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SYNTHESIS AND CHARACTERIZATIONS OF MULTIFUNCTIONAL IRON OXIDES PREPARED VIA MODIFIED CO-PRECIPITATION METHOD

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ABSTRACT

Iron oxide nanoparticles have garnered significant attention due to their unique physicochemical and magnetic properties making them suitable for diverse application in environmental remediation ,biomedical diagnostic and catalysis .this study focuses on the synthesis of iron oxide via the co precipitation method followed by surface modification using silica gel to enhance their stability and adsorption efficiency .further the silica modified iron oxide nanoparticles were doped with silver nitrate to form composite materials with improved functional properties. The synthesized materials were characterized using FTIR and the point of zero charge to investigate surface functionalities and charge behavior. Their effectiveness was assessed for the adsorption and degradation of methylene blue dye from aqueous media under various experimental condition including adsorbent dose,dye concentration , and contact time. The result demonstrated that both modified and silver dropped iron oxide naoparticles exhibited excellent dye removal efficiencies with AGMO achieving up 99% degradation within a short reaction time.this study highlight the potential of modified iron based nanomaterials as cost-effective and environmentally friendly adsorbent for wastewater .

Keywords: *Metal Sulfide , Dye, XRD ,SEM, Congo Red Dye, Degradation*

1. INTRODUCTION

The environment, human health, animals, birds, and underwater creatures are all directly affected by the introduction of various pollutants, including dyes, drugs, and pesticides, into the water as a result of industrialization, population growth, and the expansion of human activities. uses.[1-3] Water pollution is currently the most major problem since industries like textiles, printing, dyeing, spinning, leather, etc. release a lot of wastewater into the environment that contains hazardous chemicals and dyes, which contaminates and degrades water quality [4].Dye is one of the most hazardous organic pollutants in industrial effluents, particularly textile effluents. These pollutants are characterized by their high toxicity and indestructible structure, which are responsible for the development of cancer, genetic mutations, dermatitis, allergies, and skin irritation in humans.[5]Synthetic dyes used to adulterate ground and surface water pose a threat to aquatic life and humans, [6]as well as to the environment. In this study, we will synthesize a binary metal sulfide nanoparticle via co-precipitation method. Combining two metal sulfides will yield desire properties. Mixed metal sulfides cations help in various chemical processes. Due to these properties metal sulfides are used for photo catalytic degradation of organic dyes in polluted water. [7] These dyes seriously harm the environment and human health at higher concentrations. The effects of pH, catalyst dosage, and irradiation duration were evaluated in the photo degradation of organic dyes. Furthermore, the process is highly efficient, easily operatable, fast, less costly, less time and energy consuming. In addition to patient therapy, nanomaterials have long been utilized to control targeted medication distribution for better use in enhancing system performance, i.e., its efficacy, safety, and quality. In this case, iron oxide magnetic nanoparticles (IOMNPs) have been effectively used to enable the medicine to target the damaged location in a biocompatible and effective manner. These NPs undergo appropriate surface modification with either organic or inorganic substrates to achieve biocompatibility.[8]

In order to functionalize IOMNPs, organic surfactants are produced in organic solutions, with the assumption that long hydrocarbon chains may produce hydrophobic nanoparticles. Using surfactants with low critical micelle concentrations results in more effective coatings of IOMNPs. Likewise, inorganic elements such as silica, carbon, metals, and non-metals have been used in conjunction with IOMNPs to enhance their antioxidant capabilities. In the meantime, metal oxides, such ZnO-coated IOMNPs, have been shown to be useful in anticancer nanosystems.[9]

In recent years, green routes synthesis of IONPs have been adapted due to its environmental friendliness and cost effectiveness. The green routes synthesis by biocompatible reagents such as biopolymers, by microorganism e.g. bacteria, fungi, etc., synthesis from plant biomaterials i.e. from leaf extract, seed extracts etc. have been well practiced. The green synthesized IONPs are crucial for environmental remediation such as organic dyes degradation, removal of heavy metals from aqueous media etc. Previously, stable colloidal suspension of nZVI (size 5-10 nm) coated with polyphenol (green tea extract) was used for the removal of chromium from groundwater.[10]

In this study iron oxide nanoparticles were synthesized and subsequently modified with silica gel followed by silver doping to form a composite materials. The synthesized nonmaterial were characterized and evaluated for their adsorption efficiency in removing methylene blue dye from aqueous solution . the research aims to provide a sustainable and efficient method for dye degradation contributing to the development of eco- friendly water treatment technologies.

1.2 Characterizations

For the better understanding of surface properties of magnetic NPs, various surface characterization techniques are used such as XRD), TEM, SEM, AFM, FT-IR, XPS, thermal gravimetric analysis, and vibrating sample magnetometry etc.In one of the study, the magnetic iron oxide NPs embedded in

amorphous silica was analyzed by Mossbauer spectroscopy (MS) and XRD. Samples synthesized at low temperature keeping high amounts of iron oxides contained high fraction of hematite. Mossbauer spectroscopy results showed a Mossbauer spectra measured at 300 K, and two sextets, one-two doublets in spectra were used to explain the spectra. Three sextets showed the various sites of Fe in ε-Fe_2O_3, and one hematite. Doublets represented particles in paramagnetic and/or superparamagnetic states. The results obtained from high resolution transmission electron microscopy also supported the presence of hematite and small crystalline ε-Fe_2O_3 having size 5-10 nm in the samples as identified by MS and XRD [11] In a separate study, XRD technique was implemented to examine the pattern and crystallinity in the Fe_3O_4NPs synthesized using $FeSO_4.(NH_4)_2SO_4.6H_2O$ through solvo-thermal techniquesupported with microwave irradiation. XRD pattern of synthesized magnetite was almost comparable with standard lattice spacing and angular orientations. The surface area measurements of magnetite (85.97 m^2/g) was within the standard range of 4-100 m^2/g. Surface uniformity of prepared nanoparticles was studied with the help of AFM. Result of AFM showed two dimensional hexagonal and three dimensional hexagonal layered structural arrangements of magnetite NPs [12].

2. EXPERIMENTAL SECTION

2.1 Synthesis of Iron Oxides NPs and their Modification with Silica Gel

For iron oxide NPs preparation, pre-determined quantities of iron precursor(s) i.e. $FeCl_2.4H_2O$ and $FeCl_3.6H_2O$ were added into adequate amounts deionized water in separate glass vessels. The neutralization of the precursors solutions were carried out by using NH_4OH upon vigorous mixing. In the meanwhile iron (hydr)oxides and iron oxy(hydr) oxides were also formed whiche were then converted to the magnetic iron oxides NPs as reported by [13] Iron oxide (Fe_3O_4) was modified via chemical mixing method using silica gel. Three solutions of iron oxide and silica gel separately having various concentrations i.e., 0.1, 0.5, 1 M were prepared in 50 mL distilled water, respectively. Firstly, 0.1 M solution of iron oxide was prepared in a volumetric flask by introducing 1.15g iron oxide into 50mL of distilled water. Also 0.1 M solution of silica gel was prepared upon adding 0.03g silica gel into 50mL distilled water in a volumetric flask. Both the solutions were allowed to mix in beaker on a magnetic stirrer. Its pH was noted by using a pH meter. Afterwards, it was kept for agitation on hot plate and was stirred for maximum 4 hours. After stirring for 4 hours, it was allowed to cool. Then, it was washed three times with distilled water followed by drying in an oven for 1 hour. After drying, the resultant precipitates were labeled as MO1. Other solutions of concentrations 0.5 and 1M were also prepared by using above described method and desired precipitates were obtained and labeled as MO2 and MO3.

2.2 Synthesis of Composite Materials using $AgNO_3$

The composites materials of this modified iron oxide coated silica gelNPs were prepared with silver nitrate. For this purpose, an appropriate amount of silver nitrate ($AgNO_3$) was taken to prepare a 0.05 M solution by dissolving 1.27 g silver nitrate into 150 mL distilled water. Certain amounts of precipitates i.e. MO1, MO2 and MO3 solutions were taken, and divided into two parts keeping one part as un-doped while the other was used for preparing the composites. Composite with MO1 wasprepared by dissolving 0.5 g of it into the 0.05 M solution of silver nitrate. Then this solution was stirred for 4 hours and after stirring, it was allowed to cool followed by washing three times with distilled water. After washing, oven drying was carried out and precipitates of this solution were obtained which were labeled as AgMO1. Similar procedures were followed to prepare the composite materials for MO2 and MO3 whereby the resultant composite materials were labelled as AgMO2 and AgMO3, respectively.

3. RESULT AND DISCUSSION

3.1 pH of the Point of Zero Charge (pHpzc) and Mixed Oxide Nanoparticles (MO)

To determine the pHpzc of materials, 0.1 M $NaNO_3$ solution was prepared, and 20 mL portions were adjusted to pH 2, 4, 6, 8, 10, and 12 using HCl or NaOH. Each solution was mixed with the test material, and initial pH was recorded. After stirring overnight, final pH was measured, and the pH difference was plotted against initial pH to find the pHpzc. The MO sample showed a pHpzc of ~6.9, meaning its surface is positively charged below this pH and negatively charged above, affecting its adsorption of cationic pollutants like MB. Silver-doped MO (AgMO) showed a lower pHpzc of 5.8, indicating a more acidic surface due to silver, enhancing negative surface charge at lower pH compared to MO.

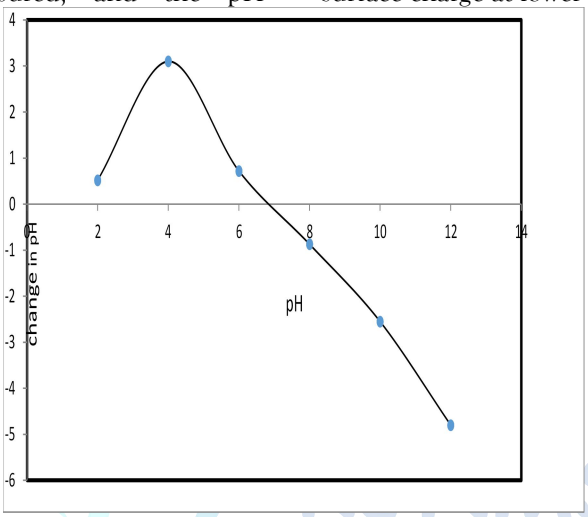

Fig.1. PH of the PZC Of the MO Naoparticles

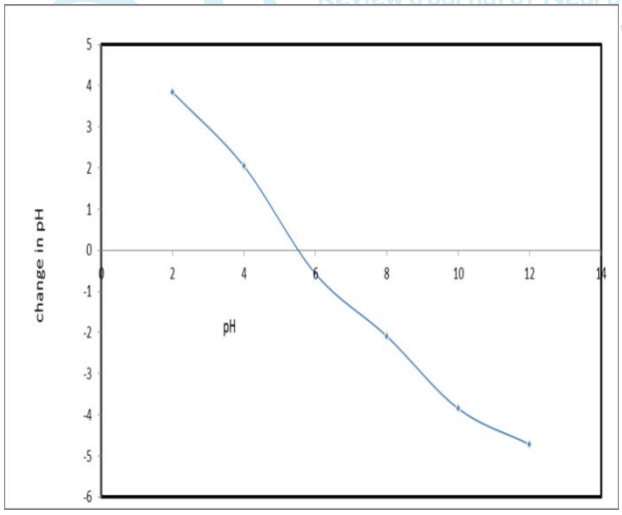

Fig .2 Phof PZC of Agmo

3.3 FTIR Analysis of Functional Groups on Modified Iron Oxide Nanoparticles

FTIR analysis was used to identify surface functional groups on the test materials. All samples showed O–H stretching peaks around 3600–3200 cm^{-1}, C–O stretching near 1000 cm^{-1}, and Fe–O vibrations below 700 cm^{-1} (especially <500 cm^{-1}, indicating magnetite). Peaks between 2500–2000 cm^{-1} were linked to C=O groups, and a band at 868 cm^{-1} confirmed Si–O–Si vibrations, showing successful silica modification. Similar spectra were observed for MO1, MO2, and MO3, with increasing intensity. Silver-doped samples (AgMO2, AgMO3) showed nearly identical peaks, with minor silver-induced changes below 1000 cm^{-1}. Dye-loaded samples (MO and AgMO after MB adsorption) showed no significant peak shifts, suggesting minimal structural or

surface functional group changes after adsorption.

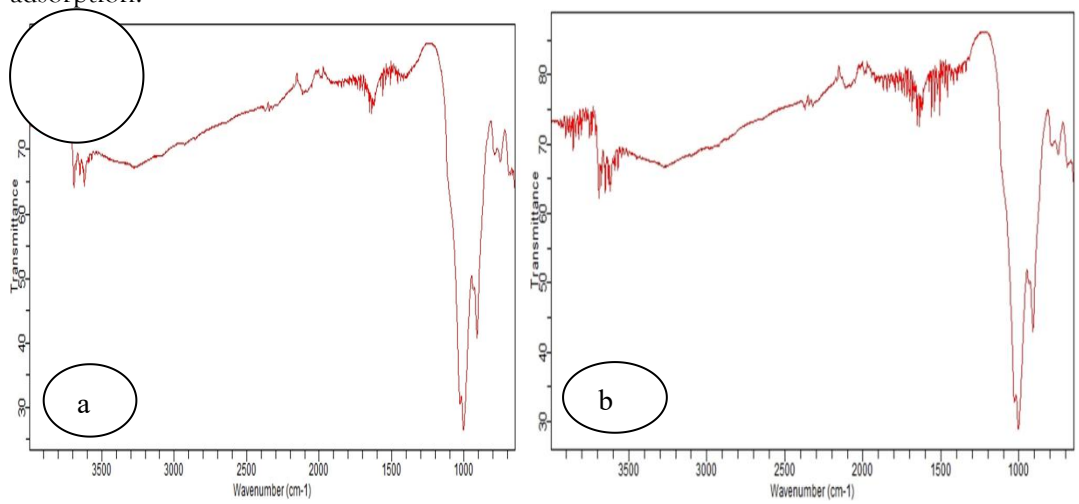

Fig 3 FTIR of MO Nanoparticles by Silica Gel of 0.1 And 0.5 m Solution

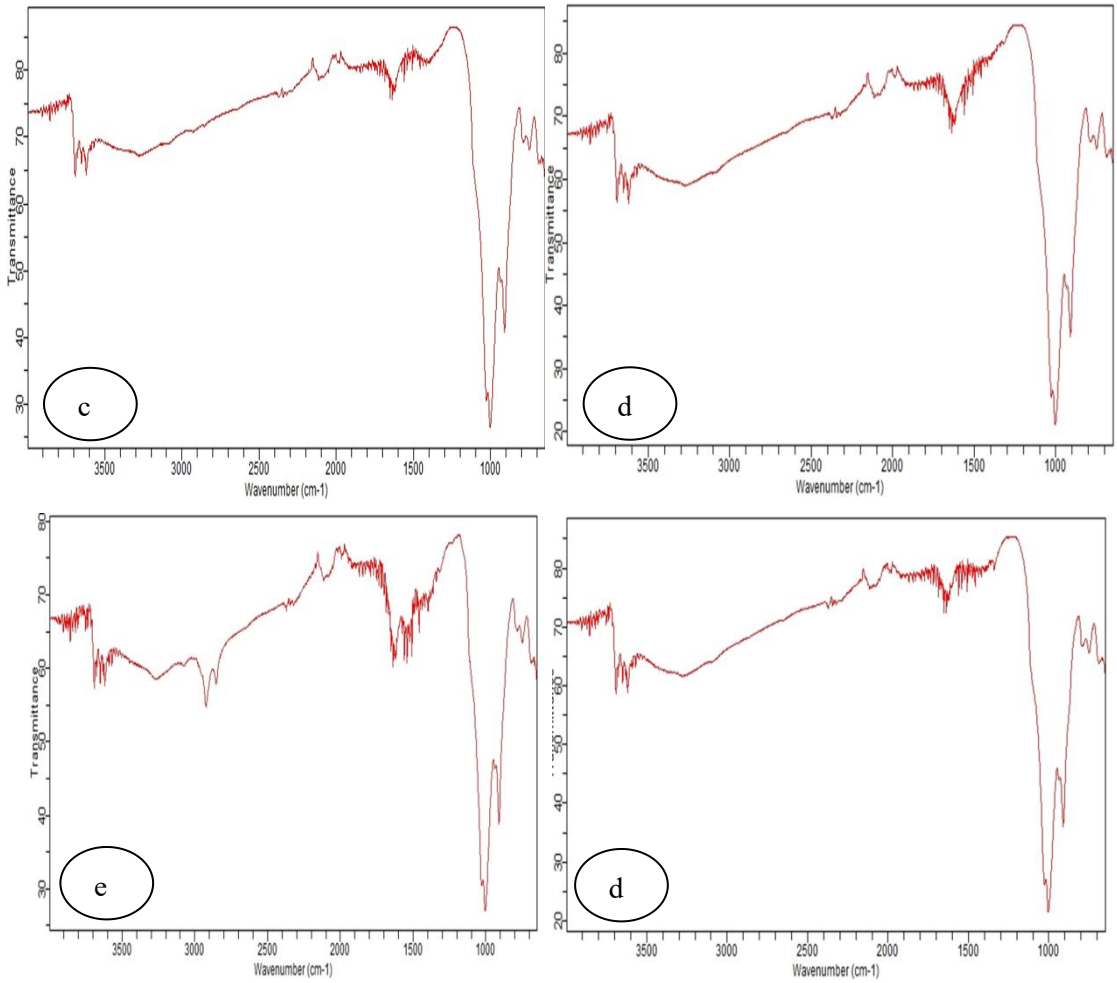

Fig 4 . FTIR Of (C) (D) (E) (F) 0.1 .0.2,0.25 G Of DYE Degraded MO and AGMO of Methyl Blue DYE

APPLICATION

4 .Prameters

4.1 Dose Study of MO and AgMO Samples for Methyl Blue Dye Degradation

To study the effect of adsorbent dose, 50 ppm methyl blue dye solution was prepared by dissolving 0.05 g dye in 1000 mL water. From this, 20 mL portions were taken into separate beakers, and varying doses (0.05, 0.1, 0.15, 0.2, and 0.25 g) of MO and AgMO samples were added. Each mixture was shaken for 3 hours to assess the degradation efficiency. Results showed that all doses contributed to dye degradation, but the highest efficiency was observed with 0.25 g of both MO and AgMO, indicating that increasing the adsorbent dose enhances dye removal .

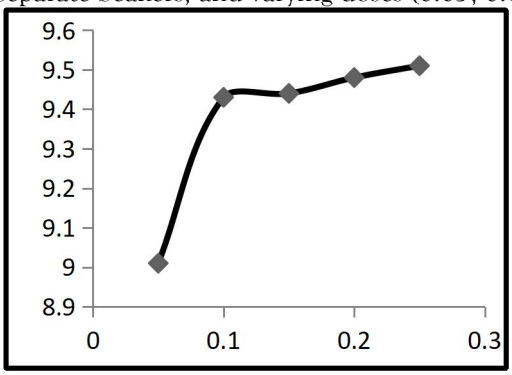

Fig 5. Dose Study Of Silica Gel Mo

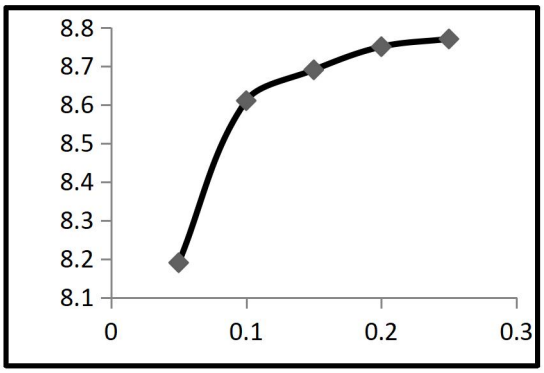

Fig 5. Dose Study Of Agmo

4.2 Effect of Dye Concentration on Degradation Efficiency Using MO and AgMO

To study the impact of dye concentration on degradation efficiency, methyl blue dye solutions of 10, 20, 30, 40, and 50 ppm were prepared. For each concentration, 20 mL of the solution was placed in separate, labeled beakers. A fixed amount (0.1 g) of MO or AgMO catalyst was added to each beaker. The mixtures were then shaken for 3 hours. Results showed that, for both MO and AgMO, the highest degradation occurred at 50 ppm concentration, indicating that higher dye concentrations enhanced the visible degradation efficiency under the same catalyst dosage.

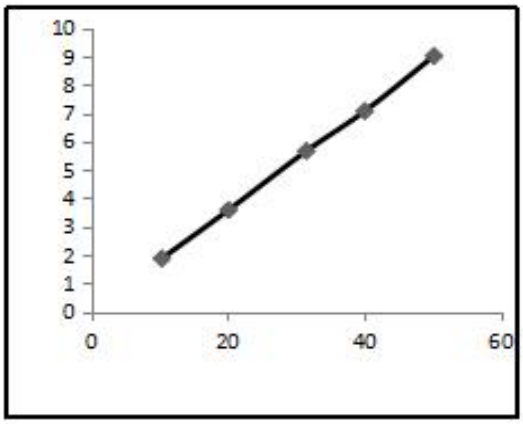

Fig 6. Dye concentration of MO nanoparticles

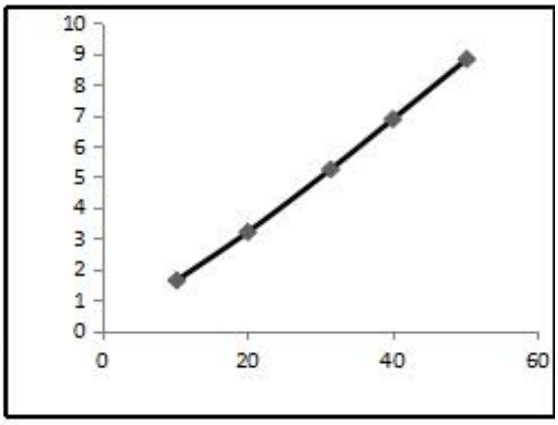

Fig 7. Dye concentration of AgMO

4.3 Kinetics of Methyl Blue (MB) Removal Using MO and AgMO

The removal kinetics of methyl blue (MB) dye using MO and AgMO samples were studied to evaluate their adsorption efficiency. For both materials, rapid decolorization was observed within the first 2 minutes, indicating a fast initial adsorption phase. However, agitation was continued for over 1 hour to ensure complete adsorption and equilibrium. For MO, the amount of MB removed ranged from 9.55 to 9.85 mg/g using 0.1 g of adsorbent. Similarly, AgMO also showed quick adsorption, reaching equilibrium within 60 minutes, with MB uptake between 9–9.5 mg/g and a removal

efficiency of up to 99%. These results confirm that both MO and AgMO are highly effective adsorbents for cationic dyes like MB, with fast kinetics and high removal capacities, making them suitable for wastewater treatment applications.

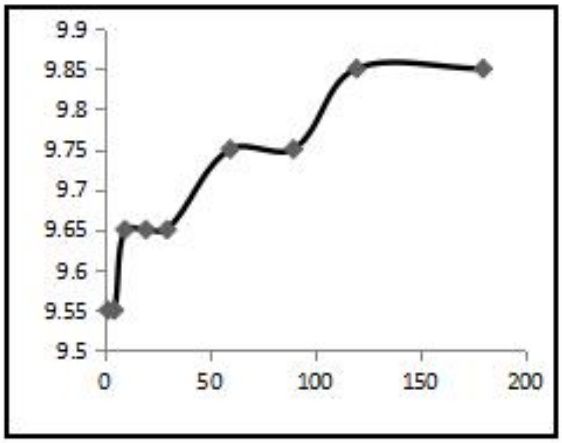

Fig 8. Kinetics study of MB removal by MO.

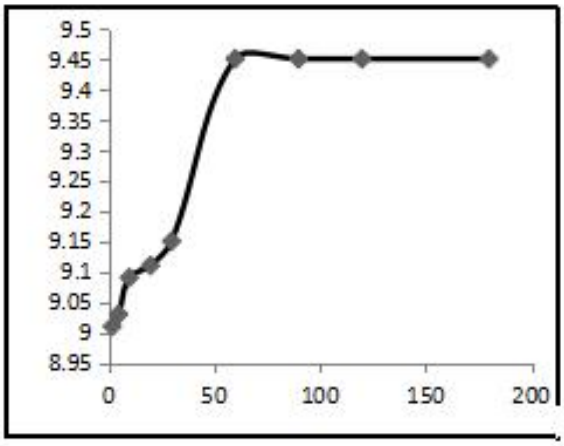

Fig 9. Kinetics study of MB adsorption by AgMO

The result showed that 0.1g of MO and AgMO was sufficient for optimal dye removal with AgMO achieving faster degradation within 2 minutes compred to MO .the kinetics followed rapid adsorption behavior suggesting that the materials are highly effective for fast pollutant removal.

CONCLUSIONS

Substantial progress has been made in the synthesis of iron oxide nanoparticles for application in various fields i.e., biomedical, agriculture, environmental applications etc. Many synthesis methods have been developed for synthesis of IONPs that control their particle size, compositions, shape, crystal size and magnetic properties. In this study co-precipitation method has been adopted for synthesis and their environmental applications were checked e.g. dye degradation. Iron oxide nanoparticle are less effective in the case if their surfaces are bare, they can be made more effective by coating them with organic molecules, polymers, surfactants, biomolecules, or inorganic layer such as silica, metal, metal sulfide, metal oxide etc. Coated surface of IONPs are less toxic, more biocompatible and allows targeted delivery. In this study silica gel was used for coating and made modified iron oxide nanoparticles (MO). Composites of modified sample were made with 0.5 M solution of silver nitrate ($AgNO_3$) and labeled as (AgMO). The materials were characterized through pHpzc and FTIR techniques describing the surface charge characters and the functionalities present on the materials. Later on, these materials were tested for the water treatment applications whereby the MB dye removal was investigated under various experimental conditions.The pHpzc results revealed that the surfaces of both of the materials were slightly acidic i.e. protonated or having more acidic functional groups present on the surface of the materials. In this connection, the pHpzc found for MO and AgMO were 6.9 and 5.8, respectively. Similarly, the major surface functional groups found on the surfaces were Fe-O, O-H, some variations from C-O and C=O in addition to the Si-O-Si linkages in both of the tested materials through FTIR.The adsorption of MB on the MO and AgMO was studied under various experimental conditions i.e. adsorbent dose, dyes initial concentration and the reaction kinetics was also studied. These results have shown a significant MB uptake by the prepared materials in all the ambient conditions applied. An optimum amount of 0.1 g, reaction time 1 h and 50 mg/L dye's concentration was found to be the working parameters. In all of the cases, up to 95%

MB removal was observed. These materials were found more effective and hence, can be applied for practical applications in the water treatment technologies being viable, feasible, less expensive and efficient in activity.

REFRENCES

1. Alageshwaramoorthy, K., et al., *Synthesis and characterization of visible-light-driven novel CuTa2O6 as a promising practical photocatalyst.* Frontiers in Chemistry, 2023. **11**: p. 1197961.
2. Bennani, L., et al., *Studies of the impact of copper doping on the electric, dielectric, and conduction mechanism of calcium titanate prepared by sol–gel process.* Journal of Materials Science: Materials in Electronics, 2025. **36**(1): p. 23.
3. Ahmadipour, M., M.F. Ain, and Z.A. Ahmad, *A short review on copper calcium titanate (CCTO) electroceramic: synthesis, dielectric properties, film deposition, and sensing application.* Nano-Micro Letters, 2016. **8**: p. 291-311.
4. (5) Eljamal, R.E., O.; Maamoun, I.; Yilmaz, G.; Sugihara, Y. Enhancing the Characteristics and Reactivity of nZVI: Polymers Effect and Mechanisms. Journal of Molecular Liquids2020, 315, 113714. and S.M. (6) Khan, A. Environmental and Health Effects of Textile Industry Wastewater. Environmental deterioration and human health: Natural and anthropogenic determinants2014, 55–71.
5. Khan, S.M., A. Environmental and Health Effects of Textile Industry Wastewater. Environmental deterioration and human health: Natural and anthropogenic determinants2014, 55–71.
6. Zafar, Z.F., R.; Kim, J.-O. Experimental Studies on Water Matrix and Influence of Textile Effluents on Photocatalytic Degradation of Organic Wastewater Using Fe–TiO2 Nanotubes: Towards Commercial Application. Environmental Research2021, 197, 111120.
7. Abo-Dief, H.M.H., O. K.; Ihsan, A.; El-Bahy, S. M.; Raslan, A. M.; Shahid, M.; Warsi, M. F. Ternary Metal Oxide WO3. NiO. ZnO Nanoparticles and Their Composite with CNTs for Organic Dye Photocatalytic Degradation. Ceramics International2022, 48 (15), 22228–22236.
8. Venkateshwarlu, V., et al., *Effect of feeding maize stover based complete rations on growth and carcass characteristics in Nellore× Deccani ram lambs.* Indian Journal of Animal Nutrition, 2014. **31**(1): p. 36-43.
9. Shukla, S.S., et al., *Removal of nickel from aqueous solutions by sawdust.* Journal of hazardous materials, 2005. **121**(1-3): p. 243-246.
10. Wang, T., et al., *Green synthesized iron nanoparticles by green tea and eucalyptus leaves extracts used for removal of nitrate in aqueous solution.* Journal of cleaner production, 2014. **83**: p. 413-419.
11. Ali, A., et al., *Synthesis, characterization, applications, and challenges of iron oxide nanoparticles.* Nanotechnology, science and applications, 2016: p. 49-67.
12. Behera, S.S., et al., *Characterization and evaluation of antibacterial activities of chemically synthesized iron oxide nanoparticles.* 2012.
13. Rashid, T., et al., *Green synthesis of Ni/Fe3O4/rGO nanocomposites for desulfurization of fuel.* ACS Applied Nano Materials, 2023. **6**(20): p. 18905-18917.
14. hommes, M. (2004). Physical adsorption characterization of ordered and amorphous mesoporous materials. *Nanoporous Materials: Science and Engineering* (pp. 317-364): World Scientific.
15. Veiseh, O., Gunn, J. W., & Zhang, M. (2010). Design and fabrication of magnetic nanoparticles for targeted drug delivery and imaging. *Advanced Drug Delivery Reviews, 62*(3), 284-304.
16. Venkateswarlu, S., Kumar, B. N., Prasad, C., Venkateswarlu, P., & Jyothi, N. (2014). Bio-inspired green synthesis of Fe_3O_4 spherical magnetic nanoparticles using *Syzygium cumini* seed extract. *Physica B: Condensed Matter, 449*, 67-71.
17. Wang, S., Dai, G., Yang, H., & Luo, Z. (2017). Lignocellulosic biomass pyrolysis mechanism: a state-of-the-art review.

Progress in Energy and Combustion Science, 62, 33-86.

18. Wang, S., Ma, Q., & Zhu, Z. (2008). Characteristics of coal fly ash and adsorption application. *Fuel*, 87(15): p. 3469-3473.
19. Wang, T., Lin, J., Chen, Z., Megharaj, M., & Naidu, R. (2014). Green synthesized iron nanoparticles by green tea and eucalyptus leaves extracts used for removal of nitrate in aqueous solution. *Journal of Cleaner Production*, 83, 413-419.
20. Wu, S., Sun, A., Zhai, F., Wang, J., Xu, W. & Zhang, Q. (2011). Fe_3O_4 Magnetic nanoparticles synthesis from tailings by ultrasonic chemical co-precipitation. *Materials Letters*, 65, 1882-1884.
21. Xie, J., Chen, K. & Chen, X. (2009). Production, modification and bio-applications of magnetic nanoparticles gestated by magnetotactic bacteria. *Nano Research*, 2, 261-278.
22. Xiong, H., Liao, Y., Zhou, L., Xu, Y. & Wang, S. (2008). Biosynthesis of nanocrystal akaganéite from $FeCl_2$ solution oxidized by *Acidithiobacillus ferrooxidans* cells. *Environmental Science & Technology*, 42, 4165-4169.