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D. Mahendiran and K. Rajendran

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Synthesis of Nickel Oxide Magnetic Nanoparticles by *Aloe vera* Plant Extract for Antibacterial Applications

D. Mahendiran[1] and K. Rajendran*[2]

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ABSTRACT

Nickel oxide magnetic nanoparticles have been successfully fabricated by heat treatment method using *Aloe vera* plant extract. In this research, the formation of NiO nanoparticles was determined by XRD and UV -Vis. The morphology and size of nanoparticles were observed by using TEM while magnetic property of NiO nanoparticles have been characterized by VSM. The results indicate that the synthesized NiO nanoparticles are in spherical shape and the sizes of nanoparticles are in range from 13.55 nm to 15.73 nm. It has been found that the NiO nanoparticles exhibit higher antibacterial efficiency against Klebsiellapneumoniae compared with other pathogens.

***Key words*:** Nickel oxide, Magnetic nanoparticles, Polyol process, Morphology, Antibacterial efficiency

Magnetic nanoparticles are known as nanomaterials that consists of magnetic elements, such as iron, nickel, cobalt, chromium, manganese, gadolinium, and their chemical compounds [1-2]. Their physical and chemical properties depend on the crystal structures, sizes, chemical components, and shapes of nanoparticles. Sometimes, these properties are also influence by the source of magnetic nanoparticles synthesized from technological process. The constant development of world population needs to increase the consumption of plant growth and rise around 8 million in 2025 and 9 million in 2050 [1]. In global agriculture, the farmers used several pesticides to enhance the productivity and protect the crops from pests [2]. Pesticides are the organic substances used to kill the targeted pests like insects, fungus, microbes, mollusks and nematodes, which cause diseases in crops and effect on production [3]. Around the world 3.42×106 t/y number of pesticides was used in 2015 with the release of toxic effects to human, animals, micro-organism and pollute the environment [4]. Organophosphate (Ops) is the worldwide used pesticides and it restricts the enzyme acetyl cholinesterase (AChE). This enzyme is responsible for regular functioning of central nervous system in insects and humans, resulting paralysis, destroys respiratory organs finally leads to death [5]. Dichlorvos (2,2-dichlorovinyl dimethyl phosphate) is frequently used Ops in Indian agriculture to regulate the pests in fruits, vegetables, households also in storage of grains. According to U.S. Environmental Protection Agency considers as class I toxic to generate chronical diseases and cancer, hence it is banned to practice as pesticide [6]. The detection of Ops is very important and has great interest in researchers. Many techniques are applied to detect the Organo pesticides for instance fluorescence sensor [7], amperometry, potentiometry, bio sensors [8], microbial biosensors, immunosensors, electrochemical sensors, conductometric detection, potentiometric detection, optical immunosensor, aptamers [9], square wave voltammetry (SWV) [10], differential pulse voltammetry (DPV), cyclic voltammetry [11] and HPLC [12]. Among this CV technique has paid more attention due to its high reliability, sensitivity, using simple instruments, easy to operate and getting immediate result [13].

In the past era, the increasing of world population, economies and developing countries are releases huge amount of toxic organic substances into water to destroy the ecosystem [14]. Per annum about 70,000 tonnes of untreated dyes with complex structures from various sectors such as textile, leather, pharmaceutical, paper, food and cosmetic industries are discharge around 1-15% of organic effluents in aquatic medium. It is least biodegradable also restrict penetration of sunlight into water bodies and spoil water ecosystem [15]. These organic pollutants have great impact on our daily life but due to fewer biodegradable natures, it causes some health hazards. Azo dyes are hard to degrade becomes extremely carcinogenic and genotoxic [16].

Nowadays, the magnetic nanoparticles are widely used in biomedicine, environmental science, mineralogy, informatics, as well as catalysis due to their attractive properties in physics and chemistry [6-7]. For biomedical uses, biomedical applications of magnetic nanoparticles have shown promise in a number of applications, including treatment of diabetes and diabetic foot ulcers [8,9], magnetic hyperthermia, enhancing magnetic resonance imaging (MRI) data, supplementing tissue engineering efforts and improving the delivery of drugs to

* **K. Rajendran**
rajendran1317@gmail.com

[1-2] Department of Chemistry, Bharath Institute of Higher Education and Research (BIHER), Chennai - 600 073, Tamil Nadu, India

difficult to reach microniches [10-14]. In additions, magnetic nanoparticles can be also used in a wide variety of other applications such as magnetic inks [15], magnetic memory devices [16], and pathogen detection in foods [17-18].

For fabrication of magnetic nanoparticles, there are many methods to produce magnetic nanoparticles such as photolytic reduction, radiolytic reduction, solvent extraction reduction, microemulsion, polyol process and alcolhol reduction have been developed for preparation of metal nanoparticles [19]. In some recent reports, heat treatment method using *Aloe vera* plant extract started receiving a significant interest from researchers because of its advance in controlling particle size and preparing solvent [20-24]. In order to synthesize nanoparticles with smaller sizes, several processes have been carried out, but they often result in the obtained nanoparticles with amorphous structure and low magnetism [25-26]. Therefore, in this study, we have synthesized nanoparticles by heat treatment method using *Aloe vera* plant extract allows us to get small -sized nanoparticles.

MATERIALS AND METHODS

The NiO nano-powders have been synthesized with the aid of using the use of precursors consisting of nickel nitrate and Aloe vera extract as a fuel that have been dissolved one at a time in 10 ml of deionized water and stirred for 15 minutes. Then it changed into located in a microwave-oven (2.forty five GHz, 750 W) for 10 minutes. Initially, the answer boiled and underwent dehydration observed with the aid of using decomposition with the evolution of gases. When the answer reached the factor of spontaneous combustion, it changed into vaporized and right away have become a strong and received with the aid of using MCM. When the solution reached the point of spontaneous combustion, it was vaporized and instantly became a solid.

Characterization studies

Morphology of the synthesized nanoparticles was determined using transmission electron microscopy (TEM, Philips, Model: CM12). The X-ray diffraction (XRD) pattern of the synthesized nanoparticles powder was done with Cu Kα radiation (λ=1.54021 nm) on an X-ray diffractometer (XRD, Siemens, Model: D5000). The UVVisible machine (UV/Vis, PerkinElmer, Model: LAMBDA 25) was also used to confirm formation of Ni magnetic nanoparticles. The absorbance spectra were determined in 10 mm optical path length quartz cuvettes with Perkin Elmer double beam spectrophotometer. The nanoparticles were taken out directly from the solution after doing hydrothermal treatment and it was dispersed in ethanol before UV-Visible test. The magnetic property of Ni magnetic nanoparticles was done with the vibrating sample magnetometer (VSM, MicroSense, Model: 10VSM). The weight of testing samples used in this study was 0.01 gram and the testing was carried out at room temperature (300 K). The magnetic properties were investigated with an applied field at -20 kOe <H< 20 kOe.

RESULTS AND DISCUSSION

XRD analysis

The (Fig)1 shows X -ray diffraction pattern of prepared nickel oxide nanoparticles. The peaks were in agreement with the published data [29-30] and belonged to JCPDS 04 -0850. These confirm that the nanoparticle powders showed the presence of pure face centered cubic (fcc) nickel oxide peaks and there are no distinct diffraction peaks other than those from fcc-NiO is found in the sample. In addition, compound of Ni_2B which often appears in the reaction between sodium borohydride and nickel salt was also not present. These indicate that there is no other impurity in synthesized product. Therefore, these results show that sodium borohydride can be used to reduce nickel ions in ethylene glycol to make high purity nickel oxide nanoparticles [31]. In addition, the width of peaks of nickel nanoparticles is narrow. This also indicates that the synthesized nanoparticles are almost in crystal form after hydrothermal treatment.

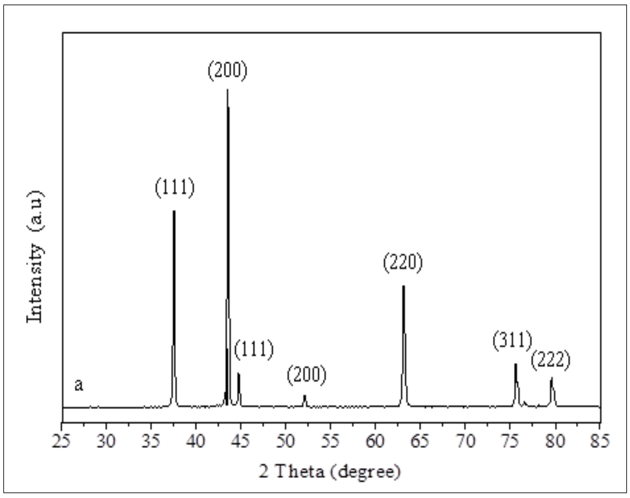

Fig 1 XRD patterns of NiO nanoparticles

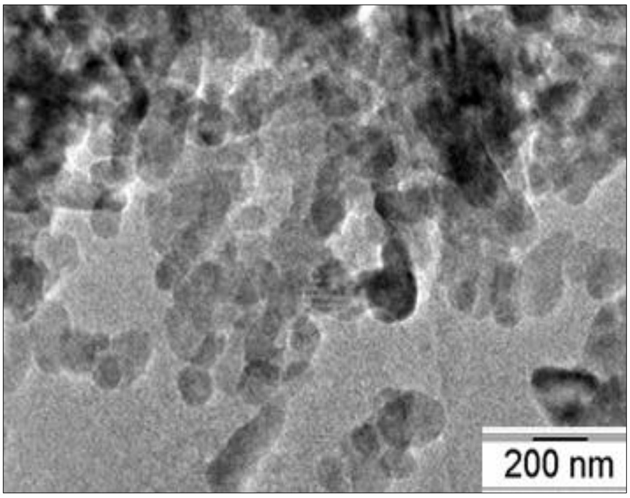

Fig 2 TEM image of NiO magnetic nanoparticles

TEM analysis

To get TEM images, the black powder of NiO magnetic nanoparticles was dispersed in ethanol. The resulting solution was then taken out by using pipette and dripped onto a copper grid of TEM. The samples were dried under vacuum condition before observation. Figure 2, TEM micrographs confirm that morphologies of NiO magnetic nanoparticles at different concentration of nickel ions are all in spherical shape. TEM Images at the same magnification also showed that difference between nanoparticle sizes in these images.

EDX analysis

NiO nano crystals was analysed by EDX as shown in Fig. 3 the presence of Ni and O by the appearance of Ni and O peaks. There is no other elements were present. Hence it was confirmed the formation of pure NiO nanoparticles.

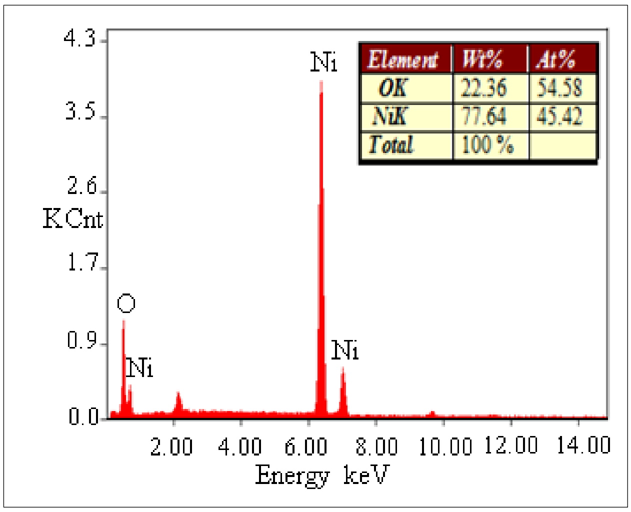

Fig 3 EDX results of NiO nanoparticles

UV -Vis results

(Fig 4) shows the UV -Visible spectrum of NiO sample. It shows that the maximum absorption of wavelength increased when particle size increased. In our opinion, for nanoparticle with larger mean size, the number of atoms required to make up the nanoparticles is enormous, so the number of conduction electrons is more than nanoparticles with smaller mean particle sizes. This results in increasing the maximum absorption of wavelength. However, difference between maximum absorptions of wavelength was not stated clearly here as the deviation between mean particle sizes of samples was too small as mentioned from the above TEM results. Besides, the UV-Vis results also showed that the only existing absorption spectrum region belongs to the NiO nanoparticles [32-35]. There are no absorption bands that belong to other types of particles. This reinforces the conclusion from the above XRD result confirming that the synthesized nanoparticles are nickel nanoparticles.

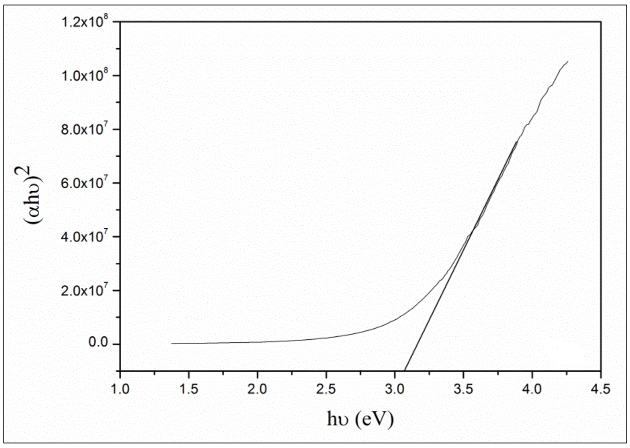

Fig 4 UV-Vis Spectra of NiO nanoparticles

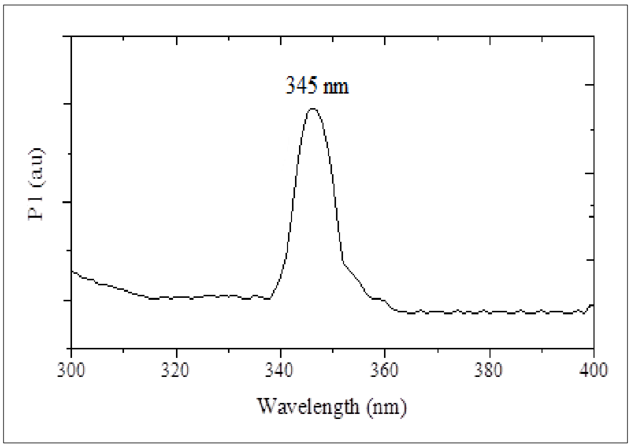

Fig 5 PL Spectra of NiO nanoparticles

PL studies

The peak located at 345 nm consistent to a near band-edge emission is observed for the samples and it should resemble to the wide band gap of the NiO nanoparticles due to the recombination of excitons (Fig 5).

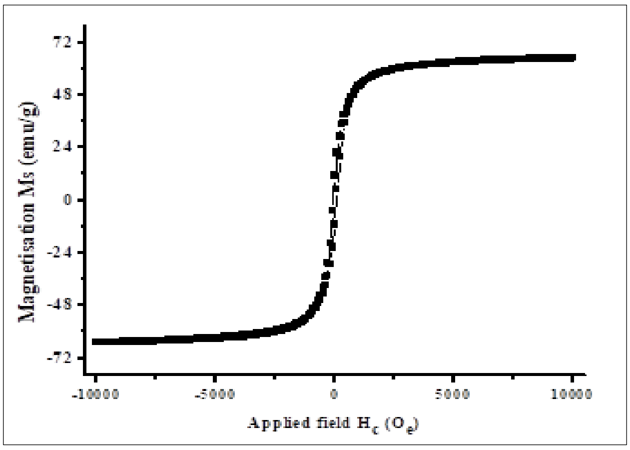

Fig 6 VSM results of NiO nanoparticles

VSM results

(Fig 6) shows the hysteresis loops of nickel oxide nanoparticle sample. At room temperature, saturation magnetization (Ms) of sample is found to be 71.8 emu/g. Results showed that magnetization is related to particle size effect [36-40]. However, the change in Ms value between samples is not much because the difference in mean particle size is not significant as mentioned in the TEM results above. It is well known that Hc of ferromagnetic nanoparticles with regular shape conforms. In addition, the hysteresis loops can also be attributed to superparamagnetic behaviour in synthesized nickel oxide nanoparticles.

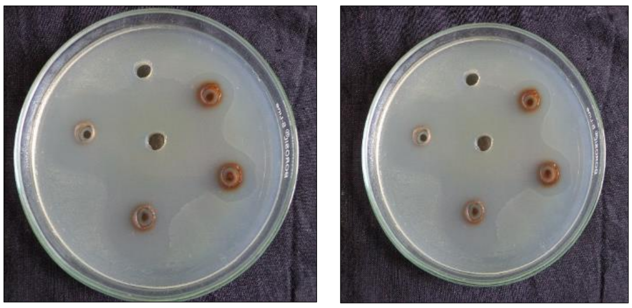

Fig 7 Antibacterial activity of NiO nanoparticles

Antibacterial activity

The (Fig 7) shows the antibacterial activity of NiO nanoparticles samples were investigated against gram negative (Klebsiellapneumoniae) and gram positive (Staphylococcus aureus) bacterial strains, respectively. From the images, it was found that there is no zone of inhibition over the control. The bar diagram of the variation in the diameter of the zone of inhibition is shown in Fig.7, which clearly shows that the zone of inhibition increases with increase in the dopant ratio [40-45]. The particle size and surface area of the samples play a vital role in the antibacterial activity of synthesized samples.

CONCLUSION

The nickel oxide magnetic nanoparticles can be fabricated using *Aloe vera* plant extract. *Aloe vera* plant extract can be used as reducing agent in synthesis of nickel oxide magnetic nanoparticles. Magnetic property of nanoparticles depends on nanoparticle size, saturation magnetization (Ms) reduces with decreasing in particle size. Magnetization study reveals that it closes to superparamagnetic state at room temperature. It has been found that the NiO nanoparticles exhibit higher antibacterial efficiency against Klebsiellapneumoniae compared with other pathogens.

LITERATURE CITED

1. Wang W, Y. Itoh, I.W. Lenggoro, K. Okuyam. 2004. Nickel and nickel oxide nanoparticles prepared from nickel nitrate hexahydrate by a low pressure spray pyrolysis, Materials Science and Engineering: B 111 (2004) 69-76.
2. T. Ahmad, K. Ramanujachary, S.E. Lofland, A.K. Ganguli, Magnetic and electrochemical properties of nickel oxide nanoparticles obtained by the reverse-micellar route, Solid State Sciences 8 (2006) 425-430.
3. H. Ni, Y. Ni, Y. Zhou, J. Hong, Microwave-hydrothermal synthesis, characterization and properties of rice-like α-Fe_2O_3 nanorods, Materials Letters 73 (2012) 206-208.
4. 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. (Impact Factor: 1.354).
5. K. Chitra, K. Reena, A. Manikandan, S. Arul Antony, Antibacterial studies and effect of poloxamer on gold nanoparticles by Zingiber officinale extracted green synthesis, Journal of Nanoscience and Nanotechnology, 15 (2015) 4984-4991.
6. K. Chitra, A. Manikandan, S. Moortheswaran, K. Reena, S. Arul Antony, Zingiber officinale extracted green synthesis of copper nanoparticles: Structural, morphological and antibacterial studies, Adv. Science, Engineering and Medicine, 7 (2015) 710-716.
7. A. P. Subramanian, S. K. Jaganathan, A. Manikandan, K. N. Pandiaraj, N. Gomathi and E. Supriyanto, Recent trends in nano based drug delivery systems for efficient delivery of phytochemicals in chemotherapy, RSC Advances, 6 (2016) 48294-48314.
8. V. Muthuvignesh, V.J. Reddy, S. Ramakrishna, S. Ray, A. Ismail, M. Mandal, A. Manikandan, S. Seal and S. K. Jaganathan, Electrospinning applications from diagnosis to treatment of diabetes, RSC Advances, 6 (2016) 83638-83655.
9. M. F. Valan, A. Manikandan, S. Arul Antony, Microwave combustion synthesis and characterization studies of magnetic $Zn_{1-x}Cd_xFe_2O_4$ ($0 \leq x \leq 0.5$) nanoparticles, Journal of Nanoscience and Nanotechnology, 15 (2015) 4543-4551.
10. V. Muthuvignesh, S. K. Jaganathan, A. Manikandan, Nanomaterials as a game changer in the management and treatment of diabetic foot ulcers, RSC Advances, 6 (2016) 114859-114878.
11. C. Barathiraja, A. Manikandan, A. M. Uduman Mohideen, S. Jayasree, S. Arul Antony, Magnetically recyclable spinel $Mn_xNi_{1-x}Fe_2O_4$ ($x = 0.0$–0.5) nano-photocatalysts: Structural, morphological and opto-magnetic properties, Journal of Superconductivity and Novel Magnetism, 29 (2016) 477-486.
12. E Murugan, G Vimala, Effective functionalization of multiwalled carbon nanotube with amphiphilic poly(propyleneimine) dendrimer carrying silver nanoparticles for better dispersability and antimicrobial activity, Journal of colloid and interface science, 2011, 357 (2), 354-365.
13. 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, Chemical Engineering Journal, 2015, 259, 266-276.
14. E Murugan, V Gopi, Amphiphilic multiwalled carbon nanotube polymer hybrid with improved conductivity and dispersibility produced by functionalization with poly (vinylbenzyl) triethylammonium chloride, The Journal of Physical Chemistry C, 2011, 115 (40), 19897-19909.
15. B. Avila Josephine, A. Manikandan, V. Mary Teresita, S. Arul Antony, Fundamental study of $LaMg_xCr_{1-x}O_{3-\delta}$ perovskites nano-photocatalysts: Sol-gel synthesis, characterization and humidity sensing, The Korean Journal of Chemical Engineering, 33 (2016) 1590-1598.
16.nB. Meenatchi, V.Renuga, A. Manikandan, Electrodeposition of nickel on glassy carbon electrode from protic ionic liquids with imidazolium cation, Journal of Inorganic and Organometallic Polymers and Materials, 26 (2016) 423-430.
17. K. Chitra, A. Manikandan, S. Arul Antony, Effect of poloxamer on Zingiber officinale extracted green synthesis and antibacterial studies of silver nanoparticles, Journal of Nanoscience and Nanotechnology, 16 (2016) 758-764.
18. V. Umapathy, A. Manikandan, P. Ramu, S. Arul Antony, P. Neeraja, Synthesis and characterizations of $Fe_2(MoO_4)_3$ nano-photocatalysts by simple sol-gel method, Journal of Nanoscience and Nanotechnology, 16 (2016) 987-993.
19. S. Rajmohan, A. Manikandan, V. Jeseentharani, S. Arul Antony, J. Pragasam, Simple co-precipitation synthesis and characterization studies of $La_{1-x}Ni_xVO_3$ perovskites nanostructures for humidity sensing applications, Journal of Nanoscience and Nanotechnology, 16 (2016) 1650-1655.
20. S Asiri, S Güner, A Demir, A Yildiz, A Manikandan, A Baykal, Synthesis and magnetic characterization of Cu substituted barium hexaferrites, Journal of Inorganic and Organometallic Polymers and Materials 28 (2018) 1065-1071.
21. H Mohandas, SK Jaganathan, MP Mani, M Ayyar, GVR Thevi, Cancer-related fatigue treatment: an overview, Journal of cancer research and therapeutics 13 (2017), 916.
22. I Pakrudheen, AN Banu, E Murugan,Cationic amphiphilic dendrimers with tunable hydrophobicity show in vitro Activity, Environmental Chemistry Letters, 2018, 16 (4), 1513-1519
23. E Murugan, A Rubavathy Jaya Priya, K Janaki Raman, K Kalpana, C R Akshata, S Santhoshkumar, S Govindaraju, Multiwalled carbon nanotubes/gold nanoparticles hybrid electrodes for enzyme-free electrochemical glucose sensor, Journal of nanoscience and nanotechnology, 2019, 19 (12), 7596-7604
24. E Murugan, R Rangasamy, Development of stable pollution free TiO2/Au nanoparticle immobilized green photo catalyst for degradation of methyl orange, Journal of biomedical nanotechnology, 2011, 7 (1), 225-228.

25. 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, Journal of colloid and interface science 2013, 396, 101-111.
26. E Murugan, RL Sherman, HO Spivey, Catalysis by hydrophobically modified poly (propylenimine) dendrimers having quaternary ammonium and tertiary amine functionality, WT Ford, Langmuir, 2004, 20 (19), 8307-8312.
27. Yassine Slimani, MA Almessiere, A Demir Korkmaz, S Guner, H Güngüneş, M Sertkol, A Manikandan, A Yildiz, S Akhtar, Sagar E Shirsath, A Baykal, $Ni_{0.4}Cu_{0.2}Zn_{0.4}Tb_xFe_{2-x}O_4$ nanospinel ferrites: Ultrasonic synthesis and physical properties, Ultrasonics sonochemistry 59 (2019) 104757.
28. P Thilagavathi, A Manikandan, S Sujatha, SK Jaganathan, S Arul Antony,Sol–gel synthesis and characterization studies of NiMoO4 nanostructures for photocatalytic degradation of methylene blue dye, Nanoscience and Nanotechnology Letters 8 (2016) 438-443.
29. V Umapathy, A Manikandan, SA Antony, P Ramu, P Neeraja, Structure, morphology and opto-magnetic properties of Bi2MoO6 nano-photocatalyst synthesized by sol–gel method, Transactions of Nonferrous Metals Society of China 25 (2015) 3271-3278.
30. MA Almessiere, Y Slimani, H Gungunes, A Manikandan, A Baykal, Investigation of the effects of Tm3+ on the structural, microstructural, optical, and magnetic properties of Sr hexaferrites, Results in Physics 13 (2019) 102166.
31. K Seevakan, A Manikandan, P Devendran, A Shameem, T Alagesan, Microwave combustion synthesis, magneto-optical and electrochemical properties of NiMoO4 nanoparticles for supercapacitor application, Ceramics International 44 (2018) 13879-13887.
32. A Shameem, P Devendran, V Siva, M Raja, SA Bahadur, A Manikandan, 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.
33. AA John, SK Jaganathan, E Supriyanto, A Manikandan, Surface modification of titanium and its alloys for the enhancement of osseointegration in orthopaedics, Current Science, 1003-1015 (2016).
34. 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, Journal of Molecular Catalysis A: Chemical, 2005, 235 (1-2), 220-229.
35. SP Ratnayake, M Mantilaka, C Sandaruwan, D Dahanayake, E Murugan, Carbon quantum dots-decorated nano-zirconia: a highly efficient photocatalyst, Applied Catalysis A: General, 2019, 570, 23-30.
36. E Murugan, P Gopinath, V Shanmugayya, N Mathivanan, Antibacterial activity of novel insoluble bead-shaped polymer-supported multiquaternary ammonium salts, Journal of applied polymer science, 2010, 117 (6), 3673-3678
37. E Murugan, I Pakrudheen, Efficient amphiphilic poly (propylene imine) dendrimer encapsulated ruthenium nanoparticles for sensing and catalysis applications, Science of Advanced Materials, 2015, 7 (5), 891-901.
38. E Murugan, JN Jebaranjitham, A Usha, Synthesis of polymer-supported dendritic palladium nanoparticle catalysts for Suzuki coupling reaction, Applied Nanoscience, 2012, 2 (3), 211-222
39. 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
40. E Murugan, SS Kumar, KM Reshna, S Govindaraju. 2019. 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
41. 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.
42. P Shanmugam, K Rajakumar, R Boddula, RC Ngullie, 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.
43. 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.
44. S Santhoshkumar, E Murugan, Rationally designed SERS AgNPs/GO/g-CN nanohybrids to detect methylene blue and Hg2+ ions in aqueous solution, Applied Surface Science, 2021, 553, 149544.
45. SP Ratnayake, C Sandaruwan, M Mantilaka, N de Silva, D Dahanayake, U.KWanninayaka, W.R.L.N. Bandara, S. Santhoshkumar, E. Murugan, G. A. J. Amaratunga, K.M. Nalinde Silv. 2021. Industrial and environmental significance of photonic zirconia nanoflakes: Influence of boron doping on structure and band states. *Journal of Industrial and Engineering Chemistry* 95: 203-214.