



## Biosynthesis and Characterization of Zinc Sulphide Nanoparticles Using Leaf Extracts of *Tridaxprocumbens*

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### ABSTRACT

In this paper, we discussed biosynthesized and characterization of zinc sulphide nanoparticles using *tridaxprocumbens* leaf extract by green synthesis method. This study focuses on the production of ZnS nanoparticles using *tridaxprocumbens* plant of methanol leaf extracts and analysis the antimicrobial activity. The formation of zinc sulphide nanoparticles was characterized by using numerous analytical techniques such as Scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FT-IR), X-ray powder diffraction (XRD) and UV-Visible spectroscopy (UV-Vis). The results showed that the leaf extract for the synthesis zinc sulphide nanoparticles to have the ability to inhibit the growth of various pathogenic microorganisms. The synthesized zinc sulphide nanoparticles can be used for various applications due to its ecofriendly, non-toxic and compatibility for pharmaceutical and other applications.

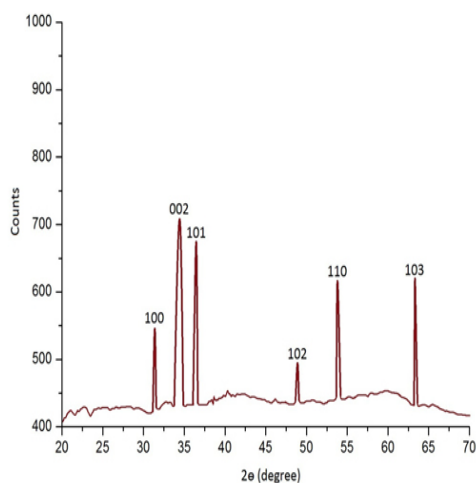
**Keywords:** Zinc sulphide nanoparticles, *Tridaxprocumbens*, Antimicrobial Activity, Scanning electron microscopy (SEM) and X-ray powder diffraction (XRD).

### INTRODUCTION

From last decades, nanoparticles have been received too much attention due to its unique properties like high surface area, magnetic, thermal, mechanical, electronic and enhanced catalytic properties. The nanotechnology has been remarkable development in the synthetic methodologies have

been developed to various nanoparticles of shape, size, dimensions and so on<sup>1-2</sup>. For instance, various types of nanoparticles (NPs) are available such as metal NP<sup>3</sup>, carbon based nanomaterials<sup>4-7</sup>, and polymers<sup>8</sup>, most commonly nanoparticles are synthesis through chemical methods<sup>9</sup>, sol-gel processing<sup>10</sup>, co-precipitation, and electro deposition method<sup>11</sup>. Zinc sulphide has been found highly

attractive because of its remarkable application potential in solar cells, piezoelectric devices, UV-vis absorbers, pharmaceutical and cosmetic industries<sup>12</sup>. Potentially, Zinc sulphide removed all the dyes and water pollutants from textile effluent under UV light have been proved<sup>13-15</sup>. Zinc sulphide nanoparticles were large particle size in bulk materials, morphology and characteristics of the particles can be derived due to their new properties<sup>16</sup>. The amount of the atoms and size approaches nanoscale materials become memorable of their properties<sup>17</sup>. In pharmaceutical applications biosynthesis of zinc sulphide nanoparticles using green chemistry, a method is very useful and ecofriendly environmental method<sup>18</sup>. Although different biological based synthetic method is known for zinc sulphide are sought by researchers. Biosynthesis of zinc sulphide nanoparticles has many advantages in pharmaceutical applications because of use nontoxic materials<sup>19</sup>. Plants become the desirable and superior option for the safe, Nontoxic method of nanoparticles synthesis because the natural capping agents are readily taken from the plants<sup>20</sup>. *Tridaxprocumbens* is belonging to family Asteraceae. It's grown on waste lands and hill stations normally, the leaves and flowers are commonly toothed and arrowhead shaped, yellow colored flowers, *Tridaxprocumbens* plant has more herbal properties in pharmaceuticals<sup>21-22</sup>. Coat buttons is a common name of *Tridaxprocumbens* in English because of its flower appearance. In India its widely used ayurvedic medicine extensively like wound healing, liver disorders. Especially leaves are used skin infection<sup>23</sup>.



**Fig. 1: XRD analysis of ZnS nanoparticles using leaf extract of *tridaxprocumbens***

Antiseptic, Parasitocidal, Insecticidal infections flowers and leaves also used<sup>24-26</sup>. *Tridaxprocumbens* full plants show various antibacterial and fungal activities against many microorganisms and various pharmacological activities<sup>27-32</sup>.

### Plant Extraction

*Tridaxprocumbens* plant fresh leaves were collected locally around the waste land of Idappadi, Salem. Collected leaves washed in DI water and placed in shadow, dried, coarsely powdered using a grinder. 250 ml of methanol solvent, 25 g of coarse leaves powder to extract by using Soxhlet apparatus and stored extracts used for further investigations.

### Alkaloids detection

The hydrochloric acid used to test a presence of alkaloids, *Tridaxprocumbens* leaf extracts were dissolved in HCL and the extract was filtered. The presence of alkaloids to expedite yellow cream formation were filtrated extracts with Mayer's reagent and the presence of alkaloids to precipitate brown or reddish brown colour formation were filtrated with Wagner's reagent.

### Flavonoids detection

The presence of flavonoids to expedite yellow colour formation were a solution of lead acetate treated with drops of extracts and H<sub>2</sub>SO<sub>4</sub> were treating with leaf extract, orange colour formation to the presence of flavonoids.

### Steroids detection

Presences of steroids were leaf extract added 1 ml of acetic anhydride with H<sub>2</sub>SO<sub>4</sub> expedite blue or green from violet colour.

### Terpenoids detection

In Salkowski's test presence of terpenoids, indicate reddish brown were leaf extract was mixed chloroform with H<sub>2</sub>SO<sub>4</sub>.

### Anthraquinones detection

In Borntrager's test presence of anthraquinones expedite pink colour, were NH<sub>3</sub> drops was adding cool

Filtered with CHCl<sub>3</sub> and before filtering extract was boiled HCL using a water bath in few minutes.

### Phenols detection

In Ferric chloride and lead acetate test presence of phenol indicates bluish black colour formation were leaf extracts mixing with ferric chloride drop by drops, yellow colour expedites were solution of lead acetate was adding in the extract

### Saponins and Tannins detection

The presence of saponins was extract shaken with DI water it indicate a frothing formation when ferric chloride added to filtrate of mixing heated water bath with extract to the formation of dark green colour indicate of tannins.

### Carbohydrates, Oil and Resins detection

DI water was dissolved in extract and filtered carbohydrate presence was tested using filtrate. Were the test solution was applied on the filter paper its form a transparent appearance to indicate the presence of oil and resins.

### Alkaloids estimation

Harborne (1973) method to use determined of alkaloid, 200 ml acetic acid was taken in 250 ml of the beaker and 4 to 5g extract were added together and the beaker allowed to stand more than 4h after that extract was filtered and treated to a water bath with original volume. Ammonium hydroxide was added drop by drop of the extract until it was complete. Once extracts were washed with dilute ammonium hydroxide solution it's filtered and weighed.

### Flavonoids estimation

The filter paper was used to dry and weighed of flavonoids, In 250 ml beaker uses to mixing 100 ml of methanol and 10g of leaf extract at room temperature.

### Steroids estimation

2 ml of sulphuric acid, 2 ml of iron chloride was added and 5 ml of potassium hexacyanoferrate solution were mixed with 10 ml of *Tridaxprocumbens* leaf extract, followed mixing were treated heating water bath at 20°C to 70°C in 30 minutes, then the mixed solution dissolved in DI water.

### Saponin determination

The saponin content calculate using the methanol, 10 ml methanol were treated with 10 ml

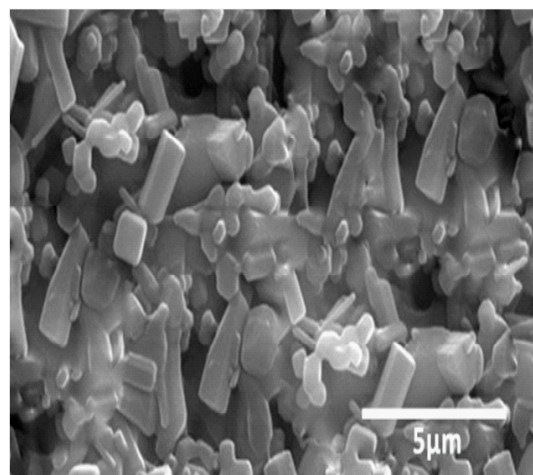
*Tridaxprocumbens* plant extract it was dispersed and solution was using continuous stirring 4 hours at 55°C With heated hot water, after complete stirring solution was filtered and again 200 ml methanol added. In that solution was heated till it reduce 90 ml using heat water bath, from the 90 ml solution 20 ml of diethyl added and shacked with separate funnel and purification process was taken. In that purified solution was adding with 10 ml of sodium chloride and remaining was treated water bathing and dried with help of oven.

### Apparatus and synthesis

The surface morphology and EDX of the composite were studied by Hitachi S-4500 scanning electron microscope (SEM). Fourier transform infrared spectroscopy (FT-IR) was measurements were carried out by using JASCO FT/1 IR-6600 instrument. XRD pattern were analyzed by X-ray diffractometer (XPRT-PRO).

### Biosynthesis of zinc sulphide nanoparticles

*Tridaxprocumbens* used to synthesis zinc sulphide nanoparticles were 14.4 g of zinc sulphate was added 100 ml DI water, one millimole aqueous solution of zinc sulphate ( $ZnSO_4$ ) was prepared, 20 ml of methanol leaf extract were added drop by drop in 80 ml (1mM) of zinc sulphate aqueous solution which was kept continuous stirring for 2 hours at room temperature, result was indicate the formation of zinc sulphide nanocolloid, then the final solution dried at 60°C for 5 hours.



**Fig. 2: SEM analyses of ZnS nanoparticles using leaf extract *Tridaxprocumbens***

## RESULT AND DISCUSSIONS

### XRD Pattern

The crystalline structural properties of prepared biosynthesis of ZnS nanoparticles powder sample is studied using X-ray diffraction figure 1 shows the different peak pattern of synthesized ZnS at  $2\theta$  value of  $31.82^\circ, 34.63^\circ, 36.07^\circ, 48.72^\circ, 54.04^\circ$  and  $63.05^\circ$  and corresponding reflection planes is 100,002,101,102,110 and 103 respectively it indicate Hexagonal Wurtzite structure.

Debye Scherrer's equations were used to calculate particle crystalline grain size of the ZnS.

$$D = \frac{K\lambda}{\beta \cos \theta} \text{ \AA}$$

Here D is the mean of grain size, K= 0.9 constant value,  $\lambda = 0.5323 \text{ \AA}$  is x-ray wavelength for CuKa radiation,  $\theta$  is the diffraction angle in radian,  $\beta$  is full width half maximum (FWHM) in radian so the average grain size of synthesis zinc sulphide is ~ 40 nm

### Morphology studies

Figure 2 clearly shows the surface morphology of biosynthesized zinc sulphide nanoparticle powder were examined and found different radial hexagonal, spherical, rod shapes and some surface areas various size of hexagonal shape particles observed and their size was found to be less than  $5 \mu\text{m}$ .

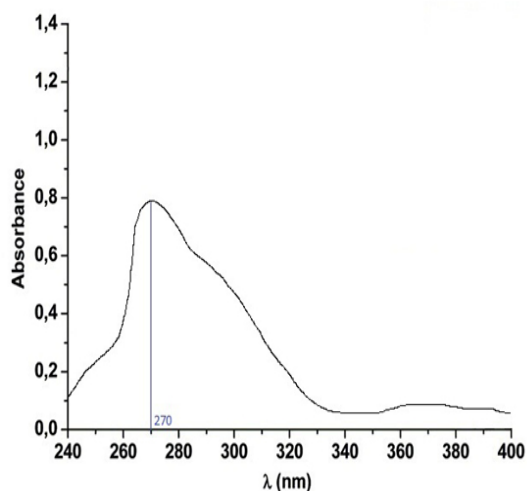


Fig. 3: UV-Visible analysis of ZnS nanoparticles using leaf extract *tridaxprocubens*

### UV- Visible analysis

The optical absorption of biosynthesized zinc sulphide nanoparticle clearly shown in figure 3 and absorption range 200 – 400 nm, the maximum absorption occurred at 270 nm.

### FT-IR Spectrum

The FTIR analysis of zinc sulphide nanoparticles synthesized using *tridaxprocubens* methanol leaf extract showed in figure 4, ranging from 500 – 4000  $\text{cm}^{-1}$  wave numbers and its produces zero to two N-H absorptions depending on its type at  $3850.50 \text{ cm}^{-1}$ , Alkenyl C-H stretch at  $3101.94 \text{ cm}^{-1}$ , Alkenyl C=C stretch at  $1642.05 \text{ cm}^{-1}$ , Aromatic C=C Bending at  $1502.47 \text{ cm}^{-1}$ , Polysaccharides at  $1091.33 \text{ cm}^{-1}$  and C-H Vibration at  $594.62 \text{ cm}^{-1}$  according to this stretching ZnS nanoparticles have degradation of TGA

### Antimicrobial activity of ZnS

Muller Hinton Agar (MHA) plate from Hi-media Mumbai very useful to screening antimicrobial activity, Here disc diffusion method was helpful to analysis antimicrobial activity. Before swap inoculums in the MHA plate surface it was treated 15 ml molten media in to sterile petriplates from pouring process and allowed 5 minutes for solidify then the minimum amount of (0.1%) microorganisms suspension was transferred uniformly in MHA plate surface and allowed 5 minute for drying , then 60 mg concentration disc placed on 6 mm sterile disc that

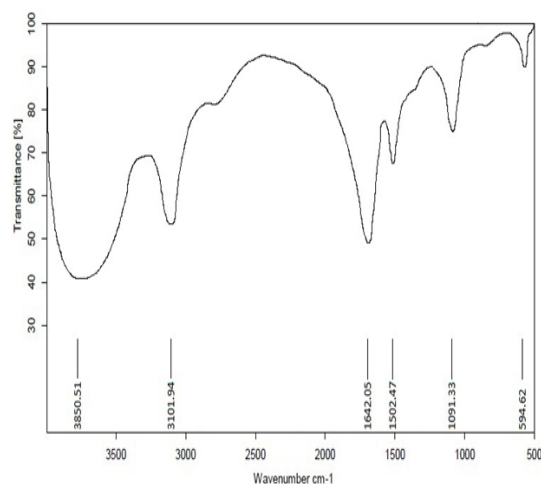


Fig. 4: FTIR analysis of ZnS nanoparticles using leaf extract *Tridaxprocubens*

was placed in surface of MHA plates for allowing 5 minutes to diffuse the ZnS leaf extract solution, after diffused that MHA plates are placed incubation at 37° C for 24 hours, during the incubation zone of inhibition formed around the sterile disc and zone was calculate by using transparent millimeter ruler. The phytochemical analysis of methanol leaf extract of *Tridaxprocumbens* zinc sulphide was clearly indicate the Table 1 and the plant found a lkaloids, flavonoids, phenol, crbohydrates, steroids were absent in Terpenoid, anthrouinone, saponins

**Table 1: Qualitative phytochemical analysis of ZnS nanoparticles using leaf extract of *Tridaxprocumbens***

Phyto chemicals	Observations	Extracts Methanol
<b>Alkaloids</b>		+
Mayer's test	Cream color	+
Wagner's test	Reddish brown solution/ precipitate	
<b>Flavonoids</b>		+
Lead acetate test	Yellow orange	+
H <sub>2</sub> SO <sub>4</sub> test	Reddish brown / Orange color precipitate	
<b>Steroids</b>		+
Liebermann-	Violet to blue or Green	
Burchard test	color formation	
<b>Terpenoids</b>		-
Salkowski test	Reddish brown precipitate	
<b>Arthroquinone</b>		-
Borntrager's test	Pink color	
<b>Phenols</b>		+
Ferric chloride test	Deep blue to Black color formation	+
Lead acetate test	White precipitate	
<b>Saponin</b>	Stable persistent	-
<b>Tanin</b>	Brownish green / Blue black	-
<b>Carbohydrates</b>	Yellow / brownish / blue / green color	+
<b>Oils &amp; Resins</b>	Filter paper method	-

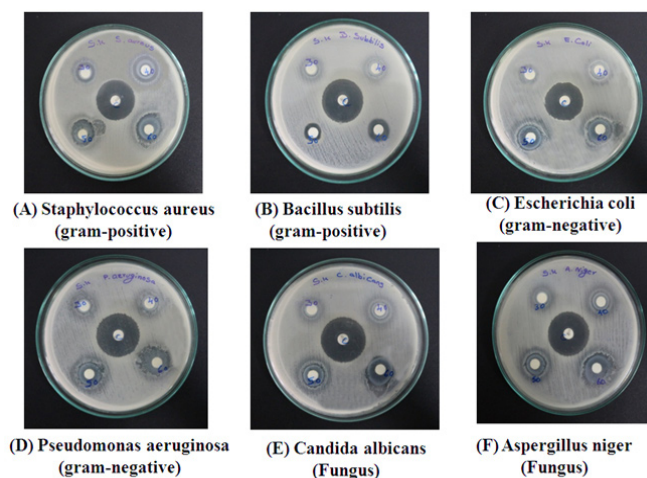
'+' Present, '-' Absent

,tannins oil and resins. In this different functional which was helpful to react with zinc sulphide and increase a zone of inhibitions, FTIR analysis shows different function group bonding to enter the cell membrane of inoculums. The quantitative analysis by presence and absence of inhibition zone, methanol leaf extract of zinc sulphide using *Tridaxprocumbens* has excellent antimicrobial activity against all tested microorganisms its clearly shown in figure 5. It was tested both gram positive, gram negative bacteria's and fungal culture by disc diffusion method the zone of inhibition is formed antibiotic action of zinc sulphide nanoparticles. Table 2 clearly indicate antimicrobial activity of all tested microorganism with different concentration at 30 µl, 40 µl, 50 µl and 60 µl, in this six microorganisms *Staphylococcus aureus*, *Bacillus subtilis* are gram positive and *Escherichia coli*, *Pseudomonas aeruginosa* are gram negative and *Candida albicans*, *Aspergillus niger* is fungus culture, at a concentration 30 µl *P.aeruginosa* start a high zone of inhibition (15 mm) followed by *S.aureus* and *E.coli* (14 mm), minimum zone of inhibition was found in *B.subtilis* (8 mm) from bacterial culture but in a fungus culture *C.albicans* (11 mm) and *A.niger* (10 mm) here *C.albicans* lead high zone of inhibition to compare *A.niger*.

The concentration 40 µl and 50 µl same zone of inhibition was increased in both bacterial and fungal culture but concentration 50 µl *B.subtilis* inhibition zone was increased as the same of *A.niger* fungus culture, *S.aureus* inhibition zone also increased to compare *E.coli*, but the maximum zone was found in *P.aeruginosa* at 40 µl and 50 µl concentration.

**Table 2: Antimicrobial activity of ZnS nanoparticles using leaf extract of *Tridaxprocumbens***

Organism	Different concentration of ZnS sampleS				
	C	30 µl	40 µl	50 µl	60 µl
<i>S.aureus</i>	26	14	15	18	20
<i>B.subtilis</i>	25	8	10	13	18
<i>E.coli</i>	24	14	16	17	20
<i>P.aeruginosa</i>	28	15	16	19	22
<i>C.albicans</i>	24	11	14	18	20
<i>A.niger</i>	25	10	11	13	15



**Fig. 5: Antimicrobial activity of ZnS nanoparticles using leaf extract of Tridaxprocumbens**

Finally the maximum zone of inhibition were observed at 60  $\mu$ l concentration for *P.aeruginosa* (22 mm) and minimum zone of inhibition was observed *B.subtilis* (18 mm) and *A.niger* (15 mm), so the in vitro antimicrobial activity of biosynthesized zinc sulphide leaf extract was high excellent activity against all tested microorganisms, the tridaxprocumbens various plant parts are used to found antimicrobial agents<sup>33</sup>there are so many plants are have high antimicrobial activity because of their zone of inhibition against bacteria and fungus<sup>34</sup>Biosynthesis is good and new field of research because it's from natural sources<sup>35</sup>.

### CONCLUSION

In conclusion, we have reported a biosynthesized of zinc sulphide nanoparticles using methanol leaf extract tridaxprocumbens for the first time, and the structural characterization and morphology of the zinc sulphide nanoparticles analyzed using XRD and SEM, the ZnS nanoparticles are Hexagonal wurtzite structure and average grain size of the nanoparticles is 40 nm and surface

morphology was found various size of nanoparticles below 5 $\mu$ m, the optical observation of the colloidal solution were studied using UV-Visible the maximum absorption frequency was 270 nm and FTIR very useful to found different functional group present in the leaf extract solution. Further antimicrobial activity of tridaxprocumbens methanol leaf extract and synthesized zinc sulphide nanoparticles were investigated in the disc diffusion method from the result it's clear to know that the zinc sulphide nanoparticles have excellent zone of inhibition in the all tested pathogenic microorganisms like *Staphylococcus aureus*, *Bacillus subtilis* are gram positive and *Escherichia coli*, *Pseudomonas aeruginosa* are gram negative and *Candida albicans*, *Aspergillusniger* is fungus culture. The maximum zone of inhibition was observed in *Pseudomonas aeruginosa* (22 mm) in 60  $\mu$ l concentration and minimum was observed in *Aspergillusniger*(15 mm), it was evident that the methanol leaf extract tridaxprocumbens can be used to synthesis ZnS nanoparticles using green chemistry method for various applications.

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