The impact of synthesis temperature on magnetite nanoparticles size synthesized by co-precipitation method

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Abstract. Ferrite nanoparticles synthesized by co-precipitation method depend mostly on parameters such as synthesis temperature, pH of the suspension, and initial molar concentration. We reported the size and morphology of nanoparticles magnetite (Fe3O4) synthesized by co-precipitation method with variation of temperature in the range between 25°C and 80°C in order to investigate the effect of synthesis temperature to its size and morphology. The nanoparticle forms are spherical, with band-gap values were 1.76 eV, 1.20 eV, 1.27 eV and 1.14 eV at synthesis temperatures of 25°C, 40°C, 60°C, and 80°C, respectively. It was found that the smallest of magnetite nanoparticles was 10.14 nm for the sample synthesized at 25°C.

1. Introduction
Research on magnetic fluid nanoparticles has attracted many interests due to the increasing valuable characteristics of nanoparticles compared with that of bulk characteristics. This characteristic difference is due to the ratio between the larger surface area and volume of nanoparticles causing more reactive and direct contact with the surface of material [1,2]. In addition, the effect of decreasing particle size or increasing surface area per unit volume improved the optical characteristics of nanoparticles as well as increased absorption in the ultraviolet (blue shift) region, and band gap values [3].

Synthesis of magnetic nanoparticles in solution phase can be performed by sol-gel, hydrothermal method and co-precipitation [4,5]. Synthesis of Fe3O4 nanoparticles by co-precipitation method depends on several parameters such as synthesis temperature, pH and initial molar concentration. Some previous result in synthesize magnetite nanoparticles with temperature variations can be seen in Table 1.

In this paper we reported the synthesis of Fe3O4 nanoparticles by co-precipitation method at pH = 10 [6] and synthesis temperature variations in the range of 25°C, 40°C, 60°C, and 80°C in order to investigate the effect of synthesis temperature on band-gap, morphology and size of Fe3O4 nanoparticles.

Table 1. Results of Fe3O4 synthesis by co-precipitation method [7-12]

| Source of Fe2⁺  | Source of Fe3⁺  | Solvent   | Temperature       | Size of Particles (nm) |
|----------------|----------------|-----------|-------------------|------------------------|
| FeCl₂          | FeCl₃          | NaOH      | RT and 80°C       | 6 and 12               |
| FeCl₂          | FeCl₃          | NH₄OH     | RT                | 12                     |
| FeCl₂          | FeCl₃          | NH₄OH     | 80°C              | 3-15                   |
| FeCl₂          | -              | NaOH      | 88°C              | 7                      |
2. Methods

The research involved two parts namely the synthesis of magnetic nanoparticles using co-precipitation method and magnetic nanoparticle characterization such as UV-Vis, TEM and Particle size Analyzer (PSA). The starting materials were FeCl3.6H2O and FeCl2.4H2O, DI-Water, ethanol, n-hexane, NH3.H2O (25%). The synthesis of magnetic nanoparticles started with the preparation of a precursor solution of FeCl3.6H2O (5.41 gram) and FeCl2.4H2O (1.99 gram) dissolved in 100 mL Di-water, then added precipitant NH3.H2O (25%) drops by drops until it reaches pH = 10. The stable solution at pH = 10 is stir red on magnetic stirrer with different temperature of 25°C, 40°C, 60°C, and 80°C for 60 minutes. To obtain good quality nanoparticles, all samples were washed with n-hexane and centrifuged for 10 minutes at 7000 rpm. This process was carried out two times following by dispersing in ethanol solution. The washed solution was then characterized by UV-Vis, TEM and PSA.

3. Results and Discussion

The UV-Vis characterization was performed in the wavelength range of 200-900 nm. The result of UV-Vis spectrum of nanoparticles synthesized by co-precipitation method at pH = 10 and synthesis temperature of 25°C, 40°C, 60°C, and 80°C can be seen in Figure 1.

![UV-Vis absorption spectrum and Tauc-plot](image)

**Figure 1.** UV-Vis absorption spectrum (a) and Tauc-plot (b) magnetic fluid nanoparticles synthesized by co-precipitation method at synthesis temperature 25°C, 40°C, 60°C, and 80°C.

The value of band-gap is determined by the Tauc-plot curve of $a\nu$ value on $h\nu$ value, where $a$ is the absorbance coefficient obtained from the UV-Vis measurement, $h$ is Planck's constant (4.14 × 10-15eV), $\nu$ is the wave frequency (Hz) and $h\nu$ is the photon energy (eV). Based on the Tauc-plot calculation, the band-gap values of Fe3O4 nanoparticles tabulated in Table 2.

| Sample | Temperature (°C) | Band-gap (eV) |
|--------|-----------------|---------------|
| A2     | 25              | 1.76          |
| B2     | 40              | 1.20          |
| C2     | 60              | 1.27          |
| D2     | 80              | 1.14          |

Table 2. The band-gap value of Fe3O4 nanoparticles synthesized by co-precipitation method at pH = 10 and synthesis temperature of 25°C, 40°C, 60°C, and 80°C.
From Table 2, it can be seen that the value of band-gap is in the range of 1.14 eV – 1.76 eV which is indicated as semiconductors band-gap.

![Figure 2](image)

**Figure 2.** Photograph of TEM of Fe₃O₄ magnetic nanoparticles with 20 nm magnification scale on synthesis temperature of 25°C (a), 40°C (b), 60°C (c), and 80°C (d).

The result of TEM measurement of Fe₃O₄ nanoparticles is shown in Figure 2. It can be seen that the form of Fe₃O₄ nanoparticles was generally spherical. The average particle size calculated from TEM measurement was 10.14 nm, 10.32 nm, 10.95 nm and 11.66 nm for synthesis temperature of 25°C, 40°C, 60°C, and 80°C, respectively as shown in Table 3. It is found that the average size of Fe₃O₄ nanoparticles increases with the synthesis temperature due to the acceleration of chemical reaction of Fe²⁺ and Fe³⁺ ions. The smallest of magnetite nanoparticles was 10.14 nm for the sample synthesized at 25°C.

**Table 3.** Average size of Fe₃O₄ nanoparticles resulting from TEM photographs.

| Particle | 25 (°C) | 40 (°C) | 60 (°C) | 80 (°C) |
|----------|---------|---------|---------|---------|
| 1        | 10.15   | 11.84   | 12.53   | 13.13   |
| 2        | 10.64   | 9.05    | 11.14   | 11.94   |
| 3        | 9.85    | 9.75    | 10.15   | 10.25   |
| 4        | 9.95    | 10.64   | 9.95    | 11.34   |
| Average (nm) | 10.14±0.24 | 10.32±0.92 | 10.95±0.89 | 11.66±0.87 |

From the PSA measurement, information about the particles size in the percentage distribution was obtained as shown in Table 4. The particle size distribution is 41nm - 63 nm, 441 nm - 2115 nm, 76 nm - 2144 nm and 66 nm - 2290 nm at synthesis temperature 25°C, 40°C, 60°C, and 80°C, respectively.

**Table 4.** Distribution percentage size of Fe₃O₄ nanoparticles synthesized at pH = 10 and synthesis temperature 25°C, 40°C, 60°C, and 80°C.

| Temperature (°C) | < 10 % | < 25 % | < 50 % | < 75 % | < 90 % |
|-----------------|--------|--------|--------|--------|--------|
| 25              | 41     | 46     | 52     | 58     | 63     |
| 40              | 441    | 542    | 1251   | 1824   | 2115   |
| 60              | 76     | 450    | 547    | 719    | 2144   |
| 80              | 66     | 223    | 331    | 490    | 2290   |

4. Conclusion
The magnetite Fe₃O₄ nanoparticles have been successfully synthesized by co-precipitation methods at the synthesis temperature of 25°C, 40°C, 60°C, and 80°C. From UV-Vis measurement, it is found that the band gap of nanoparticles magnetite were 1.76 eV, 1.20 eV, 1.27 eV and 1.14 eV at temperature
synthesis 25°C, 40°C, 60°C, and 80°C, respectively. From TEM measurement, it was observed that morphology of Fe3O4 nanoparticles was spherical form, with the average size of (10.14±0.24) nm, (10.32±0.92) nm, (10.95±0.89) nm and (11.66±0.87) nm at temperature synthesis of 25°C, 40°C, 60°C, and 80°C, respectively. From TEM measurement, it was also observed that the Fe3O4 nanoparticles distributed in the range size of (41-63) nm, (441-2115) nm, (76-2114) nm and (66-2290) nm at temperature synthesis of 25°C, 40°C, 60°C, and 80°C, respectively. It is concluded that the synthesis temperature can be able to control the size of magnetite nanoparticles. The average size of Fe3O4 nanoparticles increases with the synthesis temperature due to the acceleration of chemical reaction of Fe2+ and Fe3+ ions.

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