BIO-SYNTHESIS AND CHARACTERIZATION OF TELLURIUM DIOXIDE NANOPARTICLES USING *TEPHROSIA PURPUREA* AND ITS BIO-POTENTIALS

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ABSTRACT

Nanoparticle synthesis using plant is an alternative to conventional physical and chemical methods. In this study, we used a simple green method for preparing tellurium nanoparticles using leaf extract of *Tephrosiapurpurea.* Tellurium nanoparticles were rapidly synthesized by the reduction of potassium tellurite anhydrous solution with plant extract as a reducing agent. The synthesized Tellurium nanoparticles were characterized with different instrumental methods such as UV, FT-IR, SEM, XRD and EDAX. The powdered Tellurium Nanoparticles were applied for antimicrobial, antioxidant and anticorrosion studies. The plant mediated nanoparticles synthesis is rapid, simple, cost effective and safe method with various applications.

Keywords: *Tephrosiapurpurea,* Tellurium Nanoparticles, Ultra violet visible, Fourier Transform Infrared Spectroscopy, Scanning Electron Microscopy, X-ray diffraction, Energy-dispersive Xray analysis

1. INTRODUCTION

Green chemistry is the utilization of set of principles that reduces or eliminates the use or generation of hazardous substances in the design, manufacture and application of chemical products. Green synthesis describes the techniques to eliminate them. Green chemistry is about waste minimization of source use of renewable sources using non-toxic reagents improved atom efficiency green synthesis are required to avoid production of unwanted or harmful bi-products through the buildup of reliable, sustainable and ecofriendly synthesis procedures. The use of ideal solvent system and natural resources (such as organic systems) is essential to achieve this goal. The biosynthesis of nanoparticles has been proposed as a cost effective and environmentally friendly alternative to physical and chemical methods.

Examples of green chemistry include manufacture of ammonia manufacture of methanol, manufacture of ethene. Some reactions include water as a solvent for

example in the manufacture of inorganic compounds such as hydrogen peroxide phosphoric acid.

Green synthesis of nanomaterials has many methods namely physical method, chemical method, green method. Physical method is time and energy consuming, and it is synthesized at high temperature and pressure. Chemical method is simple, inexpensive and low temperature is required. Green method is easy, efficient and eco-friendly **[1].**

1.1. ADVANTAGES AND DISADVANTAGES:

The advantages of green synthesis include that they are environmentally friendly, easily scaled up for large synthesis of nanoparticles. They don't need any temperature, pressure energy and toxic chemicals It reduces cost of microorganism isolation and their culture media. The disadvantage is that plants cannot be manipulated as the choice of nanoparticles through optimized synthesis of genetic engineering. Another one is that plants produce low yield of secreted proteins which decrease the synthesis rate **[2].**

1.2. NANO SCIENCE:

The word **Nano science**refers to the study, manipulation and engineering of matter, particles and structures on the nanometer scale (one millionth of a millimeter, the scale of atoms and molecules). Important properties of materials. Such as the electrical, optical, thermal and mechanical properties, are determined by the way molecules and atoms assemble on the nanoscale into larger structures. Moreover, in nanometer size structures these properties often different then on macro scale, because quantum mechanical effects become important. **[3]**

1.3. NANOTECHNOLOGY:

Nanotechnology is a branch of science which deals with matter having at least One dimension sized from 1-100 nm. Nanoparticles have attracted great interest Recently due to their unique physical and chemical properties, which are different from those of either the bulk materials or single atoms. The field of nanotechnology is one of the most active researches in modern material science. Nanotechnology is emerging as a rapid growing field with its applications in science and technology for the purpose of manufacturing new materials at the nano scale level. The nanoparticles possess unique physiochemical, optical and biological properties which can be manipulated suitably for desired applications. The nanoparticles are of great interest due to their externally small size and large surface to volume ratio, and they exhibited utterly novel

characteristics compared to the large particles of bulk material. Moreover, the nanoparticles are more various in shape and size in comparison with those produced by other organisms. Synthesis of nanoparticles can be performed using a number of routinely used chemical and physical methods. The use of plant systems has been considered a green route and a reliable method for the biosynthesis of nanoparticles owing to its environmentally friendly nature **[4]**.

2. MATERIALS AND METHODS BIOSYNTHESIS OF TELLURIUM NANOPARTICLES

2.1. Collection of the plant

Tephrosiapurpurea plant was collected from our college garden in the month of December. The leaves were separated from the plant. The separated leaves were washed with distilled water and wiped to use.

2.2. Preparation of leaf extract

The cleaned leaves were chopped and ground to paste without adding water. About 20 g of leaf paste was taken in 250 mL conical flask and add 100 mL of distilled water. The mixture was kept in the ultrasonicator and heated for 30 minutes at 70°C.

2.3. Synthesis of tellurium oxide nanoparticles

Fig. 2.1 synthesis of tellurium oxide

3 gram of anhydrous potassium tellurite was dissolved in 50 mL of water. 20 mL of *Tephrosia purpura* leaf extract is taken in 100 ml beaker. To this extract, the potassium tellurite solution was added. The mixture was ultra-sonicated for 50 minutes. The light green colour of the solution turned to black. The solution was kept in hot plate for an hour and muffle furnace at 70°C for about 12 hours.

3. RESULTS AND DISCUSSION:

3.1 SOLUBILITY TEST

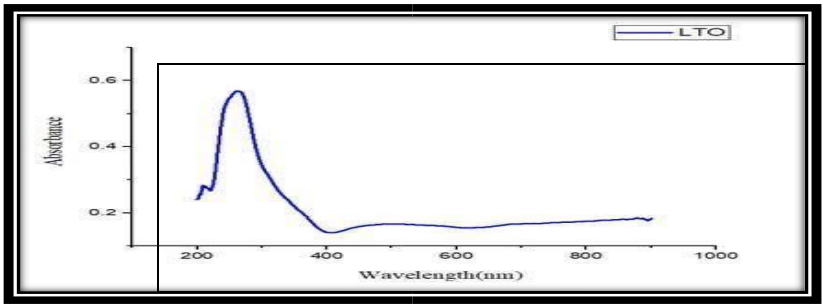
A few milligrams of substance are taken in a test tube and 1 mL of solvent was added. it was Shaken well and check for solubility.

Table 3.1. Solubility of TeNps

Solvents used	**Solubility**
Dil. HCl	Insoluble
Con HCl	Soluble
Con H_2SO_4	Insoluble
NaOH	Insoluble
Ethanol	Soluble
Benzene	Soluble
Acetone	Insoluble
Hot water	Partially soluble

3.2 UV Analysis

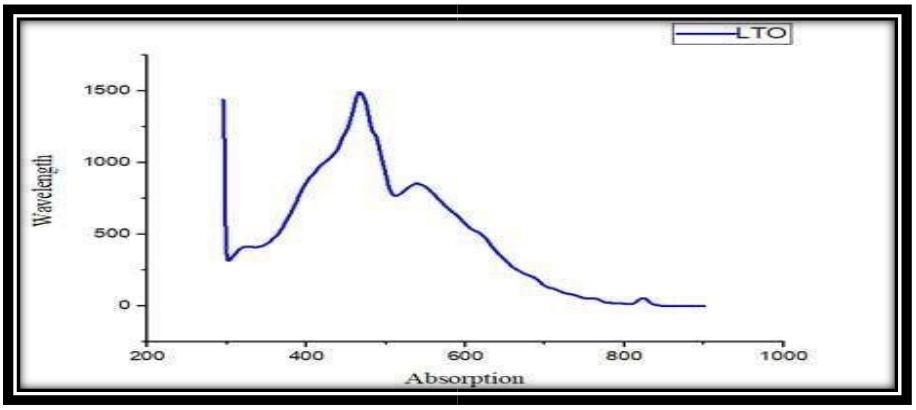
The UV visible spectrum of bio - synthesized TeO_2(LTO) nanoparticles using *Teppuriapurpurea* leaf extract was shown fig. 5.1. An adsorption band in region 200 – 900 nm is observed. The absorption bands were observed at 208 and 265 for LTO. The blue shift is to the small size of nanoparticles. The indicates the formation of smaller particles.



3.1 UV- Visible spectrum of TeO

3.3 UV – Visible Fluorescence Spectroscopy

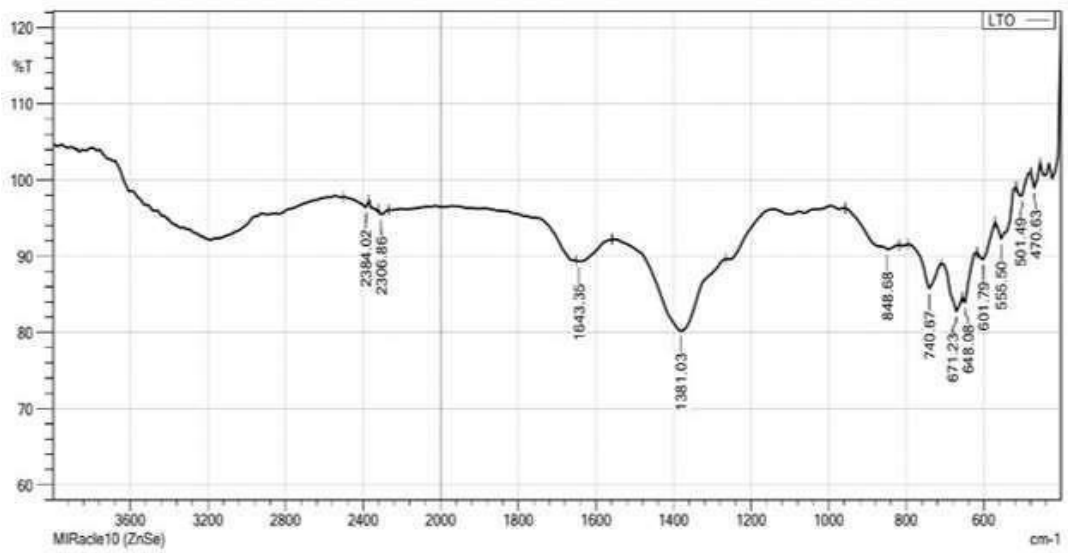
The fluorescence graph for synthesis tellurium nanoparticles recorded between the range excited at 200 – 900 nm. Solid sample emission band at with high intensity, form the emission spectra studies the sample LTO has high intensity.



3.2 UV – Visible Fluorescence Spectroscopy of TeO_2

3.4 FT-IR studies

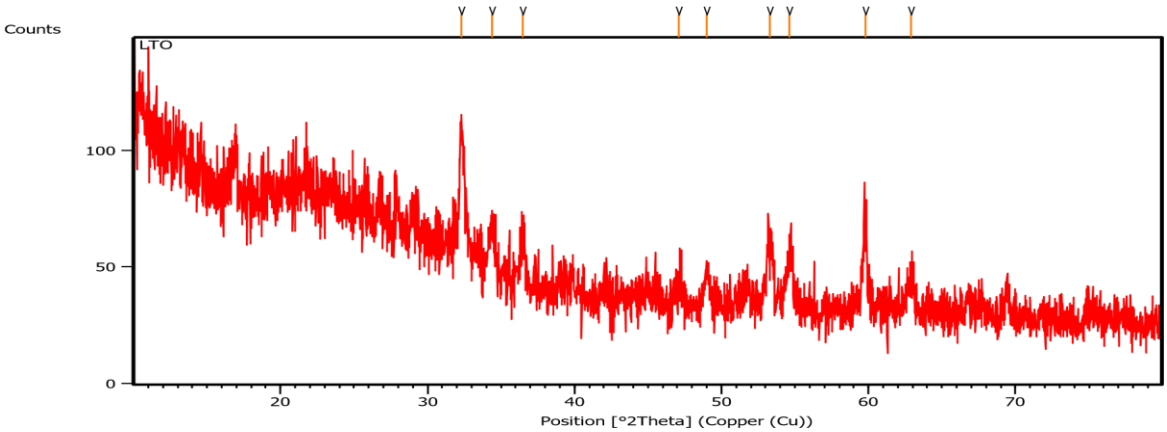
The stretching frequency involved in LTO Nanoparticles were detected by FT-IR Analysis. The IR spectra of the sample were recorded in the range 400-3800 cm^{-1}. The stretching of the peaks at 455.20$cm^{-1,}$ 424.34 cm^{-1} could be designed Te-O-Te Strechting.509.21 $cm^{-1,}$ 547.78 $cm^{-1,}$ 586.36 cm^{-1} could be designed LTO bending.



3.3. FT-IR data of TeO_2 nanoparticles

3.5 XRD Calculation

XRD diffraction with 2θ range of 20 - 70° of TeO_2 nanoparticles is using Cu ka 1 (1.540598A°) radiation. The 2θ formed are 32.30°, 34.37 47.10. Based on the Goniometer and diffractometer system Orthorhombic Tellurium based on JCPDS Card No.2106368 regarding hkl (110), (021), (200), (112). the result in that the TeO_2 exhibits a crystalline nature. The crystal phase of this type of Tellurium Orthorhombic with a lattice parameter of a-axis 5.79800 A° ,b-axis 12.2400 A° and c-axis 5.21400 A° respectively. In the high temperature influence the crystanality and structural phase of the material.



3.4 XRD spectrum of Tellurium dioxide nanoparticles synthesized using *Tephrosiapurpurea* leaf extract

The lattice value, **a = λ / 2 √k** **a = 5.6743 A°**

The calculated lattice value (a) for the synthesized nanoparticle was found to be **5.6743**

A°.

3.6 SCANNING ELECTRON MICROSCOPY (SEM)

The morphology and size distribution of the biosynthesized LTO Nanoparticles can be studied by the field emission scanning microscopy (FE-SEM). the average particle size ranges from 5 µm – 500 nm the figure a, b, c, d, shows the difference magnification.

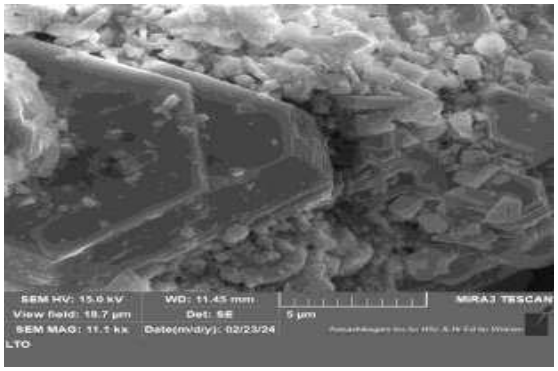


Fig (a)

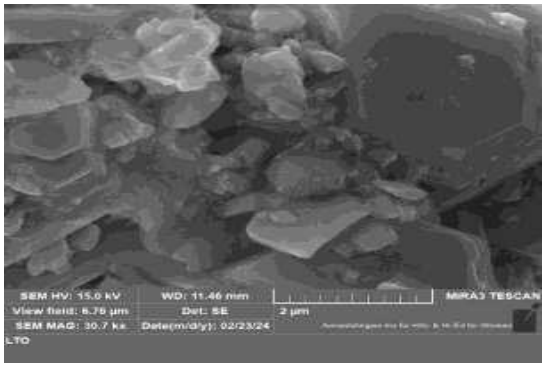


Fig (b)

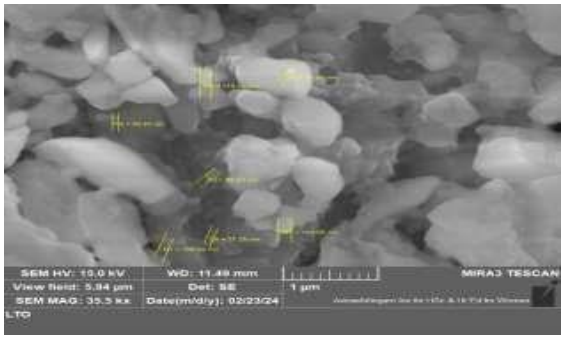


Fig (c)

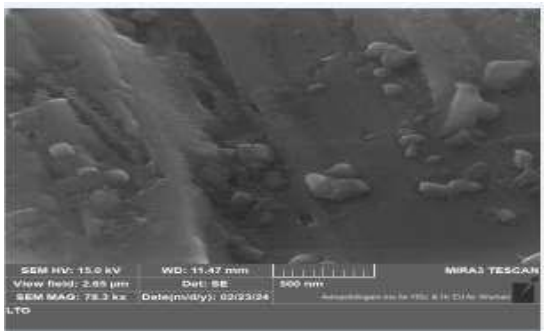
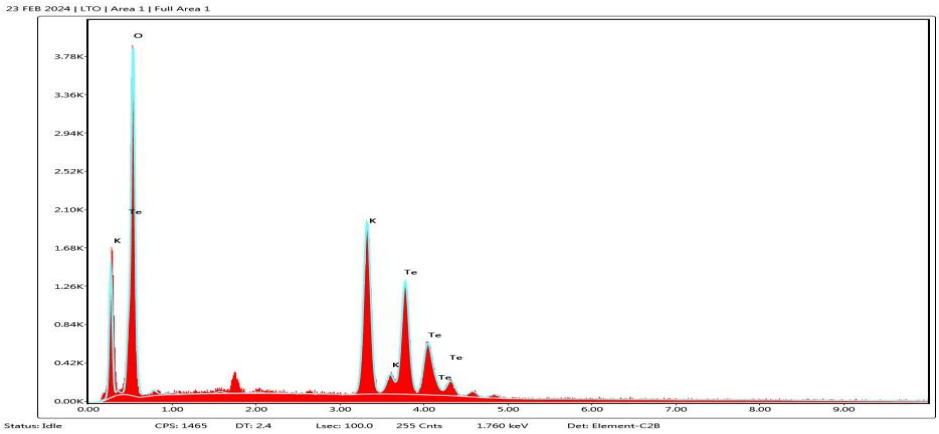


Fig (d)

Fig. 3.1 SEM images of TeNps in different magnification a) 5 µm b)2 µm, c) 1 d) 500 nm.

3.7 ENERGY DISPERSIVE X- RAY ANALYSIS (EDAX)

The composition of Te and K synthesized sample is given by energy dispersive X -ray Spectroscopy (EDAX) . The results clearly indicate the presence of the elements Te and K as the respective peaks are clearly visible.



3.5. EDAX spectrum of LTO

4.APPLICATIONS 4.1.ANTIOXIDANT ACTIVITY

4.1.1. Phosphomolybdenum assay

For the conduction of the phosphomolybdenum assay, the method of Prieto *et al*., was followed. Briefly, Tellurium nanoparticles were prepared at the concentrations of 200 to 600 µg/ml in ethanol. Then these Tellurium nanoparticles solution of different concentrations

(200 - 600 µg/ml) treated with 1 ml of reagent solution (0.6 M sulfuric acid, 28 mM sodium phosphate and 4 mM ammonium molybdate). The tubes were incubated at 95°C in a water bath for 90 min. The samples were cooled to room temperature and their absorbance was recorded at 695 nm against a reagent blank. Ascorbic acid was used as standard. Antioxidant capacity was estimated by using following equation:

$$Antioxidant\ activity\% = \frac{[(Absorbance\ control - Absorbance\ sample)]}{Absorbance\ control} \times 100$$

Table: 4.1. Phosphomolybdenum assay of Tellurium nanoparticles

Medium	**Concentration (µg/ml)**	**OD @ 695nm**
Ascorbic acid (standard)	600	0.574
Tellurium nanoparticles	200	0.249
	400	0.146
	600	0.098
Control / Blank	-	0.316

Table: 4.2. Percentage of inhibition

Concentration (µg/ml)	**Percentage of inhibition**
200	21.20%
400	53.79%
600	68.98%

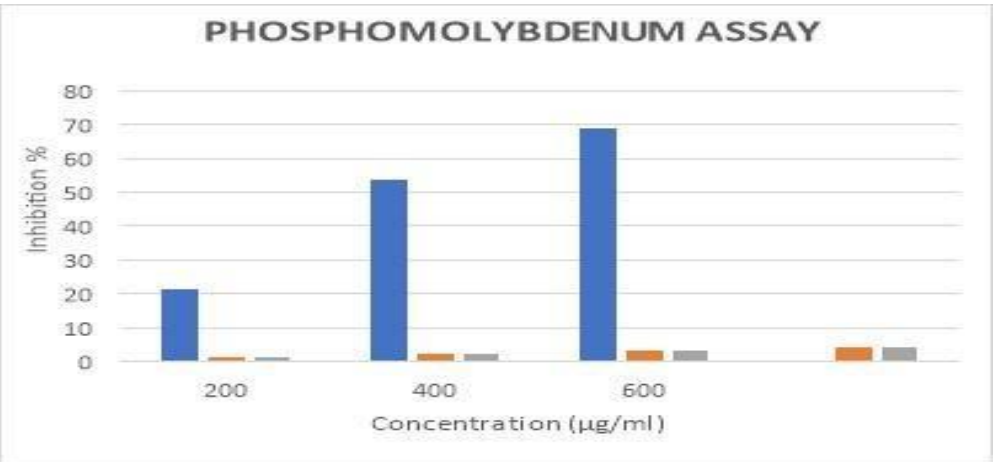


Fig. 4.1 Plot of Inhibition % vs Concentration for Phosphomolybdenum assay

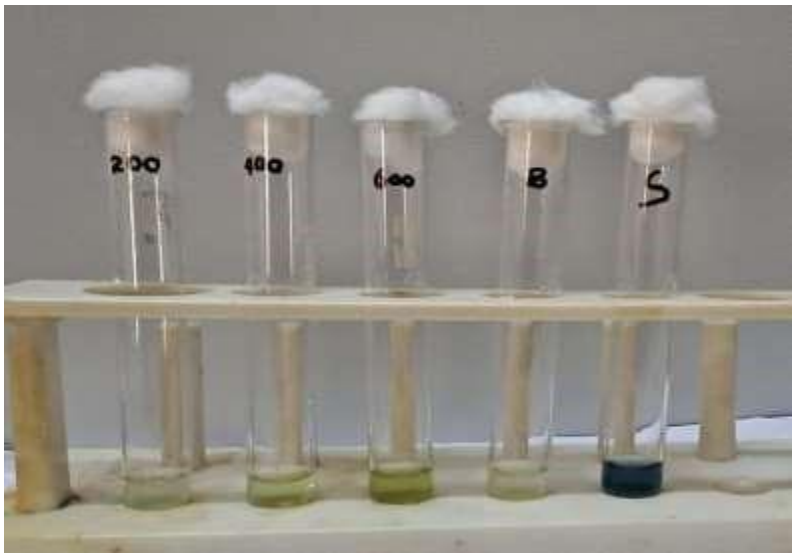


Fig. 4.1. Phosphomolybdenum assay of Tellurium nanoparticles

4.1.2. Hydrogen Peroxide Scavenging Assay:

The ability of the extract to scavenge hydrogen peroxide (H_2O_2) was determined according to the method of Ruch *et al.*, Briefly, Tellurium nanoparticles samples were prepared at the concentrations of 200 to 600 µg/ml in ethanol. Then these Tellurium nanoparticles solution of different concentrations (200 - 600 µg/ml) was transferred into the test tubes and their volume was made up to 0.4 ml with 50 mM phosphate buffer (pH 7.4) followed by the addition of 0.6 ml of H_2O_2 solution (2 mM). The absorbance was measured at 230 nm in the UV spectrophotometer against a blank after 10 minutes of incubation at 37°C. Ascorbic acid was used as standard. The ability of the extracts to scavenge the H_2O_2 was calculated using the following equation: [56]

$$Antioxidant\ activity\% = \frac{[(Absorbance\ control - Absorbance\ sample)]}{Absorbance\ control} \times 10$$

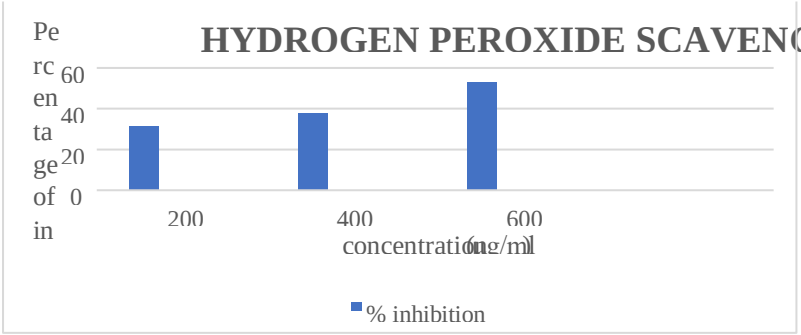
Table: 4.3. Hydrogen peroxide scavenging assay of Tellurium nanoparticles

Medium	**Concentration (µg/ml)**	**OD @ 230nm**
Ascorbic acid (standard)	600	0.355
Tellurium nanoparticles	200	0.226
	400	0.204
	600	0.155
Control / Blank	-	0.329

Table: 4.4. Percentage of inhibition

Concentration (µg/ml)	**Percentage of inhibition**
200	31.31 %
400	37.99 %
600	52.89 %

Fig. 4.2 Plot of Inhibition % vs Concentration for hydrogen peroxide assay



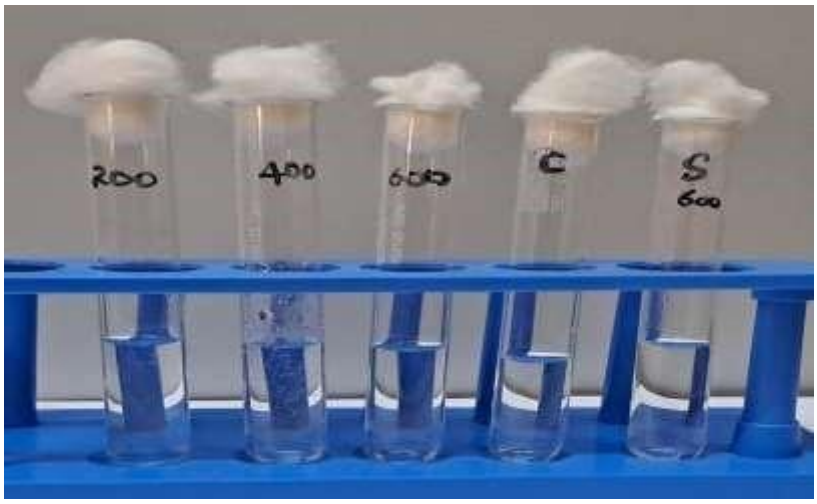


Fig. 4.2. Hydrogen peroxide scavenging assay of Tellurium nanoparticles

4.2. ANTIBACTERIAL ACTIVITY

4.2.1. Microbial strains used

Four bacterial strains namely *Escherichia coli, Staphylococcus aureus, Vibrio cholerae* and *Pseudomonas aeruginosa*were used for antimicrobial activity.

4.2.2. Inoculum preparation for bacteria

Nutrient broth was prepared and sterilized in an autoclave at 15 lbs pressure for 15 minutes. All the four bacterial strains were individually inoculated in the sterilized marine broth and incubated at 37℃ for 24 hours. The 24 hours old bacterial broth cultures *Escherichia coli, Staphylococcus aureus, Vibrio cholerae* and *Pseudomonas aeruginosa* were inoculated in the petri dishes using a sterile cotton swab.

4.2.3. Antibacterial activity assay

The antibacterial activity assay was carried out by disc diffusion method (Kirby& Bauer, 1966). Mueller hint on agar was prepared and poured in sterile petri plates. Bacterial culture was inoculated on the surface of Mueller hint on agar plates. The inoculated plates were allowed to dry for five minutes. Briefly, Tellurium nanoparticles were prepared at the concentration of 1000 µg/ml in ethanol. Sterilized paper disc prepared from what mann No.1 paper disc with 6mm diameter were loaded with Tellurium nanoparticles concentration of 1000 µg/ml and placed on the surface of inoculated petri plates along with control- streptomycin using sterile technique. The plate was incubated at 37ºC for 18-24 hours. The plate was examined for inhibitory zone, the inhibition zones were measured with the outer side of the disc to inner side of the inhibition zone and the zone of inhibition was measured in mm.

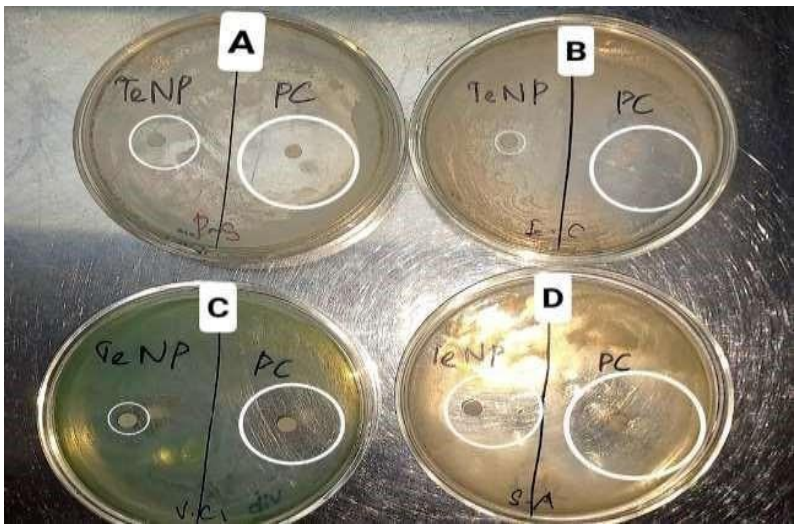


Fig. 4.3. Antibacterial activity on A) *Pseudomonas aeruginosa* B) *Escherichia coli* C) *Vibrio cholerae* D) *Staphylococcus aureus*

Table: 4.5 Study of Antibacterial activity

Bacterial strains	**Zone of inhibition (mm)**	
	Nanoparticles	**Positive – controlStreptomycin (PC)**
Pseudomonas aeruginosa	11 mm	20 mm
Escherichia coli	2 mm	18 mm
Vibrio cholerae	8 mm	16 mm

Staphylococcus aureus	17 mm	23 mm

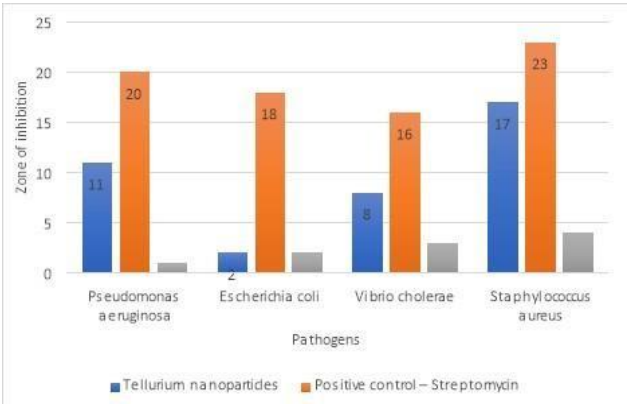


Fig. 4.3. Antibacterial activity of Tellurium Nanoparticles

4.3. Anticorrosion activity

The anticorrosion activity of synthesized TeNps is high in basic medium. On the other hand, in acidic and neutral conditions, the corrosion rate is high in the presence of TeNps. **Table 4.6. Anti corrosion activity for nano particles**

Medium	**Weight of inhibitor (g)**	**Weight of plate, g**		**Weight loss g**	**% of Corrosion rate g/*cm* h**
		Before immersion	**After immersion**		
Acidic buffer	-	0.7989	0.7986	0.0003	0.04
	0.1	0.7131	0.7123	0.0008	0.11
Basic buffer	-	0.8664	0.8653	0.0011	1.27
	0.1	0.6724	0.6679	0.0045	0.67
Sea water	-	0.7743	0.7735	0.0008	0.10
	0.1	0.6182	0.6165	0.0017	0.27

5. CONCLUSION

Tellurium dioxide nano particles were synthesized by green method using leaf extract of *Tephrosiapurpurea*. The prepared TeO_2 nanoparticles were characterized using several techniques such as UV-Visible, FT-IR, SEM, EDAX and XRD. And the anti-corrosion, antimicrobial and anti-oxidant activities were studied.

- The UV-visible absorption peak occurs at 356 nm. This confirms that the synthesized nanoparticles are TeO_2.
- The band gap energy is 3.4827 eV and hence it behaves as semi-conductor.
- The FT-IR studies showed an absorption spectrum of Te-O-Te at 871 and O-Te-O peak at 648.08cm^{-1} ,601.79^{-1} 555.50 $^{-1}$ and 470.63 $^{-1}$ which indicates the formation of Tellurium dioxide nanoparticles.
- XRD behaviour exhibits the size of Tellurium dioxide nanoparticles. The crystallite size was found to be 17 nm for TeO2 nanoparticles. The presence of several sharp peaks indicates the random orientation of crystalline nature, confirming face centered cubic structure of TeO2 nanoparticles.
- The surface morphology of TeO2 nanoparticles was characterized by SEM. The SEM images of different magnification show the flakes shape structure.
- The Energy Dispersive X-ray analysis showed the presence and distrains of Titanium and Oxygen elements in the synthesized TeO2 nanoparticles.
- The Anti-corrosion activity on mild steel was carriedout, in basic medium.
- The Antioxidant study shows the fluctuation in percentage of antioxidant activity as the concentration varies. The maximum percentage of antioxidant activity of about 0.249% was seen for the 200 µg/mL.
- The Antibacterial activity explains clearly that the prepared sample has zone inhibition with 4 pathogens namelyA) *Pseudomonas aeruginosa* B) *Escherichia coli* C) *Vibrio cholerae*D) *Staphylococcus*

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