

Special Issue on Chemistry

Microwave Synthesis and Magnetic Properties of Spinel CoFe_2O_4 Nanoparticles using Prickly Pear Cactus (*Opuntia fuliginosa*) Plant Extract

M. Lakshmi, B. Muthu and A. Manikandan

Issue Editor
Dr. A. Manikandan

Research Journal of Agricultural Sciences
An International Journal

P- ISSN: 0976-1675
E- ISSN: 2249-4538

Volume: 13
Issue: Special

Res. Jr. of Agril. Sci. (2022) 13(S): 083–087



Microwave Synthesis and Magnetic Properties of Spinel CoFe_2O_4 Nanoparticles using Prickly Pear Cactus (*Opuntia fuliginosa*) Plant Extract

M. Lakshmi¹, B. Muthu² and A. Manikandan*³

Received: 07 Dec 2021 | Revised accepted: 15 Feb 2022 | Published online: 25 Feb 2022
© CARAS (Centre for Advanced Research in Agricultural Sciences) 2022

ABSTRACT

Spinel CoFe_2O_4 nanoparticles were synthesized by microwave heating method using prickly pear cactus (*Opuntia fuliginosa*) plant extract as a bio reducing agent. The obtained samples are analyzed by chemical and physical properties. Average crystallite size (nm) is discovered to be fluctuating between 19 - 24 nm. The surface morphology was dissected by utilizing SEM and TEM analysis. The metal oxide (M-O) band extending vibrations was confirmed by infrared spectral analysis. Spinel CoFe_2O_4 NPs can be raised as a noticeable antibacterial activity against human pathogens, due to their smaller size and higher surface area of the samples.

Key words: *Opuntia fuliginosa*, Microwave heating, CoFe_2O_4 NPs, FTIR, XRD, TEM, Antibacterial activity

In recently years, spinel ferrites with general recipe AB_2O_4 (A: Zn, Co, Cu, etc.) have fascinated a lot of attention as a result of the physical, chemical and biomedical properties [1-5]. The AB_2O_4 is the overall recipe for this spinel structure. Various techniques, such as sol-gel [6], co-precipitation [7], hydrothermal [8] method, etc. have been reported. It is utilized to noticeable light photocatalysis [9]. Mixed oxides with spinel type materials are furthermore among the gigantic number of photocatalyst pieces researched. Semiconductor materials are along with the bigger part potential photocatalysts for hydrogen age since they contain sensitive constancy, fixed temperament and thermodynamically best band structures [10]. The microwave heating method is the standard procedure for the synthesis of CoFe_2O_4 [11-12]. The CoFe_2O_4 is a confined band-opening semiconductor fabric, which have gigantic view for applications in clear light photocatalysis [13–16]. The Fe-O-Fe bond point is a scale to register the degree of distortion which impacts the hidden, optical, appealing, dielectric and various properties of the compound [17]. MFe_2O_4 pottery creation have been the goal of considered experts on account of their valuable properties in different applications going from solid oxide power gadgets [18], sensors [19], and normal driving forces [20].

Researchers are looking for new methods to treatment [21]. Recent findings suggest that a number of nanomaterials

induce neuroprotective effects and preserve neuronal life. These nanomaterials are deployed due to their nanometer size, antioxidant properties [22], minimal toxicity, magnetic properties [23-25], etc. In recent decades, ferrite magnetic nanoparticles (NPs) with the chemical formula of AFe_2O_4 (A: Zn, Co, Cu, etc.) [26-30] have attracted the attention of researchers in various biomedical fields including drug delivery etc. are of particular importance in neuroprotection and also can be employed as magnetic resonance imaging (MRI) contrast agents [31-33]. Cobalt ferrite NPs are ceramic nanoscale materials [34-38], which can be deployed different biomedical and biological applications. In this study, Cobalt ferrite NPs were eco-friendly synthesized using prickly pear cactus (*Opuntia fuliginosa*) plant extract as a bio reducing agent. The physicochemical properties of CoFe_2O_4 NPs were determined by XRD, SEM, TEM, and VSM analyses. Spinel CoFe_2O_4 NPs can be raised as a noticeable antibacterial activity against human pathogens, due to their smaller size and higher surface area of the samples.

MATERIALS AND METHODS

Everyone chemicals of elevated purity marks were used without further sanitization. Iron (III) nitrate ($\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, 99.9%), Cobalt (II) nitrate ($\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$, 99.98%) were obtained from Sigma Aldrich. Prickly pear cactus (*Opuntia fuliginosa*) plant extract was used for synthesis of CoFe_2O_4 .

Iron (III) nitrate, cobalt (II) nitrate (Sigma-Aldrich, 99%), and Prickly pear cactus (*Opuntia fuliginosa*) plant extract were utilized for the green synthesis of CoFe_2O_4 NPs. Deionized water was utilized in all stages of the synthesis. Prickly pear cactus (*Opuntia fuliginosa*) plant extract were

* A. Manikandan

✉ manikandana.che@bharathuniv.ac.in

¹⁻³ Department of Chemistry, Bharath Institute of Higher Education and Research (BIHER), Chennai - 600 073, Tamil Nadu, India

washed with deionized water. Plant moisture was removed at 50°C. To each gram of watercress powder, 10 ml of deionized water was added and shaken at 50°C, overnight. Finally, the extract was separated with Whatman paper and centrifuged. Iron (III) nitrate, cobalt (II) nitrate solution was added to 50 ml of aqueous extract. The mixture was kept in a microwave oven. The final product were washed well with DI water and ethanol twice finally dried at 70°C and used for further characterizations.

RESULTS AND DISCUSSION

XRD analysis

In microwave heating helped union procedure, metal salts were blended in with urea earlier than microwave treatment. The ensuing combination was then exposed to microwave light to accomplish spinel CoFe_2O_4 as of nitrate salt of as well as iron and cobalt. The phase and immaculateness were portrayed by combined materials as displayed in Fig. 1. The pinnacles are listed to average nanoparticle design of CoFe_2O_4 alluding to the nanoparticle nature of cubic structure. The crystallite size (D) of the example is assessed utilizing Scherrer's eqn. 1 [21]:

$$D = \frac{k\lambda}{\beta \cos \theta} \text{-----(1)}$$

where L is the crystallite size, λ , the X-ray wavelength, θ , the Bragg diffraction angle and β , the full width at half maximum (FWHM). The average crystallite size ' L ' calculated from the diffraction peaks was found to be around 23 nm.

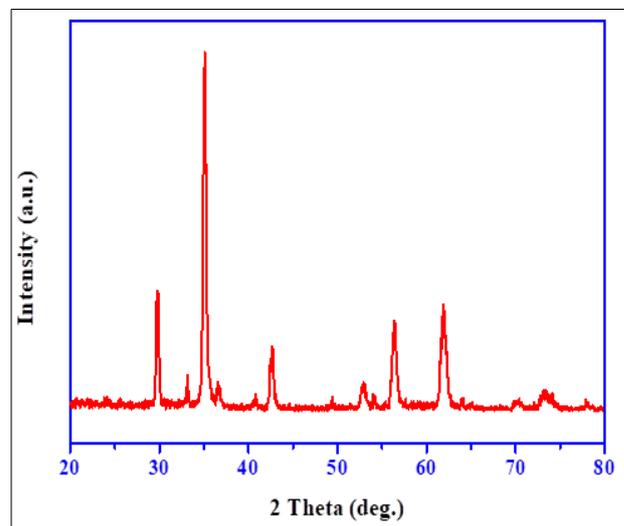


Fig 1 XRD patterns of CoFe_2O_4 nanoparticles

Morphological studies

SEM and TEM investigation (Fig. 2a and 2b respectively) takes spinel state of CoFe_2O_4 as additionally affirmed by XRD result. SEM and TEM portrays miniature construction with gem specifics of microwave incorporated CoFe_2O_4 . The cross-section planes with unidirectional course of action express by presence of crystalline nature of CoFe_2O_4 . Basic SAED examples of these examples (Fig. 2c) show ring designs, demonstrating a crystalline dependable with the XRD results.

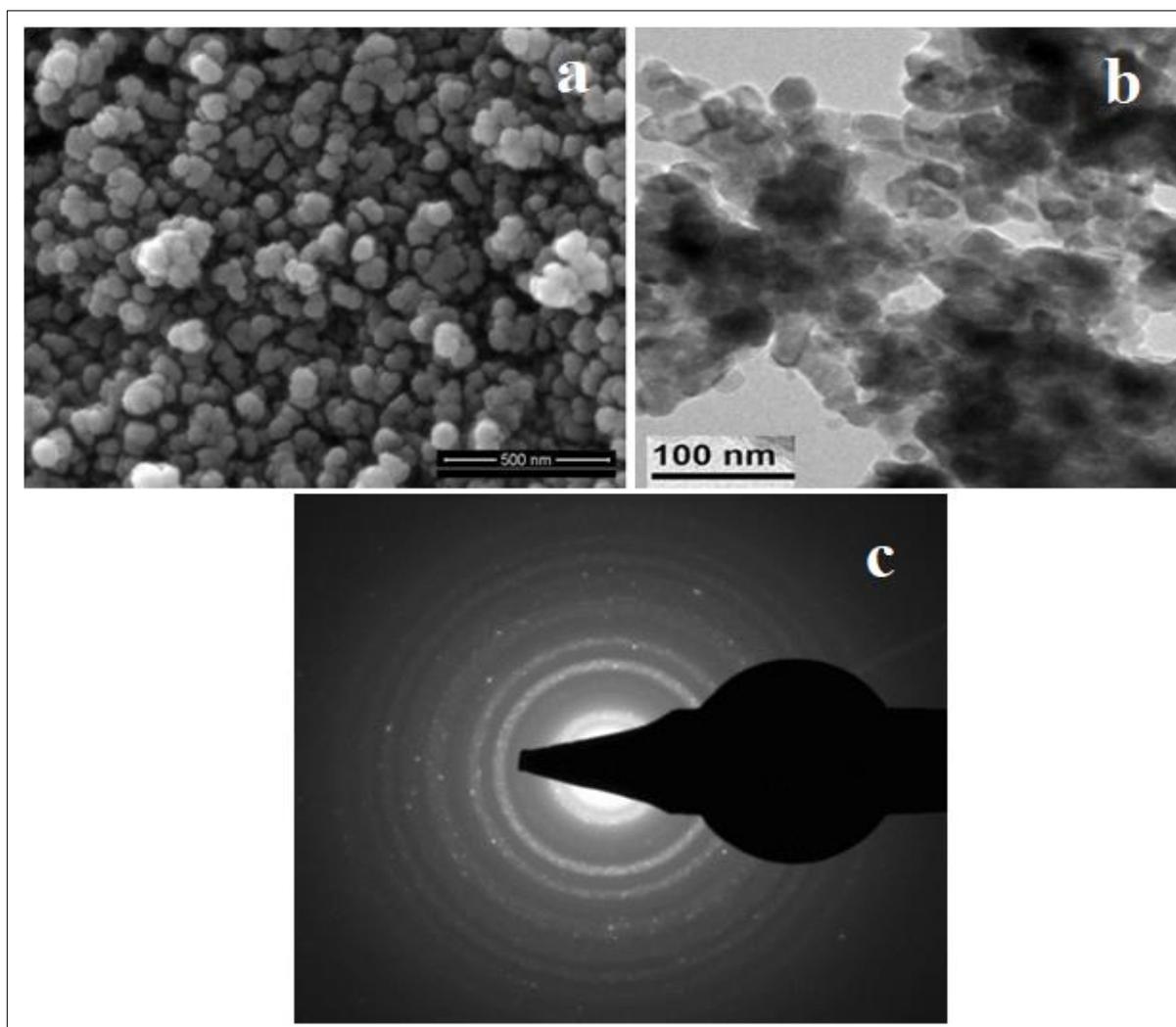
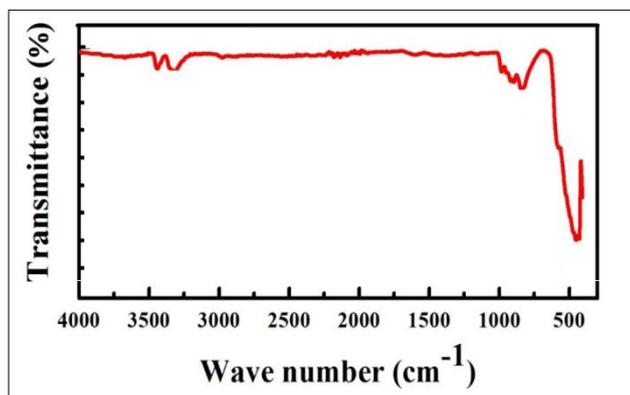


Fig 2 SEM (a), TEM (b) and SAED (c) pattern of CoFe_2O_4 nanoparticles

Fig 3 FT-IR spectra of CoFe_2O_4 nanoparticles

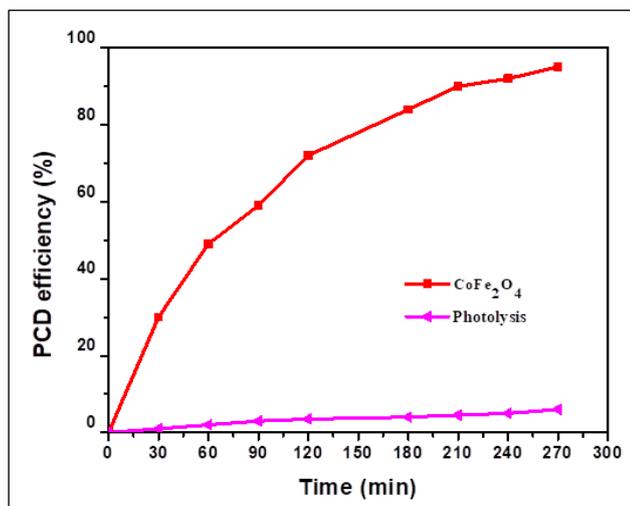
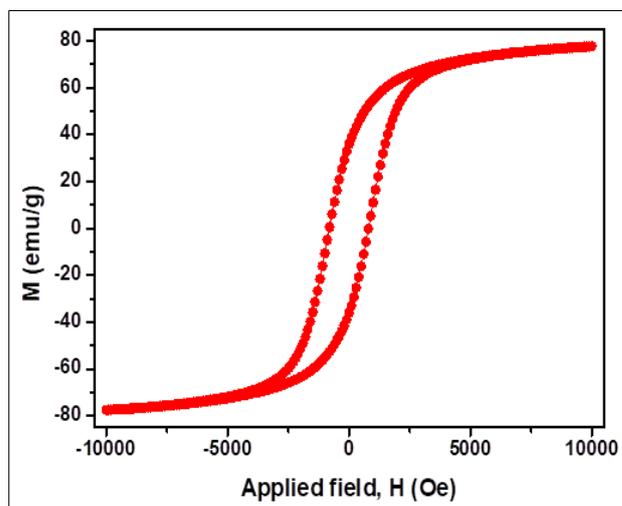
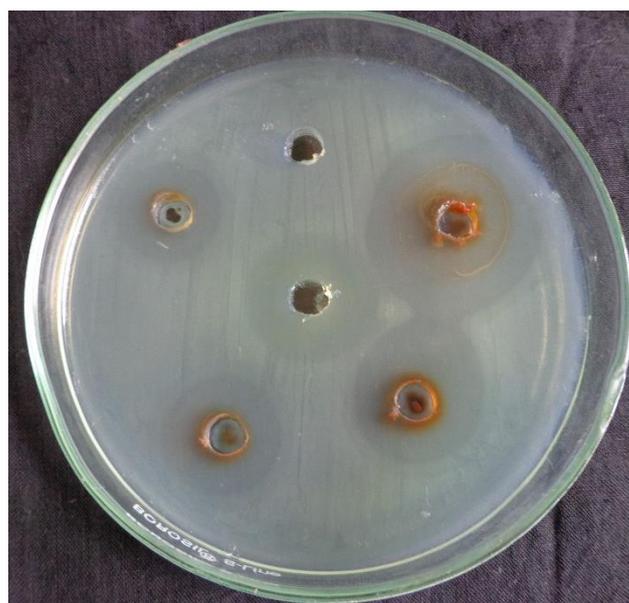
FT-IR analysis

The Fourier change infrared spectra (FTIR) of CoFe_2O_4 nanoparticles are shown in Fig. 3. The expansive retention band

around $420 - 800 \text{ cm}^{-1}$ Shows in all spectra. A little shift frequency of 546 cm^{-1} is distinguished for metal – oxide (M – O). The wave number of 3420 cm^{-1} is more ingestion band is noticed water molecules [22]. The bowing methods of O-H band are seen in roughly 1372 cm^{-1} and 1620 cm^{-1} . The metal – oxide groups presence of the little ingestion top at $420 - 800 \text{ cm}^{-1}$ which the vibrations of spinel ferrites bonds are allotted [23].

Photocatalytic evaluation

The photocatalytic dye degradation using CoFe_2O_4 nanoparticles for Congo Red (CR) and obtained results are shown in Fig 4. The interest of Congo Red dye was kept up with after lighting for 2 hrs in the shy of the photocatalyst, which affirms that there was no photolysis of Congo Red. It is remarkable that Congo Red nearly corrupted $\sim 94\%$ under noticeable light brightening inside 120 min over CoFe_2O_4 nanoparticles.

Fig 4 Photocatalytic degradation of Congo Red using CoFe_2O_4 nanoparticlesFig 5 VSM analysis of CoFe_2O_4 nanoparticlesFig 6 Antibacterial activity of CoFe_2O_4 nanoparticles

VSM analysis

Magnetic hysteresis (M-H) curves recorded at room temperature (RT) shows in Fig. 5. From the M-H loop it was observed the magnetic property of CoFe_2O_4 nanoparticles. The M_s value is 77 emu/g . However, the permeability spectrum of

general characteristics M_s remains constant in a convinced frequency range. The advent of M_s is due to the casing of the motion of the domains with the applied field. Remanent magnetization (M_r) and coercivity (H_c) values are very low, which shows the soft magnetic nature of the samples.

Antibacterial activity

The antibacterial activity was determined by well diffusion methods (Fig 6). About 25 mL of molten Mueller Hinton Agar was poured into a sterile Petri plate (Himedia, Mumbai, India). The plates were allowed to solidify, after which 18 h grown (OD adjusted 0.6) 100 μ l of above said pathogenic bacteria cultures were transferred onto plate and made culture lawn by using sterile L-rod spreader. After five min setting of the bacteria, the wells were made using sterile 5 mm cork borer and test samples were dissolved in sterile water at various concentrations (i.e., 25, 50, 75 and 100 μ l/well). The sterile water served as control. The plates were incubated at 37°C in a 40 W fluorescent light source (~ 400 nm) for 24 h. The antibacterial activity was determined by measuring the diameter of the zone of inhibition around the well using antibiotic zone scale (Himedia, Mumbai, India). The given samples were capable to kill all the tested pathogens ranging from 6 mm to 29 mm of zone of inhibition (Refer Figures). The test material highly active against all test pathogens.

CONCLUSION

In this work, CoFe₂O₄ nanoparticles can be effectively prepared by microwave method using prickly pear cactus (*Opuntia fuliginosa*) plant extract as a bio reducing agent. Powder XRD results signify the pure spinel phase CoFe₂O₄ nanoparticles. XRD, SEM, TEM, FTIR, VSM and photo degradation studies were carried out. CoFe₂O₄ nanoparticles exhibited improved absorption ability especially in the visible light region. The photocatalytic exercises of the pre-arranged photocatalysts were assessed by photocatalytic debasement of CR under apparent light illumination. It was discovered that CoFe₂O₄ nanoparticles photocatalysts showed a lot higher photocatalytic action. The ferromagnetic behaviours saw from magnetic - hysteresis loop.

Acknowledgment

The authors are thankful to Tamil Nadu State Council for Science and Technology (TNSCST), DOTE Campus, Chennai for the financial support (S&T Project: TNSCST/ STP-PRG/AR/2018-2019/9307).

LITERATURE CITED

1. A. Manikandan, E. Hema, M. Durka, K. Seevakam, T. Alagesan, S. Arul Antony, Room temperature ferromagnetism of magnetically recyclable photocatalyst of Cu_{1-x}Mn_xFe₂O₄-TiO₂ (0.0 \leq x \leq 0.5) nano-composites, J. Supercond. Nov. Magn., 28 (2015) 1783-1795.
2. V. Umapathy, A. Manikandan, S. Arul Antony, P. Ramu, P. Neeraja, Synthesis, structural, morphological and opto-magnetic properties of Bi₂MoO₆ nano-photocatalyst by sol-gel method, Transactions of Nonferrous Metals Society of China, 25 (2015) 3271-3278.
3. E Murugan, M Ariraman, S Rajendran, J Kathirvel, CR Akshata, K Kumar, Core-Shell Nanostructured Fe₃O₄-Poly(styrene-co-vinylbenzyl chloride) Grafted PPI Dendrimers Stabilized with AuNPs/PdNPs for Efficient Nuclease Activity, ACS omega, 2018, 3 (10), 13685-13693.
4. P Shanmugam, K Rajakumar, R Boddula, RC Nnullie, W Wei, J Xie, Heterogeneous form of poly (4-vinyl pyridine) beads based dendrimer stabilized Ag, Au and PdNPs catalyst for reduction of trypan blue, Materials Science for Energy Technologies, 2019, 2 (3), 532-542.
5. E Murugan, S Santhoshkumar, S Govindaraju, M Palanichamy, Silver nanoparticles decorated g-C₃N₄: An efficient SERS substrate for monitoring catalytic reduction and selective Hg²⁺ ions detection, Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy, 2021, 246, 119036.
6. A. Manikandan, M. Durka, S. Arul Antony, Magnetically recyclable spinel Mn_xZn_{1-x}Fe₂O₄; (0.0 \leq x \leq 0.5) nano-photocatalysts, Advanced Science, Engineering and Medicine, 7 (2015) 33-46.
7. V. Mary Teresita, A. Manikandan, B. Avila Josephine, S. Sujatha, S. Arul Antony, Electro-magnetic properties and humidity sensing studies of magnetically recoverable LaMg_xFe_{1-x}O_{3- δ} perovskites nano-photocatalysts by sol-gel route, J. Supercond. Nov. Magn., 29 (2016) 1691-1701.
8. B. Avila Josephine, A. Manikandan, V. Mary Teresita, S. Arul Antony, Fundamental study of LaMg_xCr_{1-x}O_{3- δ} perovskites nano-photocatalysts: Sol-gel synthesis, characterization and humidity sensing, The Korean Journal of Chemical Engineering, 33 (2016) 1590-1598.
9. D. Maruthamani, S. Vadivel, M. Kumaravel, B. Saravanakumar, B. Paul, S. Sankar Dhar, A. H. Yangjeh, A. Manikandan, G. Ramadoss, Facile synthesis of Bi₂O₃/reduced graphene oxide (RGO) nanocomposite for supercapacitor and visible light photocatalytic applications, Journal of Colloid and Interface Science, 498 (2017) 449-459.
10. A. Shameem, P. Devendran, V. Siva, M. Raja, A. Manikandan, S. A. Bahadur, Preparation and characterization studies of nanostructured CdO thin films by SILAR method for photocatalytic applications, Journal of Inorganic and Organometallic Polymers and Materials, 27 (2017) 692-699.
11. A. Silambarasu, A. Manikandan, K. Balakrishnan, Room temperature superparamagnetism and enhanced photocatalytic activity of magnetically reusable spinel ZnFe₂O₄ nano-catalysts, J. Supercond. Nov. Magn., 30 (2017) 2631-2640.
12. R. Bomila, S. Srinivasan, S. Gunasekaran, A. Manikandan, Enhanced photocatalytic degradation of methylene blue dye, opto-magnetic and antibacterial behaviour of pure and La-doped ZnO nanoparticles, J. Supercond. Nov. Magn., 31 (2018) 855-864.
13. I. J. C. Lynda, M. Durka, A. Dinesh, A. Manikandan, S. K. Jaganathan, A. Baykal, S. Arul Antony, Enhanced Magneto-optical and Photocatalytic Properties of Ferromagnetic Mg_{1-y}Ni_yFe₂O₄ (0.0 \leq y \leq 1.0) Spinel Nano-ferrites, J. Supercond. Nov. Magn., 31 (2018) 3637-3647.
14. S. Velanganni, A. Manikandan, J. Joseph Prince, C. Neela Mohan, R. Thiruneelakandan, Nanostructured ZnO coated Bi₂S₃ thin films: Enhanced photocatalytic degradation of Methylene blue dye, Physica B: Condensed Matter, 545 (2018) 383-389.
15. E Murugan, JN Jebaranjitham, KJ Raman, A Mandal, D Geethalakshmi, Insoluble dendrimer-grafted poly (vinylimidazole) microbeads stabilized with mono/bimetallic nanoparticle catalysts for effective degradation of malachite green, New Journal of Chemistry, 2017, 41 (19), 10860-10871

16. E Murugan, SS Kumar, KM Reshna, S Govindaraju, Highly sensitive, stable g-CN decorated with AgNPs for SERS sensing of toluidine blue and catalytic reduction of crystal violet, *Journal of materials science* 2019, 54 (7), 5294-5310
17. E Murugan, R Rangasamy, Development of stable pollution free TiO₂/Au nanoparticle immobilized green photo catalyst for degradation of methyl orange, *J. biomed. Nanotech.*, 2011, 7 (1), 225-228.
18. J. A. H. Sheela, S. Lakshmanan, A. Manikandan, S. A. Antony, Structural, morphological and optical properties of ZnO, ZnO:Ni²⁺ and ZnO:Co²⁺ nanostructures by hydrothermal process and their photocatalytic activity, *J. Inorg. Organomet. Polym.* 28 (2018) 2388–2398.
19. R. A. Senthil, S. Osman, J. Pan, Y. Sun, T. R. Kumar, A. Manikandan, A facile hydrothermal synthesis of visible-light responsive BiFeWO₆/MoS₂ composite as superior photocatalyst for degradation of organic pollutants, *Ceram. Int.*, 45 (2019) 18683-18690.
20. A. Manikandan, M. Durka, K. Seevakan, S. Arul Antony, A novel one-pot combustion synthesis and opto-magnetic properties of magnetically separable spinel Mn_xMg_{1-x}Fe₂O₄ (0.0 ≤ x ≤ 0.5) nano-photocatalysts, *J. Supercond. Nov. Magn.*, 28 (2015) 1405-1416
21. A. Manikandan, E. Hema, M. Durka, M. Amutha Selvi, T. Alagesan, S. Arul Antony, Mn²⁺ doped NiS (Mn_xNi_{1-x}S: x = 0.0, 0.3 and 0.5) nanocrystals: Structural, morphological, opto-magnetic and photocatalytic properties, *J. Inorg. Organomet. Polym.*, 25 (2015) 804–815.
22. R. A. Senthil, S. Osman, J. Pan, A. Khan, V. Yang, T. R. Kumar, Y. Sun, A. Manikandan, One-pot preparation of AgBr/α-Ag₂WO₄ composites with superior photocatalytic activity under visible-light irradiation, *Colloids and Surfaces A: Physicochemical and Engineering Aspects*, 586 (2020) 124079.
23. S. Rathinavel, R. Deepika, D. Panda, A. Manikandan, Synthesis and characterization of MgFe₂O₄ and MgFe₂O₄/rGO nanocomposites for the photocatalytic degradation of methylene blue, *Inorganic and Nano-Metal Chemistry*, 51, 2 (2021) 210-217.
24. A Muthukrishnaraj, SS Kalaivani, A Manikandan, Helen P Kavitha, R Srinivasan, N Balasubramanian, Sonochemical synthesis and visible light induced photocatalytic property of reduced graphene oxide@ ZnO hexagonal hollow rod nanocomposite, *J. Alloys Compds.*, 83625 (2020) 155377.
25. T. L. Ajeesha, A. Ashwini, Mary George, A. Manikandan, J. Arul Mary, Y. Slimani, M. A. Almessiere, A. Baykal, Nickel substituted MgFe₂O₄ nanoparticles via co-precipitation method for photocatalytic applications, *Physica B*, 606 (2021) 412660.
26. E Murugan, A Siva, Preparation of a novel soluble multi-site phase transfer catalyst and the kinetic study for the C-alkylation of α-pinene. *J. Mol. Catal. A: Chem.*, 2005, 235 (1-2), 220-229.
27. SP Ratnayake, M Mantilaka, C Sandaruwan, D Dahanayake, E Murugan, Carbon quantum dots-decorated nano-zirconia: a highly efficient photocatalyst, *Appl. Catal. A: General*, 2019, 570, 23-30.
28. R. Renuga, A. Manikandan, J. A. Mary, A. Muthukrishnaraj, A. Khan, S. Srinivasan, B. Abdullah M. Al Alwan and K. M. Khedher, Enhanced Magneto-Optical, Morphological, and Photocatalytic Properties of Nickel-Substituted SnO₂ Nanoparticles, *J. Supercond. Nov. Magn.*, 34 (2021) 825–836.
29. M. George, T.L. Ajeesha, A. Manikandan, Ashwini Anantharaman, R.S. Jansi, E. Ranjith Kumar, Y. Slimani, M.A. Almessiere, A. Baykal, Evaluation of Cu-MgFe₂O₄ spinel nanoparticles for photocatalytic and antimicrobial activities, *J. Phys. Chem. Solids*, 153 (2021) 110010.
30. A. Muthukrishnaraj , S. A. Al-Zahrani , A. Al Otaibi , S. S. Kalaivani, A. Manikandan, N. Balasubramanian, A. L. Bilgrami, M. A. R. Ahamed, A. Khan, A. M. Asiri, N. Balasubramanian, Enhanced Photocatalytic Activity of Cu₂O Cabbage/RGO Nanocomposites under Visible Light Irradiation, *Polymers*, 13 (2021) 1712.
31. K. Geetha, R. Udhayakumar, A. Manikandan, Enhanced magnetic and photocatalytic characteristics of cerium substituted spinel MgFe₂O₄ ferrite nanoparticles, *Physica B*, 615 (2021) 413083.
32. C. Sambathkumar, V. Manirathinam, A. Manikandan, M. Krishna Kumar, S. Sudhakar, P. Devendran, Solvothermal synthesis of Bi₂S₃ nanoparticles for active photocatalytic and energy storage device applications, *J. Mater. Sci.: Mater. Electron.*, 32 (2021) 20827-20843.
33. E Murugan, P Gopinath, V Shanmugayya, N Mathivanan, Antibacterial activity of novel insoluble bead-shaped polymer-supported multiquaternary ammonium salts, *J. Appl. Polymer Sci.*, 2010, 117 (6), 3673-3678
34. E Murugan, I Pakrudheen, Efficient amphiphilic poly (propylene imine) dendrimer encapsulated ruthenium nanoparticles for sensing and catalysis applications, *Sci. Ad. Mater.*, 2015, 7 (5), 891-901.
35. E Murugan, JN Jebaranjitham, A Usha Synthesis of polymer-supported dendritic palladium nanoparticle catalysts for Suzuki coupling reaction, *Appl. Nanosci.*, 2012, 2 (3), 211-222
36. E Murugan, G Vimala, Synthesis, characterization, and catalytic activity for hybrids of multi-walled carbon nanotube and amphiphilic poly(propyleneimine) dendrimer immobilized with silver and palladium nanoparticle, *J. Colloid Interf. Sci.* 2013, 396, 101-111.
37. E Murugan, JN Jebaranjitham, Dendrimer grafted core-shell Fe₃O₄-polymer magnetic nanocomposites stabilized with AuNPs for enhanced catalytic degradation of Rhodamine B—A kinetic study, *Chem. Eng. J.*, 2015, 259, 266-276.
38. E Murugan, RL Sherman, HO Spivey, Catalysis by hydrophobically modified poly (propyleneimine) dendrimers having quaternary ammonium and tertiary amine functionality, *WT Ford, Langmuir*, 2004, 20 (19), 8307-8312.