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Synthesis And Characterization of Flourine Doped Tin Dioxide (Sno2) Nanoparticles Using Prosopis Juliflora Leaf Extract and Their Biological Activities

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KEYWORDS

Nanoparticles**,** Characterization, *Prosopis Juliflora* leaf, F- doped SnO_2, Antimicrobial activity.

ABSTRACT:

The field of nanotechnology is one of the most active research areas in modern materials science. Types of metals used in the manufacture of nanoparticles. SnO_2 is an important n-type wide band semiconductor that appears as one of the most important classes due to the diversity of controllable physicochemical characteristics of the nanoparticles from SnO_2. Therefore, the development of highly effective nanomaterials with advanced antibacterial functions is of the utmost importance. The aim of this study was to synthesize 10% F doped tin dioxide (SnO_2) (FTO) nanoparticles using the leaf extract of *Prosopis juliflora* to determine its antibiotic activity against selected microbial strains. The characterization of the synthesized nanoparticles was studied by scanning electron microscopy, transmission electron microscopy and UV–Vis–IR spectroscopy. Particle size was obtained in the range of 60 ~ 90nm. The antibiotic activity of SnO_2 nanoparticles doped with 10%F was studied against bacteria (*Escherichia coli*, *Staphylococcus aureus* and *Bacillus subtilis*) and fungi (*Aspergillus flavus* and *Aspergillus niger*) using the standard disc diffusion method. The antimicrobial activity was increased directly proportional to dose-dependent manner. Experimental results demonstrated that 10 % F doped Tin dioxide (SnO_2) nanoparticles (F-doped SnO_2) exhibited the good antimicrobial effects were observed.

Introduction

The field of nanotechnology is one of the most active areas of research in modern materials science. Nanoparticles exhibit completely new or improved properties based on specific characteristics such as size, distribution and morphology. Nanotechnology is a field that is burgeoning day by day, making an impact in all spheres of human life. New applications of nanoparticles and nano materials are emerging rapidly (Naiwa, 2000). Nano sized tin oxide materials have been reported to possess specific properties and advantages of high sensitivity, including conductivity, transparency in the visible region, in addition to mechanical and chemical stabilities (Ayeshamariam et al., 2013). Thus, nano-materials play an important role in the basic and practical sciences as well as in bio-nanotechnology (Zhang *et al.* 2012). SnO_2 is a prominent n-type wide-bandgap semiconductor, and due to the variety of controllable physicochemical features of SnO_2-based nanostructures, they are emerging as one of the most significant classes. Many studies have recently reported the synthesis of SnO_2-based nanocomposite materials, which can significantly

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improve performance (Gebreslassie and Gebretnsae, 2021). Elemental doping is one of the most essential techniques for material modification. It is well known that fluorine is considered to be a highly efficient and inexpensive dopant in the field of materials. Fluorine is one of the most reactive elements with the highest electronegativity. F-doped SnO_2 has been synthesized by various techniques (Jiale Huo *et al*., 2023).

Biological methods of synthesis have paved way for the "greener synthesis" of nanoparticles and these have proven to be better methods due to slower kinetics, they offer better manipulation and control over crystal growth and their stabilization. This has motivated an upsurge in research on the synthesis routes that allow better control of shape and size for various nano technological applications (Vennila Raj et al., 2013). The use of environmentally benign materials like plant extract, bacteria, fungi and enzymes for the synthesis of fluoride nanoparticles offer numerous benefits of eco-friendliness and compatibility for pharmaceutical and other biomedical applications (Waikhom *et al*., 2022) as they do not use toxic chemicals for the synthesis protocol.

Bacterial and fungal infections are a major cause of chronic infections and mortality. Antibiotics have been the preferred treatment method for bacterial infections because of their cost-effectiveness and powerful outcomes. Therefore, the development of more efficient material with enhanced antimicrobial activity is of great significance. Despite the great progress in antimicrobial development, many infectious diseases like intracellular infections are difficult to treat (Matthews et al., 2010). Major reasons of difficulty are transportation through cell membranes, low activity in the cells, antimicrobial toxicity to healthy tissues and acquired resistance of infectious microbes (Deotale et al., 2010) To address these issues, nanoscale materials have been emerged up as novel antimicrobial agents. Nanoparticles (NPs) are ideal forms of antimicrobial agents because these materials exhibit large surface to volume ratio and high reactivity in comparison to bulk form (Reddy et al., 2007).

In the present study, 10% Fluorine doped Tin dioxide (SnO_2) nanoparticles have been synthesized by using *Prosopis Juliflora* leaf extract. The prepared F nanoparticles have been doped in tin oxide and determine its antimicrobial activity against selected microbial strains.

Materials and Method

Collection of plant materials

The leaves of *Prosopis Juliflora* were collected in January 2021 from Poondi, Thanjavur District, Tamil Nadu, India.

Preparation of aqueous extract of *Prosopis Juliflora* leaf

The collected leaves were washed with double distilled water to remove dust particles and then dried in the shade for two days for remove wet condition. 20 g of leaf sample was mixed 100ml of deionized water and the mixture was boiled for 30 min. after cooling the leaf extract was filtered with whatman no.1 filter paper. The filtrate was stored at 4 decree C for further uses.

Synthesis of 10% F-doped SnO_2 nanoparticles using *Prosopis Juliflora* (PJ) leaf extract

For synthesis of F doped SnO_2 NPs, 0.1 M $SnCl_2 \cdot 2H_2O$ solution and Ammonium fluoride were used as fluorine dopant (10%) was prepared with 80 mL water. Then 20 mL *Prosopis juliflora* leaf extract was added and 0.1 M NaOH solution added maintain pH level 6-7 to solution and kept under continuous stirring at 80^{o}C for 2 h. The F-SnO_2 PJ NPs were then collected with annealing the sample in a furnace at 350 deg C for 3 h. F-SnO_2 nanoparticles collected and involved character study and application. Preparation F-SnO_2 nanoparticles by co-precipitate method.

Characterization of nanoparticles

UV and FTIR Spectroscopic analysis

The nanoparticles were examined under UV and visible spectrophotometer analysis. The nanoparticles were scanned within the wavelength starting from 200-1000 nm using Perkin Elmer photometer and also the characteristic peaks were identified. FTIR analysis was performed using Spectrophotometer system, which was used to detect the characteristic peaks in ranging from 400-4000 cm^{-1} and their functional groups. The peak values of the UV and FTIR were recorded. Each and every analysis was repeated twice for the spectrum confirmation

Scanning Electron microscopy (SEM)

The particle size and morphology of nanoparticles were analysed by ZEEISS-SEM machine. The dried form of

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silver nanoparticles were sonicated with distilled water, small droplet nanoparticles were placed on glass slide and permitted to dry. The ZEEISS-SEM machine was worked at a vacuum of the order of 10-5 torr. The accelerating voltage is 20 kV. The particle size of nanoparticles can be analyzed by using image magnification software compatible with SEM.

Transmission Electron microscopy (TEM)

Transmission electron microscopy (TEM) technique was used to visualize the morphology of the nanoparticles. The make of the Transmission electron microscope (TEM; Philips model CM200) technique was to visualize the morphology of nanoparticles. The instrument was operated at an accelerating voltage of 200 kV with ultra-high-resolution of 0.2 nm and magnification of 2,000,000 X. TEM's grid size is 3 mm diameter which was prepared placinga 5µl of the nanoparticle solutions on carbon-coated copper grids and drying under mercury lamp and then analyzed

Determination of antimicrobial activity

The antimicrobial activity was performed by disc diffusion method followed by NCCLS (1993) and Awoyinka *et al.,* (2007). Antibiogram was done by disc diffusion method using test sample. Petri plates were prepared by pouring 30 ml of NA medium for bacteria. The test organism was inoculated on solidified agar plate with the help of micropipette and spread and allowed to dry for 10 mints. The surfaces of media were inoculated with bacteria from a broth culture. A sterile cotton swab is dipped into a standardized bacterial test suspension and used to evenly inoculate the entire surface of the Nutrient agar plate while PDA used for fungal study. Briefly, inoculums containing of bacteria (*Escherichia coli, Staphylococcus aureus* and *Bacillus subtilis*) and fungi (*Aspergillus flavus* and *Aspergillus niger*) were spread on agar plates. Using sterile forceps, the sterile filter papers (6 mm diameter) containing different concentration of sample (50, 100, 150 and 200 µl), 30µl deionized water (control) and 30µl Standard (Bacteria: Chloramphenicol and Fungi : Clotrimazole**)** solution were laid down on the surface of inoculated agar plate. The plates were incubated at 37 ºC for 24 h for the bacteria.

Results and Discussion

UV–Vis spectroscopy is the most important and simplest technique for verifying the formation of nanoparticles. In the UV-visible absorption spectrum of F-SnO_2 nanoparticles, a clear absorption peak was observed at a wavelength of 200 nm (Figure 1). Lisnic *et al*. (Current research agreement with Solar Cell Applications 2023) Report that absorption peaks are observed at a wavelength of 350nm from F- SnO_2 thin sheet.

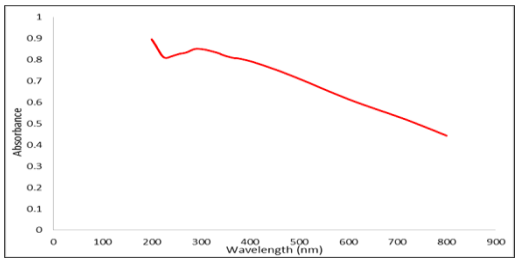

Figure 1: UV-Visible Spectroscopic analysis of 10% F-doped SnO_2 nanoparticles

FTIR Spectroscopic analysis

The FTIR spectra for the F-doped SnO_2 images are shown in Figure 2. The spectrum is formed at several absorption peaks assigned to the vibration pattern of F-SnO_2, which confirms its formation. The spectrum obtained is comparable to those reported in the literature (Kim and Riu, 2011; Kar and Kundoo, 2015). Spectrum refers to several absorption peaks that confirm the formation of the material. 500 - 1000 cm^{-1} were assigned to peak Sn-O and Sn-O-Sn vibrations (642.30cm-1) (Van Tran et al., 2010). The small peak (1024,93,1113,13 cm-1) between 600 and 1900 cm^{-1} is due to the Sn-OH vibration. The peak is located at 1024.93 cm^{-1}, but the increase in fluorine concentration can be attributed to the O-Sn-O group (Zhang et al., 2011) Oxygen Vacancies (V^{2+}),). Wide crest (3430.98 cm-1)3000 - 3500 cm^{-1} OH extension due to vibration. Si-O vibrations caused by the substrate cause a peak of 1113 cm^{-1}. The FTIR spectrum was obtained to identify potential biomolecules in extracts that could predict their role in the synthesis of nanoparticles and their role in ion reduction and agents stability capping in biogenic solutions of nanoparticles.

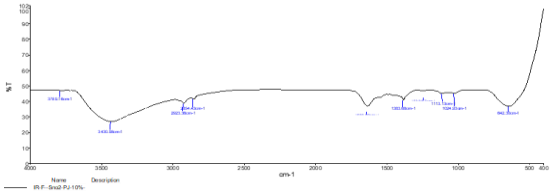

Figure 2: FTIR Spectroscopic analysis of 10% F-doped SnO_2 nanoparticles

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Scanning Electron Microscopical (SEM)

Scanning electron microscopical examination was carried out to know the topology and the NP magnitude, which showed a set of crystals of high-density multi-diffusion spherical NPs and nanoparticles of different sizes in the range of 60 to 90 nm (Figure 3). When they were found in SEM, most of the nanoparticles were collected, and only a small fraction of them were polydisper and spherical. The particle size distribution is studied using the ImageJ software, using a projected map of the obtained data, and the nanoparticles are analyzed. The average particle size was also found to be 78.03 ± 10.75 nm (Figure 4).

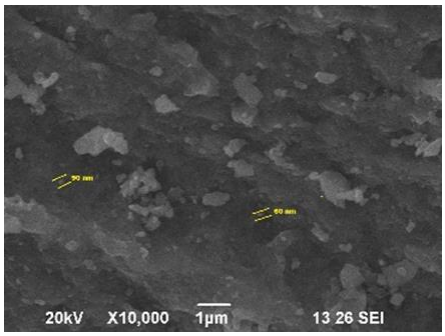

Figure 3: Scanning Electron Microscopical (SEM) analysis of F-doped SnO_2 nanoparticles

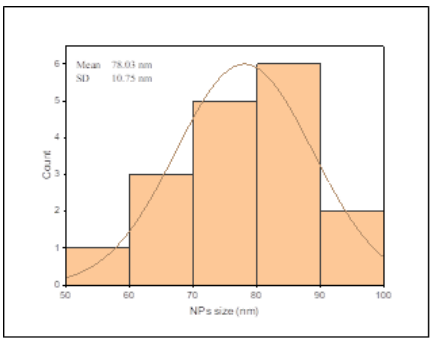

Figure 4: Histogram of showing particle size distribution of F-doped SnO_2 nanoparticles

TEM Analysis

Transmission electron microscope improved the spatial resolution compared to SEM, allowing a detailed analysis of the nanoparticles. The TEM image of the fluorine-doped tin oxide nanoparticles is shown in Figure 5. The morphology of the nanoparticles is often polydisporst and spherical. By examining the size of FTO particles in detail, it is known that they fall into nanoparticles and are integrated. In general, for nanoparticles, due to the activity of surface forces such as the Vanderwalls force, capillary and electrostatic forces, and, this size can only be transmitted to the gravitational and passive forces of particles in the range (Pugh and Bergstrom, 1994). Similar reports were found in Senthilkumar et al. (2010).

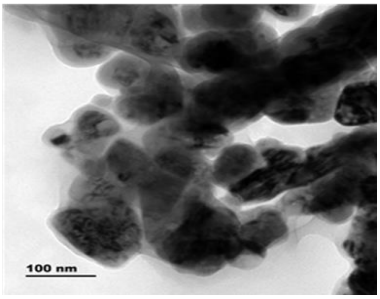

Figure 5: Transmission Electron Microscopical (TEM) analysis of F-doped SnO_2 nanoparticles

Antimicrobial activity of 10% F-doped SnO_2 nanoparticles

The 10% F-SnO_2 nanoparticles tested against bacteria (*Escherichia coli*, *Staphylococcus aureus* and *Bacillus subtilis*) and fungi (*Aspergillus flavus* and *Aspergillus niger*). The maximum zone of inhibition of 10 % F-SnO_2 nanoparticles against bacteria *Escherichia coli*, *Staphylococcus aureus* and *Bacillus subtilis* showed 22, 19 and 20 nm at the concentration of 200µl while the minimum was 6, 5 and 4 nm at the concentration of 50µl. The maximum zone of inhibition of 10 % F-SnO_2 nanoparticles against fungi *Aspergillus flavus* and *Aspergillus niger* showed 10 and 12nm at the concentration of 200µl while the minimum was 2 and 4 nm at the concentration of 50µl. The zone of inhibition was increased with increase the concentrations were noticed. The antimicrobial activity of 10 % F-SnO_2 nanoparticles are nearest to the standard (Chloramphenicol and Clotrimazole) were observed (Table 1 and Figure 6). Among the various bacteria and fungal strains, the antimicrobial property of 10 % F-SnO_2 nanoparticle has potential against *Escherichia coli* and *Aspergillus niger*.

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Table 1: Antimicrobial activity of F-SnO_2 nanoparticles against bactria and fungal strains

S.No.	Microorganisms	Zone of Inhibition (mm in diameter)					
		Control	Standard*	50µl	100µl	150µl	200µl
	Bacteria						
1	*Bacillus Subtilis*	-	13	4	10	15	20
2	*Staphylococcus aureus*	-	12	5	9	13	19
3	*Escherichia coli*	-	14	6	11	16	22
	Fungi						
1	*Aspergillus flavus*	-	19	2	4	6	10
2	*Aspergillus niger*	-	18	4	7	9	12

*Chloramphenicol (30 mcg); Clotrimazole (mcg/disc)

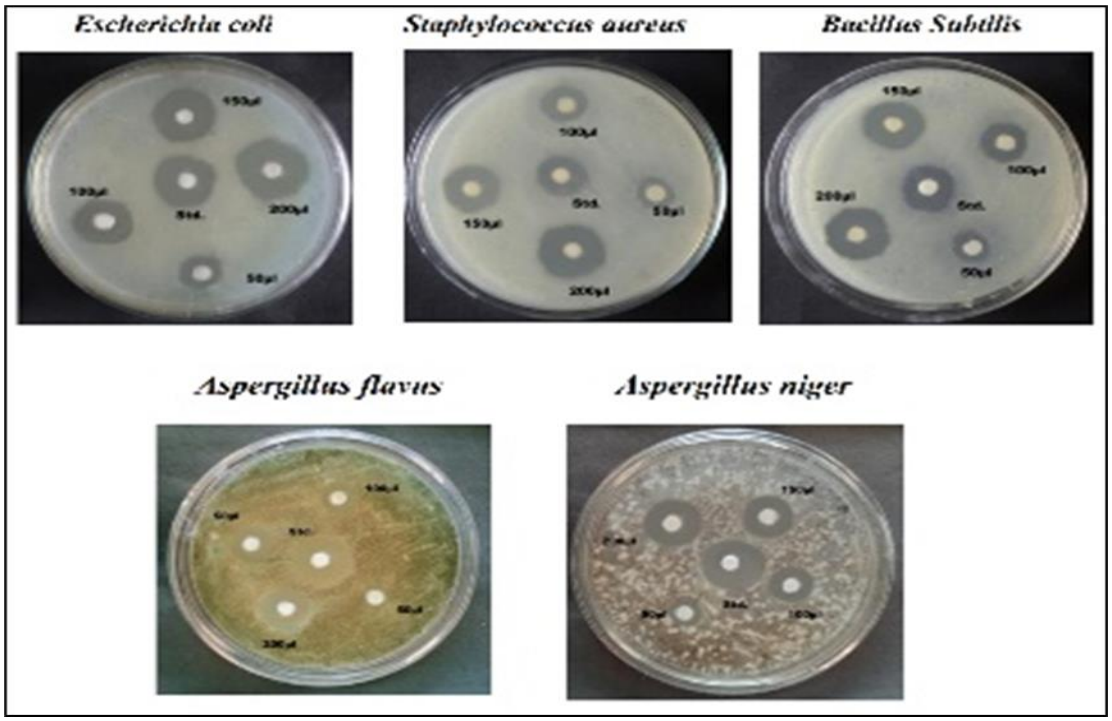

Figure 6: Antimicrobial activity of F-doped SnO_2 nanoparticles against bacterial and fungal strains

Metal nanoparticles are involved considerable attention due to their unique physical and chemical properties, which result from their high surface-to-volume ratio and high levels of surface atoms. In fact, as the diameter decreases, the available surface area of the particles increases significantly and, as a result, it increases more than the original properties of the corresponding total material. This feature makes nanoparticles an ideal candidate for biomedical applications (Sharma et al. 2009) as various biological processes occur at the nanometer level (Prabhu and Poulose, 2012). Thus, nanoparticles have an incredible potential in a variety of biomedical applications, including antibiotics. Recently, several studies have demonstrated that different nanoparticles of metal oxide have biocompatible effects on bacteria and fungi. The antibiotic activity of the nanoparticles is known as a function of the surface interacting with the microorganisms (Franci et al. 2015; Chiriac et al. 2016).

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In recent years, bacterial resistance to fungicides and antibiotics has increased. Many organic antibiotics are toxic to humans and other animals, and can cause various allergic reactions (Rajawat and Qureshi 2012; Hossain et al. 2015). sTo solve this problem, inorganic antibacterial agents have attracted interest in bacterial control because of its safety, stability, thermal resistance and improved stability under harsh processing conditions. Currently, nanotechnology provides a solid platform to adjust the physicochemical properties of a large number of materials in order to produce effective antibiotics (Beyth et al., 2015). Management of silver (Ag), gold (Au), titanium oxide (TiO_2), copper oxide (CuO), zinc oxide (ZnO) and Manganese oxides (MnO), etc, They are also the main metal nanoparticles (NPs) used as an antibacterial agent (Zhang 2015).

The antimicrobial activities of and F doped SnO_2 nanoparticles (F-SnO_2) were studied against bacteria and fungi using the standard disc diffusion method. In the present study 10% F doped SnO_2 nanoparticles tested against bacteria (*Escherichia coli*, *Staphylococcus aureus* and *Bacillus subtilis*). The antibacterial activity was increased dose-dependent manner. Experimental results demonstrated that 10% F-doped tin oxide nanoparticles (F doped SnO_2) exhibited the maximum antibacterial effect compared to standard. Related reports agreed by Algethami *et al*. (2023) who observed the antibacterial activity against *Escherichia coli* and CeO_2−SnO_2 composite nanofibers depicted excellent activity. Another study Assd et al., (2023) who showed that the most effective inhibitor of both Gram-negative and Gram-positive bacteria is SnO_2 nanoparticles produced. Many NPs have antimicrobial properties and used to control drug-resistant microbial populations. Various inorganic metal oxide NPs viz., ZnO, MnO, TiO_2 and SiO_2 exhibit considerable antimicrobial activities and used in therapeutics, diagnostics and nanomedicine-based antimicrobial agents (Mukherjee et al., 2011; Sobha et al., 2010).

Metal oxide nanoparticles have a negative zeta potential and are easily correlated to this surface energy according to their constant equilibrium. This significant bactericidal activity is due to the release of fluorine and Sn ions from F-doped SnO_2 nanoparticles and the penetration of bacterial cell membranes by these ions. The interaction of SnO_2 NP with the cell wall releases ions, forming reactive oxygen species (ROS) on the surface of the nanoparticles. Ion penetration is possible because the positive charges are attracted to the negatively charged cell walls. The binding of these ions destroyed the bacterial cell membrane. In addition, metal ions are involved in the crosslinking of nucleic acid chains by binding to the DNA molecules of the bacterium. This leads to a weak DNA structure, which leads to the disintegration of proteins and the complete destruction of bacterial cells (Reddy et al., 2007; Wang et al., 2015; and; Ahmad et al., 2017). The bactericidal effect of metal nanoparticles is attributed to their small size and high surface-to-volume ratio, and, this allows closer contact with microbial membranes and is not limited to the release of metal ions into the solution (Ruparelia et al., 2008).

Fungi are composed of different groups of different forms, such as single-celled yeast and multicellular organisms. A promising solution as an antifungal drug is the use of nanoparticles (NP). Although many studies have examined this part of nanotechnology, the mechanism of action involved in NP is used as an antifungal agent (Babele *et al*., 2018). In the present study 10% F doped SnO_2 nanoparticles tested against fungi (*Aspergillus flavus* and *Aspergillus niger*). Present study agreement with Jebril et al., (2020) who reported that metal nanoparticles synthesized with *M. charantia* and *P. guajava* extracts showed good antifungal capacity in concentrations of 20 ppm, inhibiting the growth of mycelium in fungi such as *A. niger*, *A. flavus*, and *F. oxysporum*.

Metallic nanoparticles mediated by fungal cytotoxicity damage and alter the cell wall (such as inhibition of ergosterol synthesis), genetic regulation, reproductive structures, and interactions with Haifa, ROS formation and lipid peroxidation occur through a number of mechanisms, including a cascade of damage. Mitochondrial damage.This multifaceted attack has made NP a worthy tool for future antifungal treatment (Slavin and Bach, 2022).

Conclusion

The outcomes of the present study show that 10% of F-doped SnO_2 nanoparticles have strong and potential biological properties such as antimicrobial activity (bacterial and fungal), and, which is of large area and

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10% due to the size of the larger F-doped particle. Nanoparticles. As a consequence, synthesized nanoparticles have great potential for use in cosmetics, food products and pharmaceutical industries.

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