

## Effect of Lanthanum Substituted $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$ on Change of Structure Parameter and Phase Formation

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**Keywords:**  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$ , substitution, lanthanum, crystal structure, and surface morphology.

**Abstract.** In this research,  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$ -based smart magnetic material has been developed which will be applied as a microwave absorbing material. This smart magnetic material is an artificial advanced material which has properties such as electromagnetic waves so that it is able to respond to the presence of microwaves through the mechanism of spin electron resonance and wall resonance domain. This smart magnetic material consists of a combination of rare earth metal elements (spin magnetic in the  $f$  orbital configuration) and transition metal elements (spin magnetic in the  $d$  orbital configuration) with a semi-hard magnetic structure. This semi-hard is a characteristic of magnetic properties which is between hard magnetic and soft magnetic properties. This characteristic of the semi-hard magnetic properties is needed so that this material has the ability to absorb microwaves. Substitution of lanthanum into cobalt ferrite  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$  for  $\text{La}^{3+}$  ( $x = 0 - 0.8$ ) has been synthesized using the solid reaction method through mechanical deformation techniques. The refinement result of X-ray diffraction shows that the sample contains 2 phases with increasing of  $x$  compositions. Particle morphology and elementary analysis were observed respectively by using a scanning electron microscope (SEM) and energy dispersive spectroscopy (EDS). It was concluded that the effect of La substitution on  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$  resulted in changes in the crystal structure parameters and phase transformation as a function of composition.

### Introduction

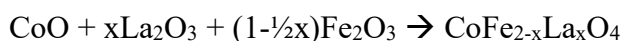
Iron oxide-based soft magnetic material commonly known as ferrite. This ferrite is ceramic and in nature it is abundant in the form of magnetite minerals ( $\text{Fe}^{2+}.\text{Fe}^{3+}_2\text{O}_4$ ). This magnetite mineral is often found in the form of iron sand. Ferrite is a hard, fragile electrical conductor. Ferrite structure is spinel with the formula  $\text{AB}_2\text{O}_4$ , where A and B represent various cations of transition metals such as iron (Fe). Ferrite spinel consists of cubic close-packed (fcc) with cation A occupying one-eighth of the tetrahedral site and cation B occupying half of the octahedral site. Another ferrite ingredient known as zinc ferrite has a formula  $\text{ZnFe}_2\text{O}_4$  normal structure of spinel ferrite, with  $\text{Fe}^{3+}$  occupy the octahedral site and  $\text{Zn}^{2+}$  occupies a tetrahedral site [1]. Zink Ferrite ( $\text{ZnFe}_2\text{O}_4$ ) composed of atoms that form a closed-packed face centred cubic (fcc) structure and are classified as soft magnets. Another form of ferrite is cobalt ferrite,  $\text{CoFe}_2\text{O}_4$  ( $\text{Co}^{2+}.\text{Fe}^{3+}_2\text{O}_4$ ), between soft magnets and hard magnets are usually classified as semi-hard materials [2]. Cobalt ferrite is mainly used for magnetostrictive applications such as sensors and actuators [3]. Magnetic anisotropy induced in cobalt ferrite is also useful for enhancing the magnetoelectric effect in composites [4]. Magnetic studies show that ferrite cobalt nanoparticles have an anisotropic field and high magnetic moment [5]. Besides cobalt ferrite is a superior magnetic because of its chemical stability and excellent thermal properties [6], uniform in terms of size [5], has a high resistivity value and a semi-hard coercivity value, but has a low dielectric loss [7]. Electromagnetic properties such as permeability

and permittivity of complexes in ferrite cobalt are in the frequency range of MHz to GHz. This indicates that cobalt ferrite has the potential to be applied as an absorbent of electromagnetic waves at high frequencies [8].

Because cobalt ferrite has a low dielectric loss, meaning that its permittivity is also low, for this reason this research has been carried out by substituting the lanthanum element which is believed to have high permittivity. In previous studies, material modification has been successfully carried out to improve the permittivity of the material by substituting Zn into ferrite cobalt so that this material has permeability and permittivity of single phase and is expected to be one of the candidates for microwave absorbing material. This material has a single phase with cubic structure formation (space group  $Fd-3m$ ), where lattice parameters, lattice strain, cell unit volume, and atomic density change as a function of composition. so that the substitution effect of Zn results in changes in the parameters of the crystal structure as a function of composition with uniform particle size distribution [9,10]. Based on this experience, this research has carried out the synthesis and characterization of the results of the modification of La substitution magnet material into ferrite cobalt with a system  $CoFe_{2-x}La_xO_4$ . The permittivity of this material is intrinsically entirely dependent on the distribution of cations at Fe crystallographic sites. That is, the intrinsic properties of this material can be adjusted to regulate the cation distribution at the Fe crystallographic site [9-11]. This means that the intrinsic properties of the ferrite cobalt material are expected to change after the Fe crystallographic site is disrupted, while its effects on the structural parameters and phase changes of this material will be discussed and become the focus of discussion in this paper. Thus, the analysis of crystal structures and their correlation with structural parameters and phase changes of this material need to be explored more deeply in this paper. The size of these particles and crystallites is also responsible for changes in their intrinsic properties [12-15]. So the purpose of this study is to determine the effect of lanthanum substitution in cobalt ferrite on changes in structural parameters and phase formation in magnetic system material  $CoFe_{2-x}La_xO_4$ .

## Experiment Procedure

Cobalt ferrite ( $CoFe_2O_4$ ) which is substituted with  $La^{3+}$  produce system  $CoFe_{2-x}La_xO_4$  prepared using a solid reaction method through mechanical deformation techniques with several raw materials namely CoO (Merck, 99.9% purity),  $La_2O_3$  (Merck, 99.9% purity), and  $Fe_2O_3$  (Sigma-Aldrich, purity 99.9%) [9]. Stoichiometric calculations have been performed for substitution variations  $La^{3+}$  of  $x = 0, 0.2, 0.4, 0.6,$  and  $0.8$  following the equation of reaction :  $\frac{1}{2}$

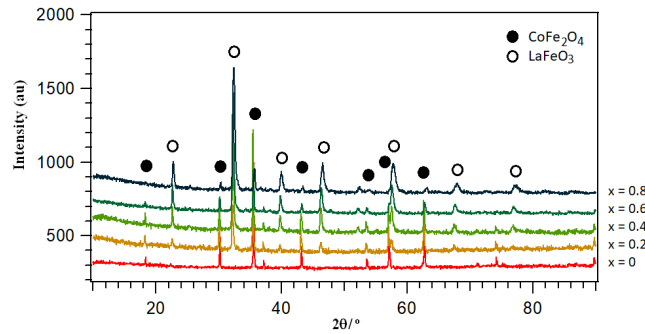


The three raw materials in accordance with their respective compositions were mixed and milled for 5 hours in a 50 ml ethanol environment using a high-energy milling device PW-1000di. This method is called solid reaction through mechanical deformation techniques. The ratio of the mass of the milling balls (10 mm diameter) to the material is 2:1 [9]. Then the milling mixture is dried in an oven at  $100^\circ C$  for 5 hours. After the sample and the milling balls are separated, the sample is crushed again manually using mortal agate. The resulting powder is compacted into tablet form with a compacting pressure of 7000psi. The tablet form samples were then sintered using a Thermolyne 6000 furnace at  $1000^\circ C$  for 2 hours. Furthermore, each sample was characterized using the XRD tool used by the Brucker brand to determine changes in structural parameters due to the influence of  $La^{3+}$  substitution. Anode X-rays use  $CuK\alpha$  ( $\lambda = 1.5406\text{\AA}$ ) with step size 0.01. Quantitative analysis was performed using general structure analysis system (GSAS) software. Surface morphology using SEM tools with JEOL JED 350 brand [15].

## Results and Discussion

The X-ray diffraction (XRD) pattern of  $La^{3+}$  substitution samples in ferrite cobalt ( $CoFe_{2-x}La_xO_4$ ) for  $x = 0 - 0.8$  is shown in Figure 1. The results of qualitative analysis using the Match program where the peaks of the XRD pattern of all samples correspond to the reference database open

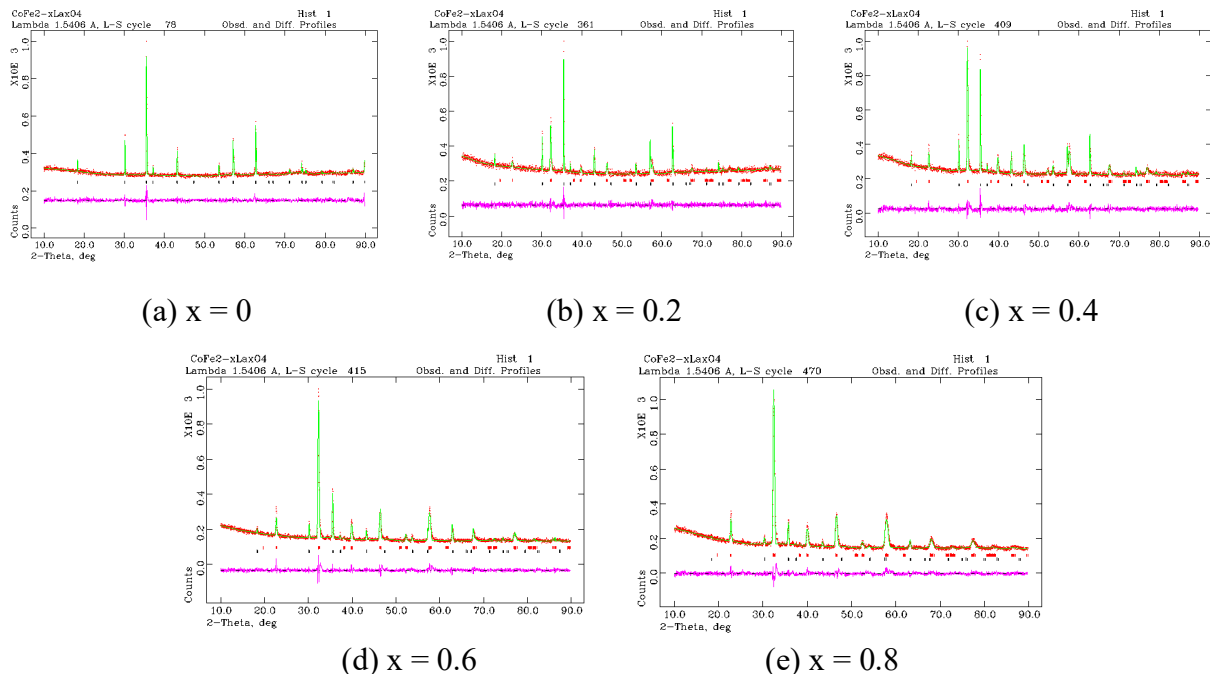
crystallography data that is  $\text{CoFe}_2\text{O}_4$  phase with cubic crystal symmetry (space group  $\text{Fd-3m}$ ) [16]. However, samples that have a single phase of  $\text{CoFe}_2\text{O}_4$  are only at the composition  $x = 0$  while  $x > 0$  has formed 2 phases, namely the  $\text{CoFe}_2\text{O}_4$  and  $\text{LaFeO}_3$  phases [15].



**Figure 1.** X-ray diffraction pattern  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$  ( $x = 0 - 0.8$ )

Based on the qualitative identification results show that  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$  single phase only in the composition  $x = 0$  and  $x > 0$  allegedly La failed to substitute Fe, meaning that the reaction process is imperfect, La comes out to form a new phase,  $\text{LaFeO}_3$ . The results of this qualitative analysis are very interesting to understand because of how many lanthanum atoms are still able to replace some of the Fe atoms in the  $\text{CoFe}_2\text{O}_4$  structure. In Figure 1 it appears that by increasing the La content in the sample, the formation of the  $\text{LaFeO}_3$  phase increases. This requires a quantitative analysis to determine changes in the parameters of the crystal structure, the number of mass fractions formed, and the cationic distribution of the substitution results of La into the Fe atom [15].

Figure 2 shows the results of refinement of X-ray diffraction patterns from  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$  samples with composition variations ( $x = 0 - 0.8$ ).



**Figure 2.** Refinement results X-ray diffraction patterns for sample  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$  ( $x = 0 - 0.8$ )

Figure 2(a) is the result of refinement of the XRD pattern for  $x = 0$  which has formed a peak of diffraction bragg with a single phase following the  $\text{CoFe}_2\text{O}_4$  structure. Figure 2(b-e) is the result of refinement of the XRD pattern for  $x = 0.2 - 0.8$  having formed a two-phase diffraction peak bragg, which follows the structure of  $\text{CoFe}_2\text{O}_4$ , and  $\text{LaFeO}_3$ . Qualitative and quantitative analysis refers to Open Database Crystallography with card numbers (COD: 1533163), and (COD: 1526450) for the  $\text{CoFe}_2\text{O}_4$ , and  $\text{LaFeO}_3$  phases, respectively [9, 15-17].

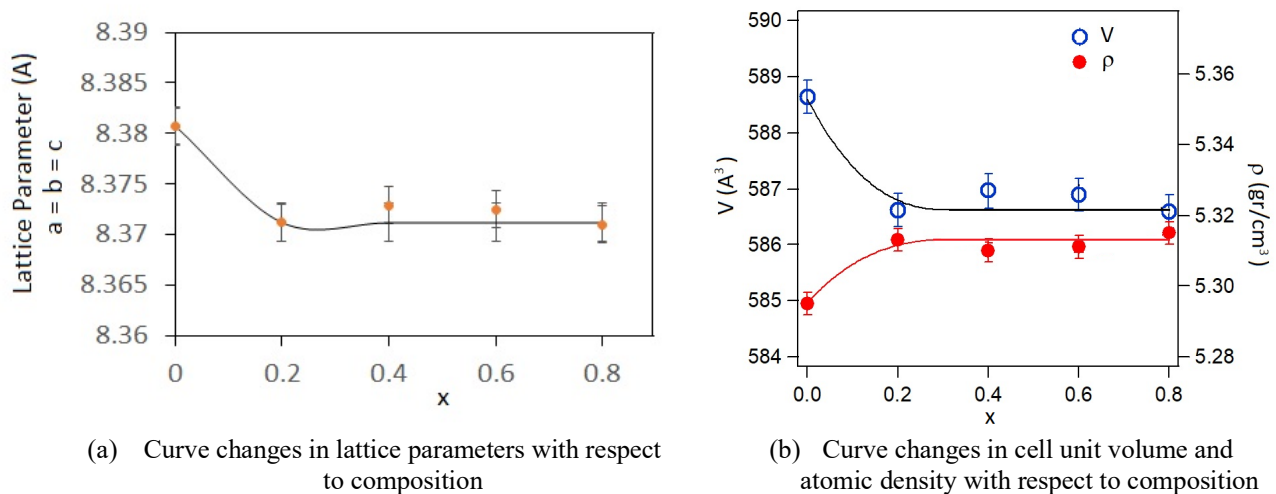
A complete summary of the results of refinement of X-ray diffraction patterns from  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$  samples with composition variations ( $x = 0 - 0.8$ ) for all compositions is shown in Table 1.

**Table 1.** Value of structure parameters, fit criteria ( $R_{wp}$ ), goodness of fit ( $\chi^2$ ) and the mass fraction formed in the  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$  sample with variations in composition ( $x = 0 - 0.8$ )

Sample (x)	Phase	Lattice parameter (Å)			V (Å <sup>3</sup> )	$\rho$ (g/cm <sup>3</sup> )	Fraction wt%	$R_{wp}$ (%)	$\chi^2$
		a	b	c					
0	CoFe <sub>2</sub> O <sub>4</sub>	8.3807(2)	8.3807(2)	8.3807(2)	588.64(4)	5.295	100	2.24	1.14
0.2	CoFe <sub>2</sub> O <sub>4</sub>	8.3712(2)	8.3712(2)	8.3712(2)	586.63(6)	5.313	73.21	3.19	1.27
	LaFeO <sub>3</sub>	7.852(2)	5.514(1)	5.547(1)	240.2(1)	6.371	26.79		
0.4	CoFe <sub>2</sub> O <sub>4</sub>	8.3729(3)	8.3729(3)	8.3729(3)	586.98(7)	5.310	49.22	3.56	1.32
	LaFeO <sub>3</sub>	7.850(1)	5.5477(7)	5.5133(6)	240.11(7)	6.378	50.78		
0.6	CoFe <sub>2</sub> O <sub>4</sub>	8.3725(8)	8.3725(8)	8.3725(8)	586.90(1)	5.311	30.52	4.51	1.32
	LaFeO <sub>3</sub>	7.8437(6)	5.5044(3)	5.5425(5)	239.30(4)	6.379	69.48		
0.8	CoFe <sub>2</sub> O <sub>4</sub>	8.371(1)	8.371(1)	8.371(1)	586.6(4)	5.315	16.95	4.38	1.35
	LaFeO <sub>3</sub>	7.820(1)	5.5269(8)	5.4808(6)	236.91(2)	6.384	83.05		

Figure 2 and Table 1 show that the results of refinement of X-ray diffraction patterns have excellent fitting quality according to fit criteria ( $R_{wp}$ ) and goodness of fit ( $\chi^2$ ) [18].  $R_{wp}$  is the weight ratio of the difference between observation patterns and XRD calculations (ideal  $R_{wp}$  value <10%). While  $\chi^2$  (*chi-squared*) is the ratio of XRD patterns of observations that is proportional to expectations.

Based on Table 1, there is an interesting phenomenon to be studied, namely changes in lattice parameters, cell unit volume and atomic density after ferrite cobalt is substituted with La atoms as shown in Figure 3.



**Figure 3.** Results of lattice parameter analysis, cell unit volume and atomic density as a function of composition

In Figure 3(a) it appears that the  $\text{CoFe}_2\text{O}_4$  phase lattice parameter decreases at composition  $x = 0.2$ , but as the composition of  $x$  increases, the lattice parameter does not change significantly. The same thing happens with the volume of cell units as in Figure 3(b), where the volume of cell units begins to shrink when the resultant La substitution starts from  $x = 0.2$ , but with the addition of  $x$  subsequently, the volume of cell units does not change significantly. And vice versa for the atomic density, where the atomic density increases when  $x = 0.2$  then does not change significantly as the composition of  $x$  increases. This shows that it is suspected that the La atom has been successfully substituted with the composition  $x < 0.2$  in the cobalt ferrite phase.

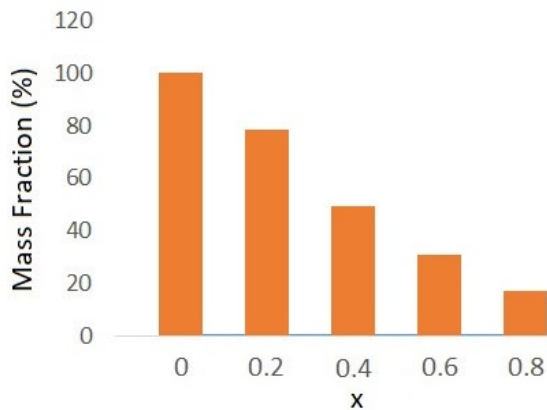
An interesting thing to learn is the ability of La atoms to substitute Fe atoms with the same structure. At the composition  $x = 0.2$ , there are 20% of La atoms which are expected to be able to partially replace Fe atoms so that they are expected to have an impact on other properties, especially

on their magnetic properties. But in reality, not all La is able to replace some Fe atoms, meaning that some La atoms have succeeded in substituting to form a spinel cubic structure and some others are bound to Fe to form another phase. In this case it is necessary to analyze the cationic distribution at the Fe atomic site by refining the atomic occupancy factor for each Fe site that has been substituted La. The  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$  structure is a cubic with a space group (Fd-3m). The Co atom position occupies the Wyckoff position at site 8a -43m symmetry, while the La and Fe atoms position occupies the Wyckoff position at the same site on site 16d. One unit of  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$  cells in which  $\text{Fe}^{3+}$  ions occupy the octahedral site (16d) and  $\text{Co}^{2+}$  occupy the tetrahedral site (8a). The La atom will occupy part of the Wyckoff position of the Fe atom on site 16d. This research will be calculated based on the simulation results using the GSAS program how much the La atom is able to substitute the Fe atom in the  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$  system [9]. This data is called the result of cationic distribution analysis. The cationic distribution of the refinement results is shown in Table 2.

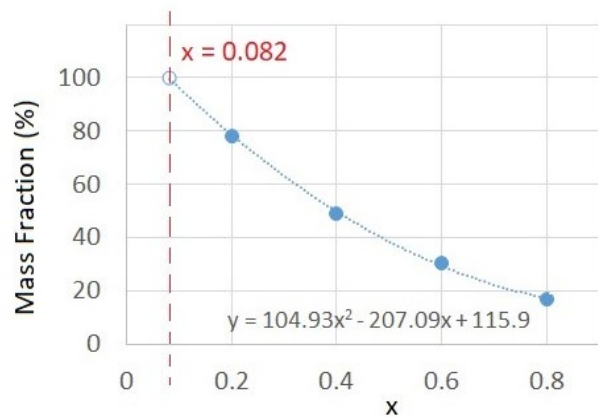
**Table 2.** Cationic distribution is calculated based on the results of XRD data refinement

X	Site	Point symmetry	Cationic distribution (Occupation factor)			Composition
			Co	Fe	La	
0	8a	-43m	1.00 (100 at.%)			$\text{CoFe}_2\text{O}_4$
	16d	-3m		1.00 (100 at.%)	-	
0.2	8a	-43m	1.00 (100 at.%)			$\text{CoFe}_{1.92}\text{La}_{0.08}\text{O}_4$
	16d	-3m		0.91 (91.46 at.%)	0.0854 (8.54 at.%)	
0.4	8a	-43m	1.00 (100 at.%)			$\text{CoFe}_{1.92}\text{Fe}_{0.08}\text{O}_4$
	16d	-3m		0.91 (91.51 at.%)	0.0849 (8.49 at.%)	
0.6	8a	-43m	1.00 (100 at.%)			$\text{CoFe}_{1.92}\text{Fe}_{0.08}\text{O}_4$
	16d	-3m		0.91 (91.58 at.%)	0.0842 (8.42 at.%)	
0.8	8a	-43m	1.00 (100 at.%)			$\text{CoFe}_{1.92}\text{Fe}_{0.08}\text{O}_4$
	16d	-3m		0.91 (91.79 at.%)	0.0821 (8.21 at.%)	

Based on Table 2 it is shown that the results of the analysis of the cationic distribution for the acquisition of La substitution into cobalt ferrite in all x compositions are an average of 0.08. This means that the La atom is able to partially replace the maximum Fe atom of  $x = 0.08$  (8 at.%) in one atomic residence. This result is supported by mathematical calculations based on the results of the analysis of the decrease in the mass fraction of the  $\text{CoFe}_2\text{O}_4$  phase as a function of composition x as shown in Figure 4.



(a)  $\text{CoFe}_2\text{O}_4$  phase mass fraction of the composition function x



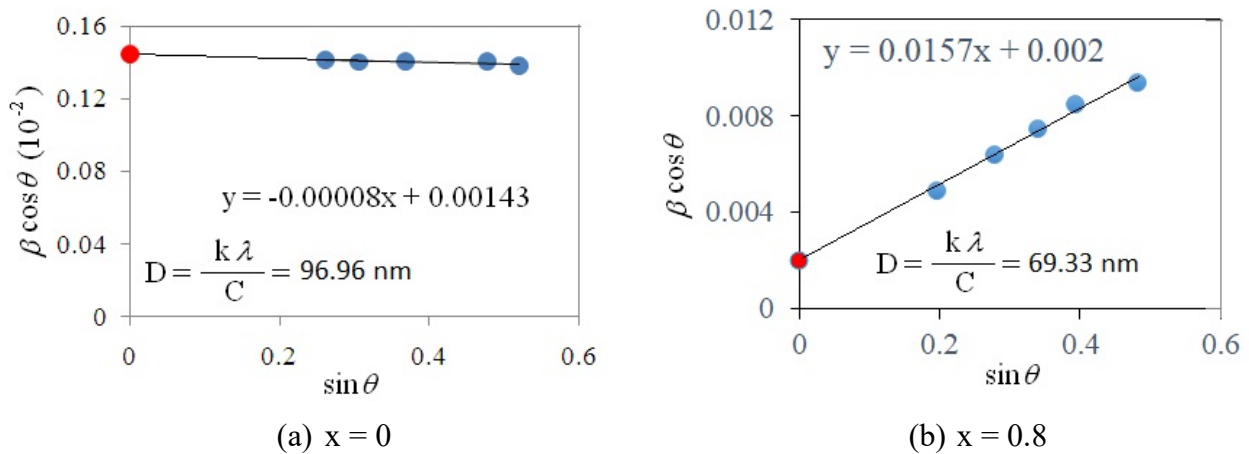
(b) Mathematical simulation of determining composition x

**Figure 4.** XRD refinement results in analyzing optimum mass fraction and composition

Figure 4(a) is the result of the analysis of  $\text{CoFe}_2\text{O}_4$  phase mass fraction based on X-ray diffraction data refinement in all x compositions, where the  $\text{CoFe}_2\text{O}_4$  phase mass fraction appears to decrease with increasing x composition. Figure 4(b) shows the mathematical calculation results for

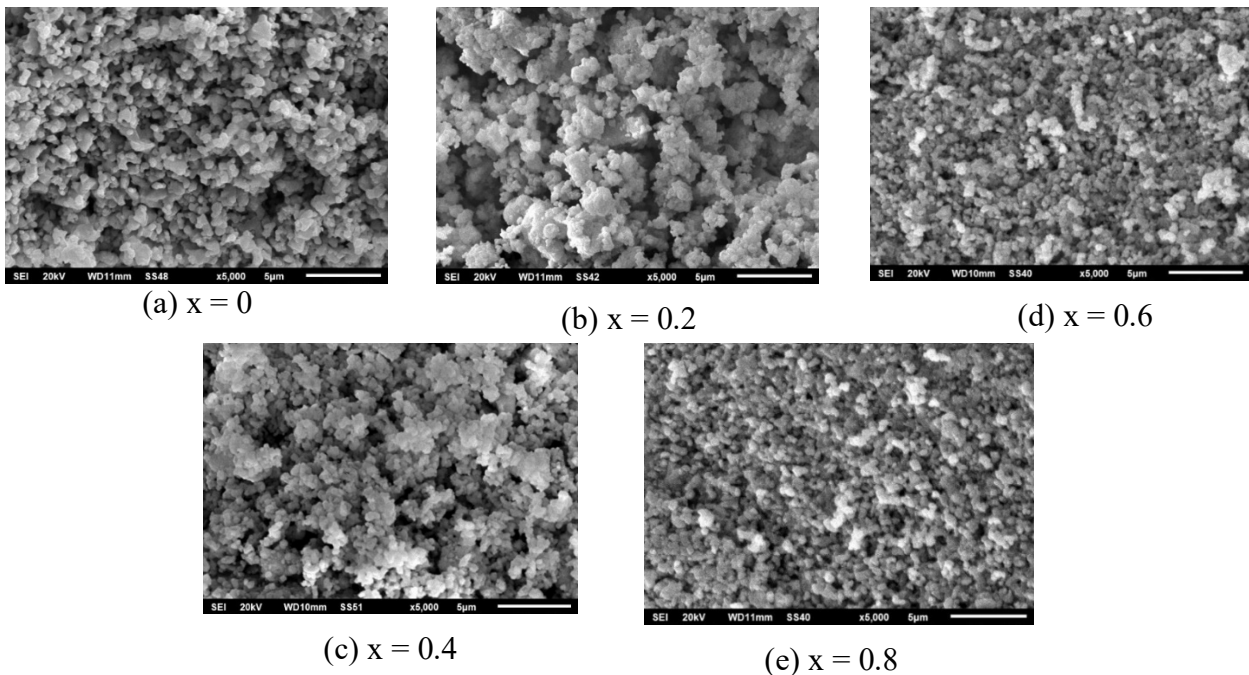
composition  $x$  which can produce a single phase  $\text{CoFe}_2\text{O}_4$  with La substitution. Thus based on the analysis of atomic occupancy factors and mass fractions it can be assumed that to obtain a single phase from the  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$  system, the substitution of La atoms is able to replace some Fe atoms of  $x = 0.08$ .

The second interesting thing to be understood in this study is the change in crystallite size of the  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$  system ( $x = 0 - 0.8$ ) which decreases with increasing composition of  $x$ . This is caused by phase changes that occur in each composition. This means that at the composition  $x = 0$ , the dominant mass fraction is the  $\text{CoFe}_2\text{O}_4$  phase, but after the composition  $x = 0.8$ , the dominant mass fraction is the  $\text{LaFeO}_3$  phase, thereby causing the crystallite size of the two to differ. The crystallite size of the  $\text{CoFe}_2\text{O}_4$  phase is greater than the  $\text{LaFeO}_3$  phase. Calculation of crystallite size using the Williamson-Hall method [9,19] as shown in Figure 5.



**Figure 5.** Calculation of crystallite size and lattice strain in a single phase  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$  sample ( $x = 0$  and  $x = 0.8$ )

Observation of particle surface morphology for all  $x$  compositions was carried out using SEM as shown in Figure 6.



**Figure 6.** Morphology of  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$  particles observed using SEM (a)  $x = 0$ ; (b)  $x = 0.2$ ; (c)  $x = 0.4$ ; (d)  $x = 0.6$  and (e)  $x = 0.8$

Figure 6 shows that the particle morphology of the composition  $x = 0$  has a very good and uniform particle homogeneity across the entire sample surface with polygonal particle shape and an

average particle size of 500 nm, then for the composition  $x = 0.2 - 0.6$  the particle morphology does not look homogeneous the particle size also seems to vary uniformly from 300-500 nm. But for composition  $x = 0.8$  the particle morphology appears homogeneous with a more uniform particle shape with an average particle size of 300 nm. The particle size for  $x = 0.8$  appears smaller compared to the composition  $x = 0$ . This is consistent with the results of measurement of crystallite size of the composition  $x = 0$  and  $x = 0.8$ . This is presumably because in the composition  $x = 0$  the phase formed is the  $\text{CoFe}_2\text{O}_4$  phase, but after the presence of La substitution with the largest composition  $x = 0.8$ , the phase has changed dominantly to produce the  $\text{LaFeO}_3$  phase. So in general it can be said that the substitution of La into cobalt ferrite can cause changes in the parameters of the crystal structure and formation of its phase.

## Conclusion

Synthesis using solid reaction method through mechanical milling technique and structural analysis of  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$  semi-hard magnetic material with composition ( $x = 0 - 0.8$ ) has been successfully carried out. The results of the refinement of the X-ray diffraction pattern show that  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$  has a single phase in the composition  $x = 0$  and  $x > 0$  consists of two phases, namely the  $\text{CoFe}_2\text{O}_4$  and  $\text{LaFeO}_3$  phases. Based on the analysis of atomic occupancy factors and mass fractions it can be assumed that to obtain a single phase from the  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$  system, the substitution of La atoms is able to replace some Fe atoms of  $x = 0.08$ . The crystallite size of the  $\text{CoFe}_{2-x}\text{La}_x\text{O}_4$  sample decreases with increasing La atom content due to phase change from  $\text{CoFe}_2\text{O}_4$  to  $\text{LaFeO}_3$ , so it appears in the results of particle morphology that the particle size of the composition  $x = 0$  and  $x = 0.8$  decreases respectively from 500 nm to 300 nm. Both compositions have good and uniform particle homogeneity throughout the sample surface with polygonal particle shapes because each composition is influenced by a different dominant phase. General conclusions indicate that the substitution of La into the cobalt ferrite can cause changes in the parameters of the crystal structure and changes in the formation of its phase.

## Acknowledgement

This work is supported by a program for research and development of magnetic materials, namely the Research and Community Service Institute of Sriwijaya University (Competitive Superior Grant for UNSRI PNPB funds in 2018) [9]. Thank you to the Laboratory for Material Physics of the Physics Department of the Faculty of Mathematics and Natural Sciences of Sriwijaya University. Akmal Johan and Wisnu Ari Adi are authors contributed equally to this work, discussed result and the writing of the manuscript.

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