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Research Article

Green Synthesis of Silver Nanoparticles Using Water Hyacinth Leaf Extract for Colorimetric Detection of Heavy Metal Ions

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Abstract

The green synthesis of silver nanoparticles have attracted many researchers due to their wide range of applications. The objective of this study is to synthesize silver nanoparticles using water hyacinth extract for the detection of metal ions in aquatic solutions. In the present study, the silver nanoparticles synthesis employing the leaf extract of water hyacinth as the capping and reducing agent has been reported. The particles showed absorption maxima at 406 nm establishing the formation of silver nanoparticles. The particles were characterized by FTIR, XRD, SEM-EDX, TEM and Zeta Potential. The polyphenols present in the leaf extract are accountable for reducing and the capping activity which was revealed in the FTIR spectra. XRD revealed the crystalline nature of the nanoparticles. The morphology, size and shape of the silver nanoparticles were investigated with the help of electron microscopy techniques. The silver nanoparticles are observed to be spherically shaped with an average diameter of 10.78 ±4.61 nm. EDX spectra established the presence of elemental silver in the nanoparticles. A zeta potential of -31.7 mV was recorded indicating that the silver nanoparticles are stable. These biosynthesized silver nanoparticles were employed to detect metal ions in aqueous solutions and two metal ions (Hg^{2+} and Fe^{3+}) at 1000 micro molar concentration were detected successfully. Thus, the results of the study indicate that the silver nanoparticles synthesized from water hyacinth leaf extract have potential application in the detection of metal ions.

Keywords

Silver Nanoparticles, Water Hyacinth, Green Synthesis, Metal Detection, Heavy Metals

1. Introduction

Materials with the dimensions in the range of 1 to 100 nm can be termed as nanoparticles. Of late, the use of nanomaterials has been on the rise due to their applicability in diverse sectors including catalysis [1-3], optics [4, 5], sensors [6, 7], energy [8], biomedical science [9-12], agriculture [13], environmental applications [14-16]. The wide range of applications of nanoparticles can be attributed to the distinct characteristics when compared to their bulk counter parts. Silver nanoparticles [AgNPs] have been receiving noteworthy attention recently, because they show strong absorption in the visible region, chemical inertness, stable dispersions and biological compatibility leading to potential applications in

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catalysis [17], biomedical [18, 19], food science [20] environmental applications [21, 22] and so on.

Physical, chemical or biological methods can be used to synthesize silver nanoparticles. Due to their advantages over the other two methods biological synthesis, often dubbed as green synthesis has garnered attention as the promising method of synthesis. The process of green synthesis is non-toxic [23], pollution-free [24], eco-friendly, less expensive [25], and more sustainable [26]. Green synthesis makes use of biological and eco-friendly materials as the reducing and capping agents to synthesize silver nanoparticles. Micro-organisms [bacteria, fungi] [27, 28] or different plant parts such as leaves [29], fruits [30], fruit peels [31], flowers [32], seeds [33] have been reportedly used for the synthesis of AgNPs via a green route of synthesis. Plant extract mediated synthesis has garnered attention as it seems to be faster than microorganisms [34]. The polyphenolic compounds and proteins present in the plant extract reduce the silver ions by acting as reducing agents [35]. Some of these bioactive compounds serve as capping and stabilizing agents in the process of synthesis. AgNPs have been successfully synthesized from extracts of different plants by researchers around the world [36, 37] and are shown in Table 1.

Table 1. *Green synthesis of silver nanoparticles from different plant parts by researchers for the detection of metal ions.*

Plant part used	*Plant*	Heavy metals detected	References
Fruits	Water apple *(Syzygium aqueum)*	Hg^{2+}	[63]
	Watermelon *(Citrullus lanatus)*	$Hg^{2+,}$ Cu^{2+}	[73]
	Kokum fruit	Hg^{2+}	[74]
	Carica papaya	Hg^{2+}, Fe^{3+}	[75]
Flower	*Acacia nilotica*	Hg^{2+}	[62]
	Moringa oleifera	Cu^{4+}	[76]
	Anchusa azurea	Hg^{2+}	[77]
Roots	*Bistorta amplexicaulis*	$Hg^{2+,}$ Pb^{2+}	[78]
	Panax ginseng	Hg^{2+}	[79]
	Soymida febrifuga	Hg^{2+}	[80]
Peel	*Allium cepa L*	Hg^{2+}	[81]
	Sapota *(Manilkara zapota L.)*	Hg^{2+}, Co^{2+}	[82]
Leaves	*Cordia myxa*	Hg^{2+}, Fe^{3+}	[51]
	Trigonella foenum-graecum L.	Hg^{2+}, Fe^{3+}	[65]
	Sonchus arvensis L	Hg^{2+}, Fe^{3+}	[66]
	Dahlia pinnata	Hg^{2+}	[70]
	Artemisia vulgaris	Hg^{2+}	[83]
	Yemeni Mistletoe *(Phragmanthera austroarabica)*	Cr (VI)	[84]
	Acacia chundra	Hg^{2+}	[85]
	Acalypha hispida	Mn^{2+}	[86]
	Lantana camara	Hg^{2+}, Cu^{2+}, Pb^{2+}, Mn^{2+}	[87]

The increasing prevalence of heavy metals in all the components of environment is a critical issue due to multiple harmful impacts on the living systems. Studies have reported that heavy metals are potential environmental pollutants even in trace amounts and cause problems to humans, animals, plants and aquatic life [38, 39]. Therefore, detection and regular monitoring of these metal ions in aqueous conditions with high sensitivity is important. Numerous techniques, including Inductively Coupled Plasma Mass Spectrometry [ICP-MS], Inductively Coupled Plasma Optical Emission Spectroscopy [ICP-OES], Atomic Fluorescence Spectroscopy [AFS], High Performance Liquid Chroma-

tography [HPLC], Flame Atomic Absorption Spectroscopy [FAAS], etc., can be used to determine specific water pollutants. However, most of these techniques demand lengthy sample preparation, expensive, high-tech equipment, and skilled employees to perform the analysis. All of these shortcomings have driven recent research toward novel sensors built on easy, rapid and affordable processes [40]. Recently, colorimetric sensors with the quick optical reaction have drawn a lot of attention as potential alternatives for such easy, affordable, and effective analytical techniques [41] that do not need complicated, expensive or large equipment. The working principle of AgNPs based upon the colorimetric detection is based on the change of absorbance when in contact with specific analytes. An efficient optical sensor may be created by using the gradual change in optical quality as an indication of pollutant level [40]. Many colorimetric based detection methods using silver nanoparticles synthesized from biological reducing agents have been studied and reported in the literature [42, 43].

Eichhornia crassipes is a perennial, free floating aquatic weed commonly known as water hyacinth, belonging to the family Pontederiaceae. Water hyacinth is considered among the most notorious invasive species due to its rapid growth and propagation. Though causing negative effects on the aquatic ecosystems as a weed, water hyacinth has drawn a lot of attention due to its tendency to accumulate toxic metal ions and for its ability to grow and flourish in polluted waters [44]. The synthesis of AgNPs from water hyacinth biomass offers a viable way to reduce the environmental contamination brought on by this invasive plant in addition to being an environmentally responsible and sustainable way to produce nanoparticles [45-47]. The presence of polyphenols and flavonoids in water hyacinth have the ability to function as both stabilizing and reducing agents making water hyacinth a viable option for the green synthesis of silver nanoparticles [48]. Studies have shown that the leaves of water hyacinth have been used for the synthesis of AgNPs [45, 49].

In this study, a green route was employed to synthesize AgNPs by utilising water hyacinth leaf extract as the reducing agent. The synthesized AgNPs were then characterized using different techniques. The ability of AgNPs for the colorimetric detection of metal ions [Hg^{2+} and Fe^{3+}] has been investigated.

2. Materials and Method

2.1. Water Hyacinth Leaf Extract Preparation

Fresh leaves of water hyacinth were collected from the local lake [Ramanthapur Chinna Cheruvu] of Hyderabad, Telangana, India. The leaves were cleansed twice using tap water followed by deionised water to get rid of the debris and dust to avoid contamination. The leaves were then cut into small pieces and weighed into 25 g portions. This 25 g of leaves were added to 100 ml of deionised water and was bought to boil for a few minutes. The solution was filtered using Whatman filter paper [pore size 11 microns] and the resulting extract was used for AgNP synthesis.

2.2. Synthesis of Silver Nanoparticles

10 ml of water hyacinth extract was taken in conical flask and 90 ml of 1 mM $AgNO_3$ solution was added. The mixture was then heated at 90 ℃ for a minute for the reduction of Ag^+ ions. The solution starts to change colour immediately after heating. The colour of the solution changed from pale green to yellowish brown indicating the synthesis of silver nanoparticles. To study the effect of concentration of Silver Nitrate on the synthesis of nanoparticles five different concentrations of $AgNO_3$ that is 0.5, 1, 1.5, 2 and 2.5 milli molar $AgNO_3$ were used for the synthesis.

2.3. Characterization of Synthesized Silver Nanoparticles

UV-Visible Spectroscopy measurements were carried out to confirm the synthesis of silver nanoparticles. UV2600 Model UV-Visible Spectrophotometer [Shimadzu] was used to record the UV-Vis Spectral Analysis. The spectral data was recorded from 200-800 nm wavelength. IR spectra was recorded by using Bruker Tensor 27 FT-IR Spectrometer which is used to identify the functional groups present in the extract that are responsible for the capping activity and reducing during the synthesis of AgNPs. FTIR spectra were recorded for extract and synthesized AgNPs separately. The surface morphology of the particles was studied using FE-SEM equipped with EDX. The AgNPs elemental composition and the presence of elemental silver were confirmed by EDX. The size and shape of the silver nanoparticles were measured using Jeol/JEM 2100 TEM. At 10000 rpm for 20 minutes the silver nanoparticles solution was centrifuged. The pellet was dried in the hot air oven after washing with deionised water. To evaluate the crystallinity of the synthesized nanoparticles XRD was performed using a Rigaku Smart Lab SE X-ray diffractometer. To analyze the stability of the nanoparticles zeta potential analysis was conducted using Anton Paar Litesizer 500.

2.4. Metal Detection Using Silver Nanoparticles

1000 micro molar solutions of were prepared to detect the metal ions in aqueous solution. 100 µl of metal ion solutions were added to 2 ml of silver nanoparticle solution individually. UV-Visible spectra was noted after 10 minutes using UV2600 Model UV-Visible Spectrophotometer [Shimadzu] from 200-800 nm wavelength range. With the addition of $Hg^{2+}$and Fe^{3+} ions, the the decrease in peak intensity and disappearance of the brown colour of AgNPs were observed. To silver nanoparticles different volumes of the two metal ion solutions were added and UV-Visible spectra was recorded.

3. Results and Discussion

3.1. UV-Visible Spectroscopy

The analysis by UV-Visible spectra of the AgNPs synthesized using water hyacinth leaves extract was recorded in the wavelength range of 300 to 800 nm. Ag-NPs have been shown to exhibit a maximum UV-visible absorption wavelength in the 400–500 nm region, due to SPR [50]. The silver nanoparticles synthesized using 1 mM $AgNO_3$ and water hyacinth leaves extract showed a maximum absorption at 406 nm indicates the reduction of silver ions [Figure 1].

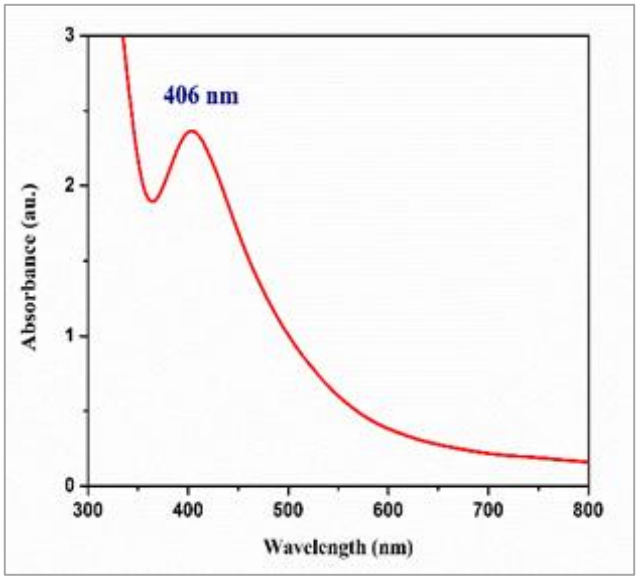

Figure 1. *The UV-Visible spectra of AgNPs synthesized from water hyacinth.*

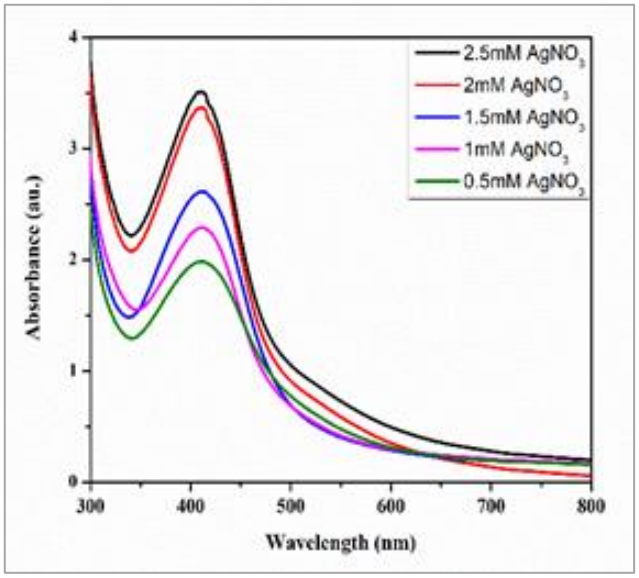

Figure 2. *UV-Visible spectra of AgNPs obtained using water hyacinth leaves extract in different concentrations of $AgNO_3$.*

To study the effect of concentration of silver nitrate on formation of AgNPs, different concentrations of $AgNO_3$ [0.5 mM, 1 mM, 1.5 mM, 2 mM, 2.5 mM] solutions were added to water hyacinth leaves extract in 9:1 ratio. By using a UV-Visible Spectrophotometer the absorbance of the resulting AgNPs was noted. As can be seen in the Figure 2, the absorbance of the silver nanoparticles increased with the increase in concentration of $AgNO_3$ indicating the increased synthesis of AgNPs.

3.2. FTIR Analysis

To identify the functional groups accountable for the reduction of silver ions FTIR analysis was performed. FTIR spectra were recorded separately for water hyacinth leaves extract and silver nanoparticles [Figure 3]. The similarity between the IR spectra of the water hyacinth extract and the AgNPs establishes the coordination of organic moieties extracted from the leaf extract to the surface of the synthesized silver nanoparticles [51]. The peaks observed in the range of 3600-3200 cm-1 were due to the O-H stretching vibrations of alcohols and 3300-2500 cm-1 represent the O-H stretching of carboxylic acids. The shift in the peaks in spectra of AgNPs occur due to the engagement of O-H bond in the reduction of Ag^+ to Ag^0 demonstrating that the polyphenols in the leaf extract primarily act as reducing agent [52]. The peaks corresponding to 2250-2100 cm-1 are due to the presence of stretching vibrations in alkynes. The IR peak at 1640-1600 cm-1 represents the C-N and C-C stretching, suggesting the presence of proteins [53, 54]. The peak observed in 1650-1580 cm-1 corresponds to N-H bending of amines. The peaks in the range of 1440-1395 and 1420-1330 correspond to O-H bending vibrations of carboxylic acids and alcohols respectively. The peaks between 700-650 cm-1 are due to the C-H bending. The production of AgNPs might be attributed to these FT-IR bands of amine and alcoholic groups, which are required for the capping of silver nanoparticles [45]. Therefore, it can be concluded that the for reduction of silver ions the organic molecules in the leaves extract are responsible.

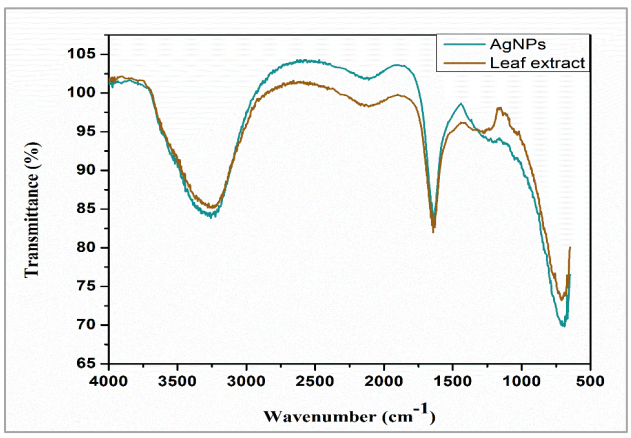

Figure 3. *FTIR spectra of biosynthesized AgNPs and water hyacinth leaves extract.*

3.3. XRD Analysis

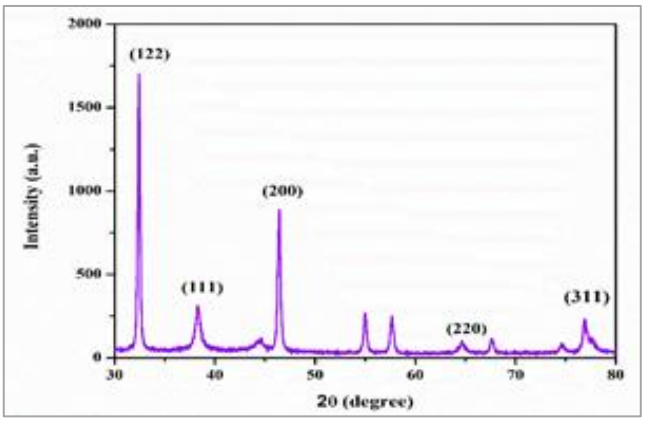

Figure 4. *XRD pattern of biosynthesized AgNPs.*

To identify the crystalline nature of the AgNPs XRD study was carried out. Five distinct diffraction peaks at 32.37 °, 38.36 °, 46.4 °, 64.5 ° and 76.78 ° have been observed in the XRD spectra which corresponds to the 122, 111, 200, 220, 311 planes respectively. Figure 4 shows the XRD spectra of the AgNPs and the peaks can be indexed as face centered cubic silver crystals and confirms that the AgNPs are crystalline in nature. Several researchers noted that the green synthesized silver nanoparticles are crystalline in nature having FCC structure [54-57].

3.4. SEM and EDAX Analysis

The AgNPs morphology and shape were analyzed by SEM. Figure 5 [a] [b] [c] shows the SEM images of AgNPs. It was observed that mostly AgNPs are spherical in shape. To assess the elemental composition, EDAX analysis was performed. The EDAX spectrum reveals the presence of silver, nitrogen, oxygen and carbon.

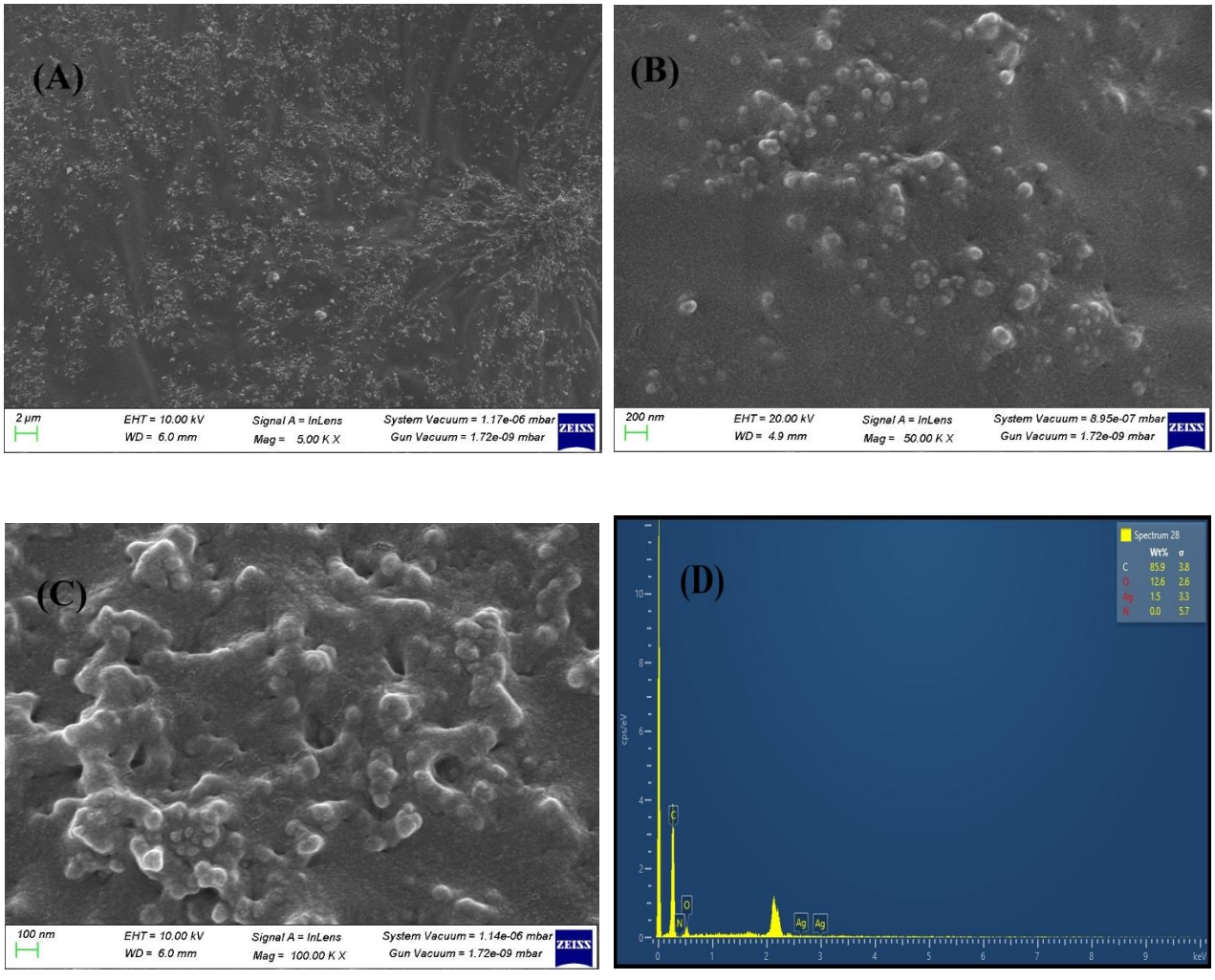

Figure 5. *[A] [B] [C] SEM images showing synthesized AgNPs [D] EDAX image of synthesized AgNPs.*

3.5. Transmission Electron Microscopy

The size, morphology, and shape of the silver nanoparticles was observed using TEM. Figure 6 shows the TEM micrograph of the synthesized AgNPs and it can also be noticed that the AgNPs are spherical in shape. The nanoparticles ranged from 5.17 nm to 31.13 nm and the nanoparticles mean diameter was found to be 10.77 nm ±4.61 nm. Figure 7 shows the synthesized AgNPs particle size distribution histogram.

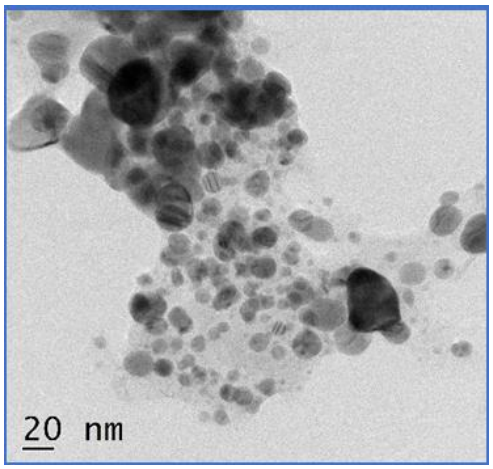

Figure 6. *TEM image of synthesized AgNPs.*

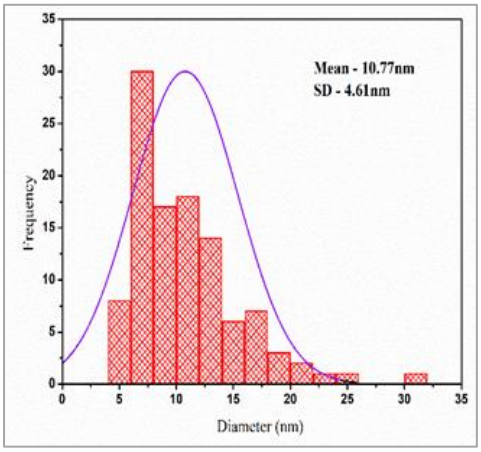

Figure 7. *Particle size distribution histogram of AgNPs.*

3.6. Zeta Potential Measurement

To assess the stability of colloidal AgNPs zeta potential was measured. Large positive or negative zeta potential metal nanoparticles have a tendency to reject one another and do not exhibit any inclination to group together. The absence of a repulsive force, however, causes these particles to flocculate and aggregate when the absolute zeta potential is low. Stable nanoparticles are those that have a zeta potential value of greater than or equal to 30 mV. [58, 59]. The zeta potential distribution of the synthesized AgNPs is observed to be -31.7 mV [Figure 8] indicating that the nanoparticles are stable.

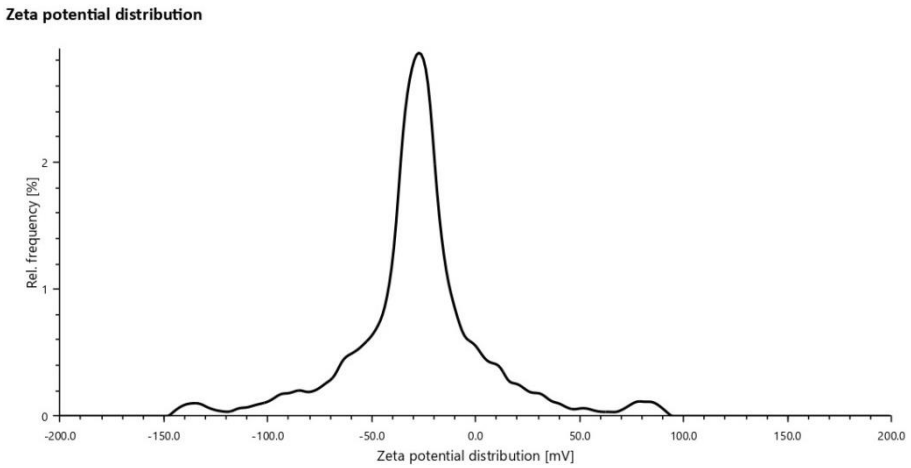

Figure 8. *Zeta potential distribution of synthesized AgNPs.*

3.7. Metal Detection

To detect metal ions in aqueous solution at 1000 micro molar concentration was analyzed colorimetrically for synthesized AgNPs. 1000 micro molar solutions of 10 different metal ions, Zn^{2+}, Sn^{2+}, Na^{+}, Mn^{2+}, K^{+}, Fe^{3+}, Hg^{2+}, Fe^{2+}, Cu^{2+} and Ba^{2+} were prepared and 100 µl of these metal ion solutions were added to 2 ml of silver nanoparticle solution individually. The silver nanoparticles displayed [brown to colorless] observable change upon the addition of Fe^{3+} and Hg^{2+} while other metal solutions had no effect. Figure 9 indicates the UV-Visible spectra of silver nanoparticles after the addition of metal solutions and the disappearance of the characteristic UV-Visible peak can be observed in case of Fe^{3+} and Hg^{2+} ions.

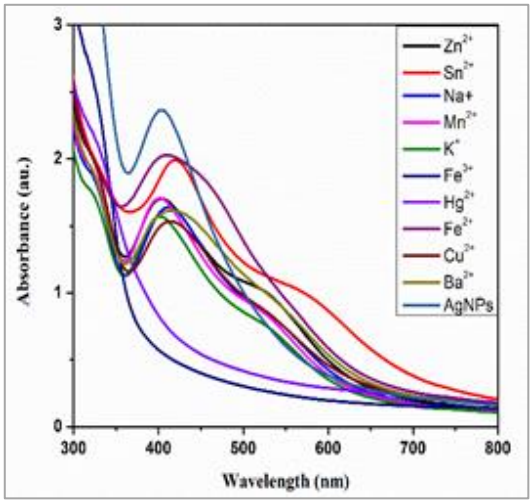

Figure 9. *Silver nanoparticles with the addition of metal ions after 10 minutes which was observed by UV-Visible spectra.*

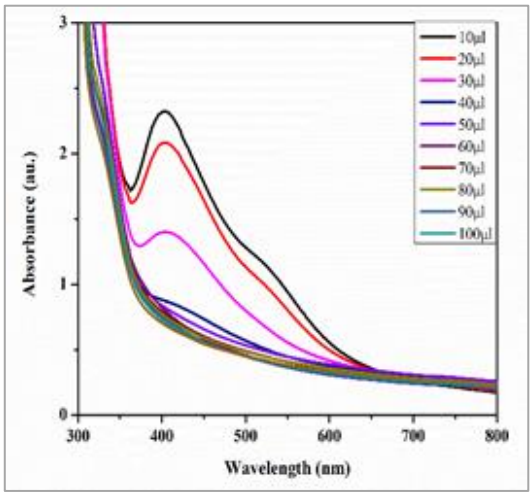

Figure 10. *AgNPs UV-Visible spectra after the addition of different volumes of Fe^{3+} solution.*

The UV-Vis spectra of AgNPs was shown in the Figures 10 and 11 after the addition of different volumes of Fe^{3+} and Hg^{2+} solutions respectively. The possible reason for the disappearance of the absorbance peak is the oxidation of Ag atoms into Ag^{+} ions by Hg^{2+} and Fe^{3+} ions resulting in the dissolution of AgNPs and the disappearance of peak [51]. It can be observed from Figures 10 and 11 that the absorbance peak disappeared gradually with the increase in the volume of Fe^{3+} and Hg^{2+} solutions respectively. The decolourization of the silver nanoparticle solution and decrease in the peak is due to the oxidation of colloidal silver particles into silver ions [60]. It can be observed that the redox reaction occurring between the silver nanoparticles and the metal ions Fe^{3+} and $Hg^{2+}$was responsible for the detection of these ions. Hg^{2+} was detected using a spectrophotometer by monitoring the decrease in peak intensity of AgNPs. As the volume of Hg^{2+} ions increased from 10 to 100 µl, the peak gradually decreased. A spontaneous redox reaction occurs between AgNPs and Hg^{2+} ions because of the positive cell potential (E^{0}cell) [61]. The loss of the typical brown colour of AgNPs demonstrates the strong oxidizing capability of mercury ions, which leads to the formation of reduced Hg^{0} [62]. Ferric ions (Fe3+) have negative E^{0}cell values, which prevent them from oxidizing elemental silver (AgNPs) like Hg^{2+} ions. The reduction potential of the silver species is considerably decreased by the presence of halide ions in the detecting medium (Cl^{-} ions produced from $FeCl_3$ metal salt solution), as they strongly coordinate with one another. By generating a positive E^{0}cell value, ferric ions are able to oxidize elemental silver as the potential of Ag^{+}/Ag decreased. This leads to the spontaneous redox interaction between AgNPs and Fe^{3+} ions [63-65].

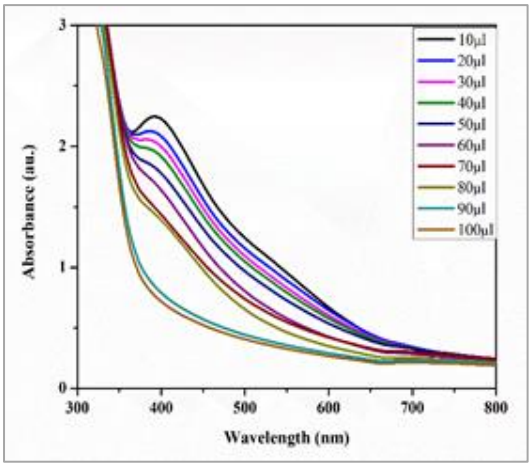

Figure 11. *AgNPs UV-Visible spectra after the addition of different volumes of Hg^{2+} solution.*

4. Discussion

A study reported the usage of Ag-NPs synthesized from *Cordia myxa* leaf extract for the Fe^{3+} and Hg^{2+} ions by colorimetric determination [51]. Silver nanoparticles (AgNPs) synthesized using the aqueous leaf extract of Trigonella foenum-graecum L. as a reducing agent were used for the colorimetric Sensing of Fe^{3+}/Hg^{2+} ions [65]. Researchers employed Ag-NPs synthesized using *Sonchus arvensis* [SA] leaf extract for the colorimetric detection of Fe^{3+} and Hg^{2+} ions [66]. Several researchers investigated the ability of Ag-NPs synthesized using green method for the colorimetric detection of iron [67-69] and mercury [41, 70, 71] ions and successfully determined Hg^{2+} ions in different water samples [72].

5. Conclusions

For the past few years, green nanoparticle synthesis has increased due to their applications in various sources. AgNPs synthesized from water hyacinth leaf extract were characterised using different techniques and are used for the detection of Hg^{2+} and Fe^{3+} ions in aqueous solution. The biosynthesized

nanoparticles were found to be reliable colorimetric sensors for the detection of Hg^{2+} and Fe^{3+} ions in aqueous solution at 1000 micro molar concentration. The reaction between bio-synthesized nanoparticles and metal ions [Hg^{2+} and Fe^{3+}] was introduced as a promising sensor for the detection of ions. The use of unmodified AgNPs for the detection of Hg^{2+} and Fe^{3+} ions is easy and simple without requiring any pre-treatment and cost-effective compared to existing methods. The simple, non-toxic, eco-friendly and cost-effective process of silver nanoparticle synthesis makes this method easy to implement. This process for the environmentally friendly synthesis of AgNPs offers a variety of appealing characteristics as well as a practical and cost-effective way for protecting the environment. The present study has provided positive results in the colorimetric detection of iron and mercury ions in aqueous solutions contributing to the existing literature. In future, researchers can use AgNP based sensors for simple, rapid, low cost and accessible applications in various areas.

Abbreviations

FTIR	Fourier Transform InfraRed Spectroscopy
XRD	X-ray Diffraction
SEM-EDX	Scanning Electron Microscopy - Energy Dispersive X-Ray Spectroscopy
TEM	Transmission Electron Microscopy
Ag-NPs	Silver Nanoparticles
UV	Ultraviolet
ICPMS	Inductively Coupled Plasma Mass Spectrometry
ICP-OES	Inductively Coupled Plasma Optical Emission Spectroscopy
AFS	Atomic Fluorescence Spectroscopy
HPLC	High Performance Liquid Chromatography
FAAS	Flame Atomic Absorption Spectroscopy

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Author Contributions

Ervaguda Revathi: Conceptualization, Data Curation, Formal Analysis, Investigation, Methodology, Writing – original draft, Writing – Review and editing

Syeda Azeem Unnisa: Conceptualization, Methodology, Supervision, Writing – original draft, Writing – Review and editing

Edupuganti Sujatha: Supervision, Writing – Review and editing

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Data Availability Statement

The data supporting the outcome of this research work has been reported in this manuscript.

Conflicts of Interest

The authors declare no conflicts of interest.

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