



STRUCTURAL ANALYSIS OF EFFECT OF DEPOSITION PARAMETERS OF COBALT DOPED FERRIC-OXIDE AND ZINC DOPED FERRIC-OXIDE IN COMPARISON WITH UNDOPED FERRIC-OXIDE USING NOVEL WET CHEMICAL SYNTHESIS

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Abstract

Aim: The aim of this work is to compare the structural analysis of zinc doped ferric-oxide, cobalt doped ferric-oxide and pure ferric-oxide components prepared by using novel wet chemical synthesis.

Materials and Methods: The research was made on the comparison of the structural analysis of the ferric-oxide and dopants as cobalt & zinc. The sample size of pure ferric-oxide, cobalt doped ferric-oxide and zinc doped ferric-oxide is 234 each, and a total sample size of 702. These samples are calculated using the clincale calculator which has a pretest power of 80 % and error correction of 0.05.

Results: As the dopants were added to the pure ferric-oxide the area of doped components, that is zinc doped ferric-oxide and cobalt doped ferric-oxide area, had been increased. The area of pure ferric-oxide components is noted as 49.599 %, the total surface area is 345.952 μm^2 , the surface area of zinc doped ferric-oxide component is 54.586 %, had a total area of 389.014 μm^2 and the area of cobalt doped ferric-oxide is noted as 52.037 % and the total surface area is noted as 366.39 μm^2 . The significance of pure iron oxide, cobalt doped iron oxide and zinc doped iron oxide are 1, 0.035 and 0.75 respectively

Conclusion: The area of the cobalt doped ferric-oxide is significantly better compared to pure ferric-oxide and zinc doped ferric-oxide.

Keywords: Scanning electron microscope, Pure ferric-oxide, Zinc doped ferric-oxide, Cobalt doped ferric-oxide, Novel wet chemical synthesis.

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1. Introduction

Nanoparticles are sub micron made of inorganic or organic materials, which have many novel properties compared with the bulk materials (Magalhães-Ghiotto et al. 2022). On this basis, magnetic Nanoparticles have many unique magnetic properties such as superparamagnetic, high coercivity, low Curie temperature, high magnetic susceptibility, etc. The three main types of ferric-oxide nanoparticles are FeO (Ferrous oxide), Fe₂O₃ (Ferric oxide) and Fe₃O₄ (Iron dioxide). They are considered to be important materials due to their catalytic activity, biocompatibility, low-cost, non toxicity and environmentally friendly nature (Kielbik et al. 2021). Among the encouraging photoanode materials, hematite has involved much consideration for PEC (PhotoElectroChemical) water splitting owing to its extensive capability of light adsorption, outstanding constancy under corrosive operative circumstances, less band gap, ecologically benign influence (Chatterjee 2012). Moreover, it has 16.8 % of the theoretical solar-to-hydrogen conversion efficiency, high photocurrent. The considerably enhanced PEC activity is ascribed to the higher conductivity, enhanced interfacial charge transfer at the surface of hematite and the synergistic effect between two metal dopants (Morkoç and Özgür 2008). Ferrites are relatively more stable and oxidized. The properties of the magnetic nano properties depend upon their chemical structure and their methods of preparation as novel wet chemical synthesis. These ferrite compounds have interesting properties as structural analysis, chemical and optical (Ahlawat 2020). Cobalt ferrites attract much attention due to their unique and excellent properties. The applications of ferrites are high saturation magnetization, high coercivity, magnetic anisotropy, good chemical stability and catalytic activity. The magnetic and electrical properties of ferrites strongly depend on cation distribution (Molakeri et al. 2018). The total number of articles that were published related to this topic are in google scholar (a total of 16000 documents) were published till now, in IEEE xplore 60 articles and in science direct we had 20 articles explaining our requirements articles publication were done from 2017 to 2021 in which all the article preparation the compound preparation processes was all with sophisticated systems which were of very high cost and the component prepared will also be of less quantity but wet chemical synthesis which is of low cost and quantity can be high (Mukhiya et al. 2021). Magnetic Nanoparticles are of great interest for researchers from a broad range of disciplines, including magnetic fluids, data storage, catalysis,

and bioapplications (Mohamad Nor et al. 2022). Some of the best reviewed articles were function gated ferric-oxide coating procedures on ferric using induction heat treatment (Fomin 2019) which had been cited by 23 members. Nanostructures have been one of the most frequently used active materials for energy storage applications. Among a wide variety of copious and eco-friendly renewable carbon reservoirs, biomass (Khatibani and Shabankhah 2021).

Our institution is keen on working on latest research trends and has extensive knowledge and research experience which resulted in quality publications (Rinesh et al. 2022; Sundararaman et al. 2022; Mohanavel et al. 2022; Ram et al. 2022; Dinesh Kumar et al. 2022; Vijayalakshmi et al. 2022; Sudhan et al. 2022; J. A. Kumar et al. 2022; Sathish et al. 2022; Mahesh et al. 2022; Yaashikaa et al. 2022). In the existing methods for the analysis of the nanomaterials area, Dynamic Light Scattering (DLS), Disc Centrifugation (DC) are used for measurement. In which the measurement of surface area takes time but in this research Scanning Electron Microscope (SEM) is used for the analysis of surface area covered.

2. Material and Methods

This research is carried out in Nanomaterials and Crystal Growth Laboratory, Department of Electronic and Communication Engineering, Saveetha School of Engineering, Saveetha Institute of Medical and Technical Sciences, Chennai. This research is based on the preparation of pure iron-oxide, cobalt doped iron-oxide and zinc doped iron-oxide and analyzing the surface area using a Scanning Electron Microscope (SEM). The work was commenced and divided into three different groups as pure iron-oxide, cobalt doped iron-oxide and zinc doped iron-oxide (Mueller et al. 2016). The pre test analysis is kept at 80 % with an error correction of 0.05 and the sample size of pure iron-oxide and cobalt doped iron-oxide was 234 each. The total sample size of doped and undoped samples was 702. This was calculated using a clincalc calculator app.

Preparation of Ferric-Oxide

The novel wet chemical synthesis and annealing processes were used for the preparation of nanopowders of pure ferric-oxide. 2.706 gms of ferric-oxide of 0.1 M (Molar) of 100 ml solution was prepared using the formula $(MW \times M \times 100) / 1000$ where MW is the molecular weight of ferric chloride and M is molarity. The obtained output is mixed using a magnetic stirrer for about 30 minutes. NaOH was taken in the 100 ml solution into a pipette. NaOH solution was

added to the ferric chloride solution drop by drop using a pipette. The pH is said to change from acidic to alkaline when the color of the mixture changes from clear to cloudy. This is followed by the washing process and subsequently the filtration using whatman Paper to get ferric-oxide. The colloidal thick viscous solution was heated in a petri dish at about 80 °C which causes it to change from liquid to solid. The solid powder thus obtained is heated for about 2 hours at a temperature of 500 °C in a muffle furnace and annealed till the temperature reaches 35 °C (Kahali, Montazer, and Kamali Dolatabadi 2022).

Preparation of Cobalt Doped Ferric-Oxide

The cobalt doped ferric-oxide was prepared by using a molarity of 0.1 M for a 10 ml distilled water. The weight of the cobalt chloride was determined as 0.237 gms by considering the molecular weight as 237.93 gms/mol. The cobalt chloride is mixed with 100 ml of ferric chloride solution. Add NaOH solution drop by drop to the cobalt mixed ferric-oxide solution using a stirrer the particles are mixed properly (Kahali, Montazer, and Kamali Dolatabadi 2022; Fang et al. 2003). The colloidal arrangement of cobalt doped ferric-oxides is red in color which indicates the formation. A similar cycle is repeated in preparation of pure ferric-oxide.

Preparation Of Zinc Doped Ferric-Oxide

The zinc doped ferric-oxide was ready with a molarity of 0.1 M for 10 ml arrangement. The heaviness of the cobalt chloride was determined as 0.217 gms. To the 100 ml ferric chloride arrangement the weighted zinc chloride is blended and smelling salts is additionally added drop by drop to it blended utilizing an attractive stirrer and the cycles referenced in the above for the planning of unadulterated ferric-oxide was rehashed to frame zinc doped ferric-oxide which was of reddish shady arrangement (Satula et al. 2008). Warming cycles were finished involving a stifle heater for around 2 hours at a temperature of 500 °C and annealed until the temperature drops to 35 °C.

SEM (Scanning Electron Microscope) is used for images of the components with different magnification then these images are kept in ImageJ software which gives all the particle sizes and this software also gives the total surface area and average size too.

Statistical Analysis

The Statistical software noted by SPSS (Statistical Package for Social Sciences) in which the dependent variable is the area and the independent variable is the count of the component, in order to measure. The significance of pure ferric-oxide,

cobalt doped ferric-oxide and zinc doped ferric-oxide is 1, 0.035 and 0.75 respectively. By the SPSS software the obtained values are mean, standard deviation and significance of doped and undoped samples by analyzing the one way anova and descriptive means (Salcedo and McCormick 2020).

3. Results

Novel wet chemical synthesis is used for the preparation of ferric-oxide, cobalt doped ferric-oxide and zinc doped ferric-oxide. Colloidal solution of pure iron-oxide stabilized using NaOH solution can be seen in Fig. 1. (a). In Fig. 2. (a) colloidal solution can be seen for cobalt doped ferric-oxide and Fig. 3. (a) represents the colloidal solution of zinc doped ferric-oxide. The annealed powder of iron-oxide can be seen in Fig. 1. (b), the cobalt doped iron oxide annealed powder can be seen in Fig. 2. (b) and zinc doped iron oxide annealed powder can be seen in Fig. 3. (b).

Pure ferric-oxide SEM (Scanning electron microscope) image of different magnification at 5 kx taken at an electron high tension of 30 KV can be seen in Fig. 4. Cobalt doped ferric-oxide Scanning Electron Microscope (SEM) image of magnification at 10.0 kx taken at an electron high tension of 30 KV is seen in Fig. 5. Zinc doped ferric-oxide SEM (Scanning Electron Microscope) image of magnification 1.00 kx taken at an electron high tension of 30 KV is seen in Fig. 6. In Fig. 7, the threshold values of the pure iron-oxide, cobalt doped iron-oxide and zinc doped iron-oxide can be seen, which are obtained from ImageJ software.

The mean surface area of the pure ferric-oxide, cobalt doped ferric-oxide and zinc doped ferric-oxide are 1.7298 μm^2 , 1.9226 μm^2 and 1.8314 μm^2 respectively. The standard deviation of pure ferric-oxide, cobalt doped ferric-oxide and zinc doped ferric-oxide is 13.9299, 22.35245 and 14.3965 respectively as shown in Table 1. The comparison between the three groups of pure ferric-oxide, cobalt doped ferric-oxide and zinc doped ferric-oxide with the significance of 1, 0.035 and 0.75 and standard error between three groups is 1.73295 as shown in Table 2. Comparison of the surface area of pure ferric-oxide, cobalt doped ferric-oxide and zinc doped ferric-oxide using ImageJ software are shown in Table 3, the total area of pure ferric-oxide, cobalt doped ferric-oxide and zinc doped ferric-oxide is 345.952 μm^2 , 389.014 μm^2 and 366.391 μm^2 respectively. The average size of three groups are 1.730 μm , 1.5322 μm and 1.831 μm and the mean of all the three is 255. In Table 4, the surface area of iron-oxide, cobalt doped iron-oxide and zinc doped iron-oxide is analyzed with 60 random samples out of 702 samples and the

corresponding threshold values are 49.94, 43.41 and 37.15 respectively.

Figure 8 the bar graph representation of iron oxide, cobalt doped iron oxide and zinc doped iron oxide are seen in which the zinc doped iron oxide produces variable results and cobalt doped iron oxide produces consistent results.

4. Discussion

From the prepared component of iron-oxide, zinc and cobalt doped iron-oxide, it is understood that the mean surface area of iron-oxide, zinc and cobalt doped iron-oxide states that cobalt doped iron-oxides has less surface area which is analyzed using Scanning Electron Microscope (SEM) and ImageJ software.

In a research by (Sekar et al. 2019) to synthesize the nanoparticles they used disc diffusion method. But in this research to synthesize the nanomaterials novel wet chemical synthesis was used. In work experience of (M. Kumar et al. 2019) zinc doped ferric-oxide has higher surface area than ferric-oxide which is due to the increase in the grain size from 1.25 μm to 3.24 μm . The surface area of the iron oxide is low and doped nanomaterials high due to the dopants occupying the area there is an increase in the grain size like 2.67 nm to 3.86 nm (Du, Wang, and Wu 2018). The work by (Anjum et al. 2017) cobalt doped ferric-oxide nanoparticles are prepared by using co precipitation method to find surface area of components. Which shows the doped component has a high surface area. In a work by (Haider et al. 2022) they heat at the temperature of 700 $^{\circ}\text{C}$ for 4 hrs to get pure nanoparticles. The temperature used in this research to get pure nanomaterials was 500 $^{\circ}\text{C}$ for 2 hours only.

The limitations of the research is that iron-oxide, cobalt doped iron-oxide and zinc doped iron-oxide consist of some impurities like chlorine ions due to which its purity reduced, so the future scope of this project is to prepare pure components without any impurities by changing the washing technique.

5. Conclusion

The surface area of Pure ferric-oxide, cobalt doped ferric-oxide and zinc doped ferric-oxide is prepared by using a novel wet chemical synthesis method using Scanning Electron Microscopy (SEM). The area of pure ferric-oxide components is noted as 49.599 %, the total surface area is 345.952 μm^2 , the surface area of zinc doped ferric-oxide component is 54.586 %, a total area of 389.014 μm^2 and the area of cobalt doped ferric-oxide is noted as 52.037 %, the total surface area is noted as 366.39 μm^2 . Cobalt doped ferric-oxide components possess

better significant surface area than other two components.

Declaration

Conflict of Interest

No conflict of interests in this manuscript.

Authors Contribution

Author PA was involved in data collection, data analysis, and manuscript writing. Author SRM was involved in conceptualization, data validation and critical review of manuscript.

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Tables and Figures

Table 1. Comparison of surface area of pure ferric-oxide, zinc doped ferric-oxide and cobalt doped ferric-oxide with mean surface area of 1.7298, 1.9226 and 1.8314 respectively. The standard deviations of pure ferric-oxide, zinc doped ferric-oxide and cobalt doped ferric-oxide are 13.929, 22.35245 and 14.3965 respectively.

Descriptive statistics						
Group		N	Minimum	Maximum	Mean	Std. Deviation
Ferric-oxide	Area	200	0.00	175.35	1.7298	13.9299

	Groups	200	1.00	1.00	1.00	0
	Valid N	200	0	0	0	0
Cobalt doped ferric-oxide	Area	200	0.00	315.10	1.9226	22.35245
	Groups	200	2.00	2.00	2.00	0
	Valid N	200	0	0	0	0
Zinc doped ferric-oxide	Area	200	0.00	176.99	1.8314	14.3965
	Groups	200	3.00	3.00	3.00	0
	Valid N	200	0	0	0	0

Table 2. The comparison of two consecutive groups with significance of 1 and the standar error is 1.73295

Post Hoc Test Multiple Comparisons						
Group number(I)	Group number(J)	Mean difference(I-J)	Std.Error	Sig	Lower bound	Upper Bound
Pure ferric-oxide	Cobalt doped ferric-oxide	-0.19280	1.73295	0.035	-4.3532	3.9676
	Zinc doped ferric-oxide	-0.10161	1.73295	1	-4.2620	4.0588
Cobalt doped ferric-oxide	Pure ferric-oxide	0.19280	1.73295	0.035	-3.9676	4.3532
	Zinc doped ferric-oxide	0.09119	1.73295	0.75	-4.0692	4.2516
Zinc doped ferric-oxide	Pure ferric-oxide	0.10161	1.73295	1	-4.0588	4.2620
	Cobalt doped ferric-oxide	-0.09119	1.73295	0.75	-4.2516	4.0692

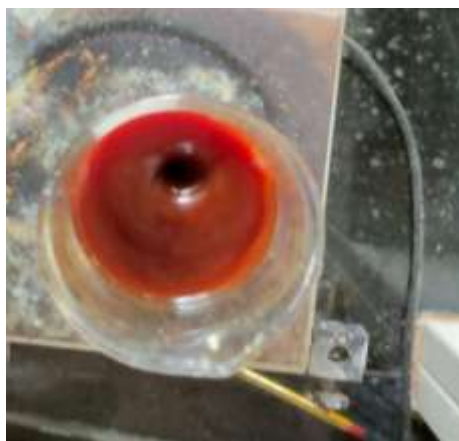
Table 3. Comparison of the surface area of pure ferric-oxide, cobalt doped ferric-oxide and zinc doped ferric-oxide using ImageJ software. The total area of pure ferric-oxide, cobalt doped ferric-oxide and zinc doped ferric-oxide is 345.952 μm^2 , 389.014 μm^2 and 366.394 μm^2 respectively and the mean of all the three is 255.

Slice	Count	Total area	Average size	Percentage	Mean
Ferric oxide	200	345.952	1.730	49.599	255
Cobalt doped ferric-oxide	254	389.014	1.5322	54.586	255
Zinc doped ferric-oxide	200	366.391	1.831	52.037	255

Table 4. The obtained values from ImageJ software of pure iron oxide, zinc doped iron oxide and cobalt doped iron oxide has a threshold of 49.94, 43.41 and 37.15 respectively.

Random Count	Pure Iron oxide	Zinc doped iron oxide	Cobalt doped Iron oxide
1	0.062	0.108	0.036
2	1.127	6.299	0.008
3	0.018	0.037	0.023
4	0.099	0.004	0.042
5	0.003	0.006	0.02
6	2.056	0.002	0.025
7	0.002	0.232	0.006
8	0.048	0.009	0.032
9	0.004	0.018	0.017
10	0.002	0.008	0.057
11	0.006	0.008	0.005
12	0.009	0.004	0.004
13	0.006	0.007	0.005
14	0.003	0.006	0.025
15	0.062	0.003	0.003
16	0.004	0.005	0.036
17	0.009	0.007	0.009

18	0.016	0.166	0.002
19	0.01	0.003	0.004
20	0.002	0.006	0.036



(a)



(b)

Fig. 1. (a) Colloidal solution of pure iron-oxide stabilize using NaOH solution (b) Annealed powder of iron-oxide at temperature of 500 °C



(a)



(b)

Fig. 2. (a) Colloidal solution of cobalt doped iron-oxide stabilize using NaOH solution (b) Heating of cobalt doped iron-oxide on petri dish at temperature of 80 °C



(a)



(b)

Fig. 3. (a) Colloidal solution of zinc doped iron-oxide stabilize using NaOH solution (b) Annealed zinc doped iron-oxide

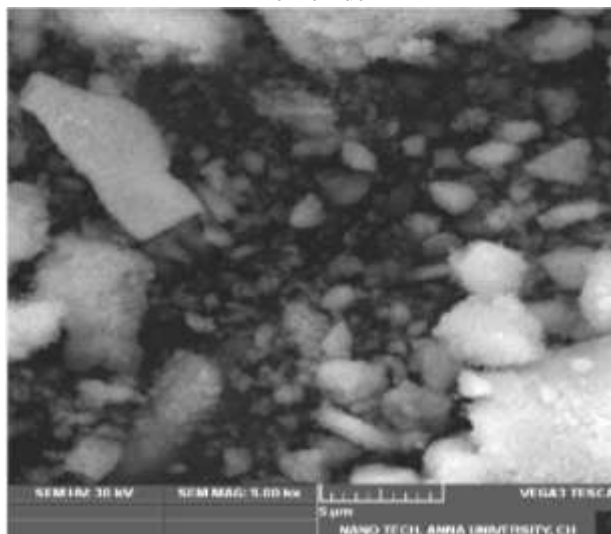


Fig. 4. Pure ferric-oxide SEM image of different magnification 1.00 kx, 2.0 kx, 5 kx, 10.0 kx and 25 kx.

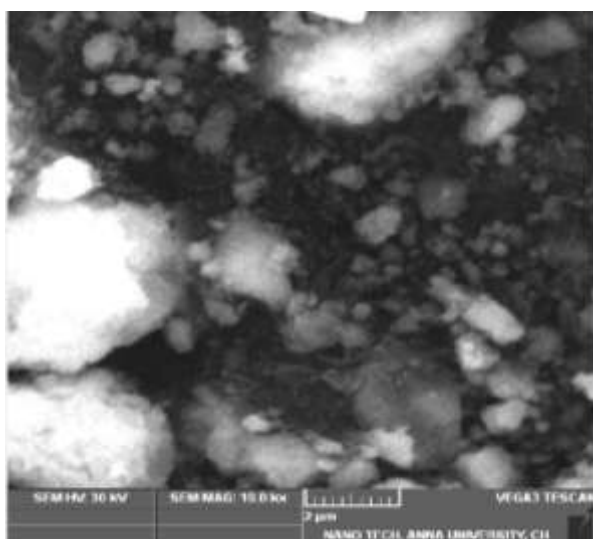


Fig. 5. Cobalt doped ferric-oxide SEM image of different magnification 1.00 kx, 2.0 kx, 5 kx, 10.0 kx and 25 kx

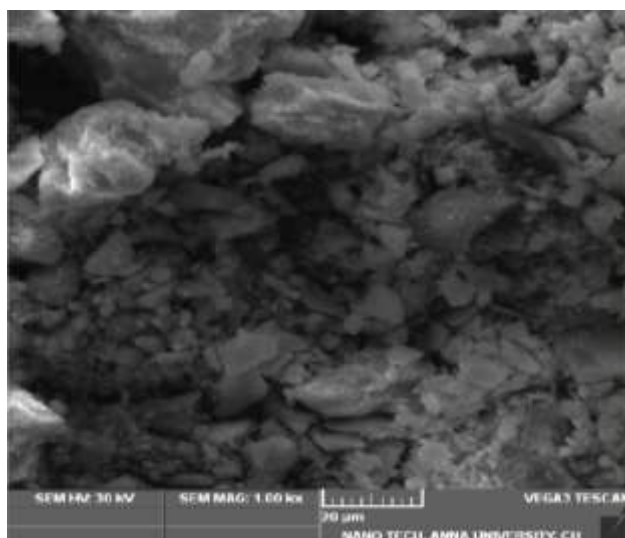


Fig. 6. Zinc doped ferric-oxide SEM image of different magnification 1.00 kx, 2.kx, 5 kx, 10.0 kx and 25 kx

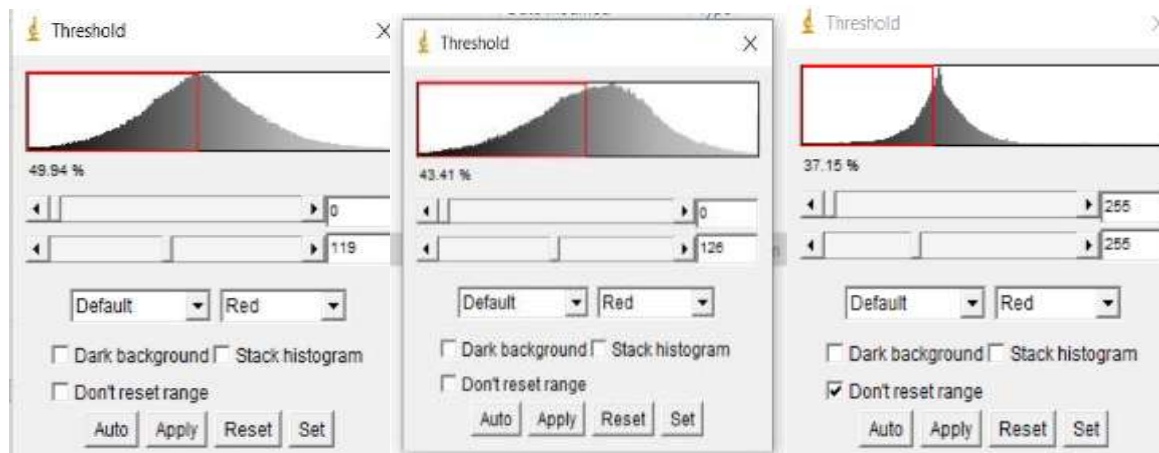


Fig. 7. The threshold for pure iron oxide, cobalt doped iron oxide and zinc doped iron oxide used in ImageJ software.

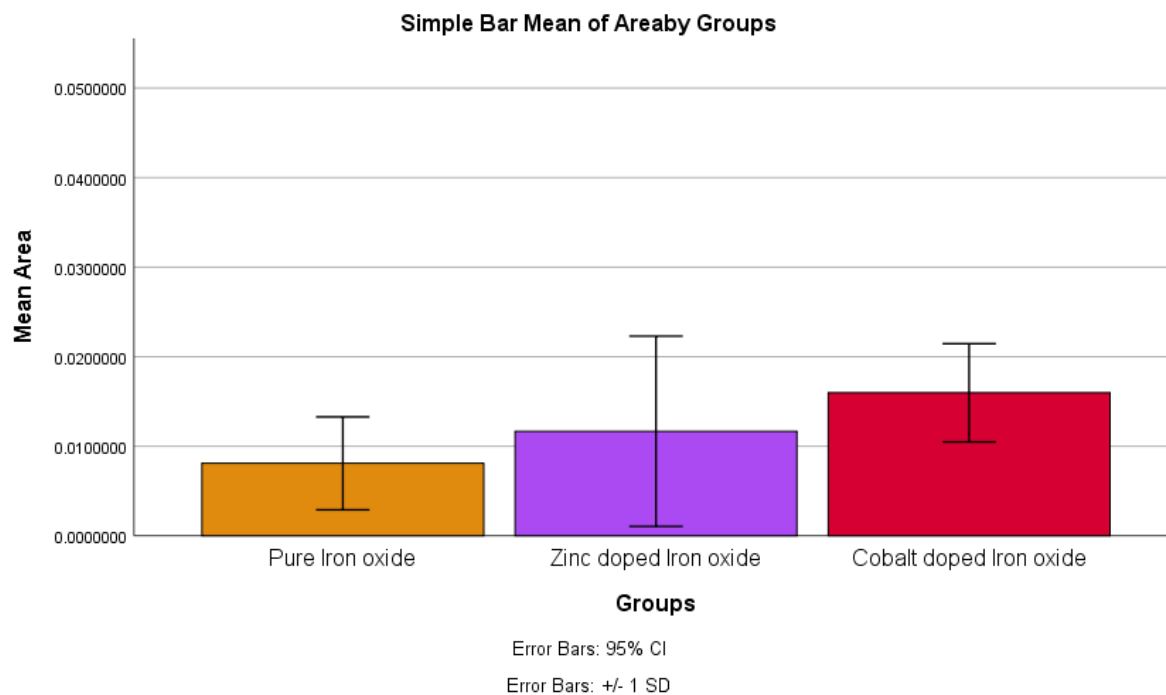


Fig. 8. Bar graph represents the variation of size or area in which it represents the iron-oxide, cobalt doped iron-oxide, zinc doped iron-oxide with standard deviation of ± 1 SD. From the graph, it can be observed that zinc doped iron oxide produces variable results and pure iron oxide produces consistent results.