Physical and Magnetic Properties Comparison of Cobalt Ferrite Nanopowder Using Sol-gel and Sonochemical Methods

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1. INTRODUCTION

In the recent years, the fast development of nanomaterial and its application depends on synthesis methods for desired properties than the base material at nanoscale compared to macro scale [1]. Industry always needs a cheap but high-quality material, and thus, research continues and progresses to produce advanced material or nanomaterial. The example is nanomaterial synthesis from the metal-oxide type that quickly develops due to its significant ability in its application [2].

Nowadays, researchers pay attention to nanomaterial ferrite because of its wide application such as in biotechnology, for catalyst, ferrofluid, and magnetic material [3]. Ferrite, as an excellent magnetic material, attracts scientists and is continuously developed. Apart from ferrite, cobalt also has some particular property as magnetic material, for example, medium magnetization saturation, strong anisotropy, high coercivity value at room temperature, stable chemical property and high curie temperature [2]. Hence, when combined into cobalt ferrite (CoFe2O4), it has a unique magnetic property and

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is applied as supercapacitor and anode in Li-ion battery [4]. Cobalt Ferrite can also be used in medical applications because of the magnetic properties. Hyperthermia is one of a modality cancer treatments at elevated temperature between 41 °C and 45 °C and treatment time of at least 30 minutes has been paid considerable attention due to its clinical efficacies, such as minimizing clinical side effects and can selectively destroy a localized or a deeply seated cancer tumor by heating with magnetic field [5]. Well-dispersed cobalt nanoparticles in the hyperthermia application give effective and controlled heat generation [6].

Cobalt ferrite (CoFe₂O₄) has unique physical and mechanical properties and widely applied in nanomedicine [6], supercapacitor and anode in Li-ion battery [4]. Cobalt ferrite is a hard magnetic material with high Curie temperature (520°C), high coercivity of around 4.3 kOe at room temperature for single domain with the size of 40 nm, medium saturation magnetization of 80 emu/g for bulk material at room temperature, high anisotropy constant (2.65 x 10⁶ erg cm⁻³) and high magnetostrictive (-225 x 10⁻⁶). Besides, cobalt ferrite shows excellent chemical stability, mechanical hardness, wear resistance, easy to synthesis, and electrical isolation. The above properties make cobalt ferrite as one of the promising candidates in various applications.

The magnetic property of CoFe₂O₄ depends on the grain size and cation distribution in the form of tetrahedral and octahedral lattice [7]. Several synthesis methods to produce CoFe₂O₄ nanomaterial in a right nanometric scale are sol-gel and sonochemical [2, 8, 9]. To date the researcher still find the best synthesis methods to obtain single phase CoFe₂O₄ resulted in the best magnetic properties. This research surprisingly describe how CoFe₂O₄ magnetic properties changing from hard magnetic to soft magnetic material as the sintering temperatures varied and also study the comparison of two synthesis methods which are sol-gel and sonochemical in term of physical and magnetic properties.

2. MATERIALS AND METHODS

2.1. Material Preparation Raw material for cobalt ferrite is Cobalt(II) nitrate hexahydrate (Co(NO₃)₂·6H₂O with 99% purity, Iron(III) nitrate nonahydrate (Fe(NO₃)₃·9H₂O with 99% purity that were purchased from Merck and ethylene glycol and was stirred for 2 hours with rotation speed of 200 rpm. Then, the solution was stirred and heated at 80°C with a rotation speed of 200 rpm until it turned to gel. Gel formation occurred in about 16.5 hours. After that, the sample was dried in the microwave oven for 7 hours at 100°C. After drying process, the sample was grinded by agate mortar for 1 hour.

2.3. The Sonochemical Method The base material in this research were 2.951 g Cobalt(II) nitrate hexahydrate (Co(NO₃)₂·6H₂O with 99% purity, 8.08 g Iron(III) nitrate nonahydrate (Fe(NO₃)₃·9H₂O with 99% purity that were bought from Merck and dissolved into 100 ml DI Water. The mixture was stirred for 1 hours with rotation speed of 200 rpm. Then, the solution was stirred and heated at 80°C with the rotation speed of 200 rpm until it turned to gel. After, the sample was sonicated for 30 minutes and titrated with 100 ml sodium hydroxide 10 M, washed with 750 ml distilled water for three times and dried in the microwave oven at 100°C. The sample was then grinding by agate mortar for 1 hour.

2.4. Characterization The powder from the sol-gel and sonochemical synthesis methods each had yellowish-brown and darkish black colour. The cobalt ferrite from two methods then characterized to find the phase identification and crystallite size using the X-Ray Diffractometer (XRD) PAN Analytical Cu Ka (λ = 1.54 Å), morphology of CoFe₂O₄ obtained from Scanning Electron Microscopy of Phenom, the functional group was observed using Fourier Transform Infrared (Shimadzu) and the magnetic properties were obtained from Vibrating Sample Magnetometer of OXFORD 1.2H at room temperature.

3. RESULTS AND DISCUSSION

3.1. Phase Characterization The polycrystalline material properties depend on the crystal size and this fact

![Figure 1](image-url)
triggered several decades of research to find the nanomaterial with the best crystal size. Therefore, it is essential to measure the crystallite size of the polycrystalline material accurately. The crystallite size was calculated using the Scherrer equation \[ D = \frac{k \lambda}{FWHM \times \cos \theta} \] (1)

Crystallinity percentage in the samples were taken into consideration because crystallinity influences the phase and physical property. Crystallinity percentage or Segal CI (Crystallinity Index) was found by Segal et al. \[12\] and formulated as peak crystalline area divided by the amorph area as shown below \[13\]:

\[ CI = \frac{I_t - I_a}{I_t} \times 100\% \] (2)

Or,

\[ CI = \frac{A_{cr}}{A} \times 100\% \] (3)

Figure 1 shows the significant crystallinity difference between both methods. As-sintered CoFe₂O₄ displays better crystallinity chart whereas the unsintered sample has lower crystallinity. Unsintered CoFe₂O₄ crystallinity percentage was 44.17% while the sintered samples had around 57% crystallinity. Although the sintered samples had different temperatures, but the percentage did not significantly change since at 500°C temperature. Figure 2 presents the calculation using Equation 3 above and calculated using OriginPro software. There are peaks on Figure 1: 220, 311, 400, and 511, that were the phase identity of CoFe₂O₄ \[14\]. The same peak of 311 from sintered samples showed that the crystallinity was single phase \[12\].

Table 1 shows the crystal size and crystallinity index of CoFe₂O₄ that were synthesized using unsintered sonochemical and sintering sol-gel for 2 hours and holding temperature variations (500°C, 800°C, and 1100°C). The smallest crystal size was found in the CoFe₂O₄ sample sintered at 500°C with the value of 13.25 nm. The most significant crystallinity percentage was found in the CoFe₂O₄ sample sintered at 1100°C, with a value of 57.71%.

Based on Figures 3(a) and (b), an increase in the temperature of sintering causing grown to the crystallite size material until 70.64 nm at sintering temperature 1100, but it did not happen with the crystallinity in the range of 500 to 1100°C sample. The crystallinity of the sample was saturated for 57% from 500°C until 1100°C but increasing the crystallinity unsintered sample which was 44.17% crystallinity to 57.04%.

### Table 1. Intensity, FWHM, d-spacing, Crystallite Size of CoFe₂O₄ and Cristalinity

| Sample Material                      | X-Ray Diffraction Corresponding to [311] peak | Crystallinity (%) |
|--------------------------------------|---------------------------------------------|-------------------|
|                                      | Intensity (counts) | FWHM (rad) | d-spacing (Å) | Crystallite Size (nm) |                      |
| CoFe₂O₄ Sonochemical                 | 24.74            | 0.0034    | 2.35897      | 42.71                 | 44.17               |
| CoFe₂O₄ Sol-Gel Sintering 500°C      | 69.05            | 0.0101    | 2.51587      | 13.25                 | 57.04               |
| CoFe₂O₄ Sol-Gel Sintering 800°C      | 226.88           | 0.0024    | 2.52141      | 60.5                  | 57.67               |
| CoFe₂O₄ Sol-Gel Sintering 1100°C     | 428.55           | 0.0021    | 2.52602      | 70.64                 | 57.71               |

### 3.2. Morphological Characterization

The observation and comparison of morphology form were conducted using SEM. This analysis aimed to find the morphological change of CoFe₂O₄ from different synthesis method and different sintering temperature.
Figure 3. Comparison Effect of Sintering CoFe₂O₄ Samples, (a) Sintering Temperature and Crystallite Size, (b) Sintering Temperature and Crystallinity

Figure 4(a) displays that unsintered sonochemical CoFe₂O₄ has morphological magnification of 25k. The grains are not homogenous but agglomerated and covered by cylindrical shape that extends into taper in various sizes, from the smallest of 34.63 nm up to the largest of 42.48 nm. Figure 4(b) presents the sintered CoFe₂O₄ at 500°C with 10k magnification, and the easiest spotted grain sizes are 65.94 nm up to 74.92 nm. Meanwhile, Figure 4(c) shows the sintered CoFe₂O₄ at 800°C with 10k magnification. The observed grains are between 59.79 nm until 73.72 nm. Figure 4(d) displays the sintered CoFe₂O₄ at 1100°C with 10k magnification. The grain sizes start from 50.98 nm up to 56.98 nm.

The SEM image analysis present that agglomeration in all samples was still high as observed the decreasing agglomeration from the sintering at 500°–1100°C due to the crystalline development in the sintering process [15].

The crystalline growth caused the grain size in SEM morphology to be small. The sintered CoFe₂O₄ at 500°C had crystal size of 13.25 nm and SEM results around 70 nm, the crystal size continued to grow up to 70.64 nm with the grain size from SEM results was 52 nm for sintered CoFe₂O₄ at 1100°C. The sonochemical CoFe₂O₄ had a crystal size of 42.71 nm, and the SEM grain results were around 27 nm. However, it had the lowest crystallinity percentage than the other three samples with a value of 44.17%. Sintering causes crystal size to grow and lower the agglomeration, as shown in Figure 4(d) where the agglomeration was reduced compared to other samples. High agglomeration was a possibility caused by Van der Waals forces that weaken the bonds between particle [16]. In general, the SEM grain was spherical, but some was elongated.

### 3.3 FTIR Analysis

The FTIR spectrum was used to identify the chemical compound structure and found the spinel phase formation of the synthesized CoFe₂O₄ [16].

Figure 4. Morphology of CoFe₂O₄, (a) Sonochemical without Sintering, (b) Sol-gel 500°C Sintering, (c) Sol-gel 800°C Sintering, (d) Sol-gel 1100°C Sintering
Figure 5 shows the comparison of the FTIR spectrum from four samples with three main zones analysis. There are several peaks based on various treatments in the first zone, 636 cm\(^{-1}\) for sonochemical method, sol-gel 500°C sintering 601 cm\(^{-1}\), sol-gel 800°C sintering 619 cm\(^{-1}\) and sol-gel 1100°C sintering 623 cm\(^{-1}\). The first zone was indicated the area stretching vibration from the metal-oxygen (M–O, where M = Co or Fe) that showed identity and fingerprint zone from the CoFe\(_2\)O\(_4\) absorption band [17].

The difference in the absorption position in the first zone that was the fingerprint of CoFe\(_2\)O\(_4\) due to the crystal size are varied. The varied crystal size will make difference distance between Fe\(^{3+}\)-O\(^{2-}\) then the absorption will slightly difference between the samples. The second zone compound several peaks based on the samples, there are 1456 cm\(^{-1}\) for sonochemical method, sol-gel 500°C sintering 1531 cm\(^{-1}\), sol-gel 800°C sintering 1427 cm\(^{-1}\), and sol-gel 1100°C sintering 1409 cm\(^{-1}\).

The second zone indicated bending vibration and stretching vibration from the hydroxyl (–OH) functional group as one of the characteristics of water absorption bands [18] the adsorbed water is featured by the bands at 1409-1531 cm\(^{-1}\) assigned to the \(\delta\) H-O-H bonding mode [19]. From the second zone, we can imply that water molecules does not get totally remove from the sample although there are several heat treatments applied to the samples.

The third zone had several differences, but for the sonochemical CoFe\(_2\)O\(_4\) peak 3423 cm\(^{-1}\) and 3412 cm\(^{-1}\) could not be observed as well as the spectrum of the sintered sample at 1100°C. The both peaks have been assigned to antisymmetric and symmetric O-H stretching vibration of lattice water. Thus, this situation followed the sintering process that omits water content because the sample was sintered at 1100°C.

Meanwhile the sonochemical sample lost the peak due to the long duration of drying. The second zone peak’s appeared on all synthesised samples because this is the water molecule identity. This peak experienced intensity decrease along with the increasing sintering temperature that proved the sintering temperature reduces water molecule as well as water absorption by other molecules and thus make the intensity to decrease [19]. Also different methods proved that sol-gel samples had higher water content as seen from the peak intensity of the second zone and the third zone because the synthesis process form sol then followed by liquid compaction into a gel that will not be separated easily from the content of water molecules in it.

\[
R = \frac{M_r}{M_s}
\]

### 3.4 Magnetic Properties Analysis

The magnetic property of CoFe\(_2\)O\(_4\) that was synthesized was obtained from the VSM test such as magnetic saturation \((M_s)\), magnetic remanence \((M_r)\), and coercivity \((H_c)\) [20]. \(M_s\) is the maximum value of magnetization that is received from the given magnetic field. The value of \(M_s\) can be taken from the intersection of curves that intersect with the vertical axis. \(H_c\) is the magnetic field intensity needed to make the magnetic sample reach zero magnetization after achieving magnetization saturation. Remanence ratio is the ability of magnetic material to change the curve in the hysteresis loop curve after the magnetic field is removed [21]. The value of parameter above was described in Table 2. Remanence ratio can be formulated [22].

![Figure 5. FTIR Spectra Analysis of CoFe\(_2\)O\(_4\) with Different Sintering Temperatures and Methods](image)

| Sample Material                  | Magnetic Saturation/M\(_s\) (emu/g) | Magnetic Remanence/M\(_r\) (emu/g) | Remanence Ratio R | Coercivity/H\(_c\) T | Coercivity/H\(_c\) Oe |
|----------------------------------|-------------------------------------|------------------------------------|-------------------|---------------------|---------------------|
| CoFe\(_2\)O\(_4\) Sonochemical   | 1.29                                | 0.06                               | 0.046             | 0.0012              | 12                  |
| CoFe\(_2\)O\(_4\) Sol-Gel Sintering 500°C | 89.02                              | 41.83                              | 0.469             | 0.1032              | 1032                |
| CoFe\(_2\)O\(_4\) Sol-Gel Sintering 800°C | 113.51                             | 23.4                               | 0.206             | 0.472               | 472                 |
| CoFe\(_2\)O\(_4\) Sol-Gel Sintering 1100°C | 4.18                               | 0.17                               | 0.040             | 0.0066              | 66                  |
Figure 6 shows the comparison of the hysteresis loop curve from the synthesized CoFe₂O₄. The hysteresis loop curve of the sintered sample at 500°C indicated hard magnetic material, sintered sample at 800°C indicated soft magnetic material, while the sintered sample at 1100°C and sonochemical sample indicated paramagnetic material [23]. The change from hard magnetic into soft magnetic occurred in 500°C to 800°C sintering temperature. Changes in the curve are thought to be due to the sintering temperature which has exceeded its temperature (520°C) [7] thereby reducing $M_s$ and $H_c$; the change was due to the transition from a single domain to multi-domain [14]. The transition change was caused by the critical diameter of CoFe₂O₄ that was 40 nm according to Figure 7, and thus, after passing the critical diameter, the coercivity was reduced [24, 25].

With the decrease of $H_c$, there was an increase of $M_s$ from 89.02 emu/g to 113.5 emu/g following the Brown’s equation as follows [14, 27]:

$$H_c = \frac{2k_1}{\mu_0M_s}$$  \hspace{1cm} (5)

An increase in sintering temperature from 500°C to 800°C made the $H_c$ value changed from 1032 Oe to 472 Oe and thus narrow down the curve. This phenomenon made the grain size uneven and agglomerated, as shown in Figure 4 where agglomeration decreases following the decreasing $H_c$. An increase in sintering temperature around 500°C to 800°C caused the crystal grain to grown from 13.25 nm to 60.5 nm, and the $H_c$ value decreased [23]. Sintered CoFe₂O₄ at 1100°C had paramagnetic curve caused by the sintering temperature that passed the Curie temperature and made the magnetic property to dissolve, whereas the sonochemical sample was thought to have paramagnetic curve because the NaOH titration that has not entirely disappeared in the sample. All samples had remanence ratio below 0.5, thus if $R < 0.5$, there was a static magneto interaction between the granules [27].

Figure 8 explains the relationship between crystal size with $M_s$ and $H_c$. Comparison of crystallite size to $M_s$ shows that there is the largest $M_s$ value in crystallite size of 60.5 nm and 113.51 emu/g. The 13.25 nm crystal size has a $M_s$ value of 89.02 but has the greatest coercivity value of all samples, which is 1032 Oe. This phenomenon occurs because the crystal size has a magnetic domain in the form of a single domain, this phenomenon has been explained in the explanation above. From Figure 8 we can say that after critical diameter the magnetic curve of $M_s$ and $H_c$ will be downward curve due the transition from single domain to multi domain magnetic. From the results above we can also conclude that cobalt ferrite sol-gel with sinterring 500°C have a high magnetic saturation and high coercivity also smaller crystallite size that can be applied to Hyperthermia.

In the application of hyperthermia, it requires several important parameters, such as high $M_s$ and $H_c$ values so that it can quickly kill cancer cells but dissolved particles must be small in size. Based on reported data by Yadavalli et al. [28] nanomaterials CoFe₂O₄ with crystallite size 19.8 nm have $M_s$ and $H_c$ about 59 emu/g and ± 300 Oe, which was the best results for their experiment to hyperthermia study. Darwish et al. [29] reported nanomaterials CoFe₂O₄ with crystallite size 32.46 nm have $M_s$ about 50.61 emu/g with applicable to hyperthermia have SLP value about 552 W/g. Rashad et al. [30] reported nanomaterials CoFe₂O₄ with crystallite size 10.7 nm have $M_s$ and $H_c$ about 36.2 emu/g and 513.2 Oe with applicable to hyperthermia have SLP value about 302 W/g. Nam et al. [31] also reported nanomaterials CoFe₂O₄ with crystallite size 11 nm have $M_s$ about 58 emu/g which is applicable to hyperthermia have SLP value about 297 W/g. Yasemian et al. [32] also reported nanomaterials CoFe₂O₄ having crystallite size 11.1 nm have $M_s$ and $H_c$ about 42.94 emu/g and 169.19 Oe with

![Figure 6. Magnetic Hysteresis Curve of CoFe₂O₄. (black) Sol-gel 500°C Sintering, (red) Sol-gel 800°C Sintering, (blue) Sol-gel 1100°C Sintering, (light green) Sonochemical without Sintering](image)

![Figure 7. Dependence of coercivity with particle diameter for cobalt ferrite](image)
The synthesized cobalt ferrite with two methods, unsintered sonochemical and sintered sol-gel at 500°C, 800°C, and 1100°C for 2 hours had cubic spinel structure with the smallest crystal size of 13.25 nm and the largest of 70.64 nm following the increasing sintering time, the lowest crystallinity of 44.16% and the highest of 57.7%. The SEM results showed that the morphology of cobalt ferrite was decreasing agglomeration and smaller grain size following the high sintering temperature. The FTIR analysis presented functional group vibration at 601-636 cm\(^{-1}\) that signified the spinel structure identity. The VSM results showed a change in hysteresis loop curve from the sintered samples at 500°C and 800°C from hard magnetic into soft magnetic material. The curve difference was due to the high temperature that passed the Curie temperature and due to the transition from the single domain into multiple domains as shown by the Brown equation. The SEM results displayed the decreasing agglomeration because of the sintering temperature that passed the Curie temperature and decreased the magnetic property. Based on research that already mentioned, cobalt ferrite sol-gel sintering 500°C had big potential to apply on hyperthermia.

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