

Investigation of Magnetic Properties and Mechanical

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Investigation of Magnetic Properties and Mechanical Responses on Hydrogel-TMAH-Magnetite

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Abstract. Hydrogel-TMAH-Magnetite (ferrogel) was fabricated by using polyvinyl alcohol (PVA) hydrogel and magnetite fluids with tetramethylammonium hydroxide (TMAH) surfactant. Iron sand as the raw material was used to synthesize magnetite nanoparticles by co-precipitation method. Magnetite nanoparticles and ferrogel were characterized using X-Ray Fluorescence (XRF) to determine the content of elements in it. To know the functional group network of magnetite nanoparticles, magnetite enclosed with TMAH and ferrogel; we investigated using Fourier Transform Infra-Red (FTIR). Meanwhile, the magnetic properties of the hydrogel-TMAH-magnetite were measured by using Vibrating Sample Magnetometer (VSM). Furthermore, the composition analysis of the ferrogels using FTIR showed that all the synthesis materials were inside the ferrogels. The saturation magnetization of the hydrogel-TMAH-magnetite with a composition of TMAH 1.2 mL ($3.95 \text{ emu}\cdot\text{g}^{-1}$) was higher than that of TMAH 0.8 mL ($0.85 \text{ emu}\cdot\text{g}^{-1}$). It exhibited that the composition of TMAH 1.2 mL was an optimum composition to produce nanoparticle magnetite-TMAH having a stable and high performance. Furthermore, the magneto-elasticity of hydrogel-TMAH-magnetite in the effect of the external magnetic field had a good response. However, the composition of the nanoparticle magnetite-TMAH in the ferrogel did not significantly affect the elongation of the gel.

Keywords. Ferrogel, hydrogel, iron sand, magneto-elasticity, and magnetite-TMAH.

1. Introduction

The use of natural material to develop the nanotechnology research has frequently been done, one of them is synthesis and characterization of magnetite nanoparticle. The researchers have successfully fabricated the magnetite nanoparticles of which raw material is local iron sand [1–3]. The magnetite nanoparticles can be synthesized through some methods such as planetary ball-milling [4, 3], co-precipitation [6, 7], hydrothermal [8], microwave [9], sol-gel, microemulsion [10, 11], polyol method



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[12, 13] or heat decomposition [14]. By the freezing-thawing route, gamma-ray irradiation, or the combination of both, the magnetite nanoparticles can be applied become a multifunction material such as ferrogel [15, 16]. From some previous reports, ferrogel can be used in health and medical fields such as medicine conduction, hyperthermia therapy, contact lens, and the others [17–19].

Furthermore, ferrogel is a unique material of which form can change with some variations of a polymer matrix and magnetic filler within it. Sahiner *et al* [20] have successfully fabricated the poly composite (acrylonitrile-co-1-vinyl imidazole) or p(AN-c-1-VI) with the magnetite nanoparticles by microemulsion method. p(AN-c-1-VI) based composite, and magnetite nanoparticles can be used as a catalyst material that can reduce the organic material. Besides, Rese'ndiz-Herna'ndez *et al* [21] also have successfully investigated the magnetization behavior of PVA polymer based ferrogel and magnetite. They reported that ferrogels with the comparison of 1:1 between filler composition and PVA polymer behaved in superparamagnetic with the value of maximum saturation magnetization was $0.6 \text{ emu}\cdot\text{g}^{-1}$. Seeing the mechanical behavior of ferrogel, the sensitivity of PVA polymer based-ferrogel move and magnetite nanoparticle move was in line with the filler concentration in the polymer network [22]. However, the investigation of PVA polymer based-ferrogel and magnetite nanoparticles enclosed with TMAH surfactant media is rarely reported.

The magnetic properties and mechanical behaviors of the PVA polymer based-ferrogel and TMAH-magnetite nanoparticles will be the focus of the study in this report. The synthesis of magnetite nanoparticles and the ferrogel fabrication in this research used co-precipitation and freezing-thawing methods, respectively. These methods were used since they were relatively fast, simple, low-cost, and easy to be conducted in the Nanomaterial Laboratory of Physics Department, State University of Malang.

2. Materials and methods

2.1. Magnetite Nanoparticles Synthesis

The sample of magnetite nanoparticles was synthesized by using co-precipitation method. It was started by the separation of natural iron sand to separate the Fe_3O_4 powder from the other particles such as Si, Ti, Mn, Zn, and so forth. The Fe_3O_4 powder as the result of separation was mixed with Hydrogen Chloride (HCl), and it was stirrer on a hotplate magnetic stirrer to fasten the separation process of Fe^{2+} ion and Fe^{3+} ion. The result of ion separation was then filtered and dropped gradually with Ammonium Hydroxide (NH_4OH) solution so that it formed a black precipitate. The precipitate as the result of titration was then washed by using distilled water so that the pH was normal. The magnetite nanoparticles as the result of synthesis were added with TMAH surfactant with the variation of 0.8 mL and 1.2 mL to prevent the aggregation of particles.

2.2. Ferrogel Synthesis

By certain comparison, PVA polymer and distilled water were mixed and stirrer on a hotplate magnetic stirrer so that the mixture became condensed. After that, the magnetite nanoparticles that had been enclosed with TMAH surfactant was put into the mixture and distributed equally over all PVA hydrogel network indicated by the change of mixture color to become black. Subsequently, the mixture was poured into a container of which length was 10 cm, and diameter was 6 mm. Finally, the Freezing-Thawing (F-T) process was conducted to get the ferrogel composite material.

3. Characterization

Magnetite nanoparticles and ferrogel were investigated by using X-Ray Fluorescence (XRF) PANalytical Minipal 4 to know the content of it. Meanwhile, to discover the functional group network of the magnetite nanoparticles, the magnetite enclosed with TMAH and ferrogel, a test was conducted by using Fourier Transform Infra-Red (FTIR) Shimadzu, Type: IRPrestige21. The magnetization properties of the ferrogel were tested by using Vibrating Sample Magnetometer (VSM) Oxford.

Meanwhile, the elongation characterization was investigated to get the mechanical behavior of ferrogel by using an electromagnetic instrument.

3. Results and discussion

3.1. XRF Characterization

Table 1 and Table 2 show the results of XRF test of magnetite as the result of co-precipitation and ferrogel synthesis. Based on XRF test, the purity of Fe contained in Fe_3O_4 nanoparticles, and ferrogel was 87.31 % and 66.3 %, respectively. Beside Fe, there was a polluter in the magnetite and ferrogel samples such as Ti, Ni, P, Ca and the others. These components are difficult to avoid since the main material used in this research was natural iron sand from Wedi Ireng Beach, Banyuwangi.

Table 1. XRF Characterization of Fe_3O_4

Comp (%)	Fe	Ti	Bi	Mn	Eu	V	Ca	Si	Re	P	Ni	Cr	Zn	Yb
Conc.	87.3	7.6	0.9	0.9	0.8	0.5	0.4	0.4	0.3	0.2	0.1	0.0	0.0	0.
Unit	1	3	5	0	2	0	9	8	5	6	0	9	9	0

Table 2. XRF Characterization of Ferrogel

Comp (%)	Fe	Ni	P	Ca	Ti	Si	Cu	Zn	Ba	Re	Mn	Cr	V
Conc.	66.3	13.2	5.0	4.81	3.30	2.7	2.0	0.77	0.6	0.5	0.36	0.34	0.32
Unit													

3.2. FTIR Characterization

The compound contained in the magnetite, the magnetite enclosed with TMAH, and ferrogel has certain energy depending on the composite compound. Every energy of the compound will be represented by a functional group of each compound. FTIR test was characterized to know and confirm the contents of the magnetite, magnetite enclosed with TMAH, and ferrogel through the amount of the energy absorbed by every functional group to vibrate.

Furthermore, this FTIR test was investigated to ensure the presence of a functional group of Fe-O that becomes a special characteristic of the Fe_3O_4 compound and ensure the other functional group that was in accordance with the additional compound in TMAH and ferrogel. Figure 1 shows the data graph as the result of FTIR test on the magnetite sample, magnetite enclosed with TMAH, and ferrogel. The functional group of Fe-O in the three samples was detected in the range of wave number for vibration of Fe-O which was $(500 \text{ to } 750) \text{ cm}^{-1}$ [23–27]. Thereby, the magnetite nanoparticles were effectively spread within the ferrogel. Besides the Fe-O group, there was also the O-H group observed in the three samples in the range of $(3000 \text{ to } 4000) \text{ cm}^{-1}$ [28–33]. This result indicated that magnetite powder used in this research still contained water compound. Although the magnetite nanoparticles had been heated to the temperature of 100°C before doing FTIR test, there was still a part of water compound included in the magnetite nanoparticles.

The spectrum of TMAH surfactant enclosing the magnetite nanoparticles was confirmed well in the wave number of 1487.12 cm^{-1} [34–36] and $(900 \text{ to } 1000) \text{ cm}^{-1}$ [37]. Each wave number showed the functional group of CH_3 and C-N that become the special characteristic of the wave number of TMAH surfactant. The result of FTIR test on the ferrogel sample also revealed the peak at the wave number of 2943.37 cm^{-1} that becomes one of the characteristics of PVA polymer compound, this obtained wave number was in accordance with the previous research that was the functional group of the compound was at the point of 2941 cm^{-1} [38] and in the range of $(2900 \text{ to } 3000) \text{ cm}^{-1}$ [39].

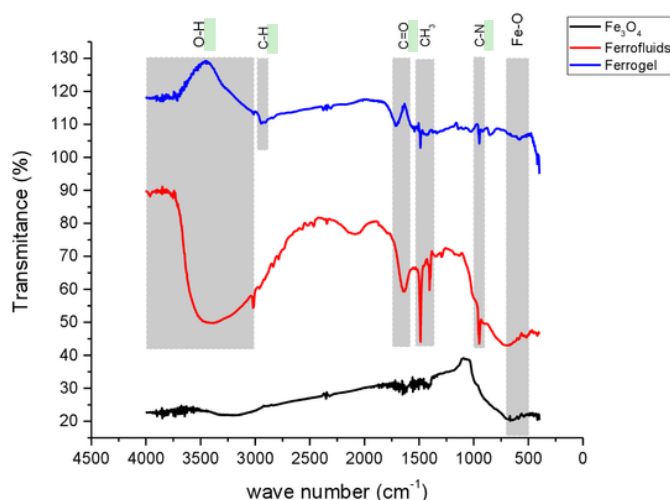


Figure 1. FTIR Characterization of the magnetite, the magnetite enclosed with TMAH, and ferrogel

3.3. VSM Characterization

VSM test was conducted to the ferrogel sample based on magnetite filler enclosed with TMAH surfactant and PVA hydrogel. This characterization aimed to know the magnetization properties of ferrogel such as saturation magnetization (M_s), the remanent magnetization (M_r), and coercivity (H_c). Besides, the magnetization data could be refined by using Langevin equation so that it resulted in magnetic moment variable (μ) and the size of magnetite nanoparticles (D).

Figure 2 displays the hysteresis curve of VSM test result in the ferrogel sample of FGT 0.8 and FGT 1.2. The sample code of FGT 0.8 and FGT 1.2 ferrogels has a meaning, each of FG means ferrogel sample, T is the TMAH surfactant enclosing magnetite nanoparticles and 0.8 and 1.2 mean the amount of TMAH concentration that was 0.8 mL and 1.2 mL enclosing the particle.

Based on the data of VSM result in Figure 2, the sample of FGT 1.2 had a higher value of saturation magnetization than the value of FGT 0.8. This case showed that the concentration of TMAH surfactant 1.2 mL had better magnetization effectiveness than the TMAH surfactant 0.8 mL. This result was also strengthened by the result of VSM data refinement showing that magnetic moment of ferrogel sample with the TMAH surfactant 1.2 mL was higher than 0.8 mL. The sample magnetization data also revealed that all ferrogel samples behaved as a superparamagnetic. This result was shown by the very small of remanent magnetization value and small coercivity field that was close to zero [40], [41]. This phenomenon was strengthened by the report of Resendiz-Hernández *et al* [21] stating that ferrogel with the value of maximum saturation magnetization was $0.6 \text{ emu} \cdot \text{g}^{-1}$ (the value that was in accordance with FGT 0.8 data) behaved as a superparamagnetic.

The result of magnetization data refinement also showed that the bigger the value of M_s the obtained particle size was getting smaller (data from Table 4). This result was confirmed well by a report stating that the increase in M_s value was caused by the decrease in the crystal size of the sample [42, 43]. If the average size of magnetite nanoparticles as the result of refinement with the XRD data or TEM data was about (11 to 15) nm [22], the value of refinement result showed the smaller value compared to XRD and TEM data. This case was possibly caused by three factors such as the presence of dead surface layer in the magnetite nanoparticles [42] and the presence of coating or enclosing the magnetite nanoparticles with another substance [44] that was present in this research in the form of TMAH surfactant and different measuring technique between XRD, TEM, and VSM.

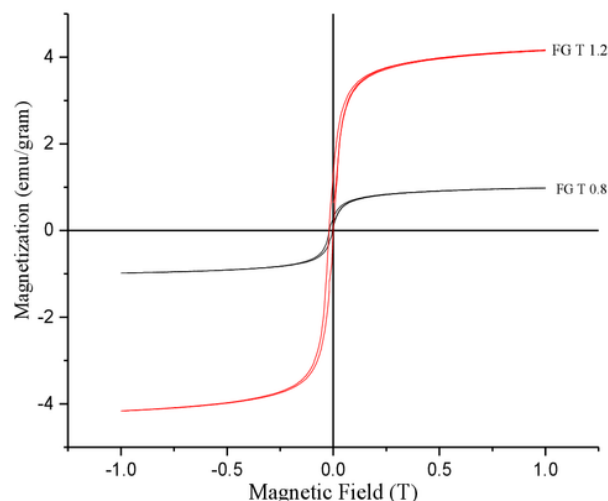


Figure 2. Hysteresis Curve of Ferrogel

Table 3. Amount of Data Fitting Result of VSM Test

Sample	M_s ($\text{emu}\cdot\text{g}^{-1}$)	M_r ($\text{emu}\cdot\text{g}^{-1}$)	H_c (T)	μ (J/T) $\times 10^{-19}$
FG T 0.8	0.8543	0.0077	0.0104	2.217
FG T 1.2	3.9495	0.0259	0.0084	2.306

Table 4. The Particle Size of Ferrogel as the Result of VSM Analysis

Sample	Particle Size (nm)
FG T 0.8	9.38
FG T 1.2	6.99

When the sample was enclosed by surfactant, the interaction between particles would decrease so that the aggregation between particles also decreased and gave the impact on the decrease in the size of magnetite nanoparticles when it was characterized by using VSM (the sample condition was in the form of a gel). For the XRD and TEM data, the condition of the particle was in the dry condition so that TMAH surfactant could not work effectively to enclose the particles and gave impact on the big aggregation of the particle so that the size was bigger.

3.4. Magneto-Elasticity Characterization

Magneto-elasticity characterization of ferrogel aimed to know the ferrogel deformation especially the elongation pattern and ferrogel response as the result of the influence of external magnetic field. Ferrogel was arranged vertically with the top tip was made constant, and the bottom tip was let to move freely. When the bottom tip of the ferrogel was given the magnetic field, the sample response could be observed from the ferrogel elongation resulted as shown in Table 5. The ferrogel sample was varied based on the concentration of magnetite filler dispersed into the ferrogel.

Based on Table 5, the elongation level happening to the ferrogel sample was very small that was about 2 mm. This result was the same for all samples with the different concentration of filler. There was no significant difference of ferrogel elongation happening with the variation of magnetite

nanoparticles filler from 10 %wt to 20 %wt. This case was not suitable for the previous research stating that the magnetic response will increase by the increase in magnetic filler within the ferrogel [22]. This phenomenon was possibly caused by some factors. For examples are the very small size of magnetic filler (in the nanometer order), the less big concentration of filler, and the too narrow area of ferrogel response. The smaller the particle size, the remanent magnetization resulted was getting smaller as well so that it caused the decrease in magnetite response. This case will be different when the filler has a micrometer size; the ferrogel response will be faster and easier in the observation process of the elongation [45, 46]. Besides, the smaller the concentration of magnetic filler and the area of magnetic response, the deformation of ferrogel move will be weaker. We know that the area of ferrogel response for the elongation only had a diameter of about 6 mm.

Table 5. Result of Elongation Test of Ferrogel with Ferrofluid Percentage

Percentage of Filler (%)	Elongation (mm)
10	0.2
12.5	0.2
15	0.2
17.5	0.2
20	0.2

4. Conclusions

Magnetite nanoparticles have been successfully synthesized from the natural sand. The magnetite nanoparticles were enclosed with TMAH surfactant to prevent and reduce the aggregation of the particles. By the variation of TMAH surfactant, the ferrogel fabrication has been successfully conducted by using the freezing-thawing method. In the result of VSM, the concentration of TMAH surfactant was 1.2 mL, and it had better magnetization effectiveness compared to 0.8 mL. This result was also strengthened by the result of VSM data refinement showing that magnetic moment of ferrogel sample with the TMAH surfactant 1.2 mL had the higher number than 0.8 mL. Referring to the data of sample magnetization, all ferrogels behaved as superparamagnetic. Meanwhile, the magneto-elasticity characterization of ferrogel showed the stable pattern; there was no significant response change between the samples of which magnetite filler was different to the influence of external magnetic field.

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