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Effect of NaOH Concentration on Magnetic Properties and Structural Studies of $MgFe_2O_4$ Based on Natural Iron Sand Synthesized by Co-Precipitation Method

Shiamny Nur Khair Tarigan, * Syahrul Humaidi, Martha Rianna, Nurul Maulida R Nasution
Departement of Physics, Faculty of Mathematics and Science, Universitas Sumatera Utara, Jl. Bioteknologi No. 1 Kampus USU, Medan, Sumatera Utara, Indonesia. Postal code: 20155

*Corresponding Author e-mail: syahrul1@usu.ac.id

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Abstract

This research was conducted to synthesize $MgFe_2O_4$ using the coprecipitation method by varying the concentration of NaOH. In this study, $MgCl_26H_2O$, iron sand, 37% HCl and variations of NaOH (5 M and 10 M) were mixed at 90 ℃ for 60 minutes, then washed using distilled water and ethanol until pH = 7, and calcined at 750 ℃. The test results using X-Ray Diffraction (XRD) showed that the diffraction pattern of phase formation identified was $MgFe_2O_4$ with hkl values (220), (311), (400), (422), (511), and (440). The resulting crystal size decreased with the addition of NaOH concentration. The test results using a Vibrating Sample Magnetometer (VSM) show that the magnetization value decreases when the crystal size decreases.

Keywords: $MgFe_2O_4$, NaOH Concentration, Co-Precipitation

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INTRODUCTION

Nanoparticles (NPs) are usually defined as particles with a size of 100 nm or less in at least one dimension. NPs exhibit unique and often improved physicochemical properties compared to their bulk counterparts.. NPs have received much attention due to their small size and high surface-to-volume ratio, which leads to their reactivity and excellent chemical, electronic, optical, magnetic and mechanical properties, (Selmani et al., 2022). Nanoparticles have different physical and chemical properties from their original structural properties. These properties can be modified by adjusting the particle size, chemical composition, surface modification and interactions between the particles (Fabiani et al., 2018).

Nanoparticles can be synthesized through various methods such as microemulsion, solvothermal, hydrothermal, sol-gel, chemical coprecipitation, laser deposition, and thermal decomposition (al Yaqoob et al., 2019). Of these methods, the coprecipitation method is the most effective and can be carried out at low temperatures. In addition, the coprecipitation method can control particle size and can be used to assess the dependence of magnetic properties on particle size (Suharyadi et al., 2018).

Indonesia has long been well known to be rich in mineral resources. Significant deposits of tin, copper, bauxite, nickel, gold, silver, uranium, granite, coal, manganese, natural asphalt and also iron sands have already been discovered over several decades. (Tapakis et al., 2018). The presence of a lot of iron sand has attracted the interest of researchers to explore this material to look for industrial opportunities that are more valuable than just construction materials with little economic value. Iron sand has been reported to contain iron oxides such as magnetite (Fe_3O_4), maghemite (γ-Fe_2O_3), and hematite (α-Fe_2O_3), which are considered as raw materials for various industrial applications. Several important physical properties such as chemical stability, high saturation magnetization, high coercivity, and corrosion resistance have led to the wide use of this material, (Togibasa et al., 2019).

$MgFe_2O_4$ is a soft magnetic material with n-type semiconductor which has low saturation magnetization, high resistivity, high stability and non-toxicity. $MgFe_2O_4$ is used in sensors, drug delivery systems and in other magnetic technologies (Hashemian & Yousofi, 2014; Jung et al., 2017). $MgFe_2O_4$ nanoparticles have good photoelectric properties [26]. The properties of ferrite materials are strongly influenced by the distribution of metal ions between crystal lattice sites and, in turn, by the synthesis method used. (Naaz et al., 2020)

The coprecipitation or precipitation method is the most commonly used method for forming magnetic adsorbents, (Meili, 2021). The coprecipitation method involves a molten metal salt (precursor) mixed with a base, which acts as a precipitating agent at sufficient temperature. The controlled release of anions and cations favors the nucleation and regulation of the particle growth rate, thereby synthesizing monodispersed nanoparticles. (Ashik et al., 2018; Bajaj & Joshi, 2021).

In this study we use Vibrating Sample Magnetometer (VSM) and X-Ray Diffraction (XRD) to report the Magnetic properties and structural studies on $MgFe_2O_4$ based on natural iron sand by co-precipitation method.

METHOD

The dissolving process of iron sand was carried out by mixing 8 grams of iron sand with 11.4 ml of 37% HCl. The mixture was mixed using a magnetic stirrer for 30 minutes at 500 rpm at room temperature. Then the mixture was filtered using Whatman 41 paper for 60 minutes to obtain $FeCl_3$ solution.

Synthesis of magnesium ferrite nanoparticles in this study was carried out using the coprecipitation method. By mixing the $FeCl_3$ solution obtained with $MgCl_2{\cdot}6H_2O$ and NaOH of varying concentrations as the main ingredients. $MgCl_2{\cdot}6H_2O$ and $FeCl_3$ were weighed according to the variations presented in Table 1. Then mixed with 100 ml of distilled water and stirred using a magnetic stirrer until homogeneous. The homogeneous solution mixture was added drop by drop slowly while stirring using a magnetic stirrer into the 5 M and 10 M NaOH solution, with a magnetic stirrer speed of 500 rpm and a temperature of 90 °C for 120 minutes.The magnesium ferrite solution is precipitated using a permanent magnet and then the precipitate and impurities are separated. The washing process was carried out using distilled water and ethanol repeatedly to neutralize the pH (pH=7) and remove the salts that were formed during the reaction. after the precipitate is clean, then the sample is dried in the oven then calcination at 750 °C for 2 hours.

Table 1. Synthesis Parameter of $MgFe_2O_4$

Sample	$MgCl_2{\cdot}6H_2O$ (gram)	Iron Sand (gram)	NaOH Concentration (M)	Stirring Duration (menit)	Temperature °C
K1	6,912	8	5	60	90
K2	6,912	8	10	60	90

X-Ray Diffraction (XRD) Characterization

Using a Rigaku D/max 2500 X-ray powder diffractometer (Rigaku, Tokyo, Japan), the XRD pattern of $MgFe_2O_4$ were studied. If a particle contains a number of small crystallites then the information provided by the Scherrer method is the size of the crystalline, not the size of the particles. For nanometer-sized particles, usually one particle contains only one crystallites. Thus, the size of the crystallinity predicted by the Scherrer method is also the particle size.

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

Vibrating Sample Magnetometer (VSM) Characterization

VSM is used to see the magnetic properties of the sample. The measurement results obtained from this test are the coercivity (Hc), remanence (r), saturation (s), and loop area values. From VSM will also produce a hysteresis curve from the sample being tested. The steps in this test are:

The sample to be tested is prepared, then the sample to be tested is weighed as much as 0.5 grams. Then the sample is put into the capsule and placed in the sample holder. Drop enough power glue so that the sample dries and hardens. After the sample is dry, the capsule containing the sample is inserted into the tester on the VSM. Then prepared software to support testing on VSM and given an external magnetic field (Hext) on the sample to get the results of testing the magnetic properties of the sample.

RESULTS AND DISCUSSION

The phase of $MgFe_2O_4$ is invistigated by XRD patterns that shown in Figure 1. The XRD patterns showed that the diffraction pattern of phase formation identified was $MgFe_2O_4$ with hkl values (220), (311), (400), (422), (511), and (440).

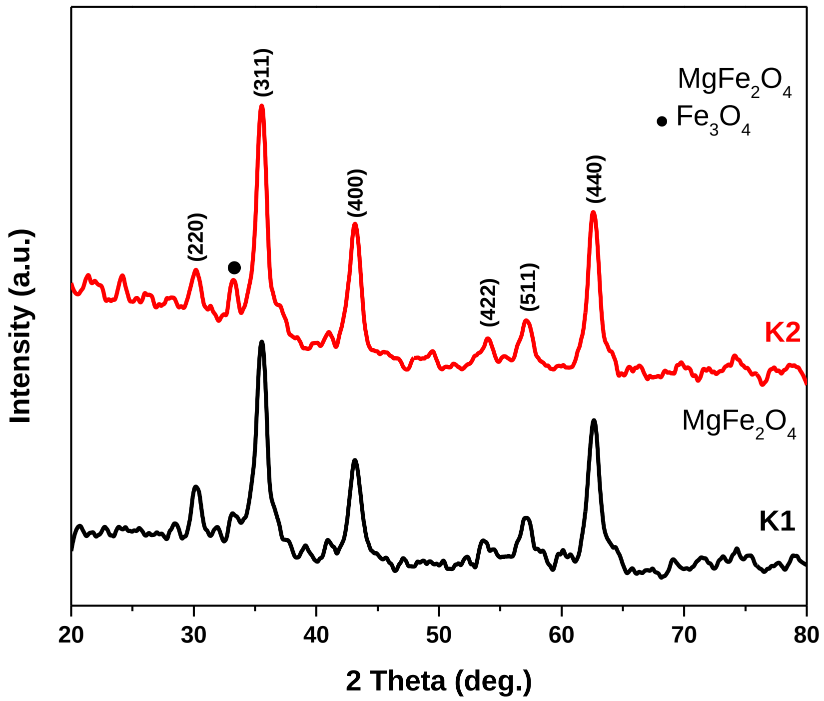

Figure 1. XRD pattern of the sample

The formed phase has a main peak (311) at 2θ=35°. Crystal size is known based on the results of calculations using the Scherrer equation. Table 2. describes the crystal size and lattice parameters obtained.

Table 2. Crystal properties on $MgFe_2O_4$

No	Sample	2 Theta (deg)	FWHM (deg)	Crystallite Size (nm)	Lattice Parameter (Å)
1	K1	35.58	5.33	1.56	8.37
2	K2	35.49	5.59	1.49	8.39

with variations in the addition of NaOH concentration produces smaller crystal grain sizes along with the addition of NaOH concentration. The grain size of the crystals produced in this study was much smaller than the results obtained in previous studies, namely 35nm (Pradeep et al., 2008). With the same lattice parameter values as previous research, Å = 8.37, this study resulted in a crystal size of 1.56 nm, while previous studies produced a size of 36.6 nm (Naaz et al., 2020). The appearance of the magnetite phase was accompanied by an increase in the number of NaOH mole concentrations in the sample. In previous research, the formation of a magnetite peak has also been reported (Ciocărlie et al., 2022). High diffraction peaks determine the formation of nano-sized particles (Vaish et al., 2019).

The samples were characterized by Vibrating Sample Magnetometer (VSM) to analized the magnetic properties of $MgFe_2O_4$. The VSM result was interpreted in a hysterisis curve. The hysteresis curve of the sample is shown in Figure 2.

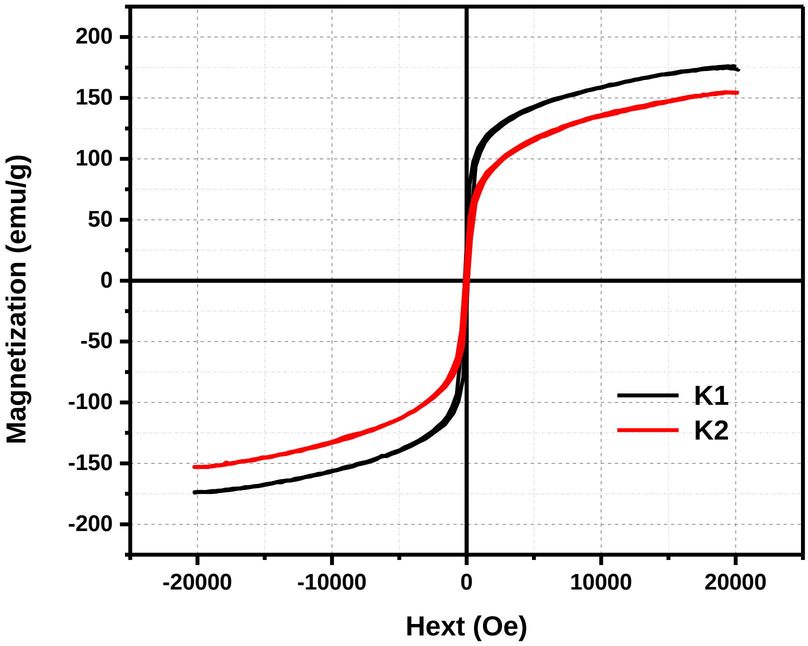

Figure 2. Hysterisis Curve of $MgFe_2O_4$

Figure 2. shows that the hysterisis curve forms a narrow area and has a small coercivity value, so it can be assumed that the synthesized $MgFe_2O_4$ sample is a soft magnetic material. The narrow area of the hysteresis curve proves that the material being synthesized is a material that is easily magnetized (does not require a large magnetic field). The magnetization values obtained from the VSM analysis shown in Figure 2. are described in Table 3.

Table 3. Magnetization value of $MgFe_2O_4$

Sample	Crystallite Size (nm)	*Hc* (Oe)	*Mr* (emu/g)	*Ms* (emu/g)
K 1	1.56	118.38	0.88	6.10
K 2	1.49	113.16	0.62	5.57

From Table 3. it can be seen that the concentration of NaOH affects the values of coercivity (Hc), remanent magnetization (Mr), and saturation magnetization (Ms). The magnetization properties are decreased as the NaOH concentration increased. The K2 sample with the smallest crystallite size. which is 1.49 nm, has the smallest coercivity (Hc) value. which is 113.16 Oe. Meanwhile. the K1 sample with the highest coercivity (Hc) value of 118.38 Oe has a crystallite size of 1.56 nm. The coercivity value gets smaller when the crystallite size gets smaller which is caused by variations in the addition of NaOH concentration. This is caused by a decrease in the barrier (energy anisotropy) in the particle. Under these conditions the magnetic moment in $MgFe_2O_4$ becomes easily magnetized by an external magnetic field and the coercivity value becomes smaller when it is demagnetized. so the magnetic properties are getting weaker. (Hermawan et al., 2015)This is caused by the effect of agglomeration (clumping) on the sample. so that the grains in the sample tend to agglomerate and cluster. thereby affecting the direction of the magnetic moment. which is not free to fluctuate. Therefore. a larger external magnetic field is needed to restore the magnetization to zero (Suharyadi et al., 2018).

The magnetic moment on magnetic nanoparticles tends to be more unstable as the magnetic particle size decreases. The instability of the magnetic moment on particles with grains with smaller grain sizes is caused by the anistrophic energy possessed by these particles which is much smaller than that of large-sized particles, as a result, if a smaller magnetic field is given, it will be more reactive in responding to the applied external magnetic field, . In addition, the magnetization value is also influenced by the presence of a primary phase, secondary phase, and the presence of impurities in the sample.

CONCLUSION

Based on XRD analysis, the diffraction pattern forming the identified phase is $MgFe_2O_4$ with a cubic crystal structure. The diffraction peaks have hkl values (220), (311), (400), (422), (511) and (440) which are confirmed according to the phase pattern database in JCPDS No. 96-900-1459. The sample forms a peak which produces a single phase $MgFe_2O_4$.

The smaller the grain size of the nanoparticles, the lower the coercivity value. This is presumably because the smaller the grain size, the smaller the energy anisotropy of the particle. Under such conditions, the magnetic moment of $MgFe_2O_4$ is easily magnetized by an external field, and when it is demagnetized, its coercivity tends to be lower than that of larger particles.

REFERENCES

al Yaqoob, K., Bououdina, M., Akhter, M. S., al Najar, B., & Vijaya, J. J. (2019). Selectivity and Efficient Pb and Cd Ions Removal by Magnetic MFe_2O_4 (M=Co, Ni, Cu and Zn) Nanoparticles. *Materials Chemistry and Physics*, *232*, 254–264. https://doi.org/10.1016/j.matchemphys.2019.04.077

Ashik, U. P. M., Kudo, S., & Hayashi, J. (2018). An Overview of Metal Oxide Nanostructures. In *Synthesis of Inorganic Nanomaterials*. Elsevier Ltd. https://doi.org/10.1016/B978-0-08-101975-7.00002-6

Bajaj, N. S., & Joshi, R. A. (2021). Energy materials: synthesis and characterization techniques. *Energy Materials*, 61–82. https://doi.org/10.1016/B978-0-12-823710-6.00019-4

Ciocărlie, L., Negrea, A., Ciopec, M., Duteanu, N., Negrea, P., Ianasi, P., Ianasi, C., & Nemes, N. S. (2022). Indium Recovery by Adsorption on MgFe2O4 Adsorbents. *Materials*, *15*(20). https://doi.org/10.3390/ma15207054

Fabiani, V. A., Sutanti, F., Silvia, D., Putri, M. A., Kimia, J., Teknik, F., & Belitung, U. B. (2018). *30533-75676594166-1-Sm*. *1*(2), 68–76.

Hermawan, A., Lismawenning, D., & Suharyadi, E. (2015). Sintesis Nanopartikel Magnesium Ferrite ($MgFe_2O_4$) dengan Metode Kopresipitasi dan Karakterisasi Sifat Kemagnetannya. *Prosiding Pertemuan Ilmiah XXIX HFI Jateng & DIY*, 178–183.

Meili, L. (2021). *Advanced Magnetic Adsorbents for Water Treatment*. Springer. https://doi.org/https://doi.org/10.1007/978/--3-030-64092-7

Naaz, F., Dubey, H. K., Kumari, C., & Lahiri, P. (2020). Structural and magnetic properties of MgFe2O4 nanopowder synthesized via co-precipitation route. *SN Applied Sciences*, *2*(5). https://doi.org/10.1007/s42452-020-2611-9

Pradeep, A., Priyadharsini, P., & Chandrasekaran, G. (2008). Sol- gel route of synthesis of nanoparticles of MgFe2O 4 and XRD, FTIR and VSM study. *Journal of Magnetism and Magnetic Materials*, *320*(21), 2774–2779. https://doi.org/10.1016/j.jmmm.2008.06.012

Selmani, A., Kovačević, D., & Bohinc, K. (2022). Nanoparticles: From synthesis to applications and beyond. *Advances in Colloid and Interface Science*, *303*, 102640. https://doi.org/10.1016/J.CIS.2022.102640

Suharyadi, E., Hermawan, A., & Puspitarum, D. L. (2018). Crystal Structure and Magnetic Properties of Magnesium Ferrite (MgFe2O4) Nanoparticles Synthesized by Coprecipitation Method. *Journal of Physics: Conference Series*, *1091*(1). https://doi.org/10.1088/1742-6596/1091/1/012003

Tapakis, U., Pariaman, P., Sumatra Adree Octova, W., Mingsi Anaperta, Y., Gina Febriandika, H., -, al, Wahyuningsih, S., Ramelan, A. H., Pranata, H. P., Rahmi, A., Rifai, H., Gunanto, Y. E., Izaak, M. P., Jobiliong, E., Cahyadi, L., & Adi, W. A. (2018). High purity Fe3O4 from Local Iron Sand Extraction. *Journal of Physics: Conference Series*, *1011*(1), 012005. https://doi.org/10.1088/1742-6596/1011/1/012005

Togibasa, O., Akbar, M., Pratama, A., & Bijaksana, S. (2019). Distribution of Magnetic Susceptibility of Natural Iron Sand in the Sarmi Coast Area. *Journal of Physics: Conference Series*, *1204*(1), 012074. https://doi.org/10.1088/1742-6596/1204/1/012074

Vaish, G., Kripal, R., & Kumar, L. (2019). EPR and optical studies of pure MgFe2O4 and ZnO nanoparticles and MgFe2O4–ZnO nanocomposite. *Journal of Materials Science: Materials in Electronics*, *30*(17), 16518–16526. https://doi.org/10.1007/s10854-019-02028-y