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Original Resarch Article

Biogenic synthesis, characterization and pharmacological study of silver nanoparticles using an extract of *Xanthium strumarium* seeds

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ABSTRACT

A novel approach for utilization of seed waste is attempted in the present investigation. As the fabrication of nanomaterial using physiochemical methods have hazardous and toxic impacts on the environment, there is a vital demand for an innovative and well organized, Eco-friendly, sustainable and greener synthetic protocol for their synthesis by applying safer, renewable and inexpensive materials. This research study discuses the rapid photosensitized biosynthesis of silver nanoparticles using aqeous extract of seeds of Xanthium strumarium. The reaction was carried out in ambient sunlight. As the pathogenic organisms are getting evolved day by day due to mutation and gaining antibiotic resistance, an important industrial sector of nanoscience deals with the preparation and plays a decisive role in study of nanoparticles. Further investigation of the formation of nanoparticle was monitored periodically by UV-Vis spectroscopy. Fourier-transform infrared (FT-IR), X-ray diffraction (XRD), energy dispersive X-ray spectroscopy (EDS), selected area electron diffraction (SAED) results confirmed the crystalline nature while transmission electron microscopy (TEM) analysis revealed the shape of polydispersed nanoparticles were predominantly spherical. The antioxidant properties were tested by free radical scavenging ability on 2,2-diphenyl-2-picrylhydrazyl (DPPH) method .The antimicrobial property of the synthesized NPs tested against pathogens such as *E.coli*, *S.typhi*, *Pseudomonas* and *S.aureus* by disc diffusion method. The EDX reveals a strong signal at 3 keV. Assembling of nanoparticles and to study the effect of rate of bioreduction of Ag^+ ions this conventional approach appears to be very cost effective.

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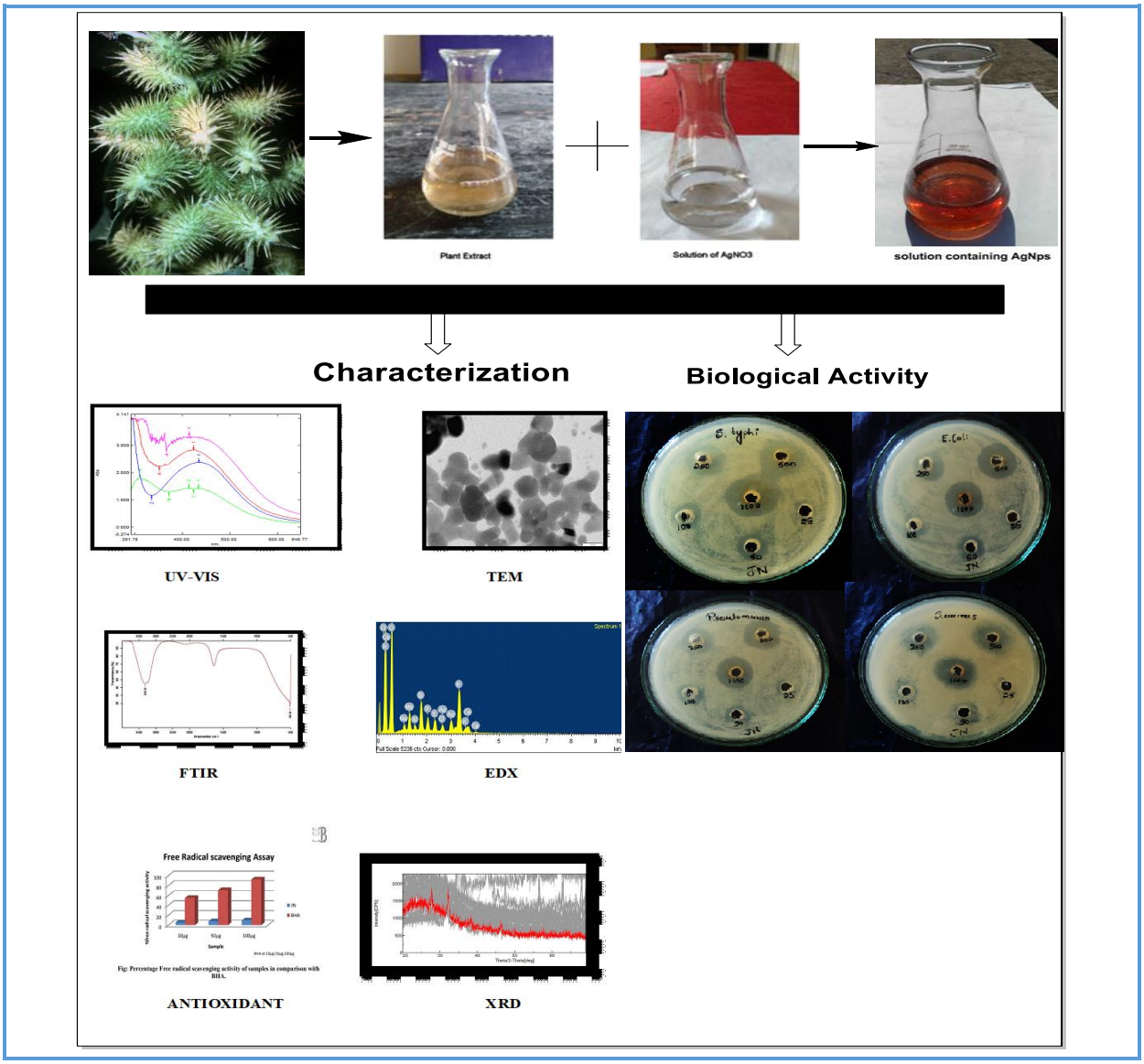
Graphical Abstract



Introduction

From the ancient Babylonians and Greek era, silver is widely used as an antimicrobial agent. Over the past decade rapid progress in the field of nanoscience has been increasingly driven attention of scientific community. Nanoparticles are ubiquitously applied in various branches of science owing to their tunable size and shape dependent properties [1]. Nanotechnology is a versatile field that exhibit new and improved properties as compared to bulk counterparts due to change in characteristics such as shape, size and size distribution. Nanotechnology has applications in many fields like wastewater treatment, dye degradation, agriculture, biosensing, medical applications, fabric and textile industry, drug delivery, cosmetics and food industry, etc. [2, 3]. Nanoscience is one of the vibrant growing fields ever known and continues to rigorously reach its branches into various modern technologies

such as hydrogen storage, photo catalysis, green energy devices, sensors, biomedical implants and photovoltaic. There are so many technological and environmental challenges in the areas of catalysis, solar conversion and medicine which can be solved by nanomaterials [4]. It is getting developed at several levels, materials, devices and systems. At present, the most advanced is the nanomaterial level in both scientific pieces of knowledge and commercial application. Due to high chemical stability, silver nanoparticles are reported to possess super capacitance properties, electron transfer capabilities and electro catalysis. It is used in biomedicines and photocatalytic, anti-inflammatory, antibacterial activities, antifungal, antiviral, antiangiogenesis and antiplatelet activity.

Nanoparticles are even less than a few 100 nm and they are the emerging area of nano science and nanotechnology. Nanotechnology matter influence with at least one dimension size 1-100 nm. This provides ability to engineer material by controlling their size. It is well established fact that, their properties can be altered by changing size at nanometer scale [5].

Synthesis of nanoparticles is possible by means of chemical, physical and biological techniques. But there are drawbacks such as long time reactions, high temperature, low yields, difficult and hazardous reaction conditions and expensive capping agents and utilization of organic solvents leading to environmental pollution. Therefore, in order to decrease environmentally toxic chemical waste [6, 7]. Thus, researchers are inclined towards the biological route of nanoparticles synthesis from the various plant parts as these procedures are simple, sustainable, ecofriendly and cost-effective [8]. Adopting the twelve basic principles of green chemistry, biological, and biometric approach is an easy and eco-friendly way to go green. Different plant extracts, such as Chenopodium murale leaf extract, have been successfully used in the biosynthesis of nano silver crystals. [9] Aloe vera, Carica papaya, Azadirachta indica (Neem), cinnamomum camphora, Geranium leaf [10]. It is already very well known that silver nanoparticles are abundantly used for their unique properties in catalysis, chemical sensing, biosensing, photonics, electronics, and pharmaceuticals Silver has been recognized as having a hindering effect on many bacterial stain and microorganisms commonly present in the medical and industrial procedures. Silver nanoparticles have a great dormant for use in biological including antimicrobial activity. The antimicrobial capability of silver nanoparticles allows them to be suitably employed in numerous household products such as textiles, food storage containers, home appliances and medical devices. Silver is an effective antimicrobial agent that are harmLess. The most important application of silver and silver nanoparticles is in the medical industry such as topical ointments to prevent infection against burn and open wounds [10–12]. Silver nanoparticles play a profound role in the field of biology and medicine due to their attractive physicochemical properties. Silver products have been used for centuries as is known to have strong inhibitory and bactericidal effects, as well as a broad spectrum of antimicrobial activities, to prevent and treat various diseases, most notably infections. Green chemistry provokes the application of renewable material to fabricate assorted nanoparticles with unique properties. Deploying renewable resources is an ecologically beneficial undertaking for formation of nanoparticles via greener approaches has gained great attention in recent years [13].

The purpose of this study is to synthesize and characterize the plant-mediated silver nanoparticles using *Xanthium Strumarium*

seeds. As the pathogenic organisms are getting evolved day by day due to mutation and gaining antibiotic resistance, our motto is to deals with the an important industrial sector of nanoscience and synthesize facile, safe nanoparticles which plays a decisive decisive role in study of nanoparticles.

Experimental

Materials and methods

Collection of plant

The plant was collected from pundlik baba Nagar, Amravati, Maharashtra. Seeds were separated and washed several times with deionized water to remove dirt particles and dried for 2 to 5 days at room temperature.

Preparation of plant extract

The dried seeds of *Xanthium strumarium* were crushed into a fine powder. 10 gm powder from this powder is mixed with 100 mL of deionized water. It was kept on a magnetic stirrer at 30 °C for 3 to 4 hours under vigorous stirring. The aqueous extract was then separated by whatsman filter paper no.1.

Biosynthesis of silver nanoparticles

Seeds extract of *Xanthium strumarium* (5 mL) was taken in a conical flask. Then this aqueous extract solution was added to freshly prepared 45 mL of $AgNO_3$ 1 mM solution, under exposure of sunlight.

Characterization of silver nanoparticles

For the characterization of the synthesized silver nanoparticles confirmed by monitoring the reaction at regular intervals and absorption peak was scanned by UV-Vis spectra. This analysis was done by using shimadzu UV-1800 spectrophotometer at room temperature between the range 190-800 nm. The crystalline nature was determined by XRD. The characterization of functional group present in the silver nanoparticles was investigated by FTIR spectrophotometer. (FT-IR analysis was done with KBr pellets and recorded in the range of 500-3500 cm^{-1} using Perkin Elmer Spectrum 100). The shape and size of resultant particles were elucidated with the help of TEM on FEI USA Technai G2 analysis.

Antimicrobial analysis

Ag-NPs synthesized from Xanthium strumarium seeds extract were tested for antibacterial activity against pathogens like Escherichia coli, Staphylococcus aureus, S.typhi and pseudomonas using agar diffusion method. Solvent used in the process was DMSO and the standard antibiotic used was Ciprofloxacin. The sample was screened at different concentrations of 1000, 500, 250, 100, 50, 25 µg agar method. Media used was peptone -10 g and yeast extract 5 g, agar 20 g in 1000 mL of distilled water. Initially the stock cultures of bacteria were revived by inoculating in broth media and grown at 37 °C for 18 h old cultures (100 µl, 10^{-4} cfu) and spread evenly on the plate. The wells were loaded with compound at various volumes after 20 min. The diameter of the inhibition zone was measured after 24 h of incubation at 37 °C.

Determination of antioxidant activity

Radical scavenging activity using DPPH method

In a series of test tubes, different concentrations of samples in Dimethyl sulfoxide (DMSO) were taken. Methanol was added to get the volume up to 500 L. These tubes were filled with 5 mL of a 0.1 mM methanolic solution of 1,1-diphenyl-2-picryl hydrazyl (DPPH; Sigma-Aldrich, Bangalore) and vigorously shaken. A control that is devoid of the test compound but

contains an equal amount of methanol was maintained. The tubes were left at room temperature for 20 minutes. The samples' absorbance was estimated at 517 nm. The reference norm was butylated hydroxy anisole (BHA). The following formula was used to quantify free radical scavenging activity:

% radical scavenging activity=100×(control OD-sample OD/control OD)

Result and Discussion

UV–Vis spectroscopy analysis

The formation of silver nanoparticles was initially observed visually due to change in color. After 24 h of addition of 1 mM silver nitrate solution to seeds extract of *Xanthium strumarium,* there is a rapid visible change in color from yellow to dark brown which designates the formation of silver nanoparticles. The absorption peak appeared at 422, 433, 439 and 446 nm at different time interval (Figure 1). Intensity of absorbance increased as reaction proceed. The peak appeared at 422 nm indicates the formation of AgNPs [14]. and the peak appeared at 446 nm confirmed stability of synthesized AgNPs after 24 h as shown in graph. This reveals that the absorbance of AgNPs gradually increases with time (Figure 2).

(a) (b) (c) (d)

Figure 1. Optical diagram of gradual color change in AgNPs at different time interval. a) after 5 min; b) after 30 min; c) after 1 hour; d) after 24 hours

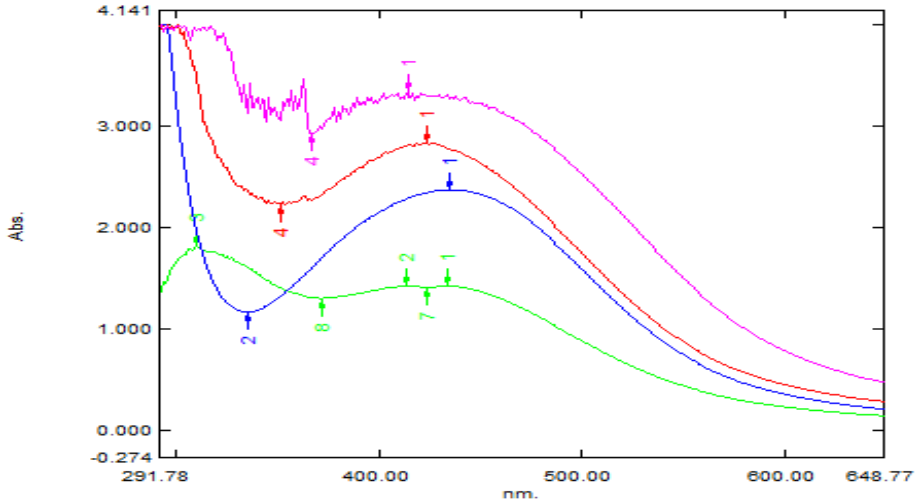


Figure 2. UV-Vis spectra of seed extract and AgNPs at different time intervals

XRD

The analysis was carried out at 2Θ value. The broad peak of silver nanoparticles appeared at 27.24°, 32.12°, 36.12°, 46.32°, 52.4°, 58° and 63.2° respectively and the broad peak of silver nanoparticles of seeds extract of *Xanthium strumarium* appeared at 27.24°, 32.12° , 36.12° ,46.32 (Figure 3). Some studies reported five intense peaks of AgNPs at 27.45°, 31.99° 45.96°, 67.24° and 76.46° [15].

The X-ray diffraction result clearly shows that silver nanoparticles formed by the reduction of Ag^+ ions by the aqueous extract of seeds *Xanthium strumarium* are crystalline in nature. The interplanar spacings morphology was determined using following Bragg's equation. $n\lambda=2d \sin\Theta$. It is in agreement with the standard diffraction spectrum (JCPDS Card No.87-0717) [16].

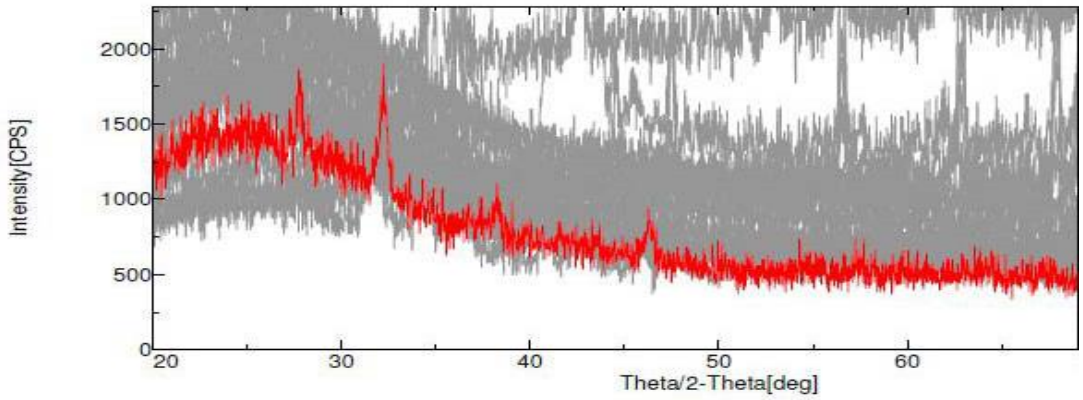
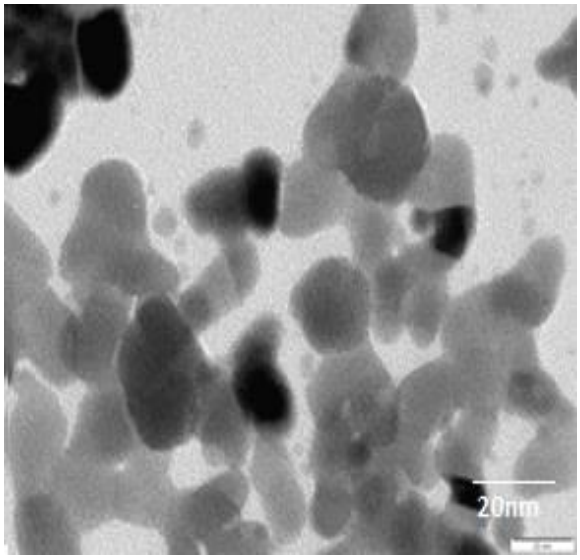
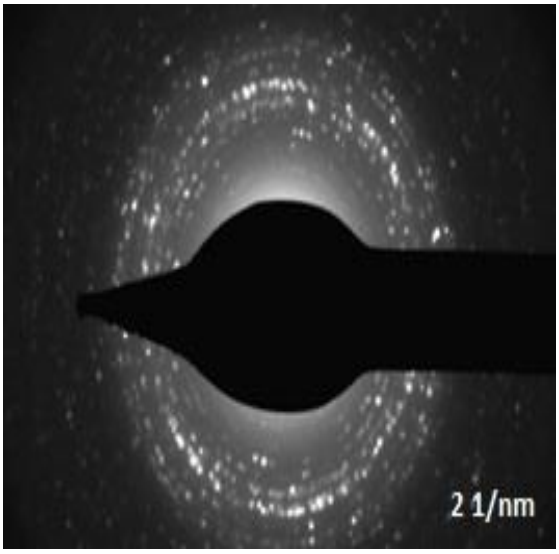


Figure 3. XRD spectrum of silver nanoparticles from seeds extracts of Xanthium strumarium

TEM (Transmission Electron Microscopy)





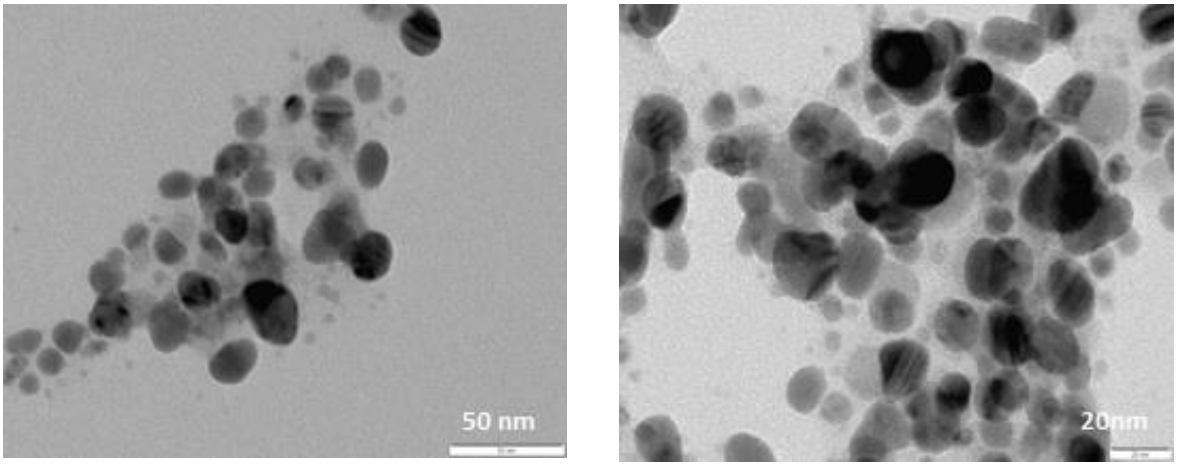


Figure 4. TEM spectrum of silver nanoparticles

The shape and size of synthesized silver nanoparticles were confirmed by TEM analysis. The TEM micrographs suggested that the synthesized silver nanoparticles were spherical and range between 20 to 50 nm [17]. It is used to obtain the measurement of a colloidal particle, its distribution and morphology (Figure 4).

Fourier-transform infrared spectroscopy (FT- IR) analysis

FT-IR was used to validate the findings. The interaction of AgNPs with aqueous seeds extract of Xanthium Strumarium was studied using FT-IR spectra. The results of this study's FT-IR analysis reveal various bond stretches at various peaks. The –N-H stretch causes the strong peak at 3418 cm^{-1}, while the band about 2071 cm^{-1} is caused by a carbon triple bond (Figure 5). The sharp peak at 1651 cm^{-1}, on the other hand, corresponds to amide. It occurs as a result of protein carbonyl stretch, suggesting the presence of surface capping organisms that are primarily responsible for stabilisation. The N-H stretching in the free amino groups of silver nanoparticles has been linked to broad asymmetric at 2100 cm^{-1} [18]. These functional groups play a critical role in the development of silver nanoparticles.

Energy dispersive X-ray (EDX) analysis

The energy dispersive X-ray analysis (EDX) of silver nanoparticles synthesized using seeds of Xanthium Strumarium seeds reveals signal in the silver region. The specific release energy for Ag has been identified, and these detection lines interact with high peaks in the spectrum. Therefore, confirming that Ag nanoparticles has been formed. Ag nanoparticles shows typical optical absorption peak at 3 keV due to surface Plasmon resonance [19]. There were also other elemental signals which may be due to thin film made on the glass slide (Figure 6).

Antimicrobial activity

Ag nanoparticles were tested for antimicrobial activity against E. coli, S. aureus, S.typhi, and Pseudomonas. It is interesting to note that the growth and reproduction of bacterial cell is inhibited as soon as it comes in contact with silver nanoparticles (Figure 7). Standard antibiotic used was ciprofloxacin. Each organism is tested at different concentration. The result are presented as diameter of inhibition zones in mm (Table 1). By considering the antimicrobial activity of silver nanoparticles it is used in treatment of cancer [20]. The pathogens S.typhi and E.coli were

particularly more susceptible to the silver nanoparticles that had been synthesized. Nanoparticles are able to damage and destroy the membrane and by penetrating into the cytoplasmic membrane show an antimicrobial activity. In this sense, the results are more consistent with Seifi Mansour and colleagues [21].

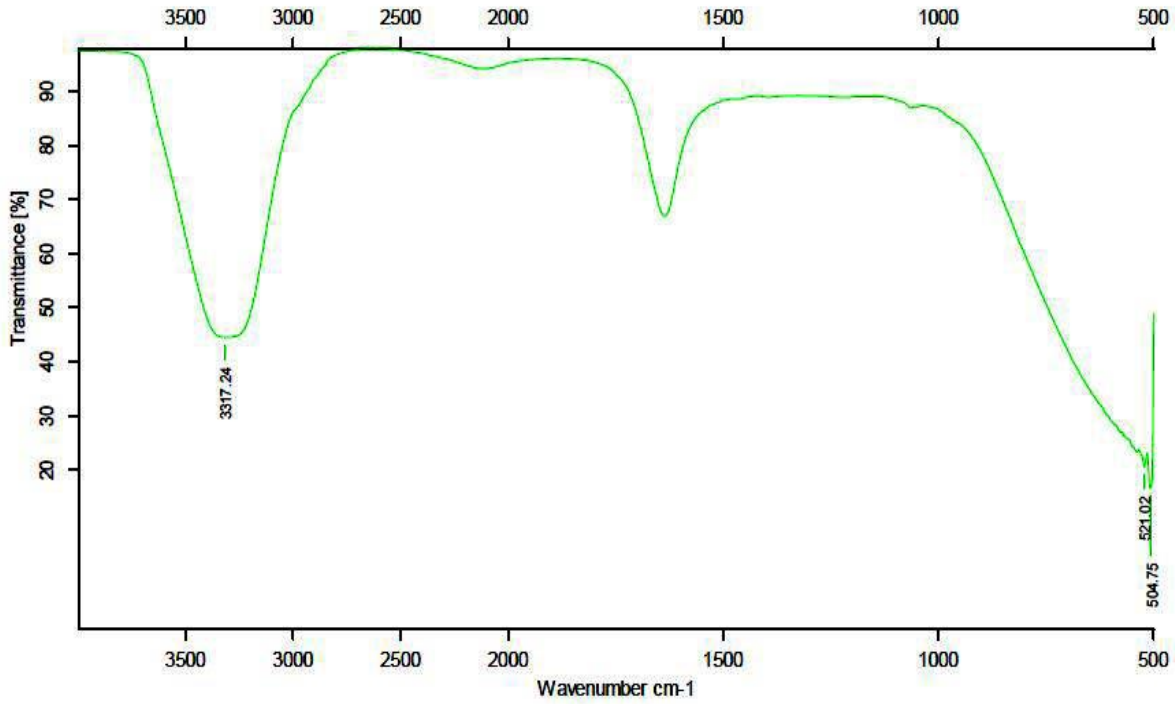


Figure 5. FT-IR spectra of synthesized silver nanoparticles from seeds extract of Xanthium strumarium

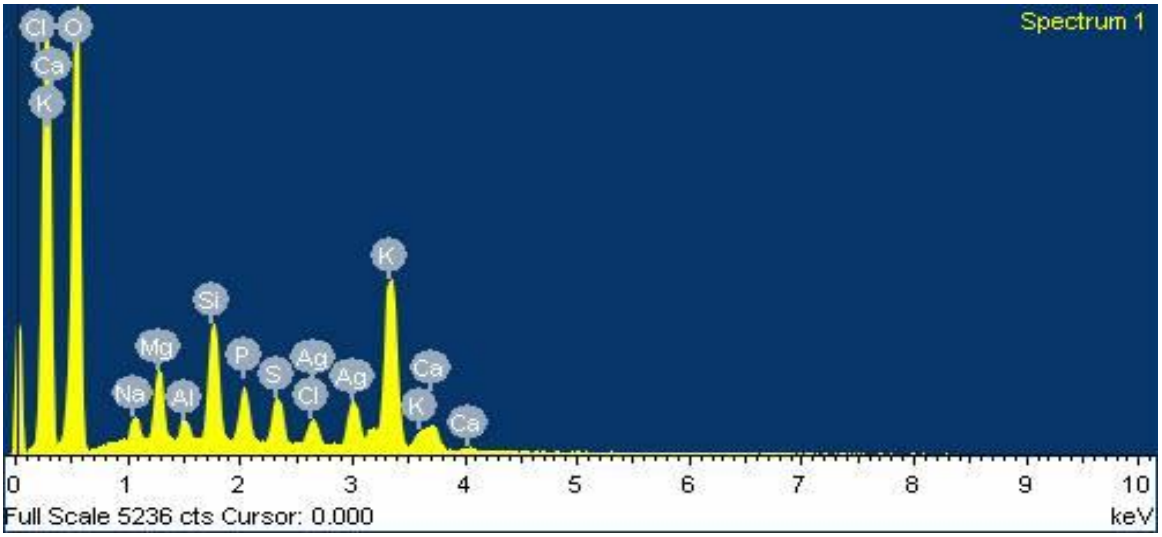


Figure 6. EDX spectrum of AgNPs

Table 1. Diameter of inhibition zone in mm at different concentration with reference to standard antibiotic

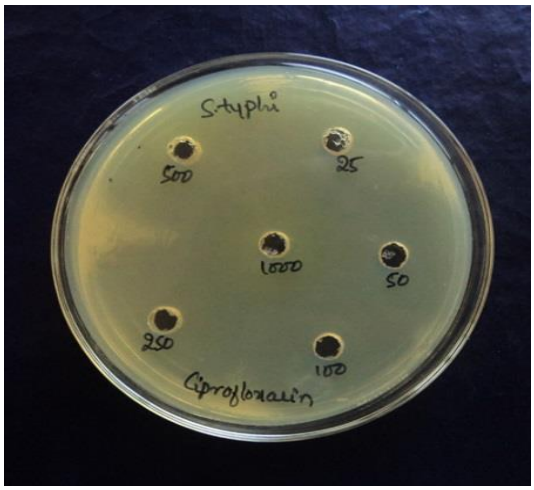
Anti-bacterial analysis
Sample:JN

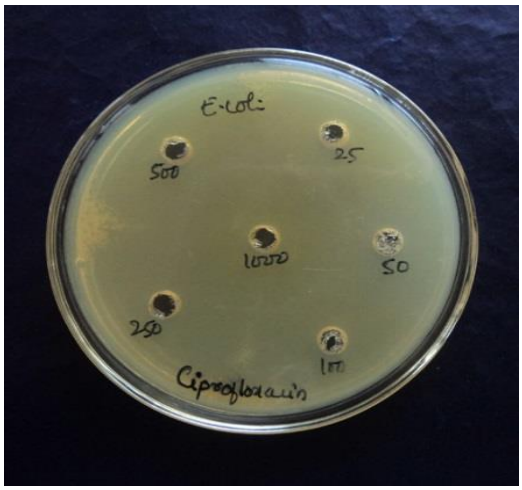
Organism	1000	500	250	100	50	25	MIC in µg
Pseudomonas	16	16	11	9	6	0	50
E.coli	17	18	12	8	9	0	50
S.typhi	21	18	14	10	9	0	50
S.aureus	16	14	14	8	5	0	50

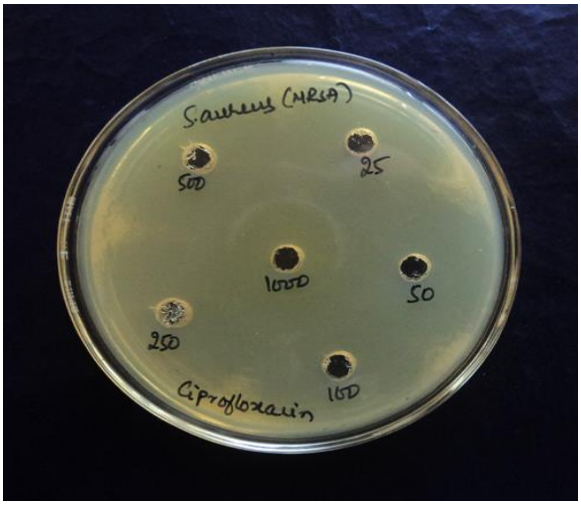
*Zones could not be measured due to margining

Standard antibiotic

Organism	25 µg	50 µg	100 µg	200 µg	400 µg	800 µg	MIC in µg
Pseudomonas	19	22	25	28	30	33	25
E.coli	18	20	23	26	28	31	25
S.typhi	2	13	16	21	25	27	25
S.aureus	13	18	21	25	27	34	25







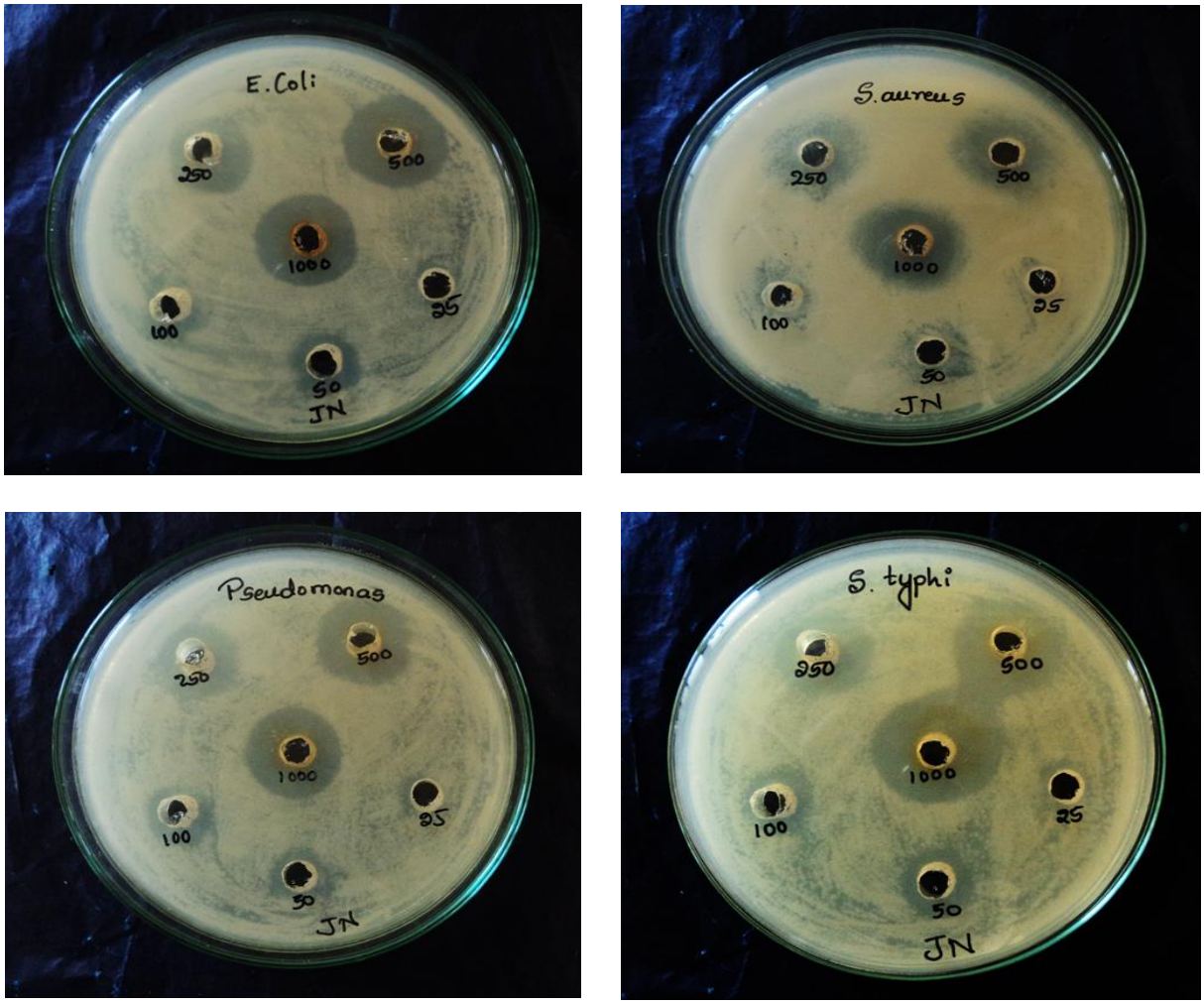


Figure 7. Antimicrobial activity of silver Nanoparticles against different bacteria at different concentration

Anti-oxidant activity

The DPPH free radical scavenging activity of AgNPs was used to assess their antioxidant potential. The AgNPs demonstrated dose dependent antioxidant activity that is as concentration increased, antioxidant activity also increased. The value of BHA and silver nanoparticles (i.e sample JN) at different concentrations are presented in (Table 2) and graphically it is represented in (Figure 8). The recorded value for the lowest concentration 10 µg is 54.27±4.7 and this value increased to 70.10±7.2 at concentration 50 µg. Similarly, at higher concentration 100 µg value noted was 91.82±8.9, respectively [22].

Table 2. Percentage Free radical scavenging activity

Concentration	%Free radical scavenging	
	JN	BHA
10 µg	4.768583	54.27
50 µg	7.223001	70.10
100 µg	8.906031	91.82

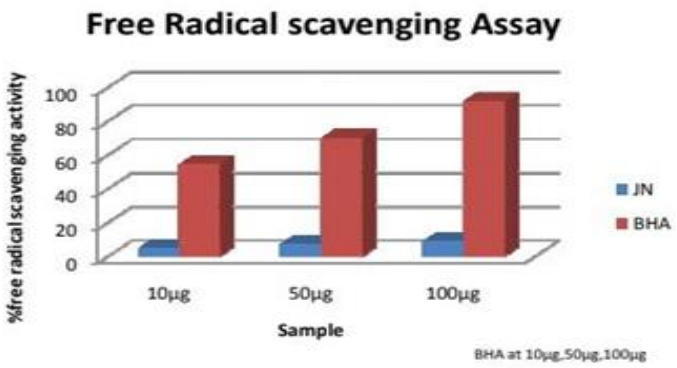


Figure 8. Percentage Free radical scavenging activity of samples in comparison with BHA

Conclusions

In the present study, we have demonstrated that the use of a natural, biological reducing agent and Xanthium strumarium seeds extracts can produce metal nanostructures, through efficient green nanochemistry methodology, avoiding the presence of toxic solvents. This is an excellent example of making most from waste. The synthesized Ag-NPs were confirmed by various analysis such as UV-Vis, XRD, EDX, FT-IR also TEM and SAED analysis revealed that shape of AgNPs are spherical and ranged between 20 nm to 50 nm. They also showed dose dependent antioxidant and good antimicrobial activity against E.coli, S.typhi, Pseudomonas and S.aureus. This method is potentially exciting for the large-scale synthesis of nanoparticles specially if they are intended for invasive application in medicine.

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Disclosure Statement

No potential conflict of interest was reported by the authors.

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