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Hibiscus rosa-sinensis Plant Extract – Assisted Synthesis, and Antibacterial Applications of CeO₂ Nanoparticles

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ABSTRACT

Hibiscus rosa-sinensis plant extract – assisted synthesis was performed to synthesize CeO₂ nanoparticles. All the produced materials were found to be dynamic under sunlight irradiation for the treatment against antibacterial activity. The synthesized CeO₂ nanoparticles were structurally characterized by FTIR and XRD techniques, which showed characteristic vibrations of successfully formed CeO₂ nanoparticles symmetry. SEM images revealed the CeO₂ nanoparticles like the spherical shaped materials. The optical response and detection of reactive species were carried out by photo-luminescence (PL) and showed emissions at 700 nm. PL data also used to calculate band gaps 3.72, eV of CeO₂ nanoparticles. UV/visible spectrophotometer scanned the photocatalytic competences of the synthesized nano materials. CeO₂ nanoparticles exhibited efficient antibacterial activity against human pathogens.

Key words: Plant extract, CeO₂ nanoparticles, Antibacterial activity, FTIR, XRD technique

The quality of water reservoirs is diminishing as a result of rising residential and industrial human needs, which has harmed aquatic life [1-2]. A large number of research investigations are now being reported to prepare suitable photocatalysts for textile industry effluents treatment [3], which are considered more harmful than other toxins. About half of the dyes amount used on textile fibers do not adhere to it and end up as a contaminant in aquatic environment [4]. Using unhygienic water resulted in a variety of ailments in human beings [5-6]. Undiagnosed and untreated dye pollutants in water not only damage the surface of water, but they also block the transmission of light, which has negative consequences for water bodies [7]. Various techniques are in practice to remove these pollutants from water such as catalysis [8], photodegradation [9-10], biodegradation [11] and adsorption [12-15]. Efficient removal of azo dyes cannot be achieved by traditional adsorption, enzymatic degradation and ion exchange procedures because they produce secondary harmful emissions such as poisonous fumes and sludge, which necessitate further processing [16-22]. Alternative strategies, such as heterogeneous photocatalysis with micro semiconductors, have emerged as potential technologies for removing dye contaminants from waste water [23-30].

MATERIALS AND METHODS

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Initially, mixing cerium nitrate, *Hibiscus rosa-sinensis* plant extract in 20 ml of water. The colloidal solution was filtered, washed (with distilled water) and dried (at 95°C for 12 hrs) to obtain product. The solid product CeO₂ nanoparticles was obtained by filtering, washing (with distilled water) and drying (at 100°C for 12 hrs). Finally, annealing of dried CeO₂ nanoparticles at 550°C conceded in furnace over 5 hrs to deduce energetic CeO₂ nanoparticles. Fourier transformed infrared (FTIR) spectrometer was used for the identification of different functional moieties in the synthesized materials. X-ray diffractometer (XRD) revealed structural characteristics of CeO₂ nanoparticles. Ultra-violet visible (Uv-vis.) spectrophotometer and Photo-luminescence (PL) studies were used to enlighten the optical nature, while EDX scanning electron microscope (EDX-SEM) examined the elemental elucidation with surface texture of CeO₂ nanoparticles.

RESULTS AND DISCUSSION

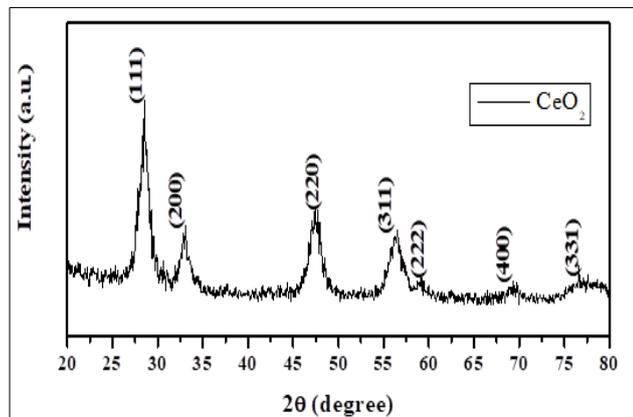
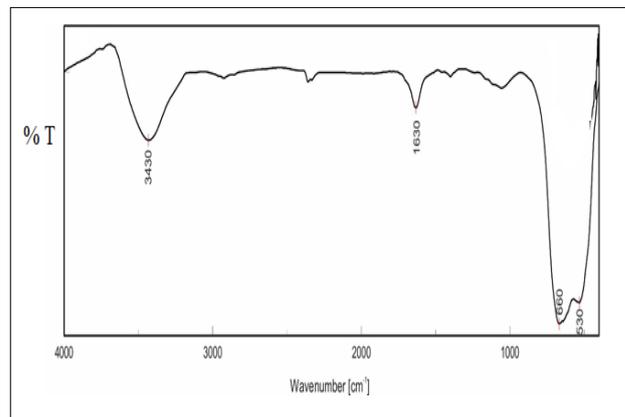
XRD analysis

XRD studies are employed for the structure demonstration of CeO₂ nanoparticles. Highly strong diffraction peaks were recorded which have 100, 200 and 220 hexagonal reflection planes [30-34]. The observed diffraction data showed that CeO₂ nanoparticles provided hexagonal foundation, which was manifested by results of CeO₂ nanoparticles in (Fig 1). Scherer's equation used to determine the crystallite size of the most intense diffractions of CeO₂ nanoparticles which was found to be 8.34 nm. These productive results showed further reduction in particle size of CeO₂ nanoparticles (average crystallite size = 12.30 nm).

FT-IR analysis

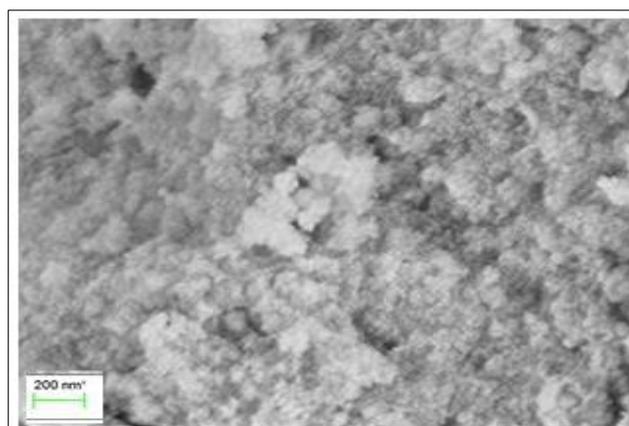
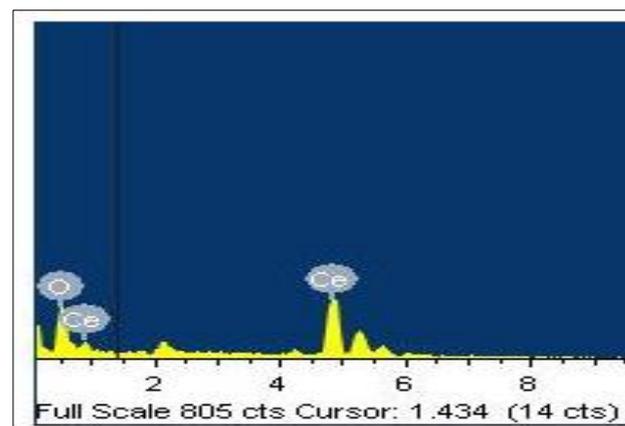
FTIR examined the characteristic peaks at 472, 519, 793, 1011, 1370 and 3415 cm^{-1} are attributed to the CeO_2 nanoparticles as shown in Fig. 2. The variation in FTIR peaks of CeO_2 nanoparticles revealed the successful formation of CeO_2 nanoparticles. The vibrations recorded ranging 472-532

cm^{-1} demonstrating the presence of cerium connections with oxygen in the material structures. However, vibration peaks at 1363-1370 cm^{-1} and 3415-3444 cm^{-1} were due to C-H bending and stretching respectively. All these spectral results were in favor of metal-oxygen interactions in organo silica framework [35-39].

Fig 1 Powder XRD patterns of CeO_2 NPsFig 2 FT-IR spectra of CeO_2 NPs*SEM analysis*

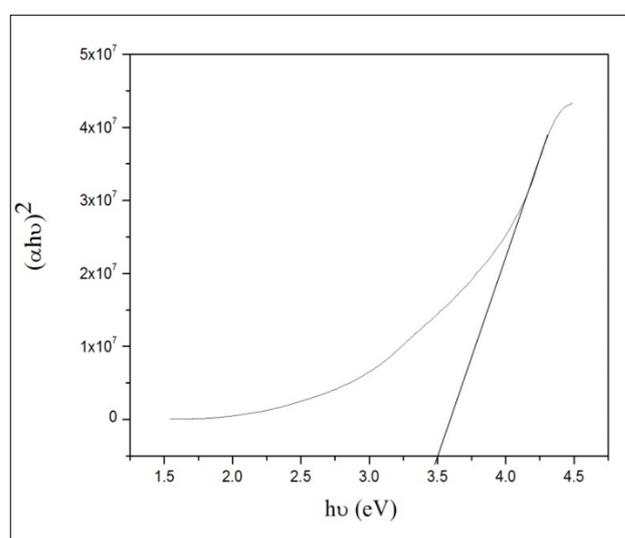
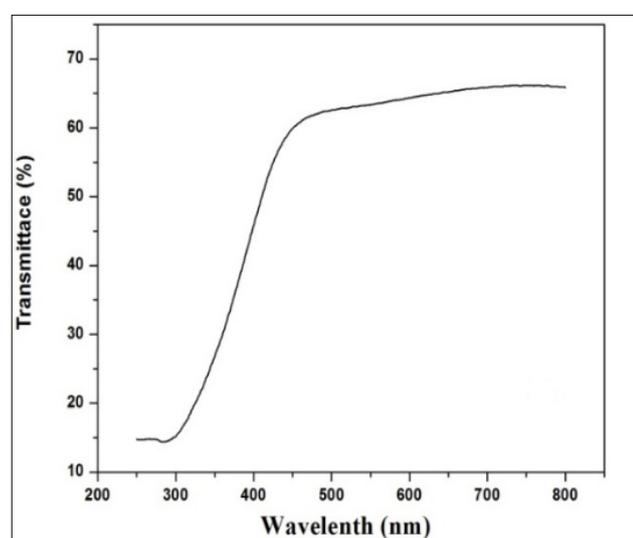
SEM images exhibited particles like organizations in CeO_2 nanoparticles as shown in (Fig 3). To further ascertain the

formation of CeO_2 nanoparticles, SEM analysis was carried out to examine the surface morphology. The tiny spheres of cerium oxide nanoparticles was formed and agglomerated.

Fig 3 SEM images of CeO_2 nanoparticlesFig 4 EDX spectra of CeO_2 nanoparticles*EDX analysis*

EDX results in (Fig 4) confirmed the CeO_2 nanoparticles formation. The EDX spectra procured during the SEM analysis

was used to confirm the elemental composition of the prepared sample. (Fig 4) depicts the EDX spectra of the CeO_2 nanoparticles.

Fig 5 UV-Vis spectra of CeO_2 NPs

UV-Vis spectroscopy studies

Tauc plots in (Fig 5) showed energy difference between valence band (VB) and conduction band (CB) in CeO₂

nanoparticles. These band gaps were determined 3.45 eV for CeO₂ nanoparticles.

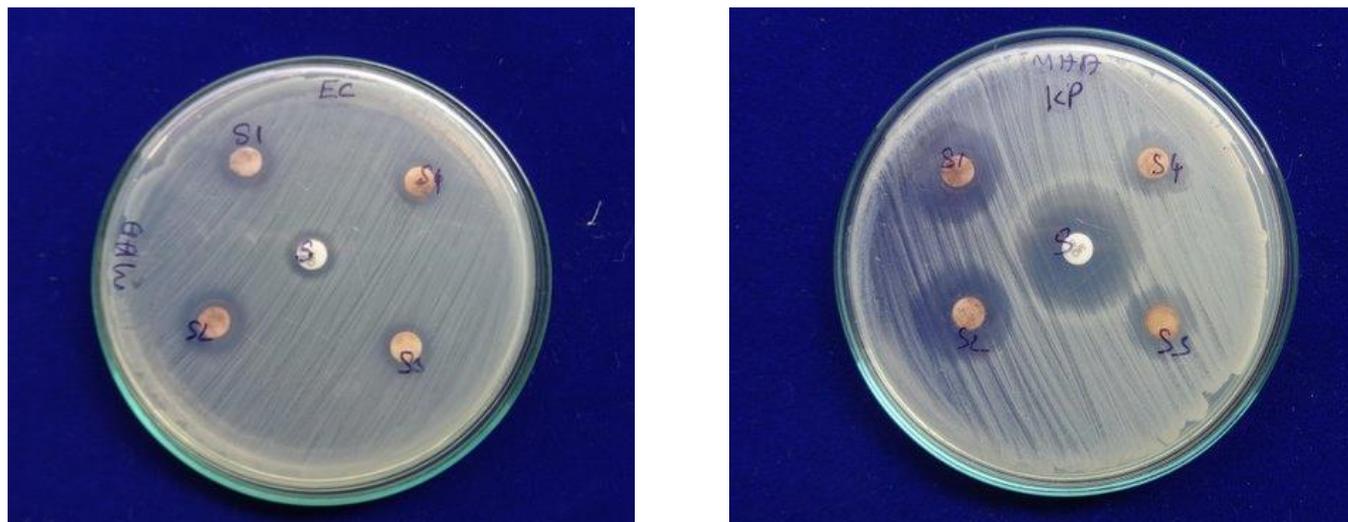


Fig 6 Antibacterial activity of CeO₂ nanoparticles

Antibacterial activity

Data presented in (Fig 6) shows the antibacterial activity of CeO₂ nanoparticles 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, which clearly shows that the zone of inhibition increases and influences higher antibacterial activity [40-46]. The particle size and surface area of the samples play a vital role in the antibacterial activity of synthesized samples.

CONCLUSION

The present research work comprised revised study of CeO₂ nanoparticles and SEM images provided foundation for the large surface area in CeO₂ nanoparticles. CeO₂ nanoparticles was validated by significant outcomes of XRD, PL, FTIR and EDX-SEM. Furthermore, the Antibacterial activity efficiencies of CeO₂ nanoparticles exhibited their improved. The distinctive approach to formulate energetic CeO₂ nanoparticles having exciting Antibacterial activity accomplished the current study more effective.

LITERATURE CITED

1. 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.
2. 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.
3. 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
4. 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.
5. 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
6. 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
7. 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
8. 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.
9. 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.
10. 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.
11. 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.
12. 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, Industrial and environmental significance of photonic

- zirconia nanoflakes: Influence of boron doping on structure and band states, *Journal of Industrial and Engineering Chemistry*, 2021, 95, 203-214.
13. I Pakrudheen, AN Banu, E Murugan, Cationic amphiphilic dendrimers with tunable hydrophobicity show in vitro Activity, *Environmental Chemistry Letters*, 2018, 16 (4), 1513-1519
 14. 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
 15. 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.
 16. 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.
 17. 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.
 18. 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.
 19. 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.
 20. 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.
 21. C. Sambathkumar, R. Ranjithkumar, S. Ezhil Arasi, A. Manikandan, N. Nallamuthu, M. Krishna Kumar, A. Arivarasan, P. Devendran, High-performance nickel sulfide modified electrode material from single source precursor for energy storage application, *Journal of Materials Science: Materials in Electronics*, 32 (2021) 20058-20070.
 22. 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, *Journal of Materials Science: Materials in Electronics*, 32 (2021) 20827-20843.
 23. V. S. P. Sakthi Sri, A. Manikandan, M. Mathankumar, R. Tamizhselvi, M. George, A. L. Bilgrami, S. A. Al-Zahrani, A. A. P. Khan, Anish Khan, A. M. Asiri, Unveiling the photosensitive, mechanical and magnetic properties of amorphous iron nanoparticles with its application towards decontamination of water and cancer treatment, *Journal of Materials Research and Technology*, 15 (2021) 99-118.
 24. S. Blessi, A. Manikandan, S. Anand, M. M. L. Sonia, V. M. Vinosel, P. Paulraj, Y. Slimani, M.A. Almessiere, M. Iqbal, S. Guner, A. Baykal, Effect of Zinc substitution on the physical and electrochemical properties of mesoporous SnO₂ nanomaterials, *Materials Chemistry and Physics*, 273 (2021) 125122.
 25. A. Alagarsamy, S. Chandrasekaran, A. Manikandan, Green synthesis and characterization studies of biogenic zirconium oxide (ZrO₂) nanoparticles for adsorptive removal of methylene blue dye, *Journal of Molecular Structure*, 1247 (2022) 131275. Impact Factor: 3.196 (Elsevier)
 26. 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
 27. S. Blessi, A. Manikandan, S. Anand, M. M. L. Sonia, V. M. Vinosel, A. M. Alosaimi, A. Khan, M. A. Hussein, A. M. Asiri, Enhanced electrochemical performance and humidity sensing properties of Al³⁺ substituted mesoporous SnO₂ nanoparticles, *Physica E: Low-dimensional Systems and Nanostructures*, 133 (2021) 114820.
 28. R. Kalidoss, K. Radhakrishnan, A. Manikandan, S.K.Jaganathan, A. Khan, A. M. Asiri, Socio-Economic Demands and Challenges for Non-invasive Disease Diagnosis through Portable Breathalyzer by the Incorporation of 2D Nanosheets and SMO Nanocomposites, *RSC Advances*, 11 (2021) 21216–21234.
 29. K. Geetha, R. Udhayakumar, A. Manikandan, Enhanced magnetic and photocatalytic characteristics of cerium substituted spinel MgFe₂O₄ ferrite nanoparticles, *Physica B: Physics of Condensed Matter*, 615 (2021) 413083
 30. S. S. Al-Jameel S. Rehman, M. A. Almessiere, F. A. Khan, Y. Slimani, N. S. Al-Saleh, A. Manikandan, E. A. Al-Suhaimi, A. Baykal, Anti-microbial and anti-cancer activities of MnZnDy_xFe_{2-x}O₄ (x ≤ 0.1) nanoparticles, *Artificial Cells, Nanomedicine and Biotechnology*, 49 (2021) 493-499.
 31. 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
 32. S. Blessi, A. Manikandan, S. Anand, M. M. L. Sonia, V. M. Vinosel, A. M. Alosaimi, A. Khan, M. A. Hussein, A. M. Asiri, Enhanced electrochemical performance and humidity sensing properties of Al³⁺ substituted mesoporous SnO₂ nanoparticles, *Physica E: Low-dimensional Systems and Nanostructures*, 133 (2021) 114820.
 33. R. Kalidoss, K. Radhakrishnan, A. Manikandan, S.K.Jaganathan, A. Khan, A. M. Asiri, Socio-Economic Demands and Challenges for Non-invasive Disease Diagnosis through Portable Breathalyzer by the Incorporation of 2D Nanosheets and SMO Nanocomposites, *RSC Advances*, 11 (2021) 21216–21234.
 34. K. Geetha, R. Udhayakumar, A. Manikandan, Enhanced magnetic and photocatalytic characteristics of cerium substituted spinel MgFe₂O₄ ferrite nanoparticles, *Physica B: Physics of Condensed Matter*, 615 (2021) 413083.
 35. S. S. Al-Jameel S. Rehman, M. A. Almessiere, F. A. Khan, Y. Slimani, N. S. Al-Saleh, A. Manikandan, E. A. Al-Suhaimi, A. Baykal, Anti-microbial and anti-cancer activities of MnZnDy_xFe_{2-x}O₄ (x ≤ 0.1) nanoparticles, *Artificial Cells, Nanomedicine and Biotechnology*, 49 (2021) 493-499.

36. P. Annie Vinosha, A. Manikandan, A. Christy Preetha, A. Dinesh, Y. Slimani, M.A. Almessiere, A. Baykal, Belina Xavier, G. Francisco Nirmala, Review on recent advances of synthesis, magnetic properties and water treatment applications of cobalt ferrite nanoparticles and nanocomposites, *Journal of Superconductivity and Novel Magnetism*, 34 (2021) 995–1018.
37. S. S. Al-Jameel, M. A. Almessiere, F. A. Khan, N. Taskhandi, Y. Slimani, N. S. Al-Saleh, A. Manikandan, E. A. Al-Suhaimi, A. Baykal, Synthesis, Characterization, Anti-Cancer Analysis of $\text{Sr}_{0.5}\text{Ba}_{0.5}\text{Dy}_x\text{Sm}_x\text{Fe}_{8-2x}\text{O}_{19}$ ($0.00 \leq x \leq 1.0$) Microsphere Nanocomposites, *Nanomaterials*, 11 (2021) 700.
38. 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, *Journal of Physics and Chemistry of Solids*, 153 (2021) 110010.
39. S. Blessi, S. Anand, A. Manikandan, M. Maria Lumina Sonia, V. Maria Vinosel, Y. Slimani, M.A. Almessiere, A. Baykal, Influence of Ni substitution on opto-magnetic and electrochemical properties of CTAB capped mesoporous SnO₂ nanoparticles, *Journal of Materials Science: Materials in Electronics*, *Journal of Materials Science: Materials in Electronics*, 32 (2021) 7630–7646.
40. 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: Condensed Matter*, 606 (2021) 412660.
41. S. Blessi, S. Anand, A. Manikandan, M. M. Lumina Sonia, V. Maria Vinosel, Y. Slimani, M.A. Almessiere, A. Baykal, Structural, optical and electrochemical investigations of Sb substituted mesoporous SnO₂ nanoparticles, *Journal of Materials Science: Materials in Electronics*, 32 (2021) 4132–4145.
42. P. A. Vinosha, A. Manikandan, A. S. J. Ceicilia, A. Dinesh, G. F. Nirmala, A. Christy Preetha, Y. Slimani, M.A. Almessiere, A. Baykal, B. Xavier, Review on recent advances of zinc substituted cobalt ferrite nanoparticles: Synthesis characterization and diverse applications, *Ceramics International*, 47 (2021) 10512–10535.
43. 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.
44. 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.
45. 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.
46. M. Vanitha, G. Ramachandran, A. Manikandan, Y. Slimani, M. A. Almessiere, A. Baykal, C. S. Dash, Effect of Sr²⁺ ions substituted nickel ferrite nanoparticles prepared by a simple microwave combustion method. *Journal of Superconductivity and Novel Magnetism* 34 (2021) 971–980.