

## Effect of ultrasonication time and peg content on the crystallite size and lattice parameter of polyethylene glycol (PEG)-coated Fe<sub>3</sub>O<sub>4</sub> nanoparticles synthesized by a ultrasound assiste co-precipitation process

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### Abstract

In this study, poly(ethylene glycol) (PEG)-coated Fe<sub>3</sub>O<sub>4</sub> nanoparticles have been synthesized by a ultrasound assisted co-precipitation method. The nanoparticles were characterized by XRD, FT-IR, FESEM and VSM techniques. The effect of ultrasonication time and content of PEG on the crystallite size and the lattice parameter of the nanoparticles was also studied using XRD. The crystalline size of bare Fe<sub>3</sub>O<sub>4</sub> and PEG-coated Fe<sub>3</sub>O<sub>4</sub> nanoparticles were about 8-40 nm. The VSM measurements at room temperature showed that the PEG-coated Fe<sub>3</sub>O<sub>4</sub> nanoparticles had superparamagnetic properties. These results indicated that ultrasonication time and PEG content greatly affect the characteristics of nanoparticles.

**Keywords.** PEG-coated Fe<sub>3</sub>O<sub>4</sub>, ultrasonication, crystallite, lattice parameter.

### 1. INTRODUCTION

Magnetite nanoparticles are one of the most important superparamagnetic materials due to their high saturation magnetization and low toxicity. Fe<sub>3</sub>O<sub>4</sub> nanoparticles can be synthesized in various sizes and morphologies and are applied in various fields such as healthcare, biology (drug delivery, magnetic resonance technology,...),<sup>[1,2,3]</sup> water treatment,<sup>[4]</sup> catalysis,<sup>[5]</sup> environmental engineering.<sup>[6,7]</sup> There are many methods to produce the nano Fe<sub>3</sub>O<sub>4</sub> materials such as coprecipitation,<sup>[8,9]</sup> solvothermal reduction,<sup>[10,11]</sup> polyol,<sup>[3]</sup> copolymer gels<sup>[12,13]</sup> ... However, Fe<sub>3</sub>O<sub>4</sub> nanoparticles is easily accumulative because of the strong bipolar interaction. In order to increase the dispersity and stability of the materials, the scientists modified the surface of materials by surfactants,<sup>[9,14,15]</sup> polymers<sup>[2,16]</sup> and inorganic compounds.<sup>[3,17]</sup> Poly(ethylene glycol) (PEG) is one of the hydrophilic, non-toxic polymers, which is importantly utilized to form the stable structures and to disperse the biological molecule of medicine, which is the vital characteristic biomedicine. For example, M. Anbarasu and his co-workers have

synthesized the nano-Fe<sub>3</sub>O<sub>4</sub> materials covered by PEG using co-precipitation method. The resulting nanoparticles had the medium size of 10-15 nm. This material has a high saturation magnetization with a potential application in biomedicine.<sup>[18]</sup> Anindita Mukhopadhyay and his co-workers have synthesized the nano-Fe<sub>3</sub>O<sub>4</sub> particles by green methods. This PEG-coated materials prevent the biomolecules from toxicity produced by nano-Fe<sub>3</sub>O<sub>4</sub>.<sup>[19]</sup>

In the sonochemical method, the reaction is assisted by ultrasonic waves. This method is also used to produce nano-Fe<sub>3</sub>O<sub>4</sub>.<sup>[3,20]</sup> The 20kHz ultrasonic waves go through liquid to produce interstices. After an appropriate time, the interstices reach limited condition and collapse to make strong local heating (~ 5000 K), high pressure (20 MPa) and rapid heating/cooling rate (~ 1010 K/s). This condition causes the heat and special heat and dispersal reaction.<sup>[21-22]</sup> Furthermore, the circulation and continuous oscillation of the interstices also causes the homogeneous concentration in liquid.<sup>[23]</sup> The reaction assisted by the sonochemical method, produce the smaller, purer and more dispersible nano-materials.<sup>[24]</sup>



In this research, we have synthesized PEG-coated  $\text{Fe}_3\text{O}_4$  nanoparticles using the coprecipitation-ultrasonication method. The effects of PEG concentration and ultrasonication time on the degree of crystalline, crystalline size and magnetic properties have been investigated.

## 2. EXPERIMENTAL

### 2.1. Materials

Ferric chloride hexahydrate ( $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ ), ferrous chloride tetrahydrate ( $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$ ) and ethanol were purchased from Merck. The PEG polymers (PEG-6000), and  $\text{NH}_3$  solution were obtained from Macklin-China, India. Deionized water was used for the preparation of all aqueous solutions.

### 2.2. Preparation of PEG-coated $\text{Fe}_3\text{O}_4$ nanoparticles

The  $\text{Fe}_3\text{O}_4$  nanoparticles and PEG-coated  $\text{Fe}_3\text{O}_4$  nanoparticles were prepared by mixing  $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$  and  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$  with ratio of 2:1 into 100 ml of deionized water. In following step, PEG (1; 3; 5 and 10 ml) was added to the solution and stirred for 10 minutes. Then, the reaction mixture was placed on a sonicator (Sonics & materials - VCX500; 500 w, 20 kHz), and 25-28 %  $\text{NH}_3$  solution was added drop wise using a syringe pump at flow rate of 50 ml/h.

These procedures resulted in the formation of black precipitates of  $\text{Fe}_3\text{O}_4$ -PEG1;  $\text{Fe}_3\text{O}_4$ -PEG3;  $\text{Fe}_3\text{O}_4$ -PEG5 and  $\text{Fe}_3\text{O}_4$ -PEG10 nanoparticles. The solid was washed with distilled water several times until the neutral pH. Before it was washed two times with ethanol. The product was dried in oven at a temperature of 65 °C.

### 2.3. Characterizations

X-ray diffraction patterns were recorded with a

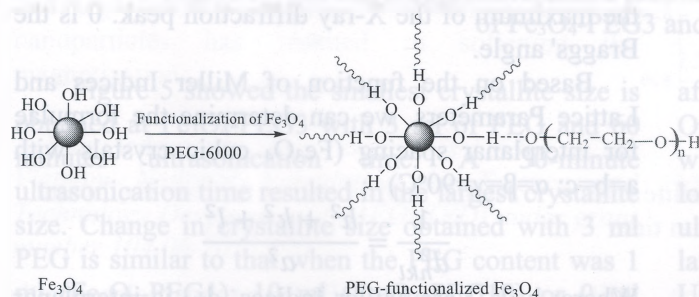


Figure 1:  $\text{Fe}_3\text{O}_4$ -PEG interaction mechanism

P'Pret Pro – PANalytical X-ray diffractometer operating at 1.8kW (40 mA/45KV) using  $\text{Cu K}\alpha$  ( $\lambda=1.5406 \text{ \AA}$ ) radiation. FT-IR spectra were recorded by KBr pellet method using a Bruker FT-IR spectrometer. FESEM were carried out using a Hitachi S-4800. The minimal amount of solid sample was dispersed in ethanol and small drops were placed on an aluminum grid. The grid was dried for 1-2 h in a vacuum over at 40 °C prior to the FESEM studies. Magnetic measurements of the solid samples were performed at room temperature (25 °C) using a Magnet B-10 Vibrating Sample Magnetometer (VSM).

## 3. RESULTS AND DISCUSSION

During the preparation of PEG-coated  $\text{Fe}_3\text{O}_4$  nanoparticles, the color of the reaction mixtures changed several times due to the influence of the compound phase formed. Ammonia solution, which was added into reaction solutions, produces  $\text{OH}^-$  anion to react with  $\text{Fe}^{2+}$  and  $\text{Fe}^{3+}$  cations to form  $\text{Fe}_3\text{O}_4$  nanoparticles. The addition of PEG to reaction mixtures resulted in coating  $\text{Fe}_3\text{O}_4$  with PEG. It happened since the interaction between O-H group of PEG compound and O-H group on the surface of  $\text{Fe}_3\text{O}_4$  nanoparticles, the H atom would be released and the C-H chain interacted with the  $\text{Fe}_3\text{O}_4$  surface [25]. Figure 1 describes  $\text{Fe}_3\text{O}_4$ -PEG interaction.

Figure 2 describes the powder X-ray diffraction (XRD) patterns of the bare  $\text{Fe}_3\text{O}_4$  and  $\text{Fe}_3\text{O}_4$ -PEG3 samples. The position and relative intensity of all diffraction peaks in all XRD patterns are fully matched. Also, all diffraction peaks were in a good agreement with the standard card of  $\text{Fe}_3\text{O}_4$  (00-072-2303). The characteristic peaks of  $\text{Fe}_3\text{O}_4$ , namely the peaks at  $2\theta$  of 30°; 35.4°; 57.2° and 62.6°, indicated that  $\text{Fe}_3\text{O}_4$  nanoparticles had a cubic spinel structure. [26]

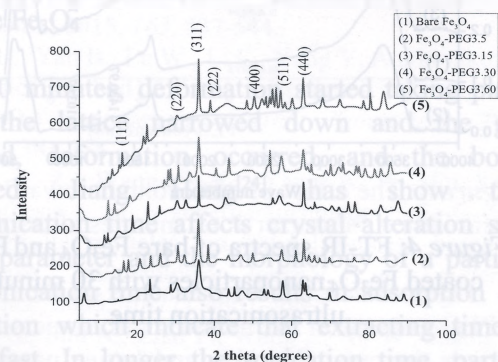


Figure 2: X-ray patterns of bare  $\text{Fe}_3\text{O}_4$  and  $\text{Fe}_3\text{O}_4$ -PEG3



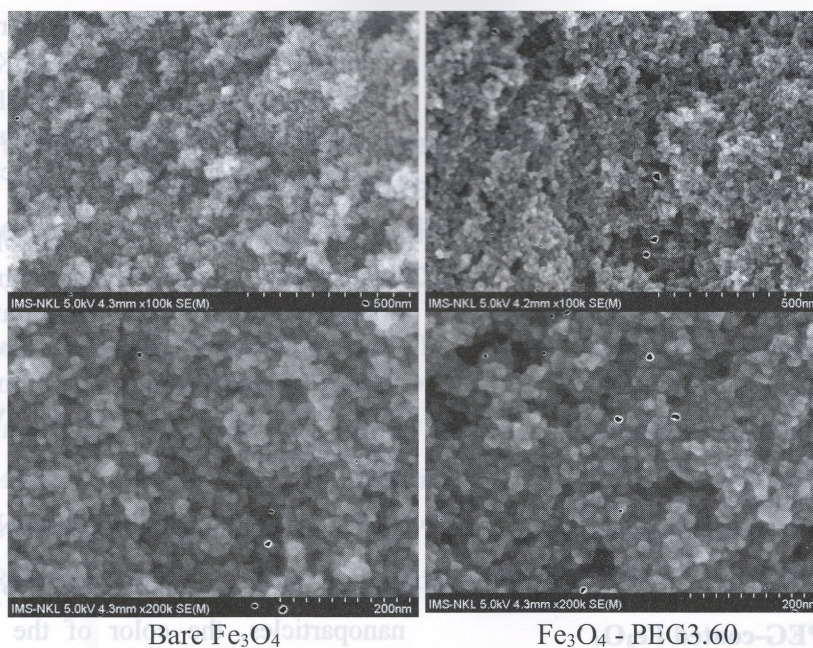


Figure 3: FESEM images of bare Fe<sub>3</sub>O<sub>4</sub> and PEG-coated Fe<sub>3</sub>O<sub>4</sub> nanoparticles

FESEM images (figure 3) showed that Fe<sub>3</sub>O<sub>4</sub> nanoparticles after surface modification with PEG have a softer surface and have a more uniform particle size distribution than the bare Fe<sub>3</sub>O<sub>4</sub> sample. Both bare Fe<sub>3</sub>O<sub>4</sub> and PEG-coated Fe<sub>3</sub>O<sub>4</sub> samples exhibit the sizes of particles in the range of 10-15 nm.

The attraction of resulting black precipitates, to a permanent magnet proves the formation of Fe<sub>3</sub>O<sub>4</sub> crystals. The products were further characterized by FT-IR; XRD and VSM methods.

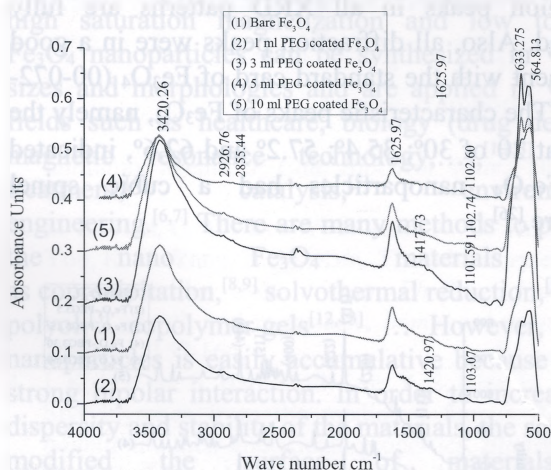


Figure 4: FT-IR spectra of bare Fe<sub>3</sub>O<sub>4</sub> and PEG-coated Fe<sub>3</sub>O<sub>4</sub> nanoparticles with 30 minutes ultrasonication time

Figure 4 shows the FT-IR spectra of bare Fe<sub>3</sub>O<sub>4</sub> and PEG-coated Fe<sub>3</sub>O<sub>4</sub> nanoparticles (Fe<sub>3</sub>O<sub>4</sub>-PEG1;

Fe<sub>3</sub>O<sub>4</sub>-PEG3; Fe<sub>3</sub>O<sub>4</sub>-PEG5; Fe<sub>3</sub>O<sub>4</sub>-PEG10) with 30 minutes ultrasonication time. In all spectra, there are two obvious bands around 3420 cm<sup>-1</sup> and 564 cm<sup>-1</sup>. The former was attributed to the O-H stretching and H-O-H bending.<sup>[27]</sup> The fingerprint range of 564 cm<sup>-1</sup> showed sharp absorption, indicating the presence of Fe-O bond in bare Fe<sub>3</sub>O<sub>4</sub> and Fe<sub>3</sub>O<sub>4</sub>-PEG samples,<sup>[28]</sup> The Fe-O-C bond interaction identified by absorption around 1100 cm<sup>-1</sup> indicates that Fe<sub>3</sub>O<sub>4</sub> has been coated with PEG.<sup>[29]</sup>

The effect of ultrasonication time on the crystallite size and lattice parameter is introduced in table 1 and figure 5.

Debye-Scherrer equation for calculating particle size:

$$d = \frac{k\lambda}{\beta \cos\theta}$$

Where: d is the mean size of crystallites (nm). k is crystallite shape factor a good approximation is 0.89. λ is the X-ray wavelength. β is the full width at half the maximum of the X-ray diffraction peak. θ is the Bragg's angle.

Based on the function of Miller Indices and Lattice Parameters, we can determine the formulae for interplanar spacing (Fe<sub>3</sub>O<sub>4</sub> cubic crystals with a=b=c; α=β=γ=90°C)

$$\frac{1}{d_{hkl}^2} = \frac{h^2 + k^2 + l^2}{a^2}$$

Where: h, k, l are Miller Indices. d<sub>hkl</sub> is interplanar spacing. a is Lattice Parameters.

Table 1 shows the changes in crystallite size and lattice parameters. The similarities between resulting patterns make them easier to observe. The lattice



parameters with crystallite size were inversely proportional to the longer duration of ultrasonic usage time in nanoparticles synthesis.

Table 1: Effect of ultrasonication time and PEG content toward crystallite size and lattice parameter of Fe<sub>3</sub>O<sub>4</sub>-PEG nanoparticles

Samples	Time (minute)	Size (nm)	Lattice parameter (Å)
bare Fe <sub>3</sub> O <sub>4</sub>	5	16.7802	8.3222
	15	20.9789	8.3081
	30	27.9666	8.2918
	60	9.3259	8.3232
Fe <sub>3</sub> O <sub>4</sub> -PEG1	5	16.7802	8.3179
	15	20.9764	8.3317
	30	41.9455	8.3466
	60	16.8292	8.3443
Fe <sub>3</sub> O <sub>4</sub> -PEG3	5	13.9893	8.2893
	15	20.9898	8.2673
	30	41.9695	8.2863
	60	8.3933	8.2914
Fe <sub>3</sub> O <sub>4</sub> -PEG5	5	41.9818	8.2632
	15	41.9111	8.3985
	30	27.9813	8.3454
	60	27.9813	8.3164
Fe <sub>3</sub> O <sub>4</sub> -PEG10	5	13.9905	8.2822
	15	20.9868	8.2783
	30	20.9765	8.3172
	60	20.9755	8.3214

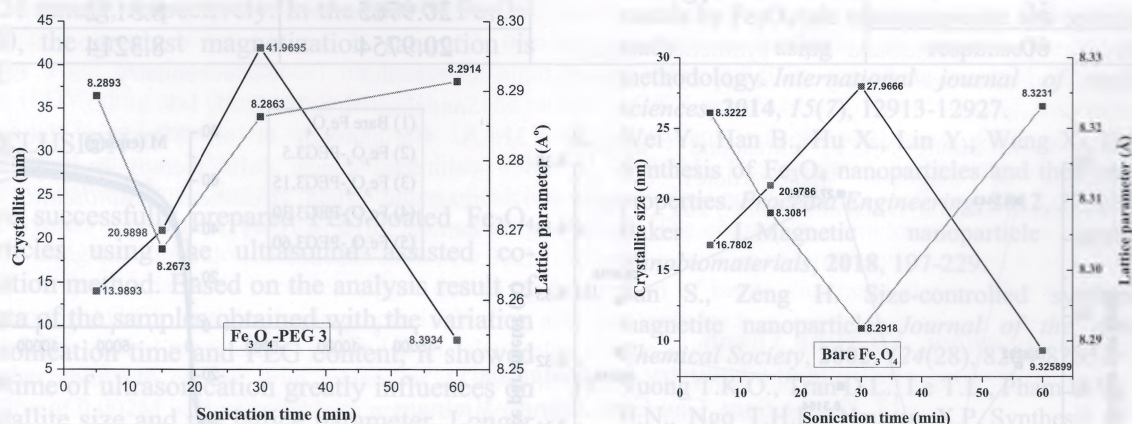


Figure 5: The effect of ultrasonication time toward crystallite size, lattice parameter of Fe<sub>3</sub>O<sub>4</sub>-PEG3 and bare Fe<sub>3</sub>O<sub>4</sub>

Figure 5 showed the smallest crystallite size is obtained at Fe<sub>3</sub>O<sub>4</sub>-PEG3 with 3 ml of PEG and 60 minutes ultrasonication time. A 30-minute ultrasonication time resulted in the largest crystallite size. Change in crystallite size obtained with 3 ml PEG is similar to that when the PEG content was 1 ml (Fe<sub>3</sub>O<sub>4</sub>-PEG1); 10 ml (Fe<sub>3</sub>O<sub>4</sub>-PEG10) or 0 ml (bare Fe<sub>3</sub>O<sub>4</sub>). This may be due to during 5-30 minutes of ultrasonication, the nucleation of Fe<sub>3</sub>O<sub>4</sub> crystals took place and followed by their growth and

after 30 minutes, deformation started taking place. Once the lattice narrowed down and the grid widened, deformation occurred, and the bonds loosened. Jiang Hong<sup>[24]</sup> has show that, ultrasonication time affects crystal alteration size, lattice parameter and the morphology of a particle. Ultrasonication time also results in adsorption and desorption which indicate that extracting time is really fast. In longer the sonication time, particle size tends to be more homogeneous and shrink



eventually leading to a stable nanoparticle size and less agglomeration. It occurs because shock wave in sonication method can separate.<sup>[30]</sup> Figure 5 shows that the lattice parameters of the Fe<sub>3</sub>O<sub>4</sub>-PEG3 nanoparticles with ultrasonication time of 5; 15; 30 and 60 minutes were 8.2892 Å; 8.2673 Å; 8.2862 Å and 8.2913 Å, respectively. The lattice parameters of the Fe<sub>3</sub>O<sub>4</sub> nanoparticles with ultrasonication time of 5; 15; 30 and 60 minutes were 8.3222 Å; 8.3081 Å; 8.2918 Å and 8.3231 Å, respectively. The smallest

crystallite size of bare nanoparticles was obtained at 60 minutes ultrasonication time. It indicated that Fe<sub>3</sub>O<sub>4</sub> sonication process has taken place from the start. However, deformation took place causing larger and longer lattice parameter as well as smaller crystallite size.

Table 2 also shows the effect of PEG content on the crystallite size and lattice parameters. With higher PEG content, crystals are more uniform and less affected by ultrasound time.

Table 2: The effect of PEG content toward crystallite size and lattice parameter of Fe<sub>3</sub>O<sub>4</sub>-PEG nanoparticles

Sonication time (min)	PEG content (ml)	Size (nm)	Lattice parameter (Å)
5	1	16.7802	8.3178
15		20.9764	8.3216
30		41.9455	8.3466
60		16.8292	8.3442
5	3	13.9893	8.2893
15		20.9898	8.2673
30		41.9695	8.2863
60		8.3933	8.2914
5	5	41.9818	8.2632
15		41.9110	8.3985
30		27.9813	8.3454
60		27.9813	8.3164
5	10	13.9905	8.2822
15		20.9868	8.2783
30		20.9765	8.3172
60		20.9754	8.3214

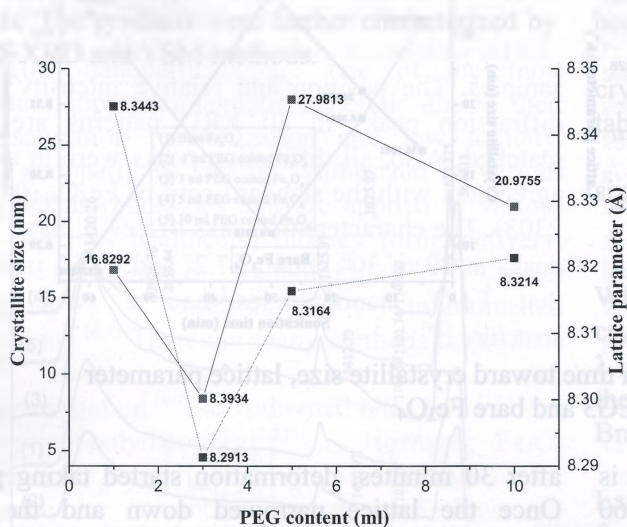


Figure 6: The effect of PEG content toward crystallite size and lattice parameter at 60 minutes ultrasonication time

Figure 6 described the analysis on the influence of PEG content at 60 minutes ultrasonication time towards the crystallite size and lattice parameter in Fe<sub>3</sub>O<sub>4</sub>-PEG nanoparticle formation. The addition of

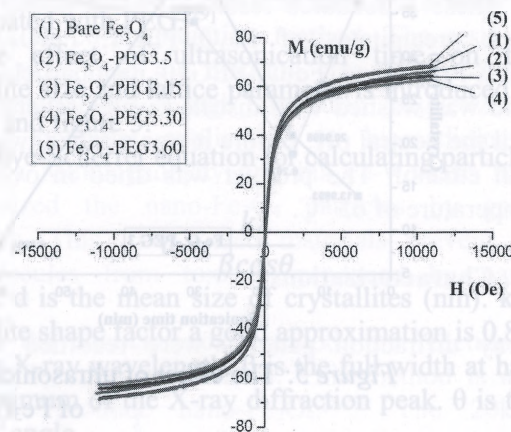


Figure 7: M-H curves for bare Fe<sub>3</sub>O<sub>4</sub> and Fe<sub>3</sub>O<sub>4</sub>-PEG3

PEG on the surface of Fe<sub>3</sub>O<sub>4</sub> nanoparticles greatly affected the lattice parameters of the nanoparticles. Larger lattice parameters indicated wider gap between Fe and O atoms. Based on the findings, it



may be concluded that there was correlation between the crystallite size and lattice parameters of the Fe<sub>3</sub>O<sub>4</sub>-PEG nanoparticles. In this case, the size of nanoparticle was inversely proportional to the lattice parameter. The smaller the crystallite size, the bigger the lattice parameter. However, the higher the PEG content was, the smaller the crystallite size would be. For instance, the 10 ml PEG resulted in smaller crystal compared to the 5 ml PEG. In 10 ml PEG, smaller crystallite size would have larger lattice parameter. A. Hardiansyah et al.<sup>[31]</sup> has show that, when using the PEG content is too large, the magnetization saturation is reduced, this is because of the thickness of the polymer cover and the formation of Fe-O-C bonding layer on the surface, which are the causes of reduction of magnetization of Fe<sub>3</sub>O<sub>4</sub> nanoparticles.

Figure 7 described the M-H curves for Fe<sub>3</sub>O<sub>4</sub>-PEG3 and bare Fe<sub>3</sub>O<sub>4</sub> samples. The saturation magnetization (M<sub>s</sub>) was obtained by extrapolation to infinite field of the experimental data obtained in the high field range where the magnetization varies linearly with the inverse of the applied field.

The results showed that the saturation magnetization of bare Fe<sub>3</sub>O<sub>4</sub> nanoparticles reached 67.77 emu/g. When using PEG as dispersant, the saturation magnetization of Fe<sub>3</sub>O<sub>4</sub>-PEG3.5; Fe<sub>3</sub>O<sub>4</sub>-PEG3.15; Fe<sub>3</sub>O<sub>4</sub>-PEG3.30 were 65.71; 64.46; 62.63 and 68.21 emu/g, respectively. In the case of Fe<sub>3</sub>O<sub>4</sub>-PEG3.60, the greatest magnetization saturation is reached.

#### 4. CONCLUSION

We have successfully prepared PEG-coated Fe<sub>3</sub>O<sub>4</sub> nanoparticles using the ultrasound assisted coprecipitation method. Based on the analysis result of XRD data of the samples obtained with the variation of ultrasonication time and PEG content, it showed that the time of ultrasonication greatly influences on the crystallite size and the lattice parameter. Longer ultrasonication time results in smaller crystallite size and larger lattice parameter. The modified surface of nanoparticles has resulted in softening the magnetization.

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