RJC

RASĀYAN *J. Chem.*
Vol. 18 | No. 4 |2044-2047| October-December | 2025
ISSN: 0974-1496 | e-ISSN: 0976-0083 | CODEN: RJCABP
http://www.rasayanjournal.com
http://www.rasayanjournal.co.in

SURFACE-FUNCTIONALIZED Fe_3O_4 NANOPARTICLES WITH IMPROVED CATALYTIC PERFORMANCE: A SUSTAINABLE APPROACH FOR INDUSTRIAL AND ENVIRONMENTAL APPLICATIONS

A.D. Kanade[1], S. Jadhav[1], B. Madje[2], D. Sonwane[3], N. Rehman[4,✉], S. Mahire[5], R. Patil[5] and D. Chavan[1]

[1]Departmnet of Chemistry, Sandip University, Nashik, 422213, MH, India
[2]Department of Chemistry, Vasantrao Naik Mahavidyalaya, Sambhajinagar, 431001, MH, India
[3]Departmnet of Chemistry, Sanjivni University, Kopergaon, 423601, MH.India
[4]Department of Microbiology, Dr. BAMU, Sambhajinagar, 431001, MH.India.
[5]Department of Chemistry, SPDM College, Shirpur, 425405, MH.India.
✉Corresponding author: nazirehman786@gmail.com

ABSTRACT

Fe_3O_4 magnetic nanoparticles were synthesized by the co-precipitation method and further surface-functionalized with silica, amine, and sulfonic acid groups to improve their stability and catalytic potential. The synthesized nanomaterials were characterized using X-ray diffraction (XRD), dynamic light scattering (DLS), and zeta potential analysis. The XRD confirmed the spinel structure of magnetite with good crystallinity. DLS analysis indicated particle sizes in the range of 180–240 nm, while zeta potential data revealed the impact of surface modification on particle charge and colloidal behavior. These functionalized Fe_3O_4 nanoparticles offer promising features for catalysis and environmental applications.

Keywords: Fe_3O_4 Nanoparticles, Silica Coating, Functionalization, Zeta Potential, DLS, Magnetite

RASĀYAN *J. Chem.*, Vol. 18, No.4, 2025

INTRODUCTION

Magnetic nanoparticles, particularly iron oxide (Fe_3O_4, magnetite), have garnered significant interest in various applications, including catalysis[1], drug delivery[2], and environmental remediation[3]. Their unique superparamagnetic properties, high surface area, and ease of separation under an external magnetic field make them highly advantageous for these applications[4]. In addition, their biocompatibility and facile synthesis further contribute to their appeal in diverse interdisciplinary domains, ranging from biomedical sciences to green chemistry. However, bare Fe_3O_4 nanoparticles often suffer from aggregation due to their high surface energy, leading to reduced stability, reactivity, and limited dispersion in aqueous or organic media[5]. This intrinsic limitation greatly impedes their performance in practical applications, especially where uniform dispersion and controlled surface chemistry are essential. To overcome these challenges, a wide range of surface modification strategies has been developed to improve their physicochemical properties and extend their functional scope. Among these strategies, silica (SiO_2) coating has emerged as a robust and versatile approach. The silica shell not only prevents agglomeration and oxidation of the magnetic core but also provides a chemically inert and porous matrix for further surface engineering[6]. The presence of abundant silanol groups on the silica surface facilitates subsequent covalent attachment of organic moieties, enabling the tailoring of surface properties for specific catalytic or biological functions. Functionalization with organosilanes such as (3-aminopropyl)triethoxysilane (APTES) introduces reactive amine groups onto the nanoparticle surface, which can serve as ligands for metal coordination, cross-linking agents, or anchoring points for catalytic sites[7]. Further derivatization using sulfonating agents like propane sultone enables the introduction of sulfonic acid (-SO_3H) groups, conferring strong Brønsted acidity to the nanocomposite and rendering it suitable for acid-catalyzed organic transformations, including esterification, hydrolysis, and multicomponent reactions[8]. Such dual-functional systems combine magnetic recoverability with high catalytic activity, offering promising prospects for recyclable

http://doi.org/10.31788/RJC.2025.1849429

heterogeneous catalysts in green and sustainable chemistry. In this study, Fe_3O_4 nanoparticles were synthesized via a conventional co-precipitation method, followed by successive silica encapsulation, amino-functionalization using APTES, and sulfonation with propane sultone. The multistep functionalization was designed to improve not only the nanoparticles' structural integrity and colloidal stability but also to impart tunable acidic functionality required for catalytic processes. The resultant hybrid nanomaterials were extensively characterized using various techniques, including XRD, particle size, and zeta potential analysis to elucidate their morphological, magnetic, and surface chemical properties. These engineered nanoparticles are anticipated to exhibit enhanced catalytic efficiency, thermal stability, and recyclability, thus holding considerable potential for application in industrial chemical synthesis and environmental pollutant degradation.

EXPERIMENTAL

General procedure for the Synthesis of Fe_3O_4 Nanoparticles

A mixture of 2.0 g of $FeCl_3 \cdot 6H_2O$ and 1.2 g of $FeSO_4 \cdot 7H_2O$ was dissolved in 100 mL of deionized water under a nitrogen atmosphere and heated to 80°C with stirring. 25% ammonia solution was added dropwise until the pH reached ~10. A black precipitate formed, indicating the formation of Fe_3O_4. The mixture was stirred for an additional 30 minutes and then cooled to room temperature. The nanoparticles were collected magnetically, washed with deionized water and ethanol, and dried in a vacuum oven at 60°C.

General Procedure for the Silica Coating of Fe_3O_4 ($Fe_3O_4@SiO_2$)

0.5 g of the dried Fe_3O_4 nanoparticles were dispersed in a mixture of 50 mL of ethanol and 10 mL of deionized water using ultrasonication for 30 minutes. Then, 5 mL of 25% ammonia solution was added under stirring, followed by dropwise addition of 1 mL of TEOS. The reaction mixture was stirred at 40°C for 6 hours. The silica-coated particles were separated magnetically, washed with ethanol and water, and dried at 60°C.

General Procedure for Amino Functionalization ($Fe_3O_4@SiO_2–NH_2$)

To 0.5 g of silica-coated Fe_3O_4 nanoparticles in 50 mL of ethanol, 1 mL of APTES was added dropwise under stirring. The reaction mixture was maintained at 60°C for 6 hours. After completion, the particles were magnetically separated, washed with ethanol and water, and dried at 60°C.

General Procedure for Sulfonic Acid Functionalization ($Fe_3O_4–SO_3H$)

0.5 g of Fe_3O_4 nanoparticles were dispersed in 50 mL of toluene via ultrasonication. Then, 1 mL of 1,3-propane sultone was added, and the mixture was refluxed at 80°C for 8 hours. After cooling, the particles were washed with ethanol and dried under vacuum at 60°C.

RESULTS AND DISCUSSION

XRD Analysis

The X-ray diffraction pattern of Fe_3O_4 nanoparticles showed peaks at 2θ = 30.1°, 35.5°, 43.3°, 53.6°, 57.1°, and 62.7°, which correspond to the (220), (311), (400), (422), (511), and (440) planes of the magnetite crystal structure, respectively (Fig.-1 and 2). The sharpness of the peaks confirmed the high crystallinity of the synthesized material and the absence of impurity phases. These peaks match well with the standard diffraction pattern of magnetite (Fe_3O_4, JCPDS No. 19-0629)[9], confirming the formation of a pure spinel ferrite structure without detectable impurities such as hematite (α-Fe_2O_3) or maghemite (γ-Fe_2O_3). The sharpness and high intensity of the diffraction peaks suggest that the nanoparticles possess a high degree of crystallinity, which is crucial for their stability and magnetic properties[10]. The absence of additional peaks indicates that the co-precipitation method employed in this study successfully produced phase-pure Fe_3O_4 nanoparticles. The crystallite size was estimated using the Scherrer equation based on the full width at half maximum (FWHM) of the most intense (311) peak, which was found to be in the nanoscale range. The narrow peak widths further support the formation of well-defined crystalline domains, consistent with previous reports on magnetite nanoparticles synthesized via similar routes[4]. Moreover, the XRD results align with previous studies where Fe_3O_4 nanoparticles were utilized in catalytic and biomedical applications, as high crystallinity enhances their chemical stability and magnetic

responsiveness[4]. The well-resolved diffraction pattern also suggests that the subsequent silica coating and functionalization steps did not disrupt the core magnetite structure, which is essential for maintaining the superparamagnetic behavior necessary for magnetic separation in catalytic and environmental applications[8].

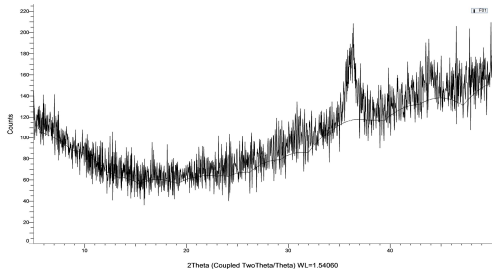

Fig.-1: XRD Pattern of Surface-Functionalized Fe_3O_4 Nanoparticles

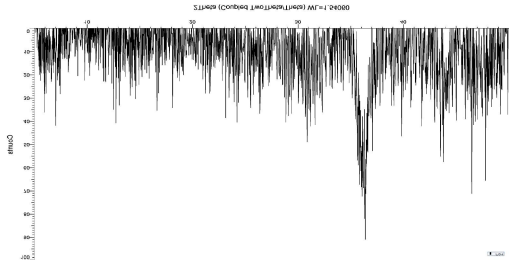

Fig.-2: XRD Pattern of Unmodified Fe_3O_4 Nanoparticles Synthesized via Co-precipitation

Particle Size Analysis

The dynamic light scattering (DLS) analysis provided insight into the hydrodynamic sizes and polydispersity of the synthesized nanoparticles at various stages of surface modification. The Z-average diameter for the bare Fe_3O_4 nanoparticles (Sample PS1) was found to be 181.9 nm with a polydispersity index (PdI) of 0.957, indicating a relatively broad size distribution likely due to aggregation tendencies. Upon silica coating and amino-functionalization (Sample PS2: Fe_3O_4@SiO_2–NH_2), the Z-average increased to 238.5 nm with a PdI of 0.933, suggesting the formation of a uniform silica shell and successful amine group incorporation. Further sulfonation to produce Fe_3O_4–SO_3H (Sample PS3) resulted in a slight increase in particle size to 241.2 nm with a PdI of 0.942. The gradual increase in Z-average values along with consistently high PdI values confirms successful surface modification steps, although the relatively broad size distributions reflect possible interparticle interactions or minor agglomeration in suspension.

Zeta Potential Analysis

Zeta potential measurements were conducted to evaluate the colloidal stability and surface charge variations resulting from successive functionalization steps. The bare Fe_3O_4 nanoparticles (ZP1) exhibited a zeta potential of –22.6 mV, indicating moderate electrostatic stability due to negatively charged surface groups. Following silica coating and amine functionalization (ZP2: Fe_3O_4@SiO_2–NH_2), the zeta potential increased to –3.56 mV, reflecting a significant reduction in surface charge, likely due to the neutral or slightly positive nature of the introduced amine groups partially neutralizing the original surface charges. Subsequent sulfonation (ZP3: Fe_3O_4–SO_3H) led to a zeta potential of –8.91 mV, suggesting the incorporation of negatively charged sulfonic acid groups, which partially restored the negative surface charge. These changes in zeta potential confirm successful surface modifications and demonstrate the influence of functional groups on the colloidal behavior of the nanoparticles. The decrease in zeta potential magnitude after silica and further functionalization reflects the surface charge alterations and is consistent with successful grafting of functional groups. These systematic changes in both particle size and surface charge provide compelling evidence for the successful stepwise functionalization of the nanoparticles. While the reduced zeta potential magnitude might theoretically decrease electrostatic stabilization, the introduced organic groups provide steric stabilization that maintains colloidal integrity[2]. This combination of properties is particularly valuable for applications requiring precise surface chemistry control, such as targeted drug delivery systems or heterogeneous catalysis. The ability to tune

both size and surface characteristics through controlled functionalization steps underscores the versatility of these nanoparticle systems for diverse technological applications.

CONCLUSION

Fe_3O_4 nanoparticles were synthesized by a simple co-precipitation method and successfully functionalized with silica, amino, and sulfonic acid groups. Characterization by XRD, DLS, and zeta potential confirmed the formation of crystalline magnetite and effective surface modifications. These functionalized nanoparticles show promise for catalytic and separation processes due to their tunable surface chemistry and magnetic recovery potential.

CONFLICT OF INTERESTS

The authors declare no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-9: Industry, Innovation, and Infrastructure.* All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

A.D. Kanade http://orchid.org/ 0009-0003-4501-3577
S. Jadhav http://orchid.org/0000-0001-3375-5318
B. Madje http://orchid.org/0000-0001-9297-4731
D. Sonwane http://orchid.org/ 0009-0001-7100-1604
N. Rehman http://orchid.org/0000-0002-1224-0656
S. Mahire http://orchid.org/0009-0002-7441-2051
R. Patil http://orchid.org/0009-0002-2842-1408
D. Chavan http://orchid.org/0009-0005-5476-0771

REFERENCES

1. A. Skiada, C. Lass-Floerl, N. Klimko, A. Ibrahim, E. Roilides, G. Petrikkos, *Medical Mycology*, **56**, S93(2018), https://doi.org/10.1093/mmy/myx101
2. A. S. Ibrahim, B. Spellberg, V. Avanessian, Y. Fu, J. E. Edwards, *Infection and Immunity*, **73**, 778(2005), https://doi.org/10.1128/IAI.73.2.778-783.2005
3. H. Prakash, A. Chakrabarti, *Microorganisms*, **9**, 523(2023), https://doi.org/10.3390/microorganisms9030523
4. H. Gogineni, W. So, K. Mata, J. N. Greene, *The Egyptian Journal of Internal Medicine*, **34**, 58(2022), https://doi.org/10.1186/s43162-022-00143-7
5. A. K. Gupta, M. Gupta, *Biomaterials*, **26**, 3995(2005), https://doi.org/10.1016/j.biomaterials.2004.10.012
6. B. Karimi, F. Mansouri, H. M. Mirzaei, *ChemCatChem*, **6**, 2126(2014), https://doi.org/10.1002/cctc.201402067
7. S. Laurent, D. Forge, M. Port, A. Roch, C. Robic, L. Vander Elst, R. N. Muller, *Chemical Reviews*, **108**, 2064(2008), https://doi.org/10.1021/cr068445e
8. A. H. Lu, E. L. Salabas, F. Schüth, *Angewandte Chemie International Edition*, **46**, 1222(2007), https://doi.org/10.1002/anie.200602866
9. M. Mahmoudi, S. Sant, B. Wang, S. Laurent, T. Sen, *Advanced Drug Delivery Reviews*, **63**, 24 (2011), https://doi.org/10.1016/j.addr.2010.05.006
10. Y.-X. J. Wang, S. Xuan, M. Port, J.-M. Idee, *Current Pharmaceutical Design*, **22**, 825(2016), https://doi.org/10.2174/1381612822666151209154706

[RJC-9429/2025]