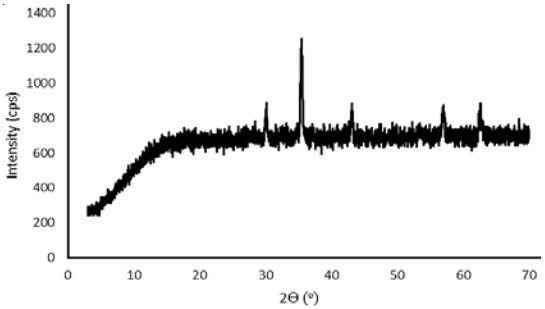

Fig. 3. Diffractogram XRD of magnetite after extraction treatment

Data of X-ray diffraction on magnetite after HEM-3D treatment with mass ratio of magnetite: ball (P/B) 1: 1, 1: 3 and 1: 5 and time collision 1, 3 and 5 h showed in Fig. 4, Fig.5 and Fig. 6. All diffraction peaks correspond to the peak diffraction at (2 2 0), (3 1 1), (4 0 0), (5 1 1), and (4 4 0). Of the highest peaks are compared with the Joint Committee of Powder Diffraction Standard (JCPDS) number. 79-0418 shows indexed to the Fe_3O_4 cubic spinel phase.

The XRD datas show that in all P / B ratio 1, 1: 1: 3 or 1: 5 with milling process for 1 and 3 h still indicates conformity with reference magnetite. When the milling for 5 h is only in P/B 1: 1 and P/B 1: 3 which still shows the suitability and even this is only at the peak of 2´ = 35.92° and 63.02° at P/B 1: 1 and 36.19° and 63.15° at P/B 1: 3, where the peak of the diffractogram is very low, whereas in P/B 1: 5 there is no correspondence with the reference

magnetite. This suggests that long-term milling treatments and strong collisions (heavier ball) can significantly reduce magnetic particle size, these treatments also decreased degrade of crystallinity. The increasing of ball to magnetite mass ratio (heavier ball) would enhancing the kinetic energy during milling. Based on kinetic energy equation:

$$E_k = {}^1/_2\ mv^2$$

in which is the kinetic energy, *m* and *v* are respectively the mass and velocity of the balls. In this research the velocity was constant.

When the colliding ball mass is heavier, so the kinetic energy increases. The high of kinetic energy would cause the particles to collide with each other, where this would decrease in particle size.

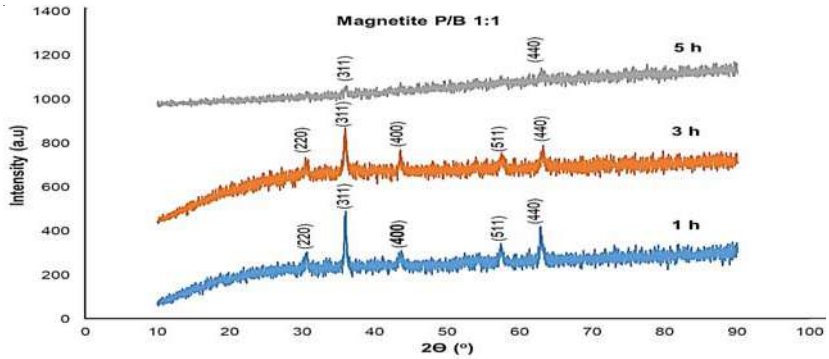

Fig. 4. Diffractogram milled magnetite by mass ratio of magnetite: ball (P/B) 1:1

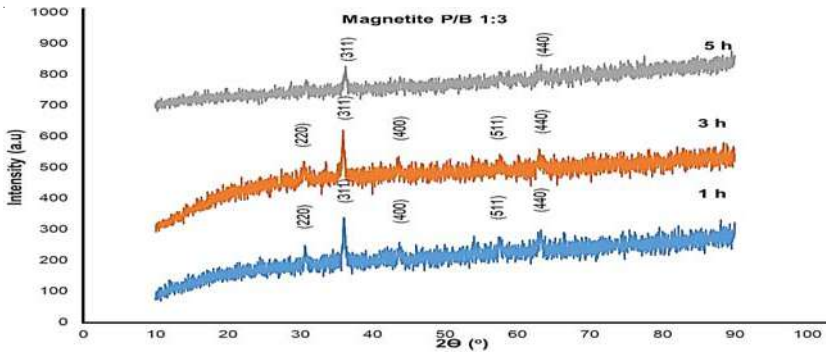

Fig. 5. Diffractogram milled magnetite by mass ratio of magnetite: ball (P/B) 1:3

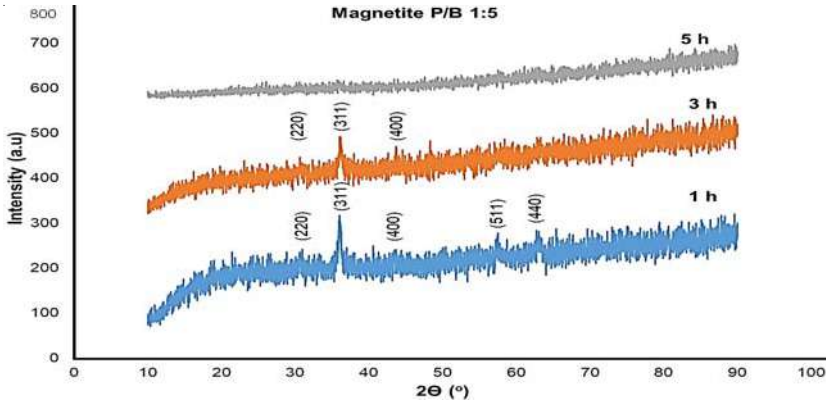

Fig. 6. Diffractogram milled magnetite by mass ratio of magnetite:ball (P/B) 1:5

This is in accordance with data that has been revealed by previous research. It was reported that the enhancing energy during milling, resulted by the increase of ball to powder weight ratio (BPR) and vial speed not only can accelerate the formation of the products but also changes the resultant phases[4]. The balls play an important role in its efficiency so that a small change in type, shape, weight or mass and size distribution of the balls can dramatically affect the milling process[5]. The increase of the number of balls at high BPR ratio, has a quite negative effect on the milling performance[6].

Particle size of magnetite

This matter proves that the milling process is done to magnetite powder can causing the destruction of the grains magnetite powder as a result collision between magnetite powder and milling balls. To know more clearly destruction of graphite powder during process milling, then the measurement magnetite particles by particle size analyzer (PSA) instrument. The choice of particle measurement methods of nanoscale and micro size is usually by using a wet method PSA (particle size analyzer) method, because it is an accurate method when compared to other methods. Small particles have a tendency for high agglomeration, the choice of wet method on PSA because the particles are dispersed into the medium so that the particles do not agglomerate (clump). Therefore the measured particle size is the size of a single particle and provides overall information on sample conditions.

Distribution particle size test by particle size analyzer (PSA) aims to determine particle size distribution after mechanical milling process by HEM-3D for 1 h, 3 h and 5 hours. The result of milled magnetite can be seen in Figure 7.

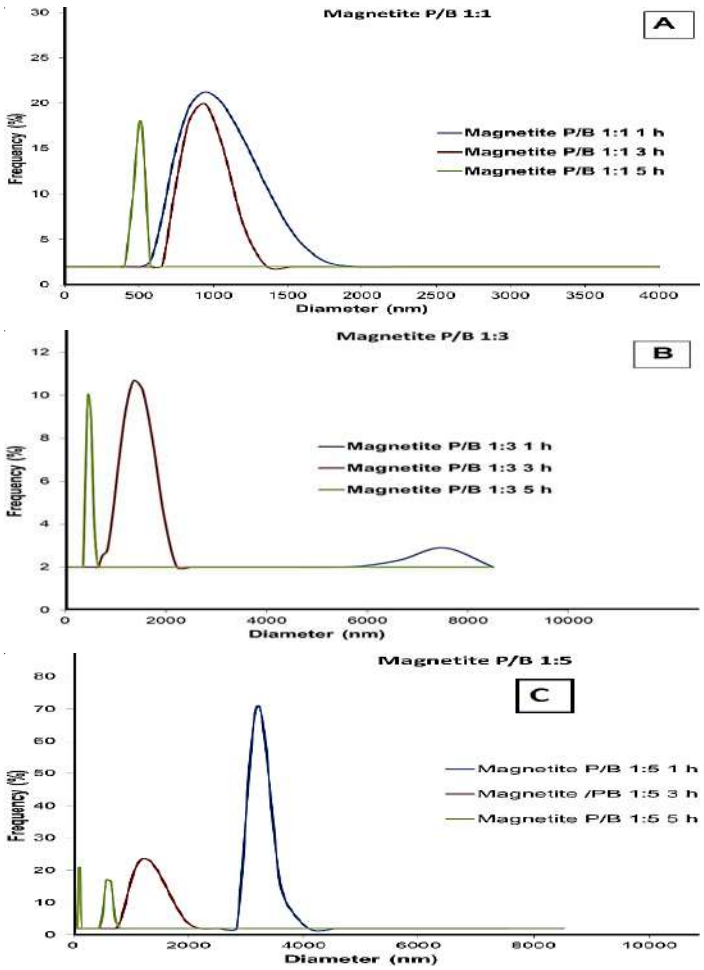

Fig. 7. Graph of magnetite size distribution on mass ratio magnetite: ball (P/B) 1: 1 (A); 1: 3 (B) and 1: 5 (C)

In Fig. 7 it is observed that the magnetite/ball mass ratio (P/B) of 1: 1 increase in time causes a significant reduction in particle size. When for 1 h milling the size range varies as well as for 3 h, however the milling is performed for 5 h gives impact to a more homogeneous magnetite size (the peak is not widened). Significant reduction in size occurred in treatment with a mass ratio of P/B 1: 3 and 1: 5. This is due to the heavier the ball and the length of time the greater the energy given to collide with the magnetite particles. Thus the magnetite treatment with HEM (high energy milling) is effective enough to reduce the size to less than 1000 nm (<1μm).

Morphology of magnetite

The surface morphology of a material can be observed using Scanning Electron Microscope. The basic principle of work on SEM is the nature of electron waves, it is diffraction at very small angles. Electrons are dissipated by a charged sample. The image formation on SEM comes from the electron beam reflected by the sample surface. If the sample used is not conductive, the sample must first be coated with gold[16].

Based on the SEM image in Fig. 8, the addition of spherical periods has an effect on the reduction of natural magnetite particle size. In the P/B ratio 1: 1 the particle size varies from small to large size. When the mass of balls increase 3 times to magnetite (P/B 1: 3), the collision between the magnetite and the ball gets stronger or the greater the energy that causes the breaking of the particles to become smaller and appear more homogeneous. In addition to the ball up to 5 times the magnetite period (P/B 1: 5) the particles also become smaller but the possibility of agglomeration appears to be larger if compared to P/B 1: 3. The size of the magnetite particles is slightly affected by the length of time the collision with the ball on the planetary ball mill. The milling process for 1 to 3 hours gives almost the same result, observed on surface morphology at P/B 1: 1 for 1 h is almost equal to 3 hours. Similarly to P/B 1: 3 for 1 hour is almost the same as for 3 hours, and P/B 1: 5 for 1 hour with 3 hours. However, when the milling for 5 h on the three variations of the ball period gives significantly different results with the previous. This is especially observed in P/B 1: 3 for 5 hours, visible particles having clear and firm shape and cleaner than others.

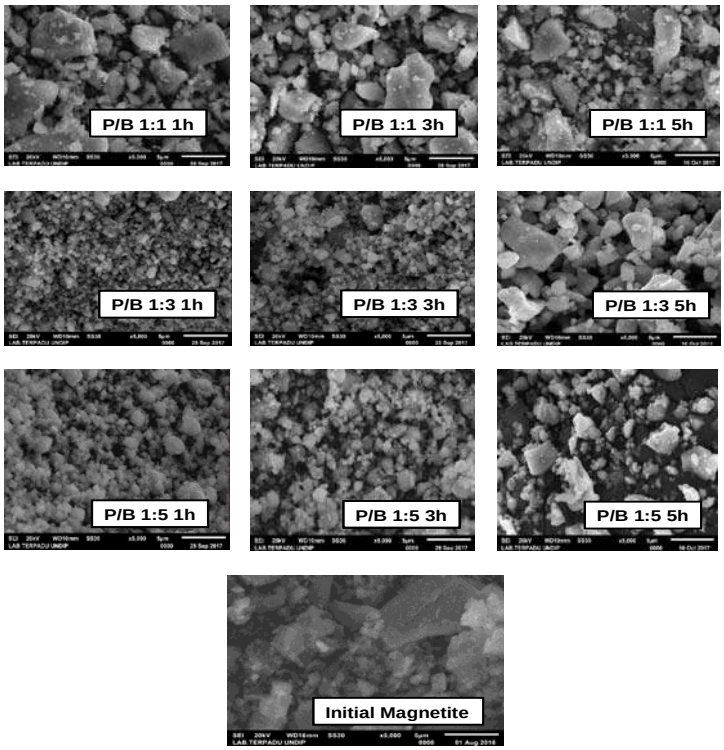

Fig. 8. Morphology of milled magnetite and initial magnetite by magnification 5000x

CONCLUSION

From the results and discussion can be concluded that the HEM-3D treatment with 400 rpm speed can reduce particle size and increase the uniformity of shape and magnetite size. The increasing of the ball mass in the milling process, this means in the mass ratio of magnetite:ball (P/B) 1: 1, 1: 3 and 1: 5 give the magnetite particles smaller, the crystallinity decreases but the phase does not change. Rising the milling time can lead to decreasing of size and crystallinity. Even milling time for 5 h on mass ratio of magnetite: ball (P/B) 1: 5 causes the magnetite phase to change to amorphous

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by Sriatun Sriatun

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Microstructure Characterization of Natural Magnetite from Sand Marina Beach by High Energy Milling

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ABSTRACT

In this work, we performed an experimental investigation the change of microstructure of magnetite by high energy milling-3D (HEM-3D) method using planetary ball milling at 400 rpm speed. The present studies mainly focusses on the effect of milling on crystallinity and phase of magnetite by XRD, particle size by PSA and the morphology by SEM. The increasing of the ball mass in the milling process, mass ratio magnetite: ball (P/B) 1: 1, 1: 3 and 1: 5 give the magnetite particles smaller (< 1μm), the crystallinity decreases but the peaks at (2 2 0), (3 1 1), (4 0 0), (5 1 1), and (4 4 0) were keep appearing. This shows that the phase of cubic spinel does not change. Rising the milling time for 1 h, 3 h and 5 h can lead to decreasing of size and crystallinity. Even milling time for 5 h on mass ratio of magnetite: ball (P/B) 1: 5 causes the magnetite phase to change to amorphous.

Keywords: Microstructure, Natural magnetite, Sand marina beach, High energy milling-3D.

INTRODUCTION

Iron sand occurs naturally in several regions throughout the world. Iron sand is one of Indonesia's natural mineral resources, which is spread over the islands along the coast of Java Island, Kalimantan and Sumatra. Iron sand is a special type of sand that's rich in the metal iron, the color is dark gray or black, consisting of (Fe) iron as a major element and a small amount of Ti, Si, Ca, Mn and Vanadium. They provide a raw material of relatively low grade, whereas in the southern coast of Yogyakarta containing 5.85 % to 95.11% of iron. In addition to magnetite in iron sand also contains other minerals such as rutile, ilmenite and hematite[1]. While most sand contains at least some trace of iron, therefore it has a distinct dark-gray or black color, which is in stark contrast to the white-yellow color of regular sand.

Iron sand is a magnetic material that is widely used in various fields such as electronics, energy, chemistry, ferrofluidics, catalysts, and medical diagnostics[2]. The application of iron sand was inseparable from the development of studies of nanomaterials demanding that they be in the order of nanometers. Magnetite or Fe_3O_4 is one of the iron oxide phases which has the greatest magnetic or ferromagnetic properties among the other phases. Iron oxide has four phases, namely magnetite (Fe_3O_4), maghemite (γ-Fe_2O_3), hematite (α-Fe_2O_3), and geotite (FeO(OH)). Only magnetite and maghemite have magnetic properties[3].

Magnetite (Fe_3O_4) is known as a class of iron oxide compound with a cubic inverse spinel structure and has face centered cubic close packed oxygen anions and Fe cations occupying interstitial tetrahedral and octahedral sites[4,5]. Nano-sized magnetite particles provide many advantages such as for the separation of magnetic contaminants in water, large of surface area and the ability to bind through electro-chemical interactions to form sludge. It is also applied to drug delivery and magnetic resonance technology and others.

For the synthesis of nanosized magnetite particles can be synthesized through various methods such as mechanical milling[6], sol-gels, direct decomposition[7], co-precipitation[8], microwave-heating[9] and solvothermal[10,11]. Mechanical milling method is one way to reduce the magnetite size is the cheapest and easy. Mechanical milling is defined as the mechanical breakdown of magnetite into smaller without changing their state of aggregation. The method was used to increase the surface area and induce defects which is needed for subsequent operations such as chemical reactions or sorption. Milling also to increase the proportion of regions of high activity in the surface[12].

Furthermore, this research the small size of magnetite from iron sand was prepared by mechanical milling method using high energy planetary ball mill. Kinetic energy of the balls depends not only on its velocity, but also on its mass and how long the collision occurred, due to in this work investigated the ratio of magnetite and ball mass in the planetary ball mill and the time of impact during collision.

MATERIALS AND METHODS

Materials

Iron sand was taken from Marina Beach in Semarang.

Instrumentations

Magnet permanent, High energy planetary ball mill-3D, X-ray diffraction (XRD) Rigaku Multiplex with Cu Ká radiation (λ = 1.54184 A°) at generator voltage 40 kV and current 40 mA, Particle Size Analyzer (PSA) Horiba SZ-100, Scanning electron microscope (SEM) JEOL JED 2300.

Procedure

Magnetite preparation

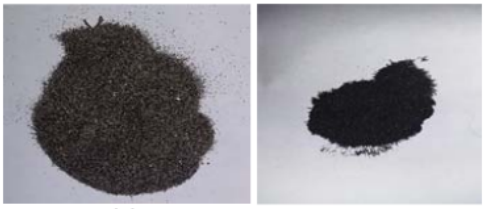
The natural iron sand from Marina Beach Semarang cleaned and washed using aquadest, dried in oven at 80 °C for 24 hours. Natural magnetite was extracted from natural iron sand using permanent magnet until 12 times. This treatment produces powder material dark gray-black color. Refinement of magnetite particles carried out by mechanical milling method using high energy planetary ball mill (HEM-E3D) instrument. The milling was done on mass ratio of magnetite: ball (P/B) 1:1, 1:3 and 1:5, speed 400 rpm. Milling of magnetite carried out for 1, 3 and 5 hours. Milled magnetite dried at 150 °C for 1.5 hours. Finally, the microstructure characterization of product was done by X-ray diffraction (XRD) to find out the structure of magnetite crystals, PSA to determine the size of magnetite particle, SEM to know the surface morphology.

RESULTS AND DISCUSSIONS

In this work the change of crystal structure, particle size and morphology of magnetite was investigated. The method is high energy milling (HEM) used planetary ball mill. The choice of this method due to it can reduce the material up to the nano order (nano particle) inside a relatively short time under conditions atmosphere at room temperature during process milling. This method using energy collision between the crushing balls and chamber walls are rotated and driven in a certain way. The change of crystal structure, particle size and morphology of magnetite was studied on variation the mass ratio magnetite:ball (P/B 1:1, 1:3 and 1:5) and milling time (1, 3 and 5 hours).

Physical changes of magnetite

The process of separation of magnetite compounds from iron sand is done repeatedly, it is intended that the compound to be obtained has a high purity. The separation process with magnets also uses a certain distance, the farther the magnet is closer to the iron sands, the less iron oxide attaches. This makes the sample (magnetite) higher purity and less impurities, although there is still the possibility of the other oxide compounds sticked to a permanent magnet. The Fig. 1 following is the embodiment of magnetite extracted from iron sand.



(a) (b)

Fig. 1. The original iron sand from marina beach before extraction treatment with permanent magnet (A) Magnetite after extraction treatment

The extracted iron sand powder then performed mechanical milling with several variations of the mass ratio of magnetite:ball (P/B) 1:1, 1:3 and 1:5 for 1, 3 and 5 h at speed 400 rpm. Magnetite obtained from the milling results has a softer texture and dark black as shown in Figure 2.

Fig. 2. Milled magnetite

It is clearly from Fig. 1A and 1B and Fig. 2, the difference in color and size of iron sand. In iron sand that has been separated with permanent magnet looks blacker than iron sand that has not been separated. This is due to the reduction of impurities from the iron sand so that the iron sand look blacker after extraction using permanent magnet as much 12 times. This shows that the separation of iron sand from impurity elements by this method more effectively. The size of iron sand after mechanical milling becomes smaller and softer than the separated iron sand. This is the advantages of mechanical milling method that ball mill is not sensitive to metal. The superiority of high energy milling is able to produce smaller particles in shorter milling time[12].

Crystal structure of magnetite

Based on the results of the analysis using X-ray diffraction on magnetite powder before milling treatment with HEM-3D obtained X-ray diffraction pattern as shown in Fig. 3. There are five highest peaks at 2è angle of 30.09°; 35.46°; 43.09°; 56.98°; and 62.59°. Furthermore the highest peaks were compared with Joint Committee of Powder Diffraction Standard (JCPDS) number 89-4319 with the highest peaks at 2θ angle of 30.083°; 35.434°; 43.064°; 56.949°; and 62.536°. Based on data obtained from XRD, the compound is magnetite.

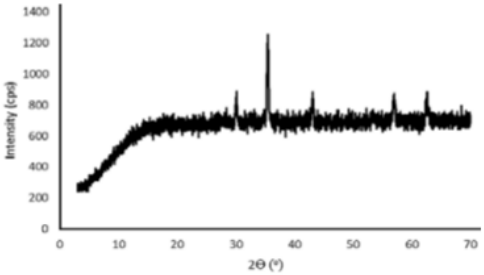


Fig. 3. Diffractogram XRD of magnetite after extraction treatment

Data of X-ray diffraction on magnetite after HEM-3D treatment with mass ratio of magnetite: ball (P/B) 1: 1, 1: 3 and 1: 5 and time collision 1, 3 and 5 h showed in Fig. 4, Fig.5 and Fig. 6. All diffraction peaks correspond to the peak diffraction at (2 2 0), (3 1 1), (4 0 0), (5 1 1), and (4 4 0). Of the highest peaks are compared with the Joint Committee of Powder Diffraction Standard (JCPDS) number. 79-0418 shows indexed to the Fe_3O_4 cubic spinel phase.

The XRD datas show that in all P / B ratio 1, 1: 1: 3 or 1: 5 with milling process for 1 and 3 h still indicates conformity with reference magnetite. When the milling for 5 h is only in P/B 1: 1 and P/B 1: 3 which still shows the suitability and even this is only at the peak of 2´ = 35.92° and 63.02° at P/B 1: 1 and 36.19° and 63.15° at P/B 1: 3, where the peak of the diffractogram is very low, whereas in P/B 1: 5 there is no correspondence with the reference

magnetite. This suggests that long-term milling treatments and strong collisions (heavier ball) can significantly reduce magnetic particle size, these treatments also decreased degrade of crystallinity. The increasing of ball to magnetite mass ratio (heavier ball) would enhancing the kinetic energy during milling. Based on kinetic energy equation:

$$E_k = {}^1/_2\, mv^2$$

which is the kinetic energy, m and v are respectively the mass and velocity of the balls. In this research the velocity was constant.

When the colliding ball mass is heavier, so the kinetic energy increases. The high of kinetic energy would cause the particles to collide with each other, where this would decrease in particle size.

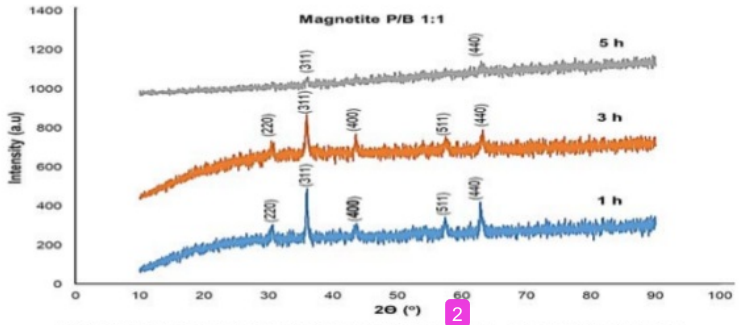


Fig. 4. Diffractogram milled magnetite by mass ratio of magnetite: ball (P/B) 1:1

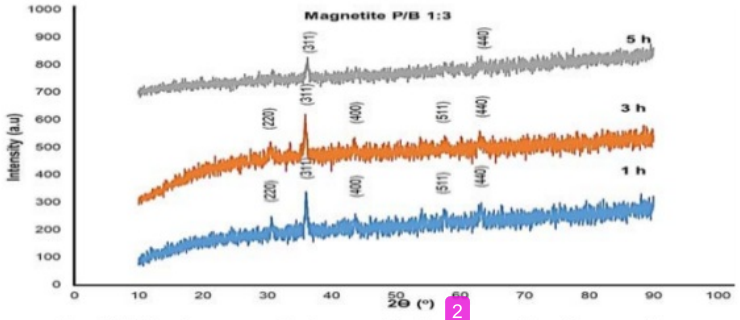


Fig. 5. Diffractogram milled magnetite by mass ratio of magnetite: ball (P/B) 1:3

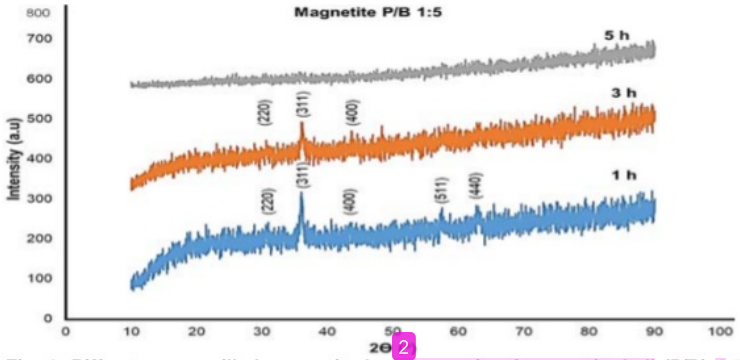


Fig. 6. Diffractogram milled magnetite by mass ratio of magnetite:ball (P/B) 1:5

This is in accordance with data that has been revealed by previous research. It was reported that the enhancing energy during milling, resulted by the increase of ball to powder weight ratio (BPR) and vial speed not only can accelerate the formation of the products but also changes the resultant phases[4]. The balls play an important role in its efficiency so that a small change in type, shape, weight or mass and size distribution of the balls can dramatically affect the milling process[5]. The increase of the number of balls at high BPR ratio, has a quite negative effect on the milling performance[6].

Particle size of magnetite

This matter proves that the milling process is done to magnetite powder can causing the destruction of the grains magnetite powder as a result collision between magnetite powder and milling balls. To know more clearly destruction of graphite powder during process milling, then the measurement magnetite particles by particle size analyzer (PSA) instrument. The choice of particle measurement methods of nanoscale and micro size is usually by using a wet method PSA (particle size analyzer) method, because it is an accurate method when compared to other methods. Small particles have a tendency for high agglomeration, the choice of wet method on PSA because the particles are dispersed into the medium so that the particles do not agglomerate (clump). Therefore the measured particle size is the size of a single particle and provides overall information on sample conditions.

Distribution particle size test by particle size analyzer (PSA) aims to determine particle size distribution after mechanical milling process by HEM-3D for 1 h, 3 h and 5 hours. The result of milled magnetite can be seen in Figure 7.

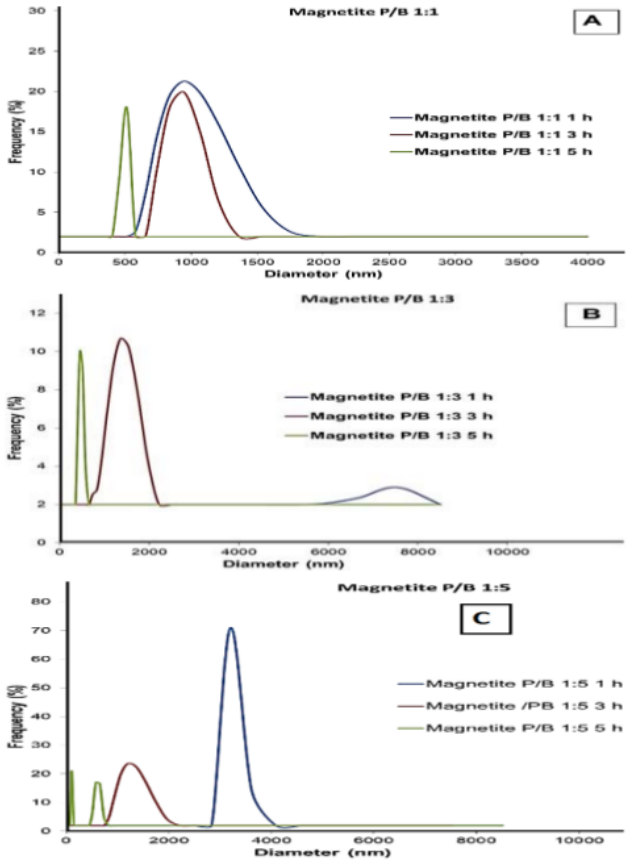


Fig. 7. Graph of magnetite size distribution on mass ratio magnetite: ball (P/B) 1: 1 (A); 1: 3 (B) and 1: 5 (C)

In Fig. 7 it is observed that the magnetite/ball mass ratio (P/B) of 1: 1 increase in time causes a significant reduction in particle size. When for 1 h milling the size range varies as well as for 3 h, however the milling is performed for 5 h gives impact to a more homogeneous magnetite size (the peak is not widened). Significant reduction in size occurred in treatment with a mass ratio of P/B 1: 3 and 1: 5. This is due to the heavier the ball and the length of time the greater the energy given to collide with the magnetite particles. Thus the magnetite treatment with HEM (high energy milling) is effective enough to reduce the size to less than 1000 nm (<1μm).

Morphology of magnetite

The surface morphology of a material can be observed using Scanning Electron Microscope. The basic principle of work on SEM is the nature of electron waves, it is diffraction at very small angles. Electrons are dissipated by a charged sample. The image formation on SEM comes from the electron beam reflected by the sample surface. If the sample used is not conductive, the sample must first be coated with gold[16].

Based on the SEM image in Fig. 8, the addition of spherical periods has an effect on the reduction of natural magnetite particle size. In the P/B ratio 1: 1 the particle size varies from small to large size. When the mass of balls increase 3 times to magnetite (P/B 1: 3), the collision between the magnetite and the ball gets stronger or the greater the energy that causes the breaking of the particles to become smaller and appear more homogeneous. In addition to the ball up to 5 times the magnetite period (P/B 1: 5) the particles also become smaller but the possibility of agglomeration appears to be larger if compared to P/B 1: 3. The size of the magnetite particles is slightly affected by the length of time the collision with the ball on the planetary ball mill. The milling process for 1 to 3 hours gives almost the same result, observed on surface morphology at P/B 1: 1 for 1 h is almost equal to 3 hours. Similarly to P/B 1: 3 for 1 hour is almost the same as for 3 hours, and P/B 1: 5 for 1 hour with 3 hours. However, when the milling for 5 h on the three variations of the ball period gives significantly different results with the previous. This is especially observed in P/B 1: 3 for 5 hours, visible particles having clear and firm shape and cleaner than others.

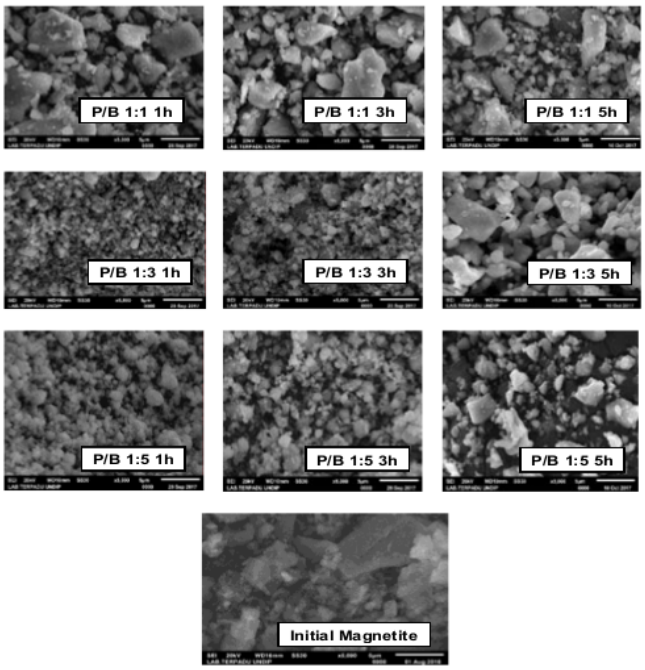


Fig. 8. Morphology of milled magnetite and initial magnetite by magnification 5000x

CONCLUSION

From the results and discussion can be concluded that the HEM-3D treatment with 400 rpm speed can reduce particle size and increase the uniformity of shape and magnetite size. The increasing of the ball mass in the milling process, this means in the mass ratio of magnetite:ball (P/B) 1: 1, 1: 3 and 1: 5 give the magnetite particles smaller, the crystallinity decreases but the phase does not change. Rising the milling time can lead to decreasing of size and crystallinity. Even milling time for 5 h on mass ratio of magnetite: ball (P/B) 1: 5 causes the magnetite phase to change to amorphous

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