

Antibacterial activity and magnetic properties of Sr-NiFe₂O₄ nanoparticles synthesized by simple microwave combustion method

S. Syed Iqbal^{1,*}, G. Padma Priya¹

¹ Department of Chemistry, Faculty of Arts and Science, Bharath Institute of Higher Education and Research (BIHER), Chennai – 600073, Tamil Nadu, India

*Corresponding Author Email addresses: ssiavsns@gmail.com (S. Syed Iqbal)

Address for Correspondence

S. Syed Iqbal^{1,*}, G. Padma Priya¹

¹ Department of Chemistry, Faculty of Arts and Science, Bharath Institute of Higher Education and Research (BIHER), Chennai – 600073, Tamil Nadu, India

*Corresponding Author Email addresses: ssiavsns@gmail.com (S. Syed Iqbal)

Abstract

Microwave combustion method (MCM) was used to synthesize of spinel Sr-NiFe₂O₄ NPs by employing the fuel Urea. The physical characteristics were obtained by following methods viz. powder XRD, SEM, EDX, FT-IR and VSM techniques. The diffraction studies revealed that the average crystallite size exist in the band of 15.22 nm to 25.55 nm. The HR-SEM pictures revealed the agglomerated and spherical morphology of spinel Sr-NiFe₂O₄ nanoparticles. Elemental analysis ensured that the existence of Ni, Sr, O and Fe ions. The broad peaks at 439 cm⁻¹ and 585 cm⁻¹ corresponds to octahedral metal stretching and tetrahedral metal stretching of Sr-NiFe₂O₄ respectively. Magnetic results revealed that the prepared NPs are ferromagnetic in nature. The antibacterial activity (ABA) of gram-positive *Staphylococcus aureus*, *Bacillus subtilis* have been investigated using Sr-NiFe₂O₄ NPs. It was found that the improved activity is intensified with smooth Sr²⁺ doping as it causes a decrease in the grain size.

Key words: Sr-NiFe₂O₄; Microwave combustion; Antibacterial activity; VSM technique.

1. Introduction

Recently, magnetic spinel ferrites are great important in the field of materials science, due to their enhanced structural, morphology, optical, magnetic and vibrational properties and also they have been widely used for applications such as magnetic fluid for the purpose of information storage, drug delivery, and many more [1-4]. The general formula for cubic spinel structure-based ferrite is MFe₂O₄. The M²⁺ and Fe³⁺ ions occupy two distinct sites namely A²⁺ and B³⁺ [4, 5]. Based on the theory of Wyckoff positions, the spinels exhibit three different

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crystal structures namely normal, inverse and mixed. In case of normal spinel structure A^{2+} and Fe^{3+} ions sits in the A- and B- sites respectively [$A^{2+}[Fe^{3+}]O^{2-}$]. While, A^{2+} and Fe^{3+} ions occupies B- and A- sites in case of inverse spinel [6-10].

Different techniques such as hydrothermal, microwave, sol-gel, ball milling, co-precipitation, solvothermal, and auto combustion [11-15] are widely utilized. However, the above techniques are high cost, difficult to synthesis, more time and even low yield [16-20]. Microwave combustion provides many advantages like gentle chemistry route, economic, simple preparation, etc. It is found that fuel plays a critical role in tuning the physical properties such as structure, morphology, magnetism etc [21-25]. In this paper Sr-NiFe₂O₄ NPs was synthesized through MCM. Further, morphology, phase, and magnetic properties were studied in detail using various physica characterization techniques.

2. Experimental**2.1 Materials and method**

Analytical grade Iron nitrate ($Fe(NO_3)_3 \cdot 9H_2O$), L-arginine ($C_6H_{14}N_4O_2$), Nickel nitrate ($Ni(NO_3)_2 \cdot 6H_2O$) and Strontium nitrate ($Sr(NO_3)_2$) attained from SD fine, India, and were utilized as it is without undergoing an additional purification process. A homogenous solution is attained by mixing precursors namely nickel nitrate and iron nitrate by maintaining a molar ratio of 1:2. It is further added to urea solution, and this solution was mixed for a period of 1hr. Here, urea is used as fuel and, nitrates precursor plays the role oxidizer [26-30]. Silica crucible was kept inside the microwave oven (SAMSUNG, India make). Then, solution went through process namely boiling and dehydration. Upon achievement of spontaneous combustion by the solution, ignition occurred which results in rapid flame fluffy production of Sr-NiFe₂O₄. Further, the obtained samples were calcined at 550 °C for 150 min.

2.2. Antibacterial activity

The antibacterial activity (ABA) of spinel Sr-NiFe₂O₄ NPs was analyzed by their zone of inhibitions on the human pathogens (HP) gram-positive *S. aureus* (*Staphylococcus aureus*), *B. subtilis* (*Bacillus subtilis*). An instant culture of all microorganisms was attuned to an OD of 0.1 and wiped onto Mueller Hilton (MH) agar plates. By a cork borer (CB), holes were stamped on the agar, followed by adding of the standard solutions containing the synthesized Sr-NiFe₂O₄ NPs (10µg/mL). The plates were incubated at 37°C for 24 h and the precinct of inhibitions on the human pathogens was dignified in diameter.

3. Results and discussion

3.1. X-ray diffraction analysis

The XRD pattern (Fig. 1) is used to determine the crystallite size and phase purity of prepared spinel Sr-NiFe₂O₄ NPs. The observed diffraction peaks at 2θ values are indexed to (220), (311), (222), (400), (422), and (622) respectively. All the observed diffraction peaks perfectly matched with JCPDS card number 44-1484 signifying the formation of NiFe₂O₄ [24].

The impurity peaks correspond to α-Fe₂O₃ phase verified by the card ICSD – 088418 data for Sr doped nickel ferrite. This impurity phase may be associated with the combustion in oxygen rich environment for during the preparation [25]. Debye Scherrer's method is used to deduce average crystallite (*L*) of the prepared NPs along the reflection plane (311) using Eq. (1). where, λ, the X-ray source wavelength (0.15406 nm); *L*, is the average crystallite size; β, the full width at half maximum (FWHM) of the observed diffraction peak; and θ, the diffraction angle. The value of (*L*) was found to exist between 14.25 nm to 27.52 nm, in range.

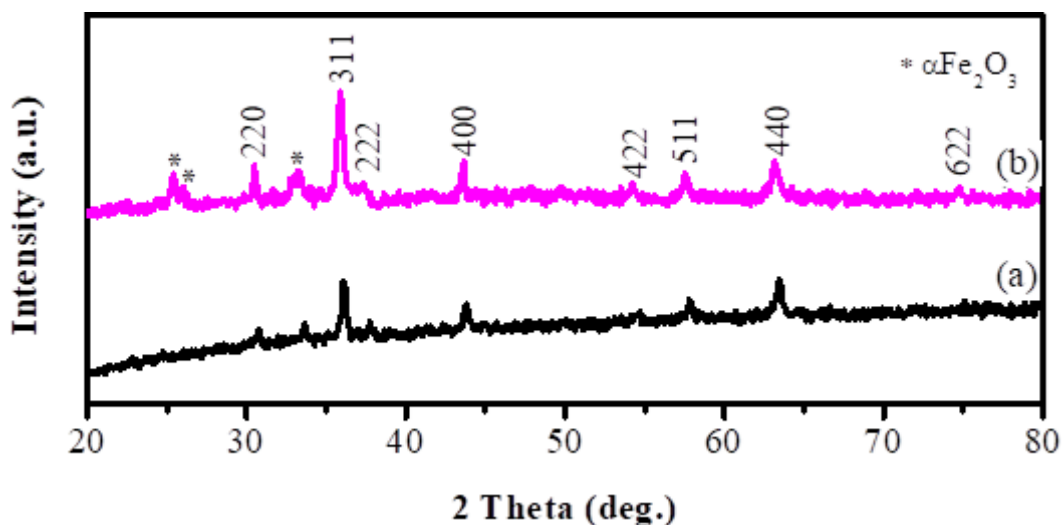


Figure 1. XRD patterns of Sr-NiFe₂O₄ nanoparticles.

3.2. Scanning electron microscopy (SEM) The HR-SEM images of spinel Ni_{1-x}Sr_xFe₂O₄ (x= 0 to 0.5) NPs revealed homogeneous spherical morphology. Further the particles exhibited

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coalescence and agglomerated, which mainly occurs due to the microwave combustion reaction during the process of synthesis [25- 30]. The energy dispersive X-ray analysis confirms that the nickel ferrite nanoparticles comprises of the elements Ni, Fe and O. Further, Fig.3 shows that all the elemental peaks of the elements Sr, Ni, Fe and O, corresponding to the formation of Sr doped NiFe₂O₄ nanoparticles.

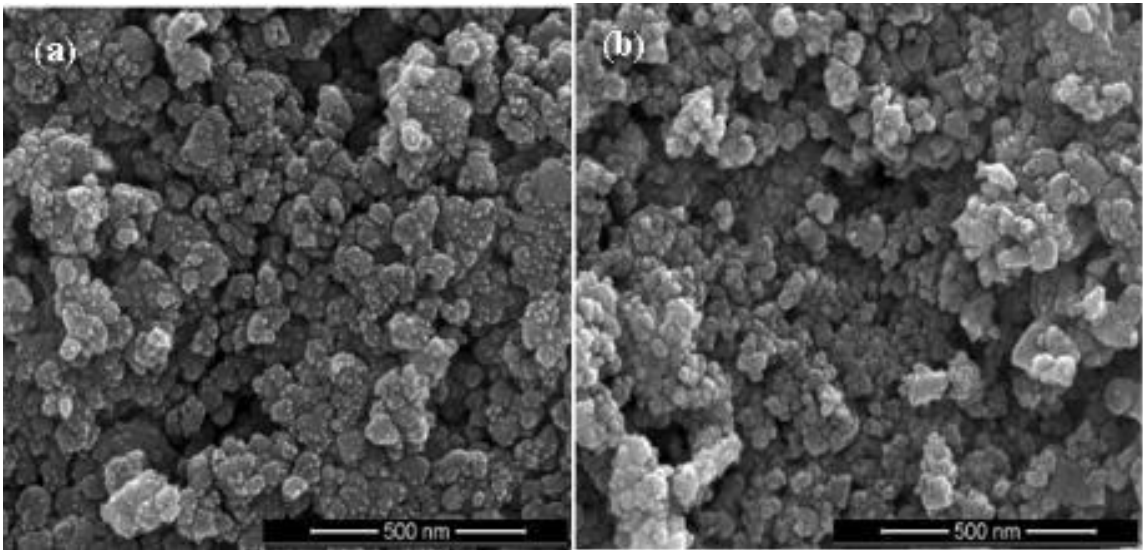


Figure 2. HR-SEM images of Sr-NiFe₂O₄ Samples

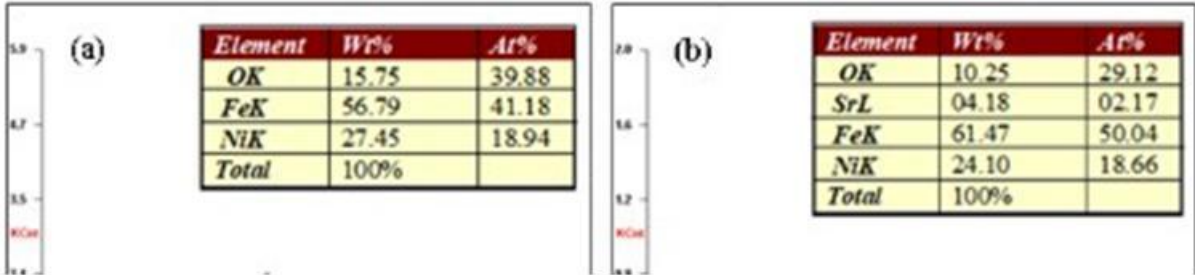


Figure 3. EDX spectra of Sr-NiFe₂O₄ samples.

3.3. FT-IR spectra

The FT-IR spectrum of Sr-NiFe₂O₄ nanoparticles was shown in Fig. 4, which was logged in the range of 4000-400 cm⁻¹ at RT. H-O stretching is due to the presence of broad peaks at 3438 cm⁻¹ [31]. C-H stretching vibration is associated with the peaks at 2925 and 285355 cm⁻¹ [4]. C-O stretching vibration on the particle surface is attributed to the absorption peaks at 1725, 1632

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and 1395 cm^{-1} [32]. The peaks at $1015\text{-}1205\text{ cm}^{-1}$ corresponds to the vibration of spinel structure NPs respectively [33]. The M-O stretching of octahedral sites is caused due to the bands at $4375, 454\text{ cm}^{-1}$. The M-O stretching at the tetrahedral sites of the spinel is due to the band at 585 cm^{-1} [34].

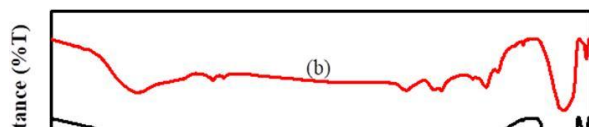


Figure 4. FTIR spectra of Sr-NiFe₂O₄ nanoparticles.

3.4. Magnetization analysis

Magnetic hysteresis curve (**Fig. 5**) of the Sr-NiFe₂O₄ NPs was measured at RT (300 K) and the stimulated field was ranged in between -15 kOe to +15kOe. The values coercivity H_c , M_r and M_s were obtained from the hysteresis curve, and it was found to be ferromagnetic in nature. The coercivity values of doped and undoped nanoparticles was found to be 295.15 Oe, 295.42, 302.92 for ($x=0.3$), further a decline in value to 285.85 Oe ($x=0.5$). This variation is due to cationic redistribution and high anisotropy [30-36].

3.5 Antibacterial activity

Antibacterial activity of spinel Sr-NiFe₂O₄ nanoparticles (**Fig. 6**) were examined beside gram-positive *S. aureus* (*Staphylococcus aureus*), *B. subtilis* (*Bacillus subtilis*) bacterial strains, respectively. From this result, it was originated that there is no zone of inhibition was obtained over the control. Moreover, a greater concentration of Sr substitution impact enhanced ABA than the lower concentration of Sr-NiFe₂O₄ products [37]. The obtained reduced particle size and higher surface area with the surface volume ratio of the samples performance a vibrant character in the antibacterial activity of samples.

4. Conclusions

Sr-NiFe₂O₄ nanoparticles were successfully synthesized by microwave combustion method. Powder XRD analysis was confirmed the cubic spinel structure. SEM pictures revealed the spherical morphology of the prepared NPs, while EDX studies further ensured

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existence of elements confirming the spinel structure. \ FT-IR bands at 436 cm^{-1} and 585 cm^{-1} , which are octahedral and tetrahedral metal stretching of ferrite NPs. The magnetization parameters are determined from the hysteresis loop. Antibacterial activity (ABA) of the samples was found that Sr-NiFe₂O₄ display enhanced antibacterial activity, due to the smaller particle size and higher surface area with the surface volume ratio of the samples.

References

1. K. Chinnaraj, A. Manikandan, P. Ramu, S. Arul Antony, P. Neeraja, Comparative study of microwave and sol-gel assisted combustion methods of Fe₃O₄ nanostructures: Structural, morphological, optical, magnetic and catalytic properties, *Journal of Superconductivity and Novel Magnetism*, 28 (2015) 179-190.
2. E. Hema, A. Manikandan, P.Karthika, M. Durka, S. Arul Antony, B. R. Venkatraman, A novel synthesis of Zn²⁺-doped CoFe₂O₄ spinel nanoparticles: Structural, morphological, opto-magnetic and catalytic properties, *Journal of Superconductivity and Novel Magnetism*, 28 (2015) 2539-2552.
3. V. Umopathy, 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.
4. A. Manikandan, S. Arul Antony, R. Sridhar, Seeram Ramakrishna, M. Bououdina, A simple combustion synthesis and optical studies of magnetic Zn_{1-x}Ni_xFe₂O₄ nanostructures for photoelectrochemical applications, *Journal of Nanoscience and Nanotechnology*, 15 (2015) 4948-4960.
5. A. Manikandan, M. Durka, S. Arul Antony, Magnetically recyclable spinel Mn_xZn_{1-x}Fe₂O₄; (0.0 ≤ x ≤ 0.5) nano-photocatalysts, *Advanced Science, Engineering and Medicine*, 7 (2015) 33-46.
6. A. Manikandan, A. Saravanan, S. Arul Antony, M. Bououdina, One-pot low temperature synthesis and characterization studies of nanocrystalline α-Fe₂O₃ based dye sensitized solar cells, *Journal of Nanoscience and Nanotechnology*, 15 (2015) 4358-4366.

Research Paper

7. M. F. Valan, A. Manikandan, S. Arul Antony, A novel synthesis and characterization studies of magnetic Co_3O_4 nanoparticles, *Journal of Nanoscience and Nanotechnology*, 15 (2015) 4580-4586.
8. M. F. Valan, A. Manikandan, S. Arul Antony, Microwave combustion synthesis and characterization studies of magnetic $\text{Zn}_{1-x}\text{Cd}_x\text{Fe}_2\text{O}_4$ ($0 \leq x \leq 0.5$) nanoparticles, *Journal of Nanoscience and Nanotechnology*, 15 (2015) 4543-4551.
9. S. Jayasree, A. Manikandan, A. M. Uduman Mohideen, C. Barathiraja, S. Arul Antony, Comparative study of combustion methods, opto-magnetic and catalytic properties of spinel CoAl_2O_4 nano- and microstructures, *Advanced Science, Engineering and Medicine*, 7 (2015) 672-682.
10. A. Mary Jacintha, A. Manikandan, K. Chinnaraj, S. Arul Antony, P. Neeraja, Comparative studies of spinel MnFe_2O_4 nanostructures: Structural, morphological, optical, magnetic and catalytic properties, *Journal of Nanoscience and Nanotechnology*, 15 (2015) 9732-9740.
11. G. Padmapriya, A. Manikandan, V. Krishnasamy, S. K. Jaganathan, S. Arul Antony, Spinel $\text{Ni}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$ ($0.0 \leq x \leq 1.0$) nano-photocatalysts: Synthesis, characterization and photocatalytic degradation of methylene blue dye, *Journal of Molecular Structure*, 1119 (2016) 39-47.
12. V. Mary Teresita, A. Manikandan, B. Avila Josephine, S. Sujatha, S. Arul Antony, Electro-magnetic properties and humidity sensing studies of magnetically recoverable $\text{LaMg}_x\text{Fe}_{1-x}\text{O}_{3-\delta}$ perovskites nano-photocatalysts by sol-gel route, *Journal of Superconductivity and Novel Magnetism*, 29 (2016) 1691–1701.
13. S. Jayasree, A. Manikandan, S. Arul Antony, A. M. Uduman Mohideen, C. Barathiraja, Magneto-optical and catalytic properties of recyclable spinel NiAl_2O_4 nanostructures using facile combustion methods, *Journal of Superconductivity and Novel Magnetism*, 29 (2016) 253–263.
14. C. Barathiraja, A. Manikandan, A. M. Uduman Mohideen, S. Jayasree, S. Arul Antony, Magnetically recyclable spinel $\text{Mn}_x\text{Ni}_{1-x}\text{Fe}_2\text{O}_4$ ($x = 0.0-0.5$) nano-photocatalysts: Structural, morphological and opto-magnetic properties, *Journal of Superconductivity and Novel Magnetism*, 29 (2016) 477-486.
15. B. Avila Josephine, A. Manikandan, V. Mary Teresita, S. Arul Antony, *Fundamental*

Research Paper

- study of $\text{LaMg}_x\text{Cr}_{1-x}\text{O}_{3-\delta}$ perovskites nano-photocatalysts: Sol-gel synthesis, characterization and humidity sensing, *The Korean Journal of Chemical Engineering*, 33 (2016) 1590-1598.
16. A. Manikandan, M. Durka, M. A. Selvi, S. Arul Antony, Sesamum indicum plant extracted microwave combustion synthesis and opto-magnetic properties of spinel $\text{Mn}_x\text{Co}_{1-x}\text{Al}_2\text{O}_4$ nano-catalysts, *Journal of Nanoscience and Nanotechnology*, 16 (2016) 448-456.
 17. A. Manikandan, M. Durka, M. A. Selvi, S. Arul Antony, Aloe vera plant extracted green synthesis, structural and opto-magnetic characterizations of spinel $\text{Co}_x\text{Zn}_{1-x}\text{Al}_2\text{O}_4$ nano-catalysts, *Journal of Nanoscience and Nanotechnology*, 16 (2016) 357-373.
 18. A. Manikandan, S. Arul Antony, Magnetically separable $\text{Mn}_x\text{Zn}_{1-x}\text{Fe}_2\text{O}_4$; ($0.0 \leq x \leq 0.5$) nanostructures: Structural, morphological, opto-magnetic and photocatalytic properties, *Synthesis and Reactivity in Inorganic, Metal-Organic, and Nano-Metal Chemistry*, 46 (2016) 1277-1297.
 19. Umapathy, A. Manikandan, P. Ramu, S. Arul Antony, P. Neeraja, Synthesis and characterizations of $\text{Fe}_2(\text{MoO}_4)_3$ nano-photocatalysts by simple sol-gel method, *Journal of Nanoscience and Nanotechnology*, 16 (2016) 987-993.
 20. S. Rajmohan, A. Manikandan, V. Jeseentharani, S. Arul Antony, J. Pragasam, Simple coprecipitation synthesis and characterization studies of $\text{La}_{1-x}\text{Ni}_x\text{VO}_3$ perovskites nanostructures for humidity sensing applications, *Journal of Nanoscience and Nanotechnology*, 16 (2016) 1650-1655.
 21. E. Hema, A. Manikandan, M. Gayathri, M. Durka, S. Arul Antony, B. R. Venkatraman, Role of Mn^{2+} -doping on structural, morphological, optical, magnetic and catalytic properties of spinel ZnFe_2O_4 nanoparticles, *Journal of Nanoscience and Nanotechnology*, 16 (2016) 5929-5943.
 22. E. Hema, A. Manikandan, P. Karthika, M. Durka, S. Arul Antony, B. R. Venkatraman, Magneto-optical properties of recyclable spinel $\text{Ni}_x\text{Mg}_{1-x}\text{Fe}_2\text{O}_4$ ($0.0 \leq x \leq 1.0$) nano-catalysts, *J. Nanoscience and Nanotechnology*, 16 (2016) 7325-7336.
 23. S. Moortheswaran, A. Manikandan, S. Sujatha, S. K. Jaganathan, S. Arul Antony, One-pot combustion synthesis and characterization studies of spinel CoAl_2O_4 nano-catalysts, *Nanoscience and Nanotechnology Letters*, 8 (2016) 424-427.

Research Paper

24. S. Moortheswaran, A. Manikandan, S. Sujatha, S. K. Jaganathan, S. Arul Antony, Selective catalytic oxidation of benzyl alcohol and characterization studies of spinel $MnAl_2O_4$ nanoparticles by a facile synthesis route, *Nanoscience and Nanotechnology Letters*, 8 (2016) 434-437.
25. P. Thilagavathi, A. Manikandan, S. Sujatha, S. K. Jaganathan, S. Arul Antony, Sol-gel synthesis and characterization studies of $NiMoO_4$ nanostructures for photocatalytic degradation of methylene blue dye, *Nanoscience and Nanotechnology Letters*, 8 (2016) 438-443.
26. S. Rajmohan, V. Jeseentharani, A. Manikandan, J. Pragasam, Co-precipitation synthesis method, characterizations and humidity sensing applications of perovskite-type mixed oxide $La_{1-x}Co_xVO_{3-\delta}$ nanocomposites, *Nanoscience and Nanotechnology Letters*, 8 (2016) 393-398.
27. K. Seevakan, A. Manikandan, P. Devendran, S. Arul Antony, T. Alagesan, One-pot synthesis and characterization studies of iron molybdenum mixed metal oxide ($Fe_2(MoO_4)_3$) nano-photocatalysts, *Advanced Science, Engineering and Medicine*, 8 (2016) 566-572.
28. G. Padmapriya, A. Manikandan, V. Krishnasamy, S. K. Jaganathan, S. Arul Antony, Enhanced catalytic activity and magnetic properties of spinel $Mn_xZn_{1-x}Fe_2O_4$ ($0.0 \leq x \leq 1.0$) nano-photocatalysts by microwave irradiation route, *Journal of Superconductivity and Novel Magnetism*, 29 (2016) 2141-2149.
29. B. Meenatchi, K. R. N. Deve, A. Manikandan, V. Renuga, and V. Sathiyalakshmi, Protic ionic liquid assisted synthesis, structural, optical and magnetic properties of Mn-doped ZnO nanoparticles, *Adv. Science, Engineering and Medicine*, 8 (2016) 653-659.
30. D. Maruthamani, S. Vadivel, M. Kumaravel, B. Saravanakumar, B. Paul, S. Sankar Dhar, A. H. Yangjeh, A. Manikandan, G. Ramadoss, Facile synthesis of Bi_2O_3 /reduced graphene oxide (RGO) nanocomposite for supercapacitor and visible light photocatalytic applications, *Journal of Colloid and Interface Science*, 498 (2017) 449-459.
31. S. Suguna, S. Shankar, S. K. Jaganathan, A. Manikandan, Novel synthesis of spinel $Mn_xCo_{1-x}Al_2O_4$ ($x = 0.0$ to 1.0) nano-catalysts: Effect of Mn^{2+} doping on structural, morphological and opto-magnetic properties, *Journal of Superconductivity and Novel Magnetism*, 30 (2017) 691–699.
32. A. Shameem, P. Devendran, V. Siva, M. Raja, A. Manikandan, S. A. Bahadur, Preparation

Research Paper

- 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.
33. B. Meenatchi, V. S. Lakshmi, A. Manikandan, V. Renuga, A. Sharmila, K. R. N. Deve, S. K. Jaganathan, Synthesis and characterizations of Co_3O_4 nanoparticles using protic ionic liquids with imidazolium cation, *Journal of Inorganic and Organometallic Polymers and Materials*, 27 (2017) 446–454.
 34. C. Neela Mohan, V. Renuga, A. Manikandan, Influence of silver precursor concentration on structural, optical and morphological properties of $\text{Cu}_{1-x}\text{Ag}_x\text{InS}_2$ semiconductor nanocrystals, *Journal of Alloys and Compounds*, 729 (2017) 407-417.
 35. P. Thilagavathi, A. Manikandan, S. K. Jaganathan, S. Arul Antony, J. Hameed Hussain, Photocatalytic activity, morphological and magneto-optical characterization studies of CoMoO_4 nanoparticles prepared by sol-gel method, *Advanced Science, Engineering and Medicine*, 9 (2017) 229-234.
 36. A. Silambarasu, A. Manikandan, K. Balakrishnan, Room temperature superparamagnetism and enhanced photocatalytic activity of magnetically reusable spinel ZnFe_2O_4 nano-catalysts, *Journal of Superconductivity and Novel Magnetism*, 30 (2017) 2631–2640.
 37. A. Manikandan, E. Manikandan, B. Meenatchi, S. Vadivel, S. K. Jaganathan, R. Ladchumananandasivam, M. Henini, M. Maaza, J. S. Aanand, Rare earth element Lanthanum doped zinc oxide (La: ZnO) nanoparticles: Synthesis structural optical and antibacterial studies, *Journal of Alloys and Compounds* 723 (2017) 1155-1161.