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Chemical Synthesis and Characterization of Magnetic Iron (Fe_3O_4) Nanoparticles Used in Catalytic Decomposition of Hydrogen Peroxide

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ABSTRACT

Magnetic nanoparticles are rapidly piquing researchers due to their numerous uses such as catalysis, magnetism, medicine, optics, antibacterial, antifungal, and larvicidal treatments, Magnetic drug targets, magnetic resonance imaging for clinical diagnosis and recording material. This paper focuses on synthesis, characterization and study of magnetic iron (Fe_3O_4) nanoparticle's catalytic properties in the decomposition of hydrogen peroxide as well as determination of reaction's order. In the current study, co-precipitation was used to create magnetic iron (Fe_3O_4) nanoparticles as a result of surface Plasmon absorptions, the reaction mixture's hue changed, indicating the production of magnetic nanoparticles. FTIR spectra of Fe_3O_4 nanoparticle revealed the presence of peaks at 3420 cm^{-1}, 2925 cm^{-1} and 500 cm^{-1} that corresponds to hydroxyl groups (O-H), CH_3 stretching vibration and Fe-O stretching vibrations on the surface of the Fe_3O_4 nanoparticle. The UV-result of synthesized Fe_3O_4 nanoparticles showed the highest peak at 300 nm due to the excitation of electrons and d-d transition ability of the metal in question. This confirms that particles were stable and well dispersed in the solution. The XRD result revealed five main diffraction peaks at 2Θ =30°, 37 .6°, 49.3°, 54°, 57.1°, that corresponds to the planes (220, 311, 400, 422, and 511 respectively) of Fe_3O_4 nanostructures. The average crystallize size of Fe_3O_4 was determined using Debye-Scherer relation $D=K\lambda/\beta\cos\theta$, and found to be 35.2 nm. Catalytic decomposition of hydrogen peroxide was carried out in an exothermic reaction in which hydrogen peroxide transforms into oxygen and water. Gasometric measurements was used to introduce various amounts of Fe_3O_4 catalyst to each reaction vessel containing hydrogen peroxide. The amount of oxygen evolved was measured on a regular basis and was observed that an increase in catalyst concentration sped up the reaction and reduced the amount of time needed for hydrogen peroxide to decompose. As a result, more oxygen gas was produced during the reaction as catalyst concentration increased. Therefore, the co-precipitation approach of creating nanoparticles is seen to be the best in terms of affordability, non-toxic nature, and uses environmentally acceptable chemicals. In the end, the decomposition of hydrogen peroxide (H_2O_2) was first order in relation to the catalyst concentration.

Keywords: Magnetic iron, co-precipitation, catalyst, synthesis, peroxide, nanoparticle

INTRODUCTION

According to Saba (2015) and Priya et al., (2021), nanotechnology is the creation and application of materials whose constituent parts are at nanoscale - typically up to 100 NM. Nanotechnology examines structural behavior at the molecular and sub-molecular levels as well as electrical, optical, and magnetic activity. It might completely transform a wide range of medical and biotechnology tools and processes, making them more transportable, affordable, secure, and simple to use. The magnetic nano particles have generated an accelerated development and research movement in the last two decades, they are solving a large portion of problems in several industries, including water remediation, photo-electronics and information storage (Jesus *et al.,* 2022). In order to reduce toxicity of nanoparticles, bimetallic nanoparticles are now synthesized from natural sources (Roopan *et al.,* 2013). Recently, Iron oxide nanoparticles have attracted much consideration due to their unique properties, such as superparamagnetism, surface to volume ratio, greater surface area, and easy separation methodology (Ali *et al.,* 2016).

Nanotechnology is the study of very small objects. It involves the usage and tinkering of matter. At this scale, atoms and molecules function differently and provide a wide range of unexpected and fascinating applications. Studies on nanotechnology and nano-science have rapidly developed during the past several years in a variety of product categories (Sovanlal *et al.,* 2011). It has a significant impact on the creation of creative ways to manufacture new goods that are compatible with existing production equipment and to reformulate new materials and chemicals with improved performance, resulting in less energy and material consumption, less environmental harm, and environmental remediation. Recently, many techniques have been used to produce nano-catalyst, which is anticipated to be a promising field. The usage of nano-catalyst in waste water management, textiles, agriculture, and medicine has also undergone a review of recent applications and research (Kandasamy *et al.,* 2015).

Faryal et al., (2021) described iron nanoparticles as the tiniest particle of iron metal with a large surface area and high reactivity. They are non-toxic. Iron nanoparticles have excellent dimensional stability and also possess high thermal and electrical conductivity, high surface area and are highly magnetic. Iron nanoparticles can oxidize immediately when exposed to water or air and produces free Fe ions. There are numerous applications of iron nanoparticles but the most promising one includes their role in catalysis (Faryal *et al.,* 2021). Loekitowaki et al., (2017) effectively produced nanoparticles using the chemical co-precipitation approach and analytical tools, the structure, morphology, and magnetic characteristics of produced nanoparticles were evaluated. He came to the conclusion that the Fe_3O_4 nanoparticles are ideal for removing dye from water using a straightforward magnetic separation technique.

According to Weihua et al., (2021) iron nanoparticles can be used as catalysts and carriers of semiconductors to improve the activity of reaction systems. Jameel and Madhloom (2016) used sulfate as a metal precursor, oleic acid as a dispersant, ethanol as a reducing agent, and NaOH as a precipitant in order to create Fe_3O_4 nanoparticles, X-ray diffraction (XRD), FTIR, and thermal analysis were used to evaluate the produced Fe_3O_4 nanoparticles. Zakiyyu et al., (2019) produced Magnetic iron nanoparticles Fe_3O_4 by annealing under vacuum at various temperatures. The prepared nanoparticle of Fe_3O_4 was characterized.

Rashid et al. (2014) used the in-situ precipitation approach and successfully synthesized nanoparticles (Fe_3O_4), and used the concentration of magnetite created during the process to gauge the overall reaction rate. The existence of a higher percentage of magnetite Fe_3O_4 in the finished product is further confirmed by X-ray diffraction, energy-dispersive X-ray spectroscopy, and Raman spectroscopy. The findings demonstrated that spherical nanoparticles produced at various points throughout the process have a relatively constrained range of particle size.

Sharifi et al. (2015) conducted research on the use of iron-manganese oxide nano-catalysts in the breakdown of hydrogen peroxide, nanocomposites made of iron-manganese oxide. The catalyst bed that was used to create the nanocomposites as a capping agent was polyvinyl pyrrolidone to prevent the nanoparticles from adhering together. FT-IR, XRD, and SEM were used to identify the nano catalysts. nanoparticles prepared using the co-precipitation method had better catalytic activity than samples obtained using other techniques (Sharifi *et al.,* 2015). Iron nanoparticles can be synthesized by three different methods namely; physical method, chemical preparation method, biological method among these methodologies, chemical-based synthesis methods are mostly adopted due to low production cost and high yield (Ali *et al.,* 2016). Above mentioned properties and their high physical and chemical stability make iron nanoparticles suitable for various purpose like catalysis, magnetic recording device as audio and video tape and digital recording disks with high density (Priyanka *et al.,* 2022).

There are various methods in which nanoparticles can be produced such as; co-precipitation synthesis, thermal decomposition, hydrothermal synthesis, sol-gel synthesis, microemulsion synthesis, sono-chemically assisted, micro-wave assisted, biological synthesis routes, surface coating, silica coating, carbon-based coating, metallic coating, polymer coatings, amongst all the methods co-precipitation which is straight forward, well known, cheap, effortless and creates abundant magnetic nanoparticles was used (Jesus *et al.,* 2022). This research studied the effectiveness of using non-toxic, inexpensive and environmentally friendly method of nanoparticle production for use in research institutions and industries, that improves the rate of production and reduce the cost of production. The presented work is about iron nanoparticles synthesis, characterization and their catalytic properties. Characterization of iron nanoparticles was done by FTIR, UV-vis and XRD. Particle size using XRD characterization was calculated by Debye-Scherrer method (Priyanka *et al.,* 2022).

MATERIALS AND METHODS

Preparation of Sodium Hydroxide Solution

In order to make a 2 M NaOH solution, 20 g of sodium hydroxide was dissolved in a small beaker of distilled water and stirred. The liquid was then transferred to a 250 ml volumetric flask and distilled water was added up to the mark while the flask was continuously shaken to create a homogeneous solution.

Synthesis of Fe_3O_4

The approach employed by Loekitowakiet al., (2017) was followed in order to synthesize Fe_3O_4 magnetic nanoparticles. All of the chemical reagents utilized in the research were of analytical quality and do not require any additional purification. The entire experiment was conducted with deionized water, co-precipitation of ferric and ferrous salts with N_2 gas was used to carry out the synthesis. In 200 mL of distilled water, 16.25 g $FeCl_3$ and 6.35 g of $FeCl_2.4H_2O$ were dissolved. Chemical precipitation was accomplished at 30 °C under vigorous stirring for 60 mins by adding 2 M NaOH solution while N_2 gas was present. The reaction system was maintained at pH of 12 and 70 °C Temperature for 5 hrs. Fe_3O_4 full precipitation is anticipated for a pH range of 8 to 14. The precipitates were removed by filtering and rinsed with distilled water until pH was neutral after the system had been cooled to room temperature. Fe_3O_4 was then dried in an oven at 70 °C after being cleaned with acetone. The chemical reaction of Fe_3O_4 precipitation is given in equations below. The overall reaction is written as follows (Chengyin and Nuggehalli, 2012):

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$$Fe^{2+} + 2OH^- \longrightarrow Fe(OH)_2 \quad (1)$$
$$Fe^{3+} + 3OH^- \longrightarrow Fe(OH)_3 \quad (2)$$
$$Fe(OH)_2 + 2Fe(OH)_3 \longrightarrow Fe_3O_4 + 4H_2O \quad (3)$$
$$Fe_3O_4 + 0.25O_2 + 4.5\,H_2O \longrightarrow 3Fe(OH)_3 \quad (4)$$
$$2Fe(OH)_3 \longrightarrow Y\text{-}Fe_2O_3 + 3H_2O \quad (5)$$

Ultraviolet-Visible Spectrophotometer

The UV-Visible Spectrophotometer model 6705 was used to analyze the supernatant liquid for wavelengths between 200 and 1000 nm. Each aliquot sample was placed in a quartz cuvette with a resolution of 1 nm, and deionized water was used as the reference or blank solvent to determine the maximum absorption wavelength. The samples were transferred into a quartz cell that measured 1 x 1 cm (Akinsiku *et al.,* 2018 and Usha et al., 2022).

Fourier Transform Infrared Spectrophotometer

Fourier Transform Infrared Spectroscopy (PerkinElmer Spectrum Version 10.03.09) was used to characterize the produced Fe_3O_4 nanoparticle and identify the distinct functional group absorptions present. In order to identify the functional groups involved in the bio-reduction process.

Catalytic Decomposition of Hydrogen Peroxide H_2O_2 Using Fe_3O_4Nanoparticles

In a test tube, 3 ml of hydrogen peroxide solution was added, distilled water was placed in a measuring cylinder, which was then inverted into a water trough with the top sealed with a finger. 0.1 g of Fe_3O_4 catalyst was measured after Bung was loosely attached into the test tube and the delivery tube was also linked to the inverted measuring cylinder. The test tube was filled with 0.1g of the catalyst, and then the bung was replaced. A timer was used to time the process up until no more oxygen was released, the volume of gas released was measured at regular intervals. The same process was carried out using catalyst weighing 0.2 g, 0.3 g, 0.4 g, 0.5 g, and 0.6 g.

RESULTS AND DISCUSSION

Fe_3O_4 Nanoparticles

After combining Fe^{2+} and Fe^{3+} solutions in the presence of ammonium solution, the color changed from bright yellow to brown, dark brown, and black. That was how Fe_3O_4 nanoparticle production was initially identified. These hues represent the iron oxide's phase transition. Iron oxide undergoes two distinct transformation processes. The production of akageneite marks the beginning of the first stage, which was followed by the formation of goethite, hematite, maghemite, and magnetite, in that order. The creation of ferrous hydroxide marks the beginning of the second stage, which later results in the formation of lepidocrite, maghemite, and magnetite (Nalle *et al.,* 2019).

FTIR spectra of Fe_3O_4

Fe_3O_4 nanoparticle spectra revealed peaks at 3420 cm^{-1}, 2925 cm^{-1} and 500 cm^{-1} that were connected to O-H vibrations in the magnet, showing that hydroxyl groups were present on the surface of the Fe_3O_4 nanoparticle. According to the literature (Jameel *et al.,* 2016), the peak at 2925 cm^{-1} was attributed to the CH_3 stretching vibration, while the absorption peak at 500 cm^{-1} corresponds to the Fe-O stretching vibration connected to the magnetite phase.

UV-visible of Synthesized Fe_3O_4 nanoparticles

According to Kumar et al., (2018) highest peak range was observed between 250-350 nm, from table 1. and figure 1 the UV-result of synthesized Fe_3O_4 nanoparticles showed the highest peak at 300 nm due to the excitation of electrons and d-d transition ability of the metal in question. This

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confirms the particles were stable and well dispersed in the solution.

XRD Result of Fe_3O_4 Nanoparticle

As presented in figure 2 the pattern of Fe_3O_4 nanoparticles obtained using co-precipitation method by using Cu K α (λ=1.54056 A.) radiation with 2θ in the range (10-60°**).** Five main diffraction peaks were observed at 2Θ =30°, 37 .6°, 49.3°, 54°, 57.1°, correspond respectively to the planes (220, 311, 400, 422, and 511 respectively) of Fe_3O_4 nanostructures which came in agreement with the card (space group: JCPDS Nos. 26-1136) The average crystallize size of Fe_3O_4 was determined using Debye-Scherer relation

$$D = K\lambda/\beta\cos\theta,$$

where, β is the full width half maximum in radian, θ is the scattering angle, λ is the X-ray wavelength of radiation with 1.54 Å, K is the correction factor and D is the crystallite size of material in nm. Then substituting the values in Debye-Scherer equation, the crystallite size of Fe_3O_4 was found to be around 35.2 nm which is similar to the result obtained by (Jameel *et al.,* 2016*)*

Table 1: UV-Result for Fe_3O_4NPs

Wavelength (nm)	Absorbance
200	0.013
300	0.248
400	0.241
500	0.218
600	0.199
700	0.180
800	0.170
900	0.157
1000	0.152

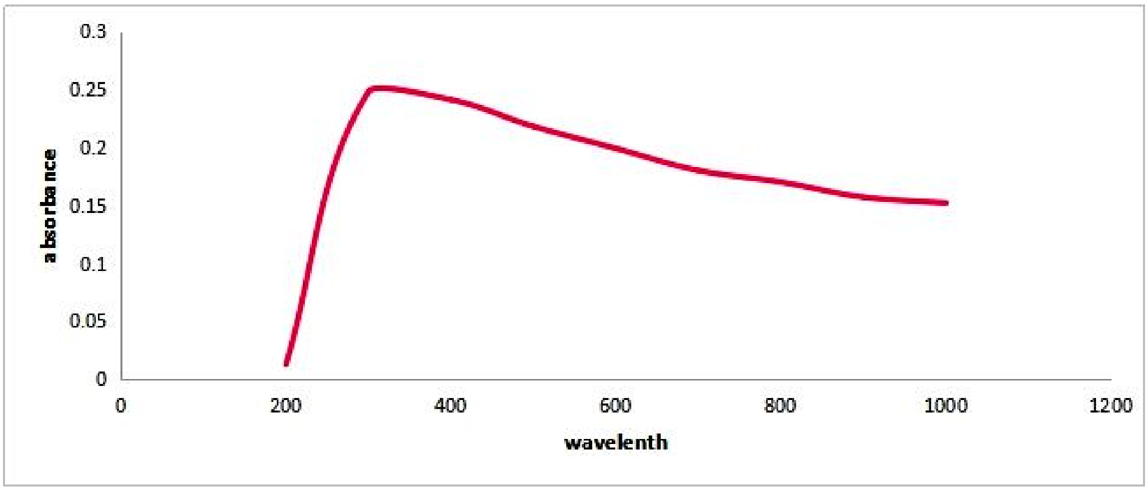

Figure 1: UV-Visible Spectrum for Fe_3O_4 NPs

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Analysis date	2021-06-30 13:20:51	Measurement start time	2021-06-30 12:28:59
Analyst	Administrator	Operator	Administrator
Sample name	Fe3o4 NPs	Comment	
Measured data name	C:\WallPaper\30-06-2021\Fe3o4 NPs_20210630_122829_G01...	Memo	

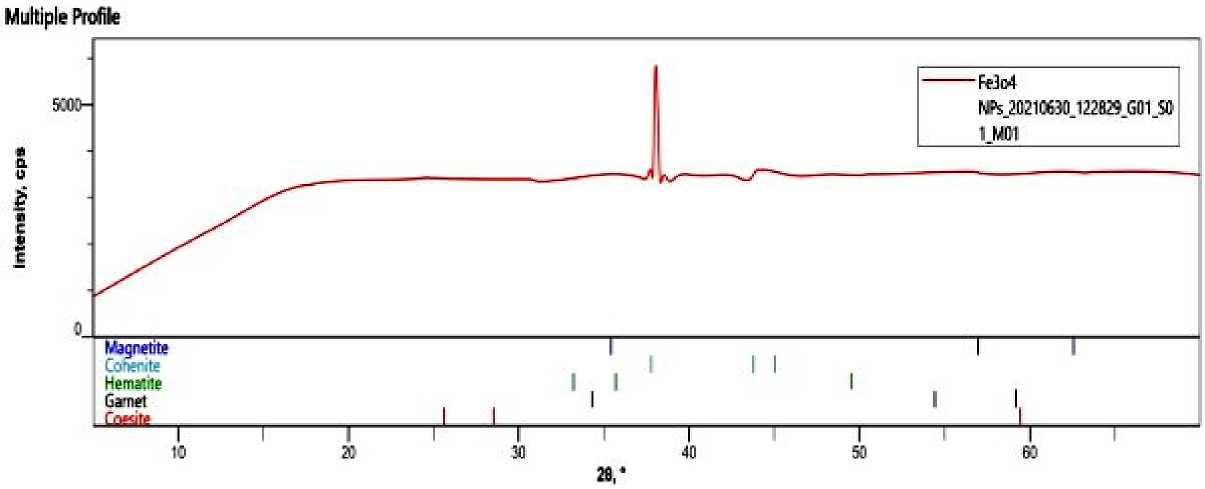

Figure 2: XRD Spectrum for magnetic Fe_3O_4 nanoparticles

Catalytic Decomposition of Magnetic Fe_3O_4 Nanoparticles

In an exothermic reaction, hydrogen peroxide transforms into oxygen and water. A catalyst made of Fe_3O_4 was used to study the breakdown of hydrogen peroxide at room temperature. Gasometric measurements were used to add various amounts of the catalyst; 0.1, 0.2, 0.3, 0.4, 0.5, and 0.6 grams to six test tubes, each containing three milliliters of hydrogen peroxide. The amount of oxygen evolved was measured on a regular basis. The table 2. and fig. 3 below demonstrates how an increase in catalyst concentration sped up the reaction and reduced the amount of time needed for hydrogen peroxide to decompose. As a result, more oxygen gas was produced during the reaction as catalyst concentration increased.

Table 2: Result for catalytic decomposition of H_2O_2 using magnetic Iron nanoparticles Fe_3O_4

Fe_3O_4NPs	TIME (sec)	volume of O_2 (ml)
0.1	1	20
0.2	1	40
0.3	1	75
0.4	1	240
0.5	1	270
0.6	1	290

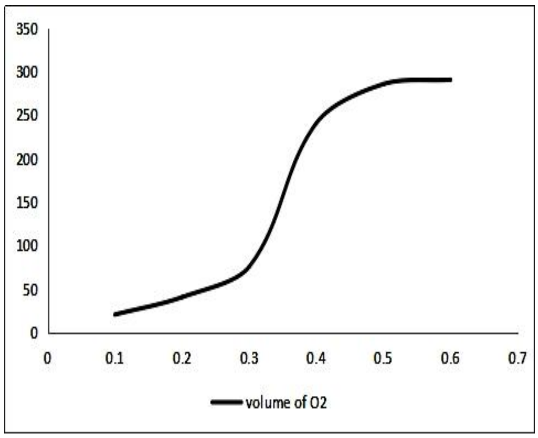

Figure 3: A Graph of volume of oxygen collected in cm^3 against the concentration of the catalyst

Table 3: Results for the Determination of Partial Order with respect to magnetic Iron Nano-particle Fe_3O_4

S/N	Mass (g)	Concentration (Moldm^{-3})	Time (s)	Rate	Log (conc)	Log (Rate)
1	0.1	0.00043	1	0.00043	-3.366	-3.366
2	0.2	0.00086	1	0.00086	-3.065	-3.065
3	0.3	0.00129	1	0.00129	-2.889	-2.889
4	0.4	0.00172	1	0.00172	-2.764	-2.764
5	0.5	0.00216	1	0.00216	-2.665	-2.665
6	0.6	0.00259	1	0.00259	-2.586	-2.586

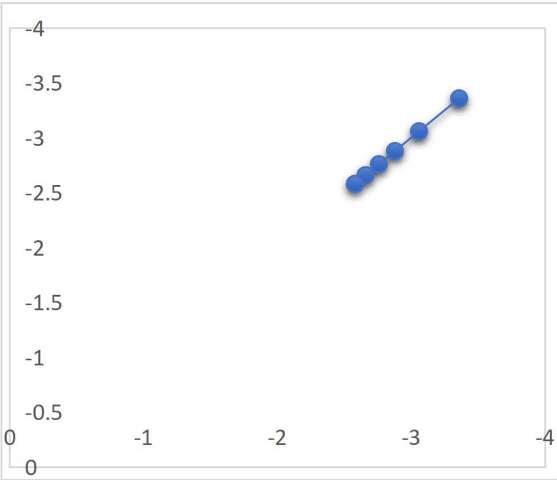

Figure 4: A graph of Log (Rate) against Log (Concentration)

From the graph in fig. 4 and table 3. the slope was approximately 1.0 and therefore first order with respect to the synthesized Magnetic Iron nano-particle concentration.

$R=K[Fe_3O_4]^1[H_2O_2]^x$

CONCLUSION

The co-precipitation approach of producing nanoparticles is said to be the best due to its affordability, non-toxic nature, and use of chemicals that are environmentally acceptable, in contrast to earlier methods that were unable to do so. Magnetic iron nanoparticles were synthesized, using co-precipitation method then characterized using FTIR, UV-vis and XRD, particle size was calculated using XRD characterization by Debye-Scherrer method. Catalytic properties of the synthesized magnetic iron nanoparticles were studied in which the reaction followed first order with respect to the concentration of catalyst synthesized. Therefore, as the concentration of the catalyst increases rate of reaction increases. Finally, the synthesized catalyst can be used in various industries and research institutions due to its friendly nature.

REFERENCES

Akinsiku A. A., Dare, E. O., Ajanaku K. O., Ajani O. O., Olugbuyiro J. A. O., (2018) Method: Optical and Biological Properties. *International Journal of Biomaterials.* 10(3): 1-18.

Ali A., Zafar H., Zia M., Haq, U., Phull A, Ali, J., Hussain A. (2016): Synthesis Characterization, Application and Challenges of Iron oxide Nanoparticles. *Nanotechnology Science and Applications.* 9(2): 23-45.

Chengyin F., and Nuggehalli M. R. (2012): Magnetic iron oxide nanoparticles; Synthesis and applications. *Bioinspired, Biomimetric and Nanobiomaterials.* 1(4):229-244.

Faryal. B., Muhammad S. I., Muhammad I. Q. (2021): Biologically synthesized iron nanoparticles (FeNPs) from *Phoenix dactylifera* have anti-bacterial activities. *Scienfic Reports.* 11: 22132.

Jameel, B. M., Lionel, F. G., and Giancarlo, E. B. (2016). Synthesis and Characterization of Fe_3O_4 Nanoparticles with Perspectives in

Biomedical Application. *Lateral Research Journal* 17(3): 542-549.

Jesus R. V., Carmen G. and Karen E. (2022): Magnetic Iron Nanoparticles; synthesis, surface enhancements and Biological Challenges. *MDPI processes.* 10(11): 2282.

Kandasamy, S., and Sorna R. P. (2015). Method of Synthesis of Nanoparticles and its Application. *Journal of Chemical and Pharmaceutical Research*, 7(3) 278-285.

Kumar, F. G., Maria, J. V., Cristiana, A., Jessica G., Monica, O., and Marta R. (2018). Characterization of Cobalt Oxide Transformation with Temperature at Different Atmospheres. *International Journal of Chemical Science* 3(17): 34-48.

Loekitowakiet, K.F., Nemade, K. R., and Waghuley, S. A. (2017). Chemical Synthesis of Cobalt Oxide Nanoparticles Using Co-Precipitation Method. *Research Journal of Chemical Sciences.* 7(1): 53-55.

Madhloom, L. F., Delphine, P., Marc, R., Alain, R., Caroline, V. E., Luce, N. M., Robert (2016). Magnetic iron Oxide Nanoparticles Synthesis Stabilization Vectorization, Physiochemical Characterization and Biological Application. *Chem, Rev.* 10(8): 2064-2072.

Nalle F. C, R. Wahid, I. O. wulandari, A. Sabarudin (2019). Synthesis and Characterization of Magnetic (Fe_3O_4) Nanoparticlas Using Oleicacid as Stabilizing Agent. *Rasayan Journal Chem*. 12(1): 14-21.

Priya, N., Kaur K and Sidhu A. (2021): Green synthesis: An eco-friendly route for the synthesis of iron oxide nanoparticles. *Frontiers Nanotechnology.* 3: 655062. http://doi.10.3389/fnano.2021.655062.

Priyanka G., Ravi. K. V., and Subhash C. (2022): Synthesis Characterization and Magnetic Properties of Nanoparticles of Cobalt Doped Ferrite. *International Journal of Chemistry, Mathematics and Physics (IJCMP).* 6(5): 6-11.

Roopan S.M., Rohit G., Madhumitha G., Abdulrahuman A. C., and Bharathi, A. T.V. (2013). Low-cost and Eco-friendly Phytosynthesis of Silver Nanoparticles Using Cocos Nucifera Coir Extract and its Larvicidal Activity. *Ind. Crop. Prod*. 43(5): 631-635.

Rashid L., Haraimi, M. F., and Ridwan, M. D. S. (2014). Synthesis and Properties of Fe_3O_4 Nanoparticles by Co-Precipitation Method to Removal of Procion Dye. *International Journal of Environment Science and Development.* 4(3): 336-340.

Saba Hassan (2015). A Review on Nanoparticles Their Synthesis and Type. *Research Journal for Recent Science*. 4(6):1-3.

Sharifi, S. L., Hossein, M. H., Mirzael, A., and Salmamani A. O. (2015). Catalytic Decomposition of Hydrogen Peroxide in the Presence of Iron Manganese Nanocomposite Via Different Method. *International Journal for Nanoscience and Technology.* 11(4): 233-240.

Sovanlal P., Utpal, J., Manna, P.K., Mohanta, G.P., and Manavalan R. (2011). An Overview of Preparation and Characterization. *Journal of Applied Pharmaceutical Science.* 1(6): 228-234.

Usha. V., Amutha. E., Pushpalaksmi. E., Jenson S., Rajaduraipandian S., Gandhimathi. S., Annadurai., G. (2022): Synthesis of Iron oxide Magnetic Nanoparticles: characterization and its biomedical application. *Journal of Applied Sciences and Environmental Management.* 26(2): 281-286. https://www.ajoil.info/index.php/jasem

Wuihura X., Ting Y., Shaobo Li., Li D., Qiang C., Xin L., Jie D., Zhuang Z., Sihui L., Youzi G., Liang Z., Yunguo L., and Xiaofei T. (2021): Insights in to the synthesis, types and application of iron nanoparticles: the overlooked significance of environmental effects. *Environment International*. 158(7):106980.

Zakiyyu, I. T., Moh'd, K. M., Saliza A., and Kharunnadim A. K. (2019). Preparation and Characterization of Magnetite (Fe_3O_4) Nanoparticles by Sol- Gel Method. *International Journal of Nanoelectronics and Materials*. 12(1): 37-46.