Frontiers in Health Informatics
ISSN-Online: 2676-7104

www.healthinformaticsjournal.com

2023; Vol 12

Open Access

Synthesis And Characterisation Of Srtio3/Fe2o3/Zno Nanocomposites And Its Antibacterial Activity Against Dental Pathogens

Srigopika T[1], Dr Vigneshwar T [2]*

[1]Undergraduate student Saveetha Dental College and Hospitals, Saveetha Institute of Medical and Technical Sciences Saveetha University, Chennai-600077.

Email ID- 152001029.sdc@saveetha.com

[2]Senior lecturer Department of Conservative Dentistry and Endodontics,Saveetha Dental College and Hospitals, Saveetha Institute of Medical and Technical Sciences, Saveetha University,Chennai-600077.

Email ID - vigneshwart.sdc@saveetha.com

CORRESPONDING AUTHOR

Dr Vigneshwar T

Senior lecturer Department of Conservative Dentistry and Endodontics,Saveetha Dental College and Hospitals, Saveetha Institute of Medical and Technical Sciences, Saveetha University,Chennai-600077.

Email ID - vigneshwart.sdc@saveetha.com

Cite this paper as: Srigopika T, Dr Vigneshwar T (2024) Synthesis And Characterisation Of Srtio3/Fe2o3/Zno Nanocomposites And Its Antibacterial Activity Against Dental Pathogens. *Frontiers in Health Informatics*, *Vol.12(2023), 703-710*

ABSTRACT

Introduction Nanoparticles are spherical particles composed of natural or artificial polymers, having size of 10 and 500 nm. These particles have a wide range of potential applications and NPs in particular have demonstrated broad-spectrum antibacterial properties against both Gram-positive and Gram-negative bacteria. Metal nanoparticles containing metals such as strontium, titanium, iron and zinc have been increasingly used for their antibacterial properties in recent times.

Materials and methods ZnO was prepared by using zinc chloride and sodium bicarbonate, $SrTiO_3$ was obtained from strontium nitrate and titanium tetrachloride in the aqueous solution of NH_3 and Fe_2O_3 was made by dissolving 10g of $FeSO_4.7H_2O$

in 100 ml of double distilled water, to which 2 to 3 drops of concentrated sulfuric acid were added. The ternary nanocomposite was prepared by taking 0.98 g of prepared $SrTiO_3$/ZnO which was dispersed in 40 mL of ethanol. 0.02 g Fe_2O_3 powder is added into the above solution and again stirred upto 12 hours. A rosy white powder is obtained after volatilization of the ethanol and drying at 80 °C in the air. , which is the nanocomposite.

Results The XRD pattern displayed several sharp peaks indicating that the material is polycrystalline, the functional groups present in the nanocomposite was studied using FTIR where ZnO showed a peak at 1134 cm^{-1}, Fe_2O_3 at 3796 cm^{-1} and $SrTiO_3$ at 580 cm^{-1}. SEM images showed $SrTiO_3$ in the form of cubic crystals, spherical/granular particles that are characteristic of ZnO. EDX spectrum results showed the presence of 27.7% zinc and 34.4% oxygen. Culture plates show significant clear zones of inhibition , confirming broad-spectrum antibacterial effect, with values of 22–25 mm.

Conclusion The synthesis and characterization of $SrTiO_3/Fe_2O_3$/ZnO nanocomposites was successful and was proved using data from XRD, SEM and FTIR. Its antibacterial activity against S.mutans and E.faecalis was also studied and at higher concentration, its zone of inhibition was similar to the control..

Keywords: Antibacterial activity, Characterisation, Ferric oxide, Innovation, Nanocomposite , Sustainability, Strontium titanate, Synthesis, Zinc oxide**.**

INTRODUCTION

Nanoparticles are spherical, polymeric particles composed of natural or artificial polymers. They range in size between 10 and 500 nm, and as a consequence of their spherical shape and high surface area to volume ratio, these particles have a wide range of potential applications [1]. In recent years, these materials have emerged as key components in modern medicine, with applications ranging from contrast agents in medical imaging to carriers for gene delivery into individual cells. Nanoparticles have a number of properties that distinguish them from bulk materials simply by virtue of their size, such as chemical reactivity, energy absorption, and biological mobility [2].

Additionally, nanoparticles are increasingly used to target bacteria as an alternative to antibiotics. NPs in particular have demonstrated broad-spectrum antibacterial properties against both Gram-positive and Gram-negative bacteria. For example, ZnO NPs were found to inhibit *Staphylococcus aureus*, and Ag NPs exhibit concentration-dependent antimicrobial activity against *Escherichia coli* and *Pseudomonas aeruginosa*. [3]. According to existing research, the major processes underlying the antibacterial effects of NPs are the disruption of the bacterial cell membrane, generation of ROS, penetration into the bacterial cell membrane and induction of intracellular antibacterial effects, including interactions with DNA and proteins [4].

Metal nanoparticles containing metals such as strontium, titanium, iron and zinc have been increasingly used for their antibacterial properties in recent times. ZnO-NPs exhibit attractive antibacterial properties due to increased specific surface area as the reduced particle size leads to enhanced particle surface reactivity. ZnO-NPs are reported by several studies as non-toxic to human cells and this aspect necessitated their usage as antibacterial agents and hold good biocompatibility to human cells [5,6]. Similarly, iron oxide NPs have antimicrobial activity against both Gram-positive (including *Staphylococcus aureus*) and Gram-negative (including *Escherichia coli*) bacteria.

Strontium based nanoparticles are also continuously increasing in numerous fields including medicine and biology, drug delivery, biosensors and catalysts. Conventionally, strontium is used in bone regeneration, as a growth stimulant, and has the ability to stimulate calcium signaling [7]. Strontium-conjugated nanomaterials exhibit antimicrobial ability and are used in targeted drug delivery and can elicit a prolonged immune response [8]. They are also efficient in the removal of toxic contaminants from industrial wastewater. In conclusion, it can act as a good immunotherapeutic agent [9].

Iron oxide nanoparticles are gaining momentum in recent years due to their significant physicochemical properties. Among the transition metal oxides, iron oxide nanoparticles have drawn tremendous interest from researchers from the last decade. This can be attributed to their chemical stability, cheap, and non-toxic nature [10]. Hematite nanoparticle, which is a type of iron oxide NP, is more popular than other types due to its cost-effective synthesis, non-toxic nature, stability at room temperature, environment friendliness, a reusable and has a wide range of applications such as catalysts, contrast agents in Magnetic Resonance Imaging (MRI), anticancer therapeutics drug delivery, pigments, gas sensors and biosensors [11].

Iron oxide nanoparticles have also been used for delivering medicines and viral vectors to target cells. The antibacterial activity of iron oxide nanoparticles is of special interest, as the emergence of antibiotic-resistant strains is a serious problem for world public health [12]. Keeping all the various properties of the above nanocomposites in mind, this study aims to synthesize and characterise $SrTiO_3/Fe_2O_3/ZnO$ nanocomposites and check its antibacterial activity against dental pathogens.

Materials and methods

Materials used

Zinc chloride, sodium bicarbonate, strontium nitrate, titanium tetrachloride, aqueous ammonia, ferrous sulfate heptahydrate, sulphuric acid, ethanol, oxalic acid, sodium hydroxide, 0.1 M Hydrochloric acid.

Preparation of ZnO

ZnO was prepared by using zinc chloride and sodium bicarbonate. 10g of Zinc chloride weighed and transferred into a 250 ml beaker, which dissolved in 100 ml of distilled water. 6.2g of sodium bicarbonate was added to that portion with vigorous stirring for a few minutes until precipitate was formed. The precipitate was washed three times with distilled water to remove sodium chloride that was formed. It was dried at 100ºC to remove the water. The solid was transferred into agate mortar and ground up to fine powder. The powder was calcinated at 500ºC in muffle furnace using ceramic crucible.

Preparation of $SrTiO_3$

$SrTiO_3$ was obtained from strontium nitrate and titanium tetrachloride in the aqueous solution of NH_3. 10g of strontium nitrate was carefully dissolved in 100 ml of distilled water. 10 ml of $TiCl_4$ was added into 100 ml of ice cold distilled water while stirring for 2 hours and a clear solution was obtained after stirring. To that solution, an already prepared strontium solution was added drop wise, following which an aqueous solution of NH_3 brown precipitate was formed. The precipitate was washed several times and then dried at 100°C to remove the water. The dried solid was grinded agate mortar and pressed into a ceramic crucible. The material was calcinated at

600°C for 4 hours.

Preparation of Fe_2O_3

10g of $FeSO_4.7H_2O$ was dissolved in 100 ml of double distilled water. To that solution, 2 to 3 drops of concentrated sulfuric acid were added up to get a clear solution and then an aqueous solution of NH_3 precipitate was added to get precipitate. The precipitate was washed several times and then dried at 100°C to remove the water. The dried solid was calcinated at 550°C in 5 hours.

Fabrication of $SrTiO_3/Fe_2O_3/ZnO$ ternary composite

The typical preparation of $SrTiO_3/Fe_2O_3/ZnO$ ternary composites photocatalysts are as follows: firstly, 0.98 g of prepared $SrTiO_3/ZnO$ binary composite are dispersed in 40 mL of ethanol and stirred for 1 hour . Then, 0.02 g Fe2O3 powder is added into the above solution and again stirred upto 12 hours. After volatilization of the ethanol, dark greenish powder is obtained after drying at 80 °C in the air. The rosy white powder is the $SrTiO_3/Fe_2O_3/ZnO$ ternary composites.

RESULTS

Figure 1 presents the results of X-ray diffraction, which is performed with the intention of substantiating various phases of the nanoparticles. The graph confirms the successful synthesis and coexistence of three distinct crystalline phases within a single nanocomposite material. The XRD pattern displays several sharp, well-defined peaks, indicating that the material is polycrystalline. Figure 2 shows the FTIR spectrum, which identifies the diverse functional groups and chemical bonds present in the synthesized nanocomposite. The functional groups are indicated using peaks. In our study, ZnO shows a peak at 1134 cm^{-1}, Fe_2O_3 shows a peak at 3796 cm^{-1} and SrTiO3 shows peak at 580 cm^{-1}. The presence of these specific peaks confirms that all three metallic components are chemically active and bonded with oxygen.

Figure 3 shows SEM images that provide visual confirmation of the physical structure and surface arrangement of the nanocomposites. The images show a heterogeneous morphology, as it is a ternary nanocomposite. The cubic structure represents $SrTiO_3$, which naturally forms cubic crystals. The spherical/granular particles are characteristic of ZnO and Fe_2O_3 nanoparticles that have decorated the surface of the larger cubic structures. Figure 4 shows the EDX spectrum that provides a quantitative elemental analysis of the nanocomposite. The peaks on the graph correspond to the characteristic X-rays emitted by specific atoms when hit by the electron beam. The spectrum confirms the presence of all primary expected elements - zinc and oxygen. These show high-intensity peaks, aligning with the high weight percentages (27.7% and 34.4%, respectively). This suggests ZnO is a major component of the composite. The peaks of iron , titanium, and strontium are visible but much smaller (0.2% for Fe and slight traces for Ti and Sr).

Images in figure 5 demonstrate the antibacterial efficacy of the nanocomposites using the Agar Well Diffusion method. The test was conducted against two clinically significant bacteria *Streptococcus mutans*, the primary bacteria responsible for tooth decay and dental plaque and *Enterococcus faecalis*, that is associated with root canal infections. Both plates show significant clear zones, confirming that the nanocomposite has a broad-spectrum antibacterial effect. Figure 6 shows the graph which measures the Zone of Inhibition (ZOI) in millimeters. The control, ciprofloxacin, represents the gold standard and shows the highest ZOI, roughly 26–28 mm. The NCs synthesized in the study, at the higher concentration are reaching values near 22–25 mm. This indicates that the NCs have high antibacterial potency, as they are approaching the effectiveness of the gold standard .For both *E. faecalis* and *S. mutans,* there is a significant jump in the ZOI diameter when moving from the lower to higher concentration, suggesting that the effect is dose dependent.

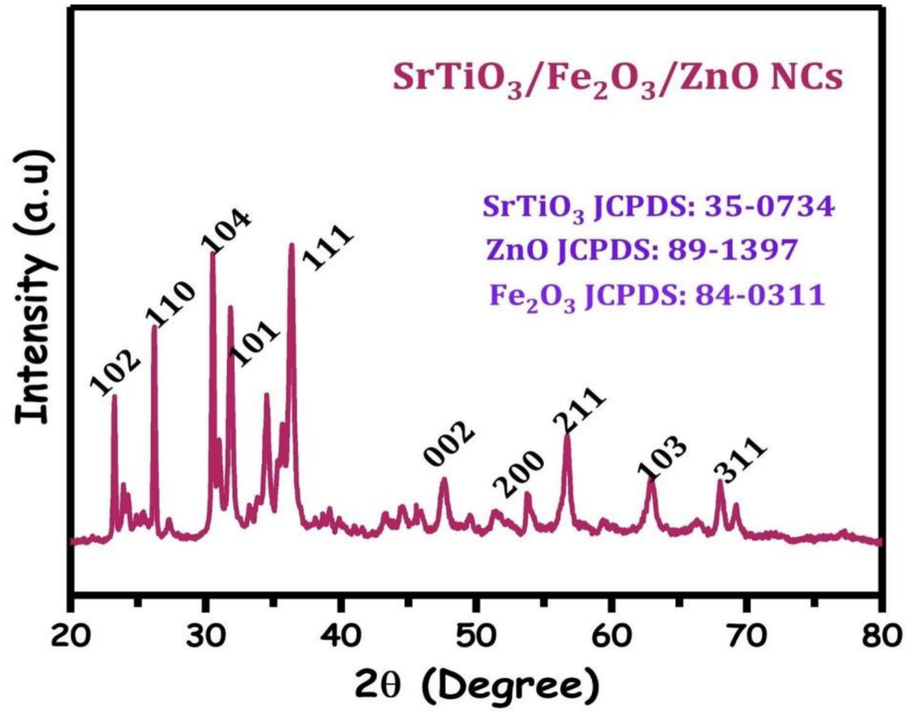


Figure 1 -XRD graph of $SrTiO_3/Fe_2O_3/ZnO$ NCs.

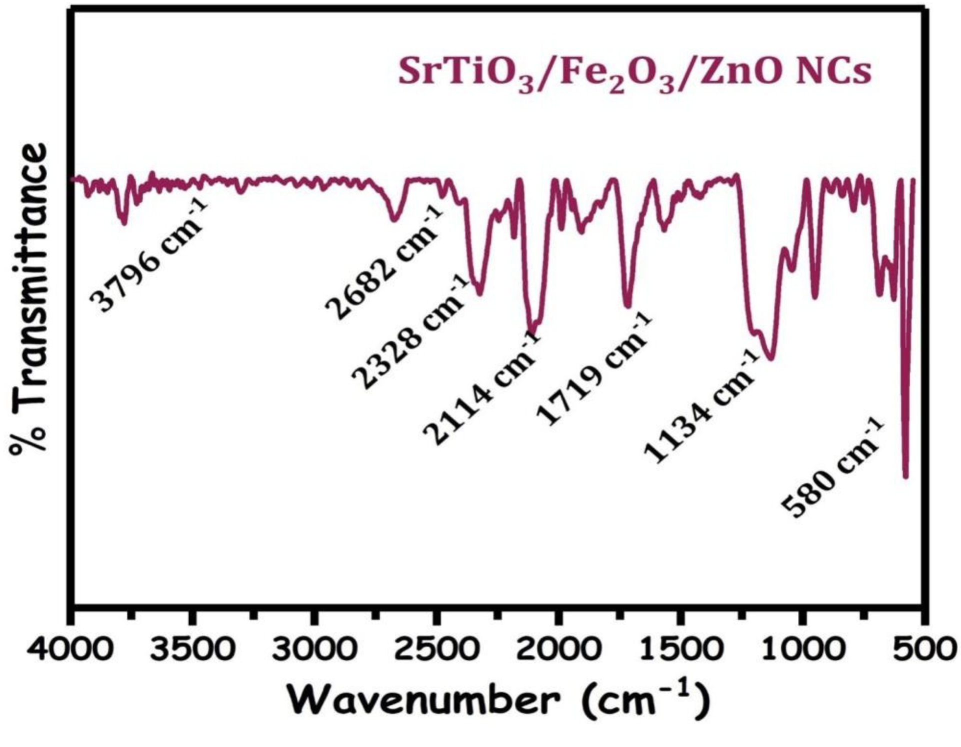


Figure 2 - FTIR spectrum of $SrTiO_3/Fe_2O_3/ZnO$ NCs.

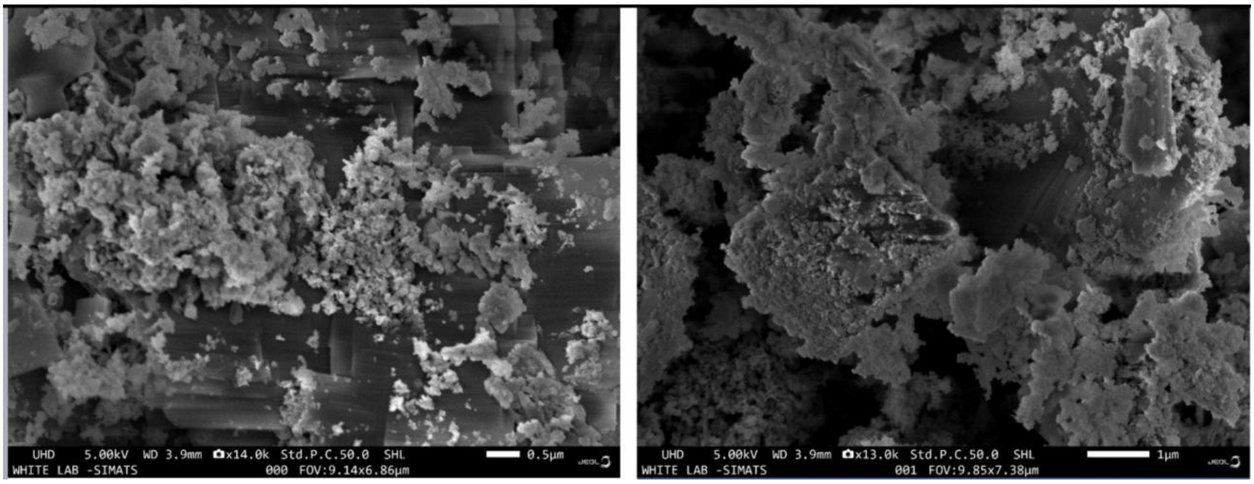


Figure 3 - SEM Images of $SrTiO_3/Fe_2O_3/ZnO$ NCs.

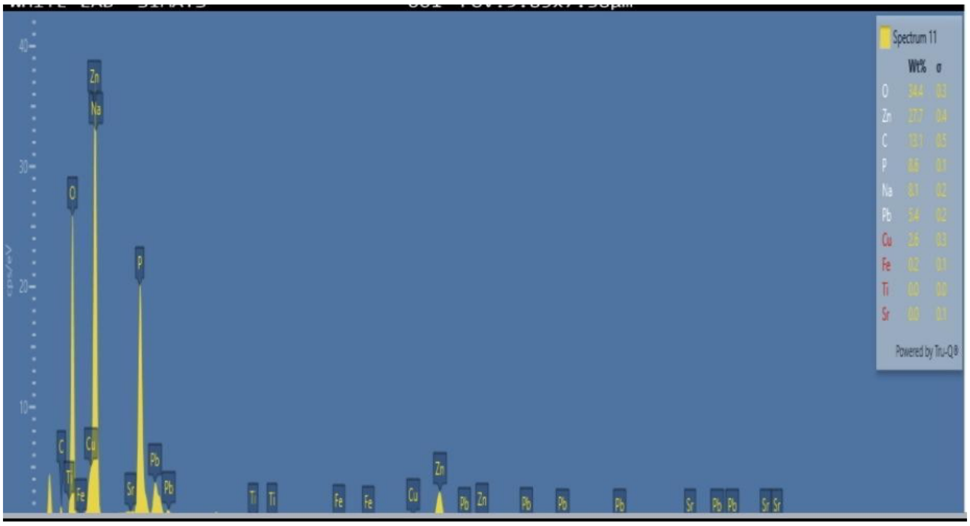
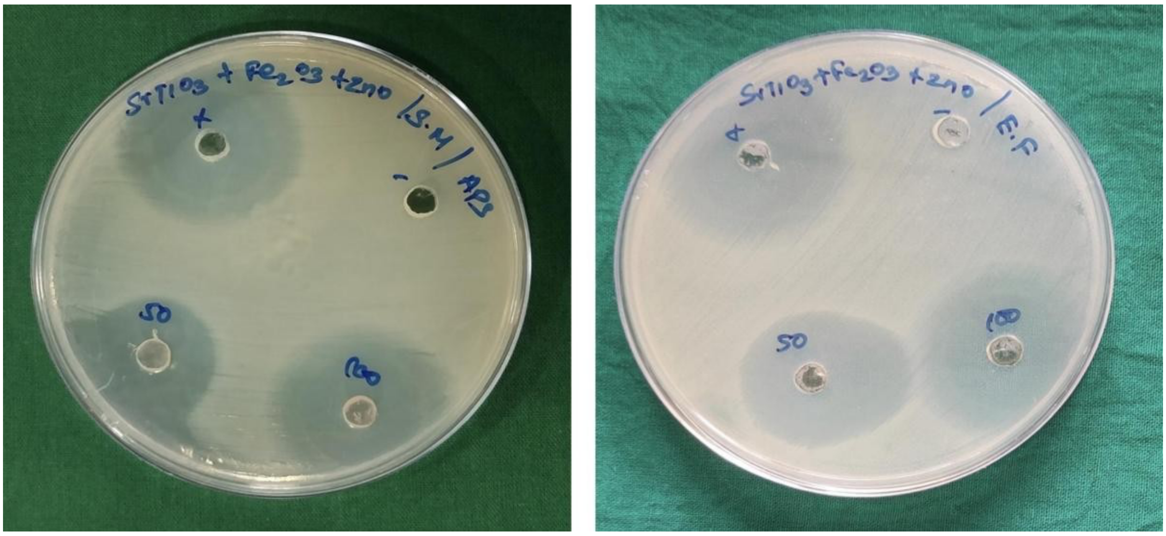


Figure 4 - EDX spectrum of $SrTiO_3/Fe_2O_3/ZnO$ NCs.



Streptococcus Mutans **Enterococcus faecalis**

Figure 5 - Antibacterial activity of $SrTiO_3/Fe_2O_3/ZnO$ NCs against *S. mutans* and *E. faecalis* bacterial strains in comparison to ciprofloxacin.

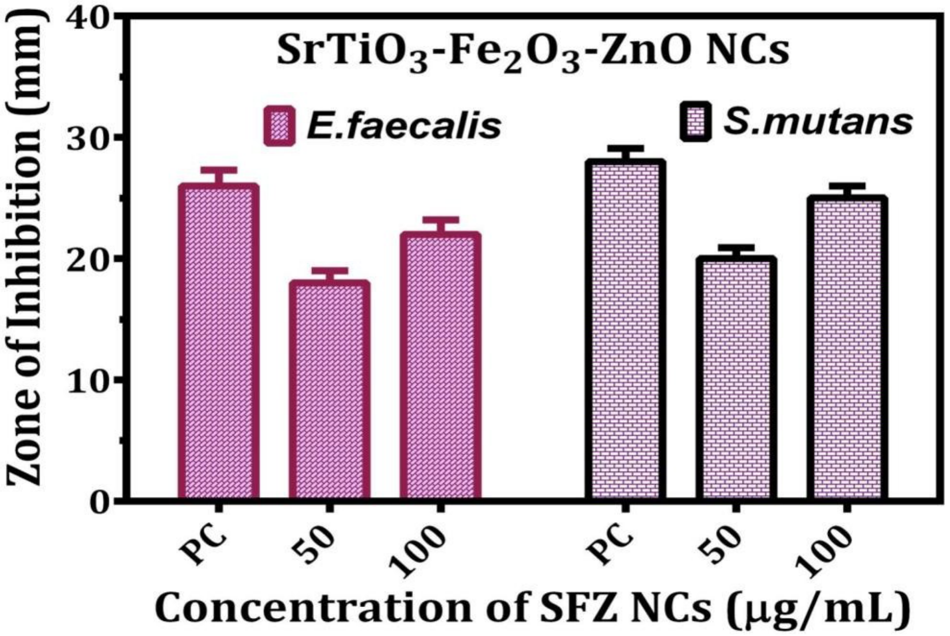


Figure 6 - Mean diameter of the zone of Inhibition (in mm) of different antimicrobials performed in duplicates, error bars indicate +SD.

Discussion

From the above results, we conclude that we have successfully synthesised the ternary nanocomposite, which has been confirmed via tests such as XRD, FTIR and SEM. Apart from its synthesis, we have also tested this nanocomposite's antibacterial activity against *E. faecalis* and *S.mutans* with the gold standard and have found satisfactory results.

In a similar research, where ZnO was synthesized, the average crystallite size of ZnO NPs was estimated by Scherrer's formula was found to be 41 nm, which is similar to that of this study [13]. A study that also synthesized mesoporous zinc oxide nanoparticles concluded that characterization of ZnO NPs revealed that all the particles were in a more or less spherical shape with a wide particle size distribution of 70-100 nm. TEM image showed uniform and aggregated ZnO NPs with a typical size of 10-15 nm [14]. By studying and characterising ZnO NPs using *Phlomis* leaf extract, a study concluded that its size was found to be around 79 nm by FESEM, whereas the hydrodynamic radius of nanoparticles was estimated to be around 165 ± 3 nm. FTIR spectra revealed the formation of ZnO bonding and surfactant molecule adsorption on the surface of ZnO NPs [15].

Looking at the antibacterial activity of ZnO NPs, a study concluded that the biogenic ZnO nanoparticles exhibited strong antibacterial activity against *E. coli* and *S. aureus* with a zone of inhibition of 16 mm and 14 mm respectively. This high antibacterial and catalytic activity of biogenic ZnO nanoparticles can be attributed to its small size, good dispersion, and well-defined morphology [16].

In a previously published article , SEM images of strontium oxide nanoparticles at room temperature showed pseudo spherical shape. As the temperature increases up to 400^0C it starts agglomeration and the shape becomes cubic. Finally it becomes cylindrical on further increase of temperature up to 600^0C [17]. It was concluded in another study that the particles in the samples were compactly arranged and were almost spherical in shape [18].

A study that synthesized strontium oxide nanoparticles by *Elodea canadensis* extract and studied it's antibacterial activity concluded that the XRD pattern revealed a face-centered cubic crystalline structure and the presence of both strontium and oxygen was verified in the synthesized NPs. The antibacterial potential of the NPs against *Escherichia coli* and *Bacillus subtilis* was evaluated. The largest inhibitory zone against *E. coli* (diameter, 22 mm) and *B. subtilis* (diameter, 20 mm) was observed at a concentration of 24 $\mu g \cdot mL^{-1}$ [19].

In a similar study that characterized iron nanoparticles, Fe_3O_4-NPs exhibited a granular, homogenous, spherical-shaped structure with an average diameter of approximately 16 nm [20]. Previous studies that synthesized ferric oxide NCs studied its antibacterial activity, concluded the broad spectrum antibacterial activity of the tested nanomaterials against both Gram positive (*S. aureus*) and Gram negative (*E. coli*) bacteria with increased activity against Gram positive bacteria than Gram negative one [21]. Another study that also studied the antimicrobial activity of iron oxide nanoparticles, concluded that antibacterial properties against Gram-positive bacteria (*Staphylococcus epidermidis*, *Staphylococcus aureus*, *Micrococcus luteus*, *Enterococcus faecalis*, *Bacillus cereus*) were better and had a MIC value of 2.5-10 mg/mL to 0.313-1.25 mg/mL [22].

Although the above results show us a promising future for $SrTiO_3/Fe_2O_3/ZnO$ NCs, it is important to note that more studies are required to understand various other applications such as antioxidant, anti inflammatory, antifungal properties, etc. Since this is an in vitro study, its side effects and limitations are unknown and in order to study this, more research, especially in vivo studies are required. But collectively, the findings of this research show that the synthesis of $SrTiO_3/Fe_2O_3/ZnO$ NCs is a probable option for developing novel materials for biomedical applications.

Conclusion

From this study, it can thus be concluded that we have successfully synthesized and characterized $SrTiO_3/Fe_2O_3/ZnO$ nanocomposites and proved its antibacterial activity against *S.mutans* and *E.faecalis*. However, since this is an in vitro study, it has to be studied in detail in vivo to know its true advantages, as well as limitations. By incorporating these nanoparticles in commercial products, they will help in reducing the risks of oral diseases and progression of the bacterial growth.

Acknowledgement:

We express our sincere gratitude to Saveetha Dental College and Hospitals and Saveetha Institute of Technical and Medical Sciences for their constant support and encouragement.

Conflict of Interest:

Nil.

Author Contribution:

Ms. Sri Gopika T: Literature search, data collection, manuscript writing.

Dr. Vigneshwar : Study design, data verification, manuscript drafting

REFERENCES

1. Berry CC, Curtis ASG. Functionalisation of magnetic nanoparticles for applications in biomedicine. J Phys D Appl Phys. 2003 Jul 7;36(13):R198–206.
2. Murthy SK. Nanoparticles in modern medicine: state of the art and future challenges. Int J Nanomedicine [Internet]. 2007 [cited 2023 Nov 14];2(2). Available from: https://pubmed.ncbi.nlm.nih.gov/17722542/
3. Ramalingam B, Parandhaman T, Das SK. Antibacterial Effects of Biosynthesized Silver Nanoparticles on Surface Ultrastructure and Nanomechanical Properties of Gram-Negative Bacteria viz. Escherichia coli and Pseudomonas aeruginosa. ACS Appl Mater Interfaces. 2016 Feb;8(7):4963–76.
4. Wang L, Hu C, Shao L. The antimicrobial activity of nanoparticles: present situation and prospects for the future. Int J Nanomedicine [Internet]. 2017 Feb 14 [cited 2023 Nov 14];12. Available from: https://pubmed.ncbi.nlm.nih.gov/28243086/
5. Padmavathy N, Vijayaraghavan R. Enhanced bioactivity of ZnO nanoparticles-an antimicrobial study. Sci Technol Adv Mater. 2008 Jul;9(3):035004.
6. Colon G, Ward BC, Webster TJ. Increased osteoblast and decreased Staphylococcus epidermidis functions on nanophase ZnO and TiO2. J Biomed Mater Res A. 2006 Sep 1;78(3):595–604.
7. Mukherjee S, Mishra M. Application of strontium-based nanoparticles in medicine and environmental sciences. Nanotechnology for Environmental Engineering. 2021 Apr 24;6(2):25.
8. Kumar P, Dash SK, Ray S, Parween S. Biomaterials-Based Sensors: Recent Advances and Applications. Springer Nature; 2023. 416 p.
9. The strontium inorganic mutant of the water oxidizing center (CaMn4O5) of PSII improves WOC efficiency but slows electron flux through the terminal acceptors. Biochimica et Biophysica Acta (BBA) - Bioenergetics. 2016 Sep 1;1857(9):1550–60.
10. Hydrothermal synthesis of hematite (α-Fe2O3) nanoparticle forms: Synthesis conditions, structure, particle shape analysis, cytotoxicity and magnetic properties. Journal of Alloys and Compounds. 2019 Jul 5;792:599–609.
11. Preparation, characterization and antifungal activity of iron oxide nanoparticles. Microbial Pathogenesis. 2018 Feb 1;115:287–92.
12. Functionalized nanoparticles enable tracking the rapid entry and release of doxorubicin in human pancreatic cancer cells. Micron. 2017 Jan 1;92:25–31.
13. Biosynthesis of zinc oxide nanoparticles using culture filtrates of Aspergillus niger: Antimicrobial textiles and dye degradation studies. OpenNano. 2018 Jan 1;3:48–55.
14. Jowkar Z, Moaddeli A, Shafiei F, Tadayon T, Hamidi SA. Synthesis and characterization of mesoporous

zinc oxide nanoparticles and evaluation of their biocompatibility in L929 fibroblasts. Clin Exp Dent Res. 2024 Feb;10(1):e844.

15. Alyamani AA, Albukhaty S, Aloufi S, AlMalki FA, Al-Karagoly H, Sulaiman GM. Green Fabrication of Zinc Oxide Nanoparticles Using Leaf Extract: Characterization and In Vitro Evaluation of Cytotoxicity and Antibacterial Properties. Molecules [Internet]. 2021 Oct 11;26(20). Available from: http://dx.doi.org/10.3390/molecules26206140

16. Ali J, Irshad R, Li B, Tahir K, Ahmad A, Shakeel M, et al. Synthesis and characterization of phytochemical fabricated zinc oxide nanoparticles with enhanced antibacterial and catalytic applications. J Photochem Photobiol B. 2018 Jun;183:349–56.

17. Alimuddin, Rafeeq M. Synthesis and Characterization of Strontium Oxide Nano Particle by Sol-Gel Method. Oriental J Chem. 2021 Feb 28;37(1):177–80.

18. Ahmad J, Wahab R, Siddiqui MA, Saquib Q, Ahmad N, Al-Khedhairy AA. Strontium-Doped Nickel Oxide Nanoparticles: Synthesis, Characterization, and Cytotoxicity Study in Human Lung Cancer A549 Cells. Biol Trace Elem Res. 2022 Apr;200(4):1598–607.

19. Website [Internet]. Available from: Anbu, P., Gopinath, S.C.B., Salimi, M.N. et al. Green synthesized strontium oxide nanoparticles by Elodea canadensis extract and their antibacterial activity. J Nanostruct Chem 12, 365–373 (2022). https://doi.org/10.1007/s40097-021-00420-x

20. Kiwumulo HF, Muwonge H, Ibingira C, Lubwama M, Kirabira JB, Ssekitoleko RT. Green synthesis and characterization of iron-oxide nanoparticles using Moringa oleifera: a potential protocol for use in low and middle income countries. BMC Res Notes. 2022 Apr 25;15(1):149.

21. Mohamed A, Atta RR, Kotp AA, Abo El-Ela FI, Abd El-Raheem H, Farghali A, et al. Green synthesis and characterization of iron oxide nanoparticles for the removal of heavy metals (Cd and Ni) from aqueous solutions with Antimicrobial Investigation. Sci Rep. 2023 May 4;13(1):7227.

22. Flieger J, Pasieczna-Patkowska S, Żuk N, Panek R, Korona-Głowniak I, Suśniak K, et al. Characteristics and Antimicrobial Activities of Iron Oxide Nanoparticles Obtained via Mixed-Mode Chemical/Biogenic Synthesis Using Spent Hop (L.) Extracts. Antibiotics (Basel) [Internet]. 2024 Jan 23;13(2). Available from: http://dx.doi.org/10.3390/antibiotics13020111.

..