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ICAMST 2019

The 7th International Conference on Advanced Materials Science and Technology 2019

Aula Barat dan Aula Timur Institut Teknologi Bandung, 25-26 September 2019

Website: <http://kosatem.org/icamst2019>

Email: icamst2019@kosatem.org

Date: 19 August 2019

Letter of Invitation

Dear Authors: E. H. Sujiono*), Muthmainnah Muchtar, Vicran Zharvan, Sultra Adi Poetra, Abdi Manab Idris, and Muhammad Yusriadi Dahlan

We are pleased to inform you that your abstract (ABS-102, Oral Presentation), entitled:

"Synthesis of Neodymium Ferrite Oxide doped Ytterbium by Using Solid State Reaction Method and Its Characterization"

has been reviewed and accepted to be presented at ICAMST 2019 conference to be held on 25-26 September 2019 in Bandung, Indonesia.

We cordially invite you to attend our conference and present your research described in the abstract.

Please submit your full paper and make the payment for registration fee before the deadlines, visit our website for more information.

Thank You.

Best regards,

A handwritten signature in blue ink, appearing to read "Khairurrijal", on a light-colored background.

Prof. Dr. Eng. Khairurrijal
ICAMST 2019 Chairperson

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Letter of Acceptance for Abstract

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Today: Proceedings

Manuscript Draft

Manuscript Number:

Title: Synthesis of Ytterbium-doped Neodymium Ferrite Oxide Using Solid-State Reaction Method and Its Characterization

Article Type: 7th_ICAMST

Keywords: Ytterbium; NdFeO₃; Solid State Reaction Method; Structure; Morphology

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Corresponding Author's Institution: Universitas Negeri Makassar

First Author: Eko Hadi Sujiono, Dr.

Order of Authors: Eko Hadi Sujiono, Dr.; Vicran Zharvan, M.Sc.; Muthmainnah Muchtar, B.Sc; Sultra Ade Poetra, B.Sc; Abdi Manab Idris, B.Sc; Muhammad Yusriadi Dahlan, B.Sc; S Samnur, Dr.

Abstract: The Yb doped NdFeO₃ using a solid-state reaction method has successfully synthesized. In this paper, Nd_{1-x}Yb_xFeO₃ samples were synthesized by varying the molar ratio of Yb at $x = 0.01$, $x = 0.05$, and $x = 0.10$ using solid-state reaction with two routes of heat treatment processes. Results of X-ray diffraction show that all samples have an orthorhombic structure with two phases: NdFeO₃ as a major phase and Nd₂O₃ as a minor phase. The average crystal size is 40 nm, with the dominant peak corresponding to hkl (121). Morphology properties used SEM Image shows grain size of all sample estimated at 0.4 μm . The presence of Yb is quantitatively confirmed based on the EDS result.

POINT TO POINT RESPONSE TO REVIEWER

RESPONSE TO REVIEWER 1

Reviewer point #1: Many grammatical errors are found in this manuscript.

Author response #1: We agree with the reviewer and in terms of grammatical errors have been improved. For English improvement as a whole can be seen in the last revision of the manuscript.

Reviewer point #2: In the abstract section, please delete the first sentence. The second sentence is more complete.

Author response #2: We disagree with the reviewer. Because the first sentence in the abstract section illustrates what becomes the objective of this research.

Reviewer point #3: In the abstract section, please change the last sentence with the detail results.

Author response #3: We agree with the reviewer and we have written more regarding detailed results of this research in the abstract section.

Reviewer point #4: Please explain in more detail the novelty of this work.

Author response #4: We agree with the reviewer and we have explained more detail regarding the novelty of this research in the introduction section.

Reviewer point #5: In the experimental method section, please combine this paragraph with the previous one.

Author response #5: We agree with the reviewer and we have combined the sentences.

Reviewer point #6: The authors should analyze the XRD data using Rietveld analysis to calculate the lattice parameters. Then explain the data and make comparison with other related works

Author response #6: The main objective of this research is to synthesis the high quaility of Yb doped NdFeO₃ using a solid-state reaction method, so that we focus on the

synthesis process. In terms of data analyzing using Rietveld method can be seen in our another article as title “Structure Identification of $\text{Nd}_{1-x}\text{Yb}_x\text{FeO}_3$ ($x=0.01, 0.05$ and 0.10) Using Rietveld Refinement Method”.

Reviewer point #7: In the analysis of SEM-EDAX section, the quality of all EDS figures should be increased.

Author response #7: We agree with the reviewer and we have changed the figures with better resolution quality.

RESPONSE TO REVIEWER 2

Revision required and comments:

- This manuscript describes Synthesis of Neodymium Ferrite Oxide doped Ytterbium by Using Solid State Reaction Method and Its Characterization.
- The title is suggested to be Synthesis of Ytterbium-doped Neodymium Ferrite Oxide Using Solid State Reaction Method and Its Characterization.
- This manuscript still contains some grammatical and typographical errors. Please fix them.
- The section Introduction explains quite well the background and motivation of this work.
- The section Experiments gives quite good explanation.
- The results have been well discussed.
- The conclusion is rewritten accordingly.

Author responses :

- In terms of grammatical and typographical errors, we agree with the reviewer and for improvements in English as a whole can be seen in the last revision of the manuscript.
- In terms of the suggested title, we agree with the reviewer and we have changed the title of this article with Synthesis of Ytterbium-doped Neodymium Ferrite Oxide Using Solid-State Reaction Method and Its Characterization.

7th ICAMST

Synthesis of Ytterbium-doped Neodymium Ferrite Oxide Using Solid-State Reaction Method and Its Characterization

Eko Hadi Sujiono^{a,*}, Vicran Zharvan^a, Muthmainnah Muchtar^a, Sultra Ade Poetra^a, Abdi Manab Idris^a, Muhammad Yusriadi Dahlan^a, Samnur^a

^aLaboratory of Materials Physics, Department of Physics, Universitas Negeri Makassar, Makassar 90224, Indonesia

Abstract

The Yb doped NdFeO₃ using a solid-state reaction method has successfully synthesized. In this paper, Nd_{1-x}Yb_xFeO₃ samples were synthesized by varying the molar ratio of Yb at x = 0.01, x = 0.05, and x = 0.10 using solid-state reaction with two routes of heat treatment processes. Results of X-ray diffraction show that all samples have an orthorhombic structure with two phases: NdFeO₃ as a major phase and Nd₂O₃ as a minor phase. FWHM value for a maximum peak is 0.2°, so the estimated crystal size is 40 nm, with the dominant peak corresponding to hkl (121). Morphology properties used SEM Image shows grain size of all sample estimated at 0.4 μm. The presence of Yb is quantitatively confirmed based on the EDS result.

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Peer-review under responsibility of the scientific committee of The 7th International Conference on Advanced Materials Science and Technology 2019, 7th ICAMST.

Keywords: Ytterbium; NdFeO₃; Solid State Reaction Method; Structure; Morphology

1. Introduction

ReFeO₃ elements that (Re: rare-earth elements) are known as the rare-earth orthoferrites having orthorhombic structure derived from a perovskite structure [1], and they have attracted much interest due to their properties such as magnetic and magneto-optic [2]. Among these ReFeO₃ elements, NdFeO₃ material useful as a raw material of many applications such as gas sensors, fuel cells, and catalyst material gas sensors [3,4,5,6]. The preparation of NdFeO₃ material can realize by several methods [7,8]. The solid-state reaction method has used because this method is cheap and easy to implement. The high purity and crystallinity materials also can be achieved by this method [9].

Present-day, many researchers are working on ReFeO₃ to obtain ideal materials for Adsorbent [10], photocatalytic Material [11], solid-state devices, and gas sensors application [12]. RFeO₃ has a characteristic feature of the presence of two magnetic subsystems of Re³⁺ and Fe³⁺. The interactions of Fe-Fe, Re-Fe, and Re-Re lead to a few interesting phenomena. ReFeO₃ has the special characteristic of spin reorientations $\Gamma_4(G_y, F_z) \rightarrow \Gamma_{24}(G_{xz}, F_{xz}) \rightarrow \Gamma_2(G_z, F_x)$ [13]. ReFeO₃ shows interesting gas sensitivity properties, gas sensors based on LaFeO₃ show good results to detect ethanol, acetone, H₂S, CO, and NO₂ [14,15]. In the few past years, NdFeO₃ material as a gas sensor to detect C₂H₅OH [16]. Gas sensors based on ReFeO₃ are developed to detect acetone [17]. The properties of ReFeO₃ elements for gas sensors such as their response and selectivity increased by doping to another oxide material [18] but less information using Ytterbium (Yb) as a dopant element for NdFeO₃.

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In this paper, presented produced the Ytterbium (Yb_x) doped $\text{Nd}_{1-x}\text{FeO}_3$ samples by using a solid-state reaction method by varying the molar ratio of Yb_x from $x = 0.01$, $x = 0.05$ and $x = 0.10$, respectively. All samples then characterized to obtain their crystal structure with qualitative analysis, morphology, and elemental composition properties.

2. Experimental method

The Yb doped NdFeO_3 sample was synthesized using the best parameters found by previous research [18-23]. Raw materials of Nd_2O_3 (Strem Chemicals, 99.99%), Yb_2O_3 (Sigma-Aldrich, 99.99%) and Fe_2O_3 (Sigma-Aldrich, 99.99%) were mixed according to stoichiometric calculation and grinded using mortar for 3 hours. That mixed material then calcined using the furnace at temperature 700°C for 4 hours. After that, the temperature increased to 950°C by keeping the temperature constant for 2 hours. The heating then decreased to 475°C and kept it continued for 2 hours. After this thermal process, the material was grinded for 6 hours.

Material that was obtained then calcined at 950°C for 4 hours. After that, the temperature was increased to 1050°C for 2 hours then decreased it to 525°C for 2 hours. All of the processes repeated by varying the Yb content at $x = 0.01$, $x = 0.05$, and $x = 0.10$. The $\text{Yb}_x\text{-Nd}_{1-x}\text{FeO}_3$ samples then characterized using XRD (Rigaku Mini Flex II $\text{CuK}\alpha$, $\lambda = 0.154 \text{ nm}$) to obtain the phase of crystallographic and SEM-EDAX (FEI Inspect S50) to analyze the morphology and elemental composition, respectively.

3. Results and discussion

3.1. Analysis of x-ray diffraction

Characterization results of x-ray diffraction for Yb doped NdFeO_3 samples can show in Fig.1. It shows that the pattern has narrow peaks indicating that the samples are in crystalline form. Further analysis using X'Pert High Score Plus software shows the presence of NdFeO_3 and Nd_2O_3 phases. Those phases give information that samples are polycrystalline material [9] with the highest peak lead to the NdFeO_3 phase at the plane (121) in orthorhombic structure, which is similar to other results [9,16,24]. The presence of Nd_2O_3 phase due to the low temperature during the calcination process [9]. There is no additional peak of impurity observed, which indicates that the samples consist of pure phases [25].

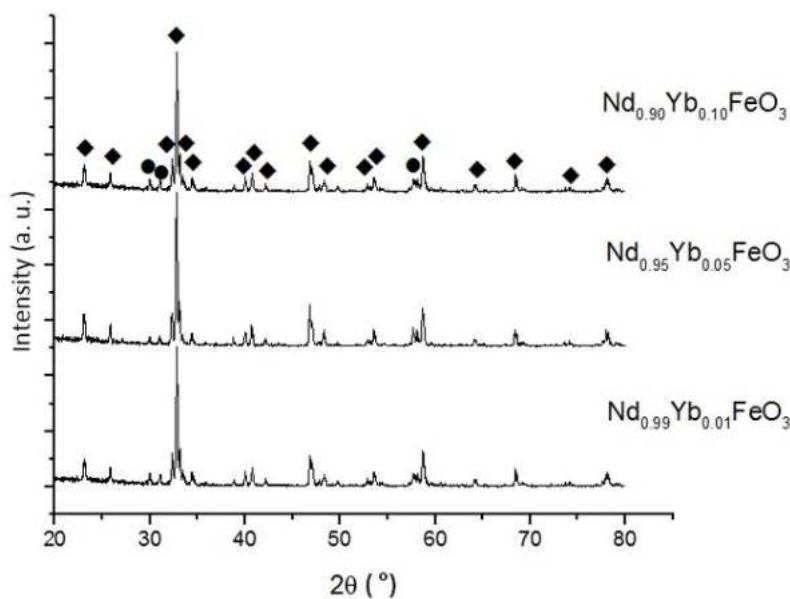


Fig. 1. X-ray diffraction pattern of Yb doped NdFeO_3 ($\blacklozenge = \text{NdFeO}_3$, $\bullet = \text{Nd}_2\text{O}_3$).

Further analysis of Yb doped NdFeO_3 describes in Table 1. It shows that the 2θ for peak (121) gradually shift into a lower degree, comparing to undoped NdFeO_3 [26]. This phenomenon can occur due to lattice distortion [24, 27], further analysis of FWHM, 2θ , and crystalline size shown in Table 1.

Table 1. X-ray analysis results of Yb_x doped $\text{Nd}_{1-x}\text{FeO}_3$ at plane (121)

X	2θ (degree)	FWHM (degree)	Crystalline Size* (nm)
0.01	32.8294	0.20197	40.56±0.02
0.05	32.8081	0.20279	40.40±0.02
0.10	32.7940	0.20138	40.68±0.02
0**	32.8600	0.20000	67.00

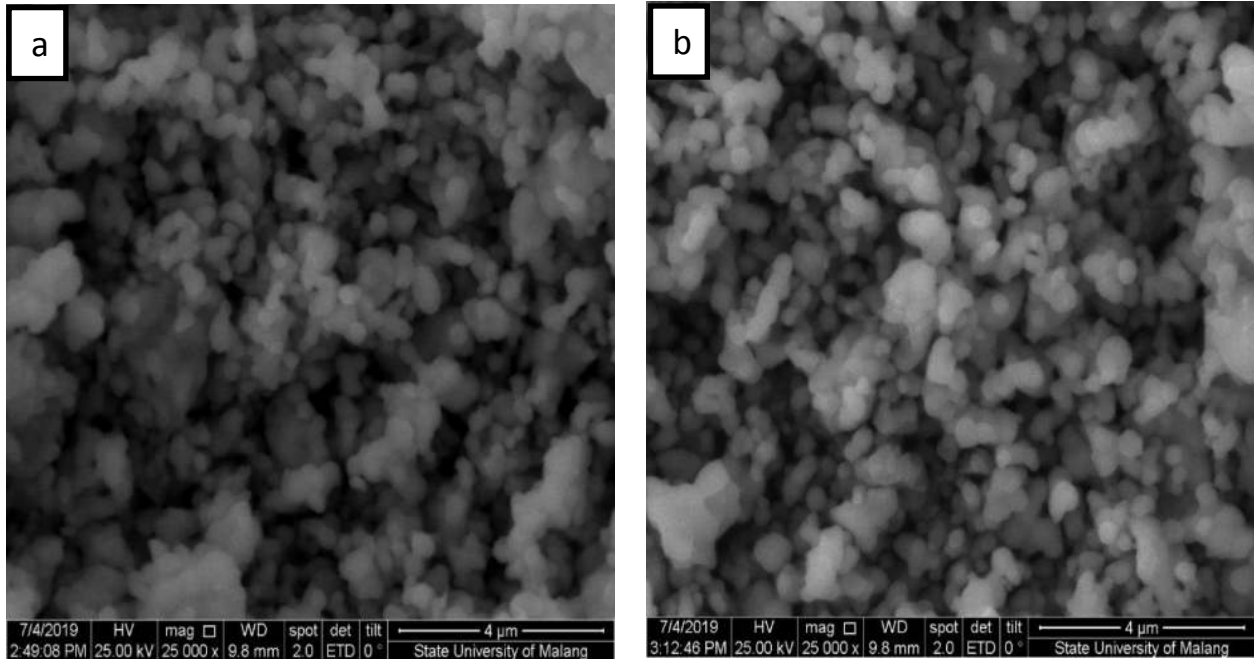
*) Calculated by Debye-Scherrer method

**) Khorasani, et al. [26]

It can be seen that increasing of Yb content gradually shift the 2θ degree into the lower degree and decrease the value of crystalline size from 67 nm as reference value to 40 nm due to the diffusion of ion Yb^{3+} with ionic radii (0.98 Å) less than ionic radii of Nd^{3+} (1.11 Å) as donor into the lattice of NdFeO_3 as acceptor [28].

3.2. Analysis of SEM-EDAX

The influence of the Yb dopant on the morphology of NdFeO_3 shown in Fig.2.



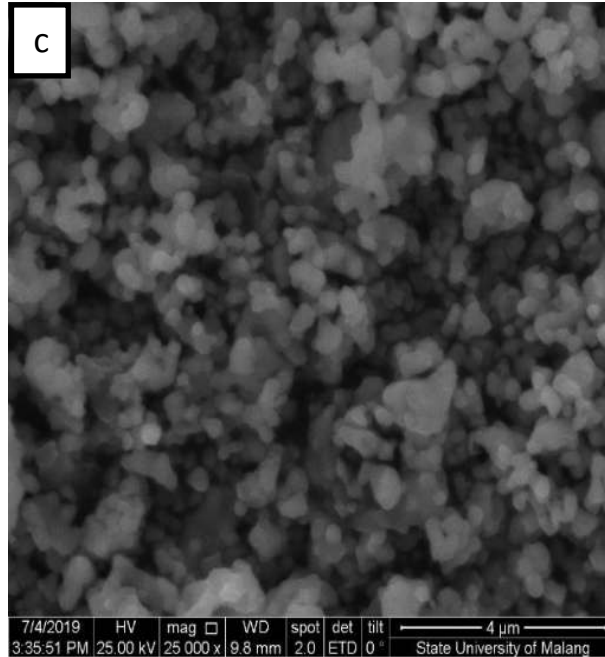
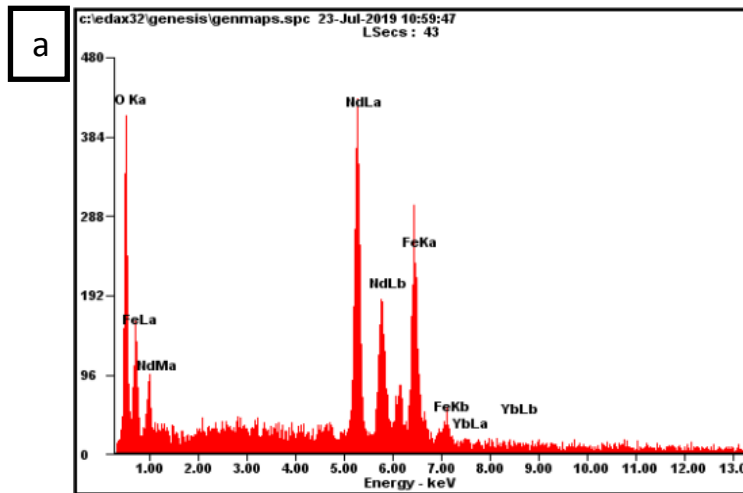


Fig. 2. Morphology of sample Yb_x doped $\text{Nd}_{1-x}\text{FeO}_3$: (a) $x = 0.01$, (b) $x = 0.05$ and (c) $x = 0.10$, respectively.

The images show all of the Yb-NdFeO_3 powders are quite uniform in shape and size. It gives information that increasing Yb does not provide a significant effect on morphology. The agglomeration also exists for all powders due to the factor of temperature and mechanical treatment during processes [18]. The particle size also can be estimated an average at $0.4 \mu\text{m}$ from the images. The presence of Yb_x in the $\text{Nd}_{1-x}\text{FeO}_3$ sample can be confirmed based on the results of the EDS spectrum. It can show in Fig. 3.



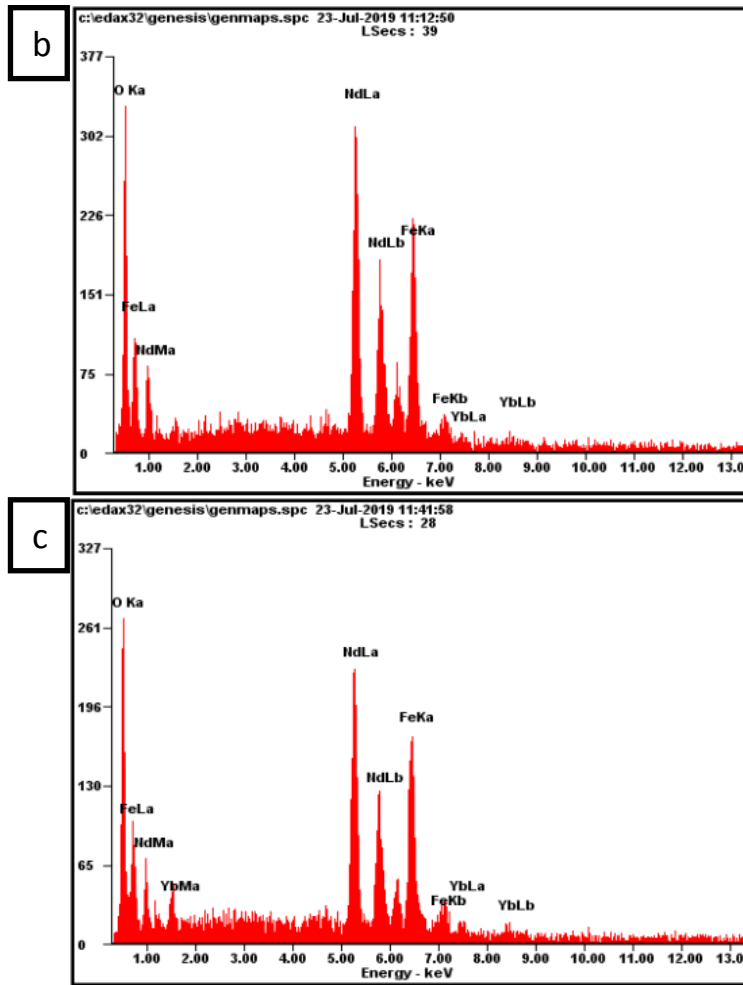


Fig. 3. EDS spectrum of sample Yb_x doped $\text{Nd}_{1-x}\text{FeO}_3$: (a) $x = 0.01$, (b) $x = 0.05$ and (c) $x = 0.10$, respectively.

Based on Fig. 3 shows the presence of Yb ions in the NdFeO_3 sample. The maximum peak of the NdLa spectrum decreases with the increasing YbLa spectrum so that the doping process of the Yb^{3+} ion to the $\text{Nd}_{1-x}\text{FeO}_3$ is successful. Table 2 shows the EDS results for each sample to convince the presence of Yb on the sample and their composition. These results confirmed that the increasing ratio molar of Ytterbium-doped to NdFeO_3 related to Yb amount increase and Nd decreases in elements (wt%), respectively.

Table 2. EDS results of the presence of Yb doped NdFeO_3

X	Elements (wt%)			
	O	Yb	Nd	Fe
0.01	16.66	1.84	60.45	21.05
0.05	16.40	3.15	58.64	21.82
0.10	17.78	8.52	52.18	21.52

4. Conclusion

The Yb-doped NdFeO_3 samples successfully synthesized by using a solid-state reaction method. Data from XRD analysis show that the presence of Yb shifted the 2θ to a lower degree. It can explain because the Yb^{3+} ion, which has less ionic radii than Nd^{3+} ion, successfully substitutes into NdFeO_3 resulting in the distortion on the NdFeO_3

lattice but do not give a significant effect on their morphology as SEM images confirmed. The grain size of the Yb-NdFeO₃ estimated at 0.4 μm. EDS results show the presence of Yb ions in the NdFeO₃ sample

Acknowledgment

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CRedit author statement

Eko Hadi Sujiono contribute to Conceptualization; Formal analysis; Funding acquisition; Methodology; Resources; Software; Supervision; Validation; Visualization; Roles/Writing - original draft; Writing - review & editing.

Vicran Zharvan contribute to performed Data curation; Formal analysis; Investigation; Methodology; Supervision; Validation; Visualization; Roles/Writing - original draft.

Sultra Ade Poetra contribute to performed the experiments; Data curation; Formal analysis; Investigation; Methodology; Visualization.

Muthmainnah Muchtar contribute to the experiments; Data curation; Formal analysis; Investigation; Methodology; Visualization.

Abdi Manab Idris contribute to Data curation; Formal analysis; Investigation; Methodology; Visualization; Roles/Writing - original draft.

M. Yusriadi Dahlan contribute to Data curation; Formal analysis; Investigation; Methodology; Visualization.

Samnur contribute to Funding acquisition; Project administration; Resources; Software; Supervision; Validation; Writing - review & editing.

Declaration of interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

The authors declare the following financial interests/personal relationships which may be considered as potential competing interests:

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Synthesis of Ytterbium-doped Neodymium Ferrite Oxide Using Solid-State Reaction Method and Its Characterization

Eko Hadi Sujiono^{a,*}, Vicran Zharvan^a, Muthmainnah Muchtar^a, Sultra Ade Poetra^a, Abdi Manab Idris^a, Muhammad Yusriadi Dahlan^a, Samnur^a

^aLaboratory of Materials Physics, Department of Physics, Universitas Negeri Makassar, Makassar 90224, Indonesia

Abstract

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Comment [A1]:

Author response point 2:
We disagree with the reviewer. Because the first sentence in the abstract section illustrates what becomes the objective of this research.

Comment [A2]:

Author response point 3:
We agree with the reviewer and we have written more regarding detailed results of this research in the abstract section.

Comment [A3]:

Author response point 4:
We agree with the reviewer and we have explained more detail regarding the novelty of this research in the introduction section.

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2. Experimental method

The Yb doped NdFeO_3 sample was synthesized using the best parameters found by previous research [18-23]. Raw materials of Nd_2O_3 (Strem Chemicals, 99.99%), Yb_2O_3 (Sigma-Aldrich, 99.99%) and Fe_2O_3 (Sigma-Aldrich, 99.99%) were mixed according to stoichiometric calculation and grinded using mortar for 3 hours. That mixed material then calcined using the furnace at temperature 700°C for 4 hours. After that, the temperature increased to 950°C by keeping the temperature constant for 2 hours. The heating then decreased to 475°C and kept it continued for 2 hours. After this thermal process, the material was grinded for 6 hours.

Material that was obtained then calcined at 950°C for 4 hours. After that, the temperature was increased to 1050°C for 2 hours then decreased it to 525°C for 2 hours. All of the processes repeated by varying the Yb content at $x = 0.01$, $x = 0.05$, and $x = 0.10$. The $\text{Yb}_x\text{-Nd}_{1-x}\text{FeO}_3$ samples then characterized using XRD (Rigaku Mini Flex II $\text{CuK}\alpha$, $\lambda = 0.154 \text{ nm}$) to obtain the phase of crystallographic and SEM-EDAX (FEI Inspect S50) to analyze the morphology and elemental composition, respectively.

3. Results and discussion

3.1. Analysis of x-ray diffraction

Characterization results of x-ray diffraction for Yb doped NdFeO_3 samples can show in Fig.1. It shows that the pattern has narrow peaks indicating that the samples are in crystalline form. Further analysis using X'Pert High Score Plus software shows the presence of NdFeO_3 and Nd_2O_3 phases. Those phases give information that samples are polycrystalline material [9] with the highest peak lead to the NdFeO_3 phase at the plane (121) in orthorhombic structure, which is similar to other results [9,16,24]. The presence of Nd_2O_3 phase due to the low temperature during the calcination process [9]. There is no additional peak of impurity observed, which indicates that the samples consist of pure phases [25].

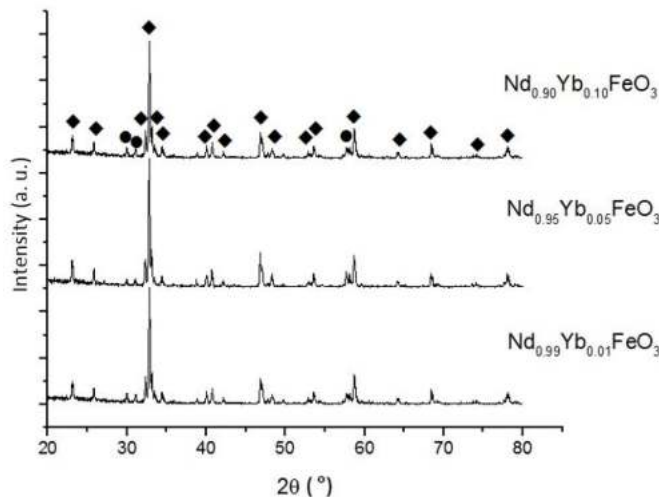


Fig. 1. X-ray diffraction pattern of Yb doped NdFeO_3 ($\blacklozenge = \text{NdFeO}_3$, $\bullet = \text{Nd}_2\text{O}_3$).

Comment [A4]:

Author response point 5:
We agree with the reviewer and we have combined the sentences.

Comment [A5]:

Author response point 6:
The main objective of this research is to synthesis the high quality of Yb doped NdFeO_3 using a solid-state reaction method, so that we focus on the synthesis process. In terms of data analyzing using Rietveld method can be seen in our another article as title "Structure Identification of $\text{Nd}_{1-x}\text{Yb}_x\text{FeO}_3$ ($x=0.01, 0.05$ and 0.10) Using Rietveld Refinement Method".

Further analysis of Yb doped NdFeO₃ describes in Table 1. It shows that the 2θ for peak (121) gradually shift into a lower degree, comparing to undoped NdFeO₃ [26]. This phenomenon can occur due to lattice distortion [24, 27], further analysis of FWHM, 2θ, and crystalline size shown in Table 1.

Table 1. X-ray analysis results of Yb_x doped Nd_{1-x}FeO₃ at plane (121)

X	2θ (degree)	FWHM (degree)	Crystalline Size* (nm)
0.01	32.8294	0.20197	40.56±0.02
0.05	32.8081	0.20279	40.40±0.02
0.10	32.7940	0.20138	40.68±0.02
0**	32.8600	0.20000	67.00

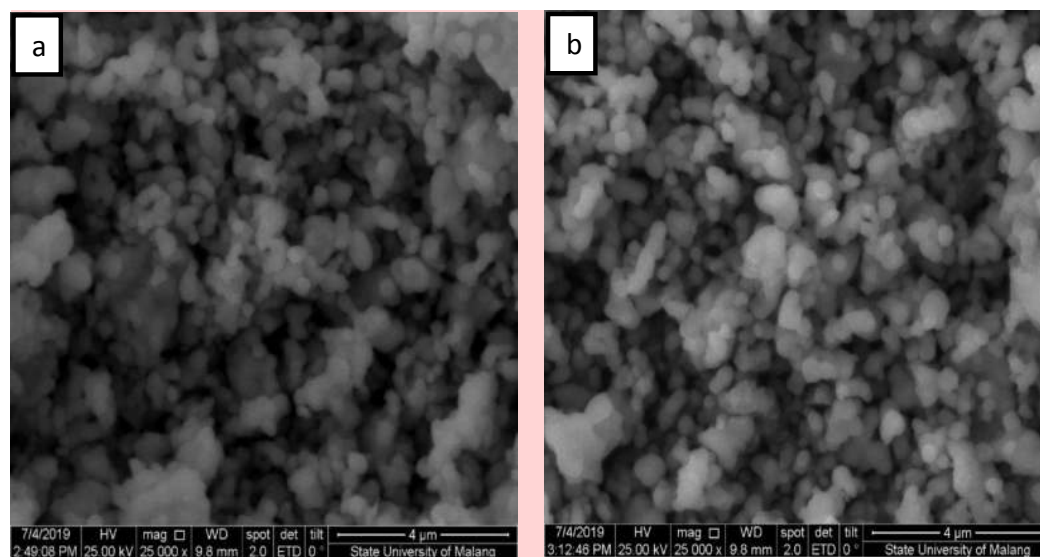
*) Calculated by Debye-Scherrer method

**) Khorasani, et al. [26]

It can be seen that increasing of Yb content gradually shift the 2θ degree into the lower degree and decrease the value of crystalline size from 67 nm as reference value to 40 nm due to the diffusion of ion Yb³⁺ with ionic radii (0.98Å) less than ionic radii of Nd³⁺ (1.11Å) as donor into the lattice of NdFeO₃ as acceptor [28].

3.2. Analysis of SEM-EDAX

The influence of the Yb dopant on the morphology of NdFeO₃ shown in Fig.2.



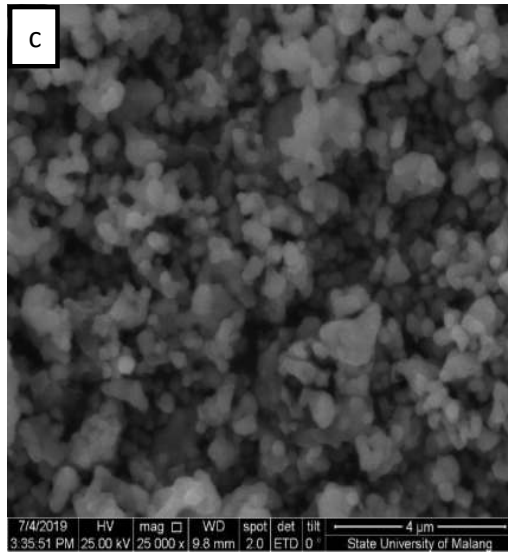
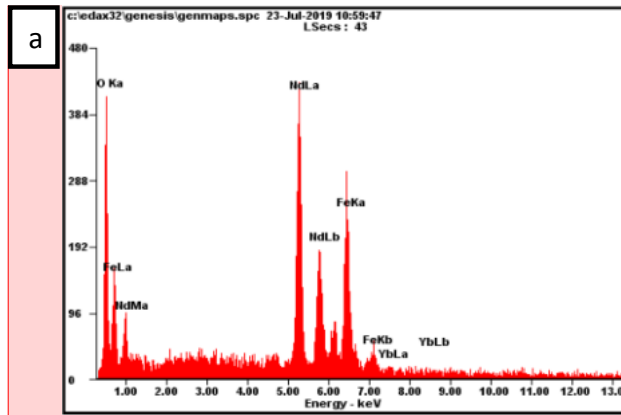


Fig. 2. Morphology of sample $\text{Yb}_x\text{Nd}_{1-x}\text{FeO}_3$: (a) $x = 0.01$, (b) $x = 0.05$ and (c) $x = 0.10$, respectively.

The images show all of the Yb-NdFeO_3 powders are quite uniform in shape and size. It gives information that increasing Yb does not provide a significant effect on morphology. The agglomeration also exists for all powders due to the factor of temperature and mechanical treatment during processes [18]. The particle size also can be estimated an average at $0.4 \mu\text{m}$ from the images. The presence of Yb_x in the $\text{Nd}_{1-x}\text{FeO}_3$ sample can be confirmed based on the results of the EDS spectrum. It can show in Fig. 3.



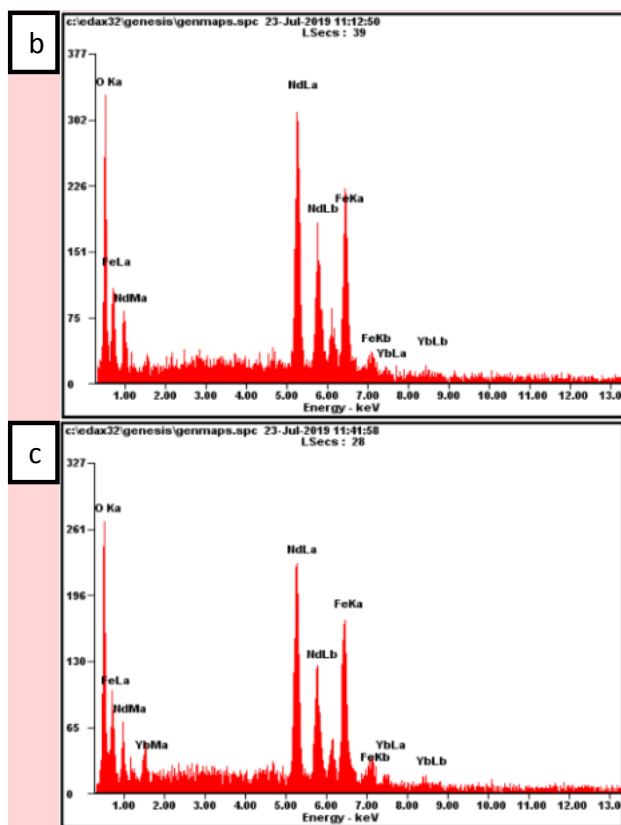


Fig. 3. EDS spectrum of sample Yb_x doped Nd_{1-x}FeO₃: (a) x = 0.01, (b) x = 0.05 and (c) x = 0.10, respectively.

Comment [A6]:
 Author response point 7:
 We agree with the reviewer and we have changed the figures with better resolution quality.

Based on Fig. 3 shows the presence of Yb ions in the NdFeO₃ sample. The maximum peak of the NdLa spectrum decreases with the increasing YbLa spectrum so that the doping process of the Yb³⁺ ion to the Nd_{1-x}FeO₃ is successful. Table 2 shows the EDS results for each sample to convince the presence of Yb on the sample and their composition. These results confirmed that the increasing ratio molar of Ytterbium-doped to NdFeO₃ related to Yb amount increase and Nd decreases in elements (wt%), respectively.

Table 2. EDS results of the presence of Yb doped NdFeO₃

X	Elements (wt%)			
	O	Yb	Nd	Fe
0.01	16.66	1.84	60.45	21.05
0.05	16.40	3.15	58.64	21.82
0.10	17.78	8.52	52.18	21.52

4. Conclusion

The Yb-doped NdFeO₃ samples successfully synthesized by using a solid-state reaction method. Data from XRD analysis show that the presence of Yb shifted the 2θ to a lower degree. It can explain because the Yb³⁺ ion, which has less ionic radii than Nd³⁺ ion, successfully substitutes into NdFeO₃ resulting in the distortion on the NdFeO₃.

lattice but do not give a significant effect on their morphology as SEM images confirmed. The grain size of the Yb-NdFeO₃ estimated at 0.4 μm. EDS results show the presence of Yb ions in the NdFeO₃ sample

Acknowledgment

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Comment [A7]:

Author response point 1:
In terms of grammatical errors have been improved. For english improvement as a whole can be seen in the last revision of the manuscript.

Synthesis of Ytterbium-doped Neodymium Ferrite Oxide Using Solid-State Reaction Method and Its Characterization

Eko Hadi Sujiono^{a,*}, Vicran Zharvan^a, Muthmainnah Muchtar^a, Sultra Ade Poetra^a, Abdi Manab Idris^a, Muhammad Yusriadi Dahlan^a, Samnur^a

^aLaboratory of Materials Physics, Department of Physics, Universitas Negeri Makassar, Makassar 90224, Indonesia

Abstract

The Yb doped NdFeO₃ using a solid-state reaction method has successfully synthesized. In this paper, Nd_{1-x}Yb_xFeO₃ samples were synthesized by varying the molar ratio of Yb at x = 0.01, x = 0.05, and x = 0.10 using solid-state reaction with two routes of heat treatment processes. Results of X-ray diffraction show that all samples have an orthorhombic structure with two phases: NdFeO₃ as a major phase and Nd₂O₃ as a minor phase. FWHM value for a maximum peak is 0.2°, so the estimated crystal size is 40 nm, with the dominant peak corresponding to hkl (121). Morphology properties used SEM Image shows grain size of all sample estimated at 0.4 μm. The presence of Yb is quantitatively confirmed based on the EDS result.

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Peer-review under responsibility of the scientific committee of The 7th International Conference on Advanced Materials Science and Technology 2019, 7th ICAMST.

Keywords: Ytterbium; NdFeO₃; Solid State Reaction Method; Structure; Morphology

5. Introduction

ReFeO₃ elements that (Re: rare-earth elements) are known as the rare-earth orthoferrites having orthorhombic structure derived from a perovskite structure [1], and they have attracted much interest due to their properties such as magnetic and magneto-optic [2]. Among these ReFeO₃ elements, NdFeO₃ material useful as a raw material of many applications such as gas sensors, fuel cells, and catalyst material gas sensors [3,4,5,6]. The preparation of NdFeO₃ material can realize by several methods [7,8]. The solid-state reaction method has used because this method is cheap and easy to implement. The high purity and crystallinity materials also can be achieved by this method [9].

Present-day, many researchers are working on ReFeO₃ to obtain ideal materials for Adsorbent [10], photocatalytic Material [11], solid-state devices, and gas sensors application [12]. RFeO₃ has a characteristic feature of the presence of two magnetic subsystems of Re³⁺ and Fe³⁺. The interactions of Fe-Fe, Re-Fe, and Re-Re lead to a few interesting phenomena. ReFeO₃ has the special characteristic of spin reorientations $\Gamma_4(G_x F_z) \rightarrow \Gamma_{24}(G_x F_x) \rightarrow \Gamma_2(G_x F_x)$ [13]. ReFeO₃ shows interesting gas sensitivity properties, gas sensors based on LaFeO₃ show good results to detect ethanol, acetone, H₂S, CO, and NO₂ [14,15]. In the few past years, NdFeO₃ material as a gas sensor to detect C₂H₅OH [16]. Gas sensors based on ReFeO₃ are developed to detect acetone [17]. The properties of ReFeO₃ elements for gas sensors such as their response and selectivity increased by doping to another oxide material [18] but less information using Ytterbium (Yb) as a dopant element for NdFeO₃.

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E-mail address: e.h.sujiono@unm.ac.id

Comment [A8]:

Author response:
In terms of the suggested title, we agree with the reviewer and we have changed the title of this article with Synthesis of Ytterbium-doped Neodymium Ferrite Oxide Using Solid-State Reaction Method and Its Characterization.

In this paper, presented produced the Ytterbium (Yb_x) doped $\text{Nd}_{1-x}\text{FeO}_3$ samples by using a solid-state reaction method by varying the molar ratio of Yb_x from $x = 0.01$, $x = 0.05$ and $x = 0.10$, respectively. All samples then characterized to obtain their crystal structure with qualitative analysis, morphology, and elemental composition properties.

6. Experimental method

The Yb doped NdFeO_3 sample was synthesized using the best parameters found by previous research [18-23]. Raw materials of Nd_2O_3 (Strem Chemicals, 99.99%), Yb_2O_3 (Sigma-Aldrich, 99.99%) and Fe_2O_3 (Sigma-Aldrich, 99.99%) were mixed according to stoichiometric calculation and grinded using mortar for 3 hours. That mixed material then calcined using the furnace at temperature 700°C for 4 hours. After that, the temperature increased to 950°C by keeping the temperature constant for 2 hours. The heating then decreased to 475°C and kept it continued for 2 hours. After this thermal process, the material was grinded for 6 hours.

Material that was obtained then calcined at 950°C for 4 hours. After that, the temperature was increased to 1050°C for 2 hours then decreased it to 525°C for 2 hours. All of the processes repeated by varying the Yb content at $x = 0.01$, $x = 0.05$, and $x = 0.10$. The $\text{Yb}_x\text{-Nd}_{1-x}\text{FeO}_3$ samples then characterized using XRD (Rigaku Mini Flex II $\text{CuK}\alpha$, $\lambda = 0.154 \text{ nm}$) to obtain the phase of crystallographic and SEM-EDAX (FEI Inspect S50) to analyze the morphology and elemental composition, respectively.

7. Results and discussion

7.1. Analysis of x-ray diffraction

Characterization results of x-ray diffraction for Yb doped NdFeO_3 samples can show in Fig.1. It shows that the pattern has narrow peaks indicating that the samples are in crystalline form. Further analysis using X'Pert High Score Plus software shows the presence of NdFeO_3 and Nd_2O_3 phases. Those phases give information that samples are polycrystalline material [9] with the highest peak lead to the NdFeO_3 phase at the plane (121) in orthorhombic structure, which is similar to other results [9,16,24]. The presence of Nd_2O_3 phase due to the low temperature during the calcination process [9]. There is no additional peak of impurity observed, which indicates that the samples consist of pure phases [25].

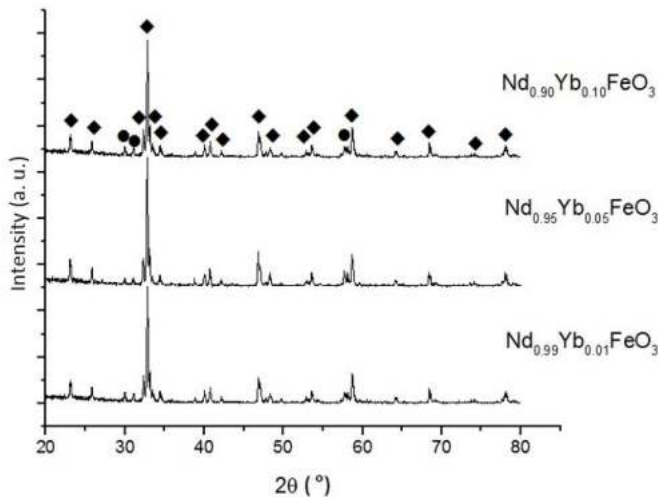


Fig. 1. X-ray diffraction pattern of Yb doped NdFeO_3 (◆ = NdFeO_3 , ● = Nd_2O_3).

Further analysis of Yb doped NdFeO₃ describes in Table 1. It shows that the 2θ for peak (121) gradually shift into a lower degree, comparing to undoped NdFeO₃ [26]. This phenomenon can occur due to lattice distortion [24, 27], further analysis of FWHM, 2θ, and crystalline size shown in Table 1.

Table 1. X-ray analysis results of Yb_x doped Nd_{1-x}FeO₃ at plane (121)

X	2θ (degree)	FWHM (degree)	Crystalline Size* (nm)
0.01	32.8294	0.20197	40.56±0.02
0.05	32.8081	0.20279	40.40±0.02
0.10	32.7940	0.20138	40.68±0.02
0**	32.8600	0.20000	67.00

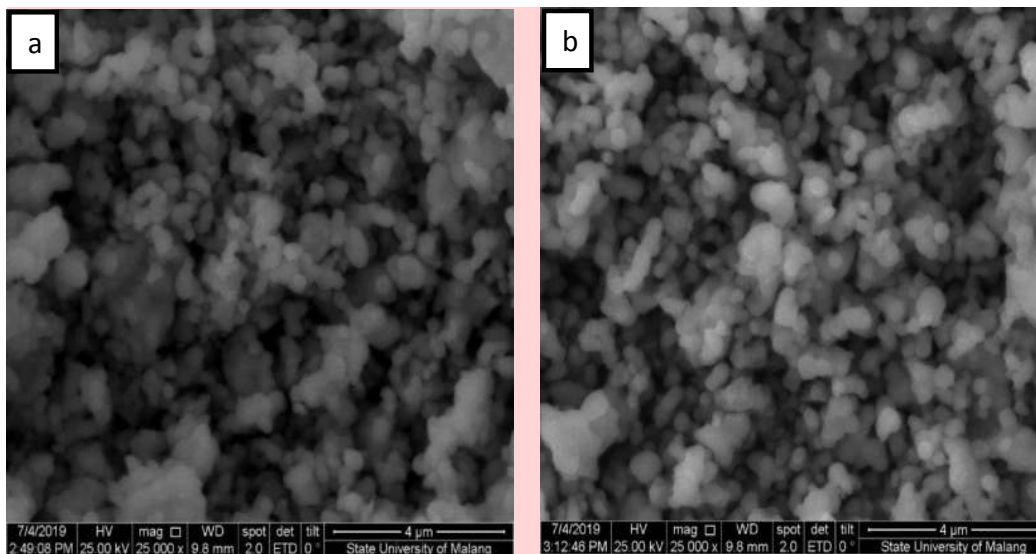
*) Calculated by Debye-Scherrer method

**) Khorasani, et al. [26]

It can be seen that increasing of Yb content gradually shift the 2θ degree into the lower degree and decrease the value of crystalline size from 67 nm as reference value to 40 nm due to the diffusion of ion Yb³⁺ with ionic radii (0.98Å) less than ionic radii of Nd³⁺ (1.11Å) as donor into the lattice of NdFeO₃ as acceptor [28].

3.2. Analysis of SEM-EDAX

The influence of the Yb dopant on the morphology of NdFeO₃ shown in Fig.2.



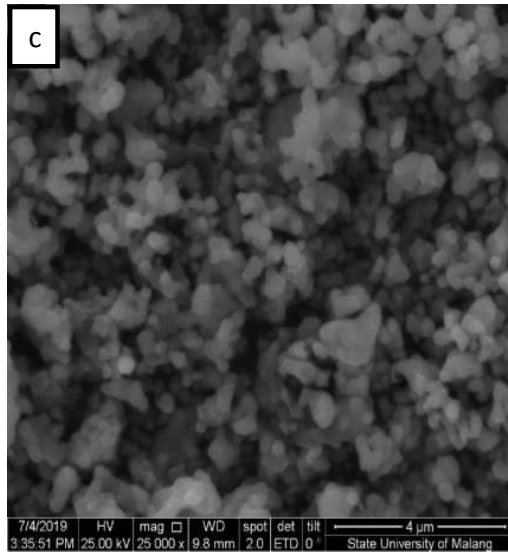
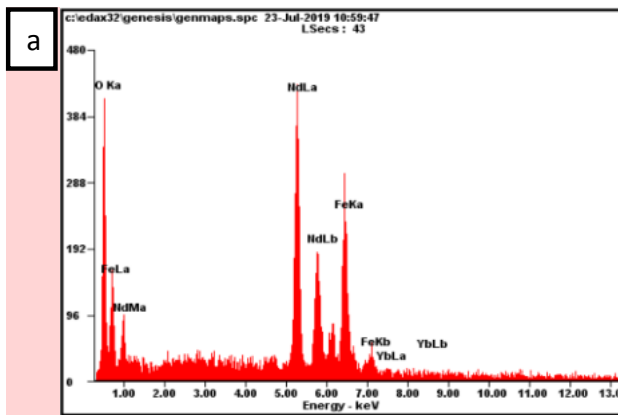


Fig. 2. Morphology of sample Yb_x doped Nd_{1-x}FeO₃: (a) x = 0.01, (b) x = 0.05 and (c) x = 0.10, respectively.

The images show all of the Yb-NdFeO₃ powders are quite uniform in shape and size. It gives information that increasing Yb does not provide a significant effect on morphology. The agglomeration also exists for all powders due to the factor of temperature and mechanical treatment during processes [18]. The particle size also can be estimated an average at 0.4 μm from the images. The presence of Yb_x in the Nd_{1-x}FeO₃ sample can be confirmed based on the results of the EDS spectrum. It can show in Fig. 3.



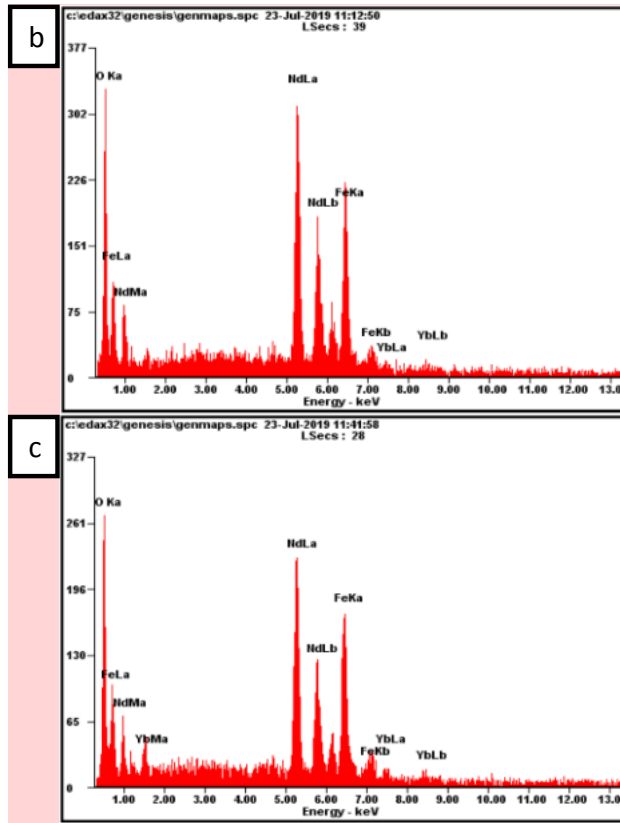


Fig. 3. EDS spectrum of sample Yb_x doped Nd_{1-x}FeO₃: (a) x = 0.01, (b) x = 0.05 and (c) x = 0.10, respectively.

Based on Fig. 3 shows the presence of Yb ions in the NdFeO₃ sample. The maximum peak of the NdLa spectrum decreases with the increasing YbLa spectrum so that the doping process of the Yb³⁺ ion to the Nd_{1-x}FeO₃ is successful. Table 2 shows the EDS results for each sample to convince the presence of Yb on the sample and their composition. These results confirmed that the increasing ratio molar of Ytterbium-doped to NdFeO₃ related to Yb amount increase and Nd decreases in elements (wt%), respectively.

Table 2. EDS results of the presence of Yb doped NdFeO₃

X	Elements (wt%)			
	O	Yb	Nd	Fe
0.01	16.66	1.84	60.45	21.05
0.05	16.40	3.15	58.64	21.82
0.10	17.78	8.52	52.18	21.52

8. Conclusion

The Yb-doped NdFeO₃ samples successfully synthesized by using a solid-state reaction method. Data from XRD analysis show that the presence of Yb shifted the 2θ to a lower degree. It can explain because the Yb³⁺ ion, which has less ionic radii than Nd³⁺ ion, successfully substitutes into NdFeO₃ resulting in the distortion on the NdFeO₃

lattice but do not give a significant effect on their morphology as SEM images confirmed. The grain size of the Yb-NdFeO₃ estimated at 0.4 μm. EDS results show the presence of Yb ions in the NdFeO₃ sample

Acknowledgment

This research funded by Directorate research and Community Services, Directorate General of Research and Development, Ministry of Research, Technology and Higher Education, Republic of Indonesia, under research scheme of Basic Research Program fiscal year of 2019.

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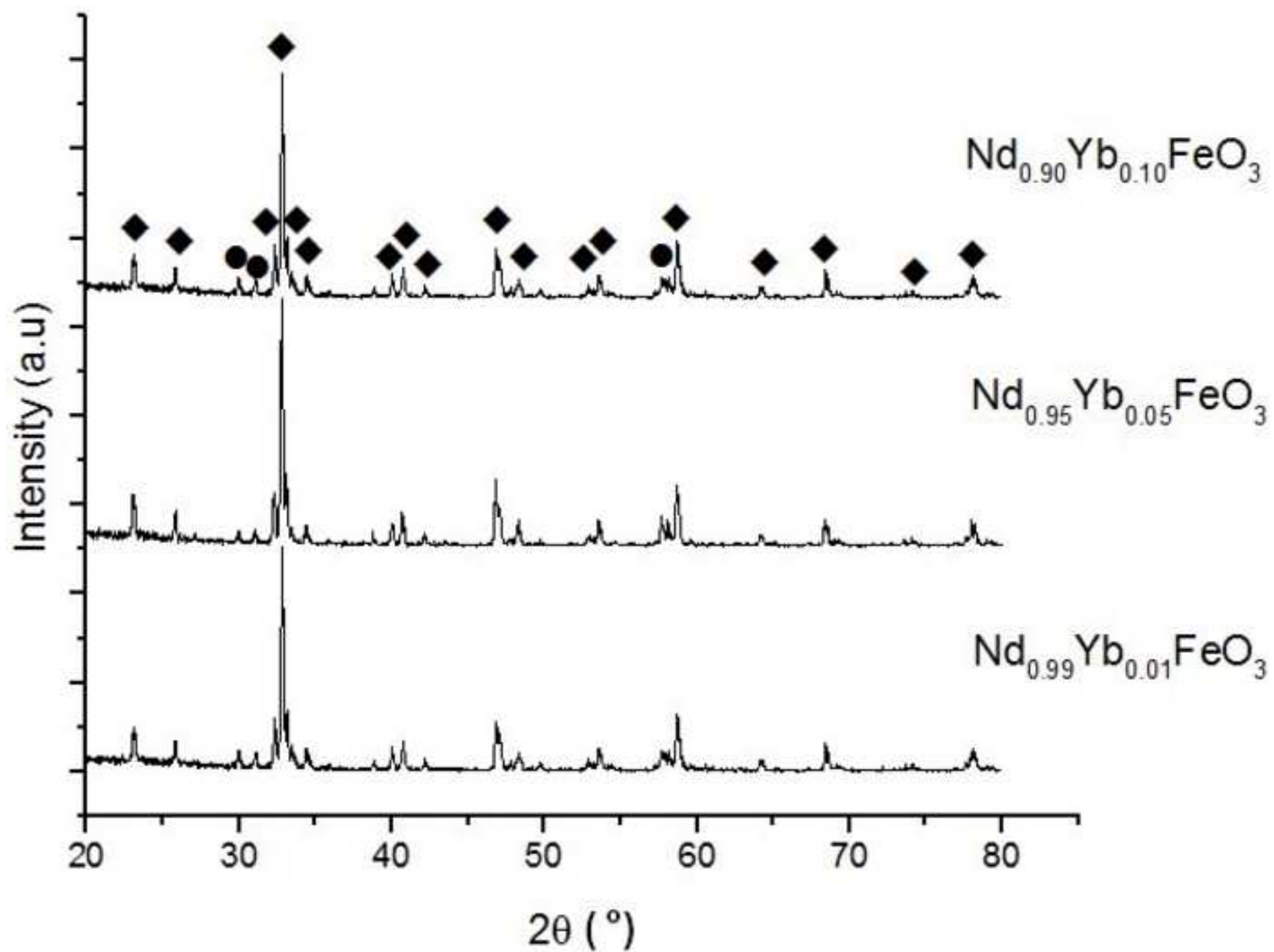
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Comment [A9]:

Author response:

In terms of grammatical and typographical errors, we agree with the reviewer and for improvements in English as a whole can be seen in the last revision of the manuscript.

*Figure 1
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*Figure 2a

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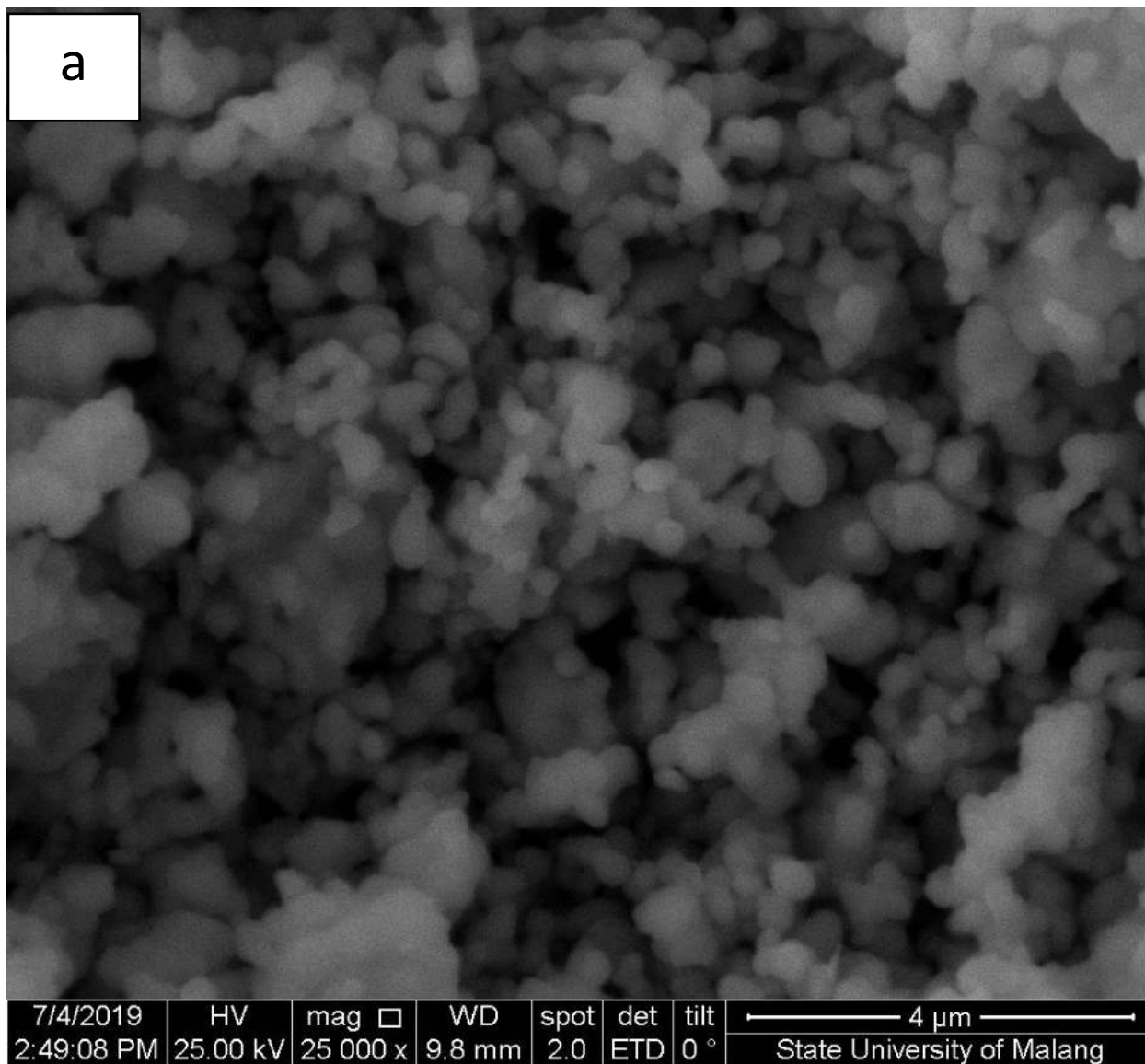
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*Figure 2



*Figure 2b

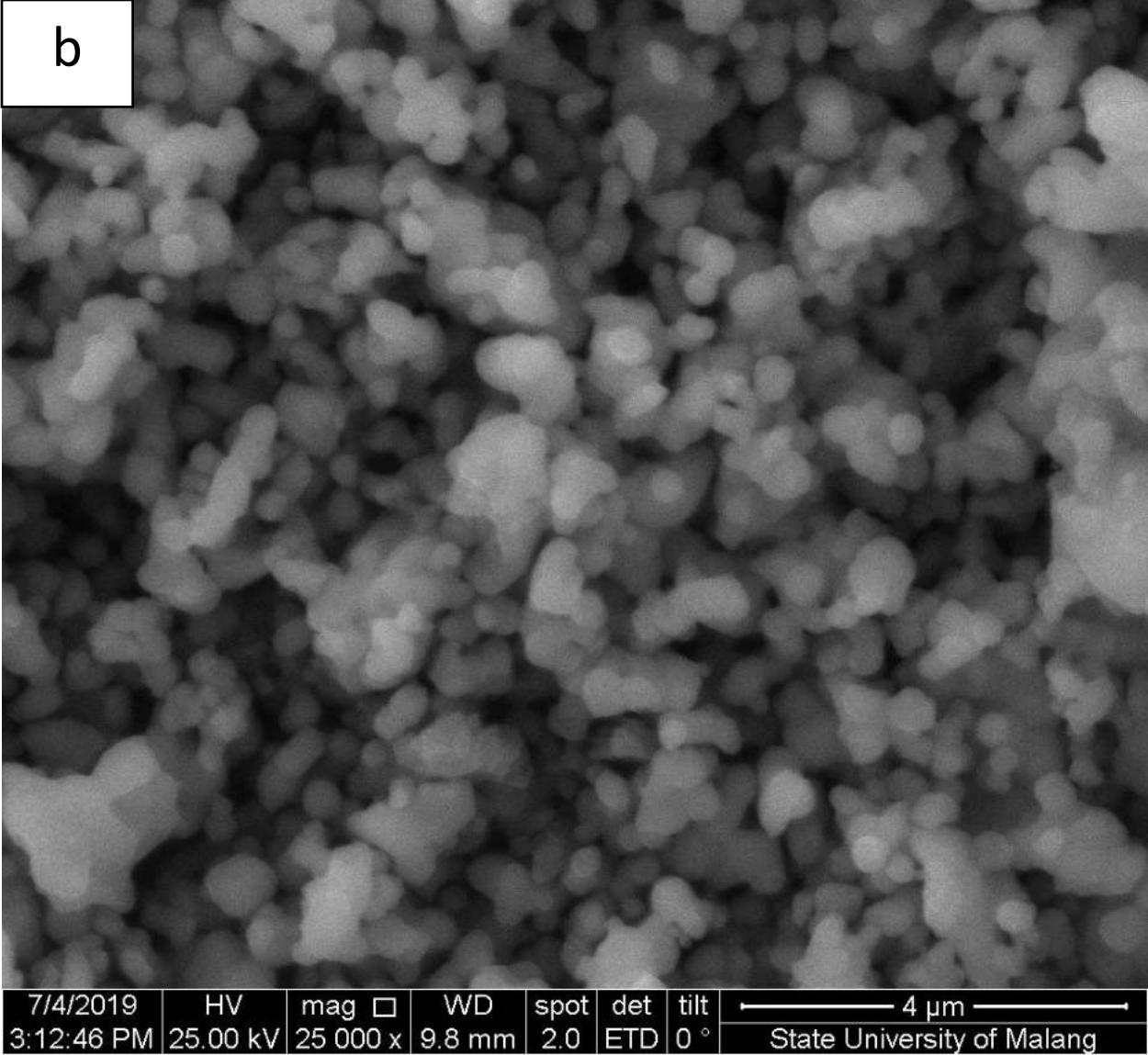
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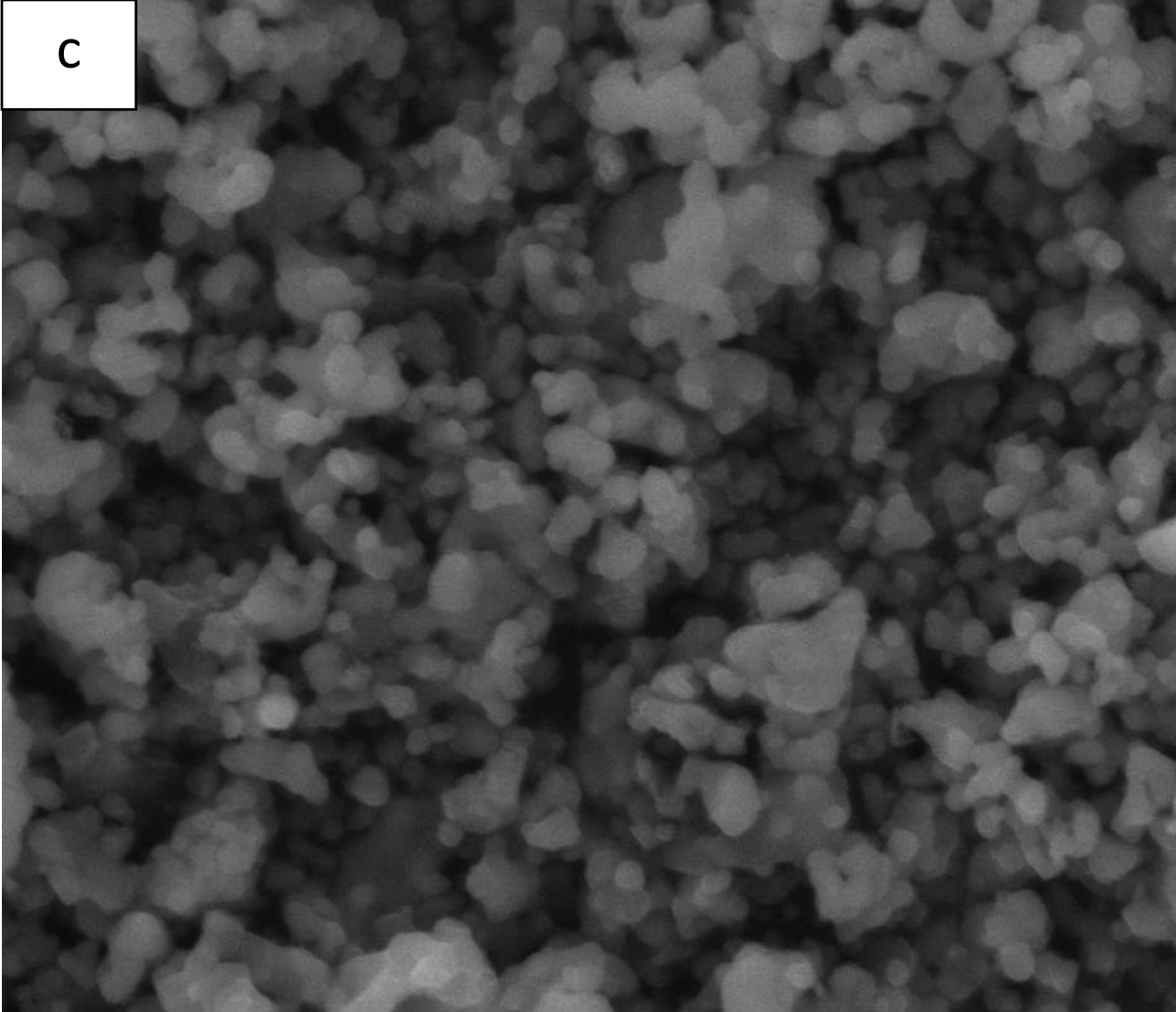
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*Figure 3a

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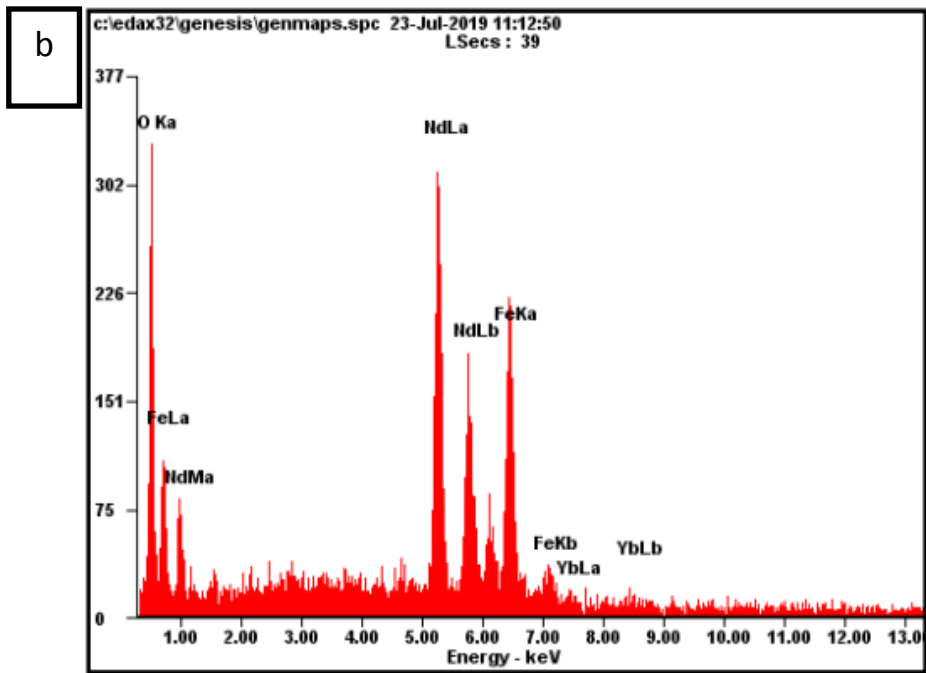
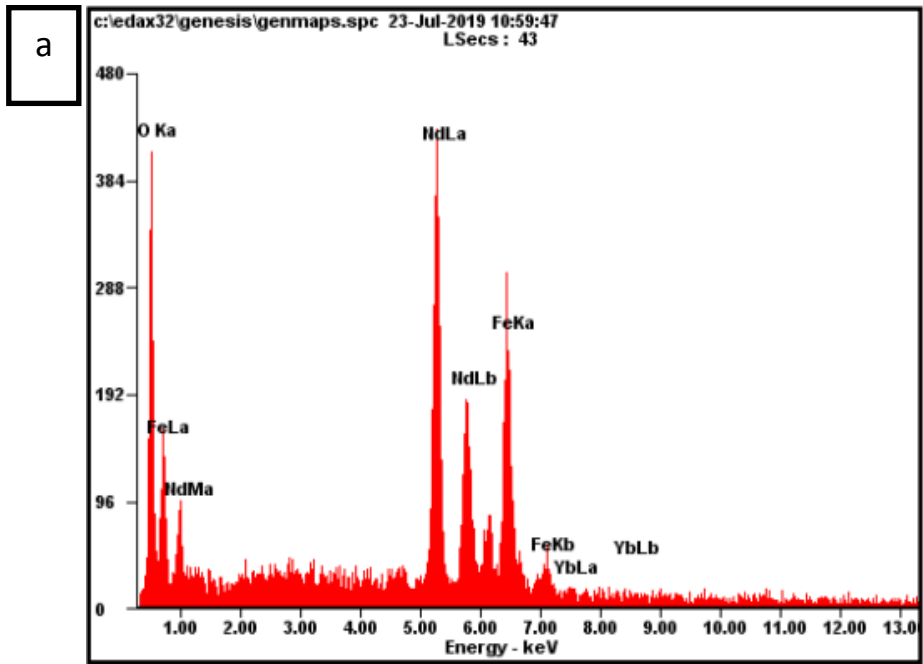
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*Figure 3



*Figure 3c

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