Original Article

Green Synthesis Of Silver Nanoparticles From Chrysanthemum Indicum And Its Characterization

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Abstract

Background: Chrysanthemum indicum is an Indian flowering plant within the family Asteraceae and genus Chrysanthemum. The Chrysanthemum flower is being used to treat chest pain(angina) high blood pressure, fever, cold, headache, dizziness and swelling. This plant is used to treat prostate cancer too. The Chrysanthemum flower is used as a tea in East and Southeast Asia. Therefore, this study investigates the synthesis of silver nanoparticles from aqueous extract of Chrysanthemum indicum and its characterization. Phytochemical analysis was carried out qualitatively and quantitatively to find various compounds present in the flower extract. Characterization of Silver nanoparticles was carried out through UV-vis spectrometry, FT-IR, XRD, TEM, Zeta potential sizer, FESEM and EDX. The Silver Nanoparticles were in crystalline irregular shape in size from 71 nm to 180 nm. The UV-Vis Spectroscopy peak was observed at 436 nm. The PDI value is 0.731, it indicates good monodispersed and zeta potential indicates the capping of Silver Nanoparticles by negatively charged groups. The particles size of Silver Nanoparticles was in the range of 36nm to 180nm. The mean value of distribution is 211nm. TEM analysis reveals that the synthesized nanoparticles are polycrystalline, polydisperse in nature and spherical in shape. The FTIR analysis showed the presence of alcohols, phenols, carboxylic acids, primary amines, aliphatic amines, aromatics, and alkyl halides groups.

KEYWORDS: UV-vis spectrometry, Zeta potential sizer, Transmission electron microscopy, Scanning electron microscopy, Fourier Transform- Infrared Spectroscopy, Phytochemical

INTRODUCTION

Silver Nanoparticles are widely used and studied nano materials due to their physiochemical properties such as high ratio of surface area to mass, electric optical, catalytic, and particularly antimicrobial properties [1]. Metallic Nanoparticles may be synthesized by physical, chemical, and organic methods. This physical method is expensive and chemical method is extremely hazardous to the environment. So, the biological method is preferable because of its eco-friendly, rapid, dependable, nontoxic, ecofriendly, simple, and low cost [2,3]. Chrysanthemum indicum is an important medicinal plant in China and other South Asian countries. It has very wide range of pharmacological effects including antidiabetic, Anti cancerous, anti-allergic etc. Nanoparticles have been used in

catalysis, ceramics, drug delivery, diagnostics, and oncology therapies [4]. Various physical and chemical methods were used to synthesize monodispersed Silver Nanoparticles by reduction of aqueous Ag+ ions using plant extract has been reported in literatures. In this present study the Silver Nanoparticles synthesized from Chrysanthemum indicum includes low cost, nontoxic, and ability to prepare compounds with varying morphologies having different prosperities by utilizing reducing and capping potential.

MATERIALS AND METHODS

Preparation of extract

About 50 grams of dried petals of Chrysanthemum indicum were proceeded for Soxhlet extraction. Extraction is proceeded with solvent -Aqueous (100%) for 24 hrs. Extracts were filtered using Whatman No.1 filter paper. Extracts were refrigerated at 4˚C for further studies.

Preliminary phytochemical analysis

Phytochemical analysis were performed both qualitatively and quantitatively. Qualitative analysis were performed accordingly to the methodology of Sofowara, Trease and Evans[5,6]. Aluminium chloride colorimetric method of Lin and Tang 2007 is used to quantify the total flavonoid content. Total tannin content is estimated by UV-vis spectrophotometric method proposed by Bajaj and Devasharma 1977 [7]. Total steroid content is quantified accordingly to the methodology of Attarde Daksha et al., 2010 [8].

Synthesis of silver nanoparticles

5ml of Aqueous extract of Chrysanthemum indicum flower is added to 45ml of 1 mM aqueous $AgNO_3$ solution in a flask. The flask is incubated at room temperature in dark for 5 hrs. The Chrysanthemum indicum extract facilitates the reduction of silver ions into smaller sized silver particles due to the electron donating ability of these phenolic compounds. The reduction of silver ions into silver nanoparticles was confirmed by the change of the silver nitrate solution to a reddish-brown color at the room temperature.

Characterization of Synthesized Ag-NPs

Synthesized nanoparticles were characterized by techniques like UV-vis spectrometry, Zeta potential sizer, FT-IR, TEM, FESEM, XRD and EDX. Using a Shimadzu UV-2450 spectrometer, the UV-visible spectra of the synthesized nanoparticle were recorded at room temperature. Spectra were detected in the wavelength range of (λ) 200 to 800 nm. FT-IR analysis of Ag-NPs with KBr was performed using a Shimadzu 8400s (Japan) with a resolution of 4 cm^{-1} in the spectrum range of 4000-400 cm^{-1} in order to determine the functional groups. Zeta potential sizer study is performed accordingly to the methodology of Soheyla Honary et al., 2013 [9]. Using a Field emission scanning electron microscope coupled with an Energy Dispersive X-ray analyzer, the shape, sizes, and atomic weight composition of the synthesized Ag-NPs were analyzed. The TESCAN Vega TS 5136LM generally operates at 20 kV at a working distance of 20 mm, and an 80 kV voltage Tecnai F20 transmission electron microscope was utilized. Shimadzu XRD model 6000 diffractometer (Japan) with graphite monochromator was used to obtain x-ray powder diffraction (XRD) patterns under Cu-Kα radiation. JCPDS-International Center for Diffraction Data was employed to analyse XRD data in order to identify the crystalline phases (Abiola Grace et al., 2018) [10].

RESULTS AND DISCUSSION

Qualitative and Quantitative analysis of phytochemicals

The aqueous extract of Chrysanthemum indicum shows the presence of phytochemicals such as tannin, saponin, flavonoids, steroids, terpenoids, alkaloids, phenols, coumarin and phytochemicals like anthraquinone, emodins and anthocyanins were absent. The Quantitative analysis shows high phenolics content (100.5±1.1GAE/g) (Gallic Acid Equivalents), Flavonoids content (13.47±4.78MgQE/g) (Quercetin Equivalent), Steroids content

(17.04±3.7MgBSE/g) (β-sitosterol Equivalents), Tannin content (29.93±4.2MgTAE/g) (Tannic Acid Equivalents).

Synthesis of silver nanoparticles

The high contents of phenolic compounds in the Chrysanthemum indicum extract facilitates the reduction of silver ions into smaller sized silver particles due to the electron donating ability of these phenolic compounds [11-13]. The reduction of silver ions into silver nanoparticles was confirmed by the change of the silver nitrate solution to a reddish-brown color at the temperature. Whereas the control $AgNO_3$ without flower extract showed no change in colour. The presence of flower extract converts the AgNO3 solution into reddish brown color solution due to the excitation of the surface plasmon resonance in the synthesized nanoparticle.

Characterization of nanoparticles

UV-Vis spectroscopy: UV-Vis spectroscopy indicates the formation of Silver Nanoparticles with the highest peak observed at 436nm.

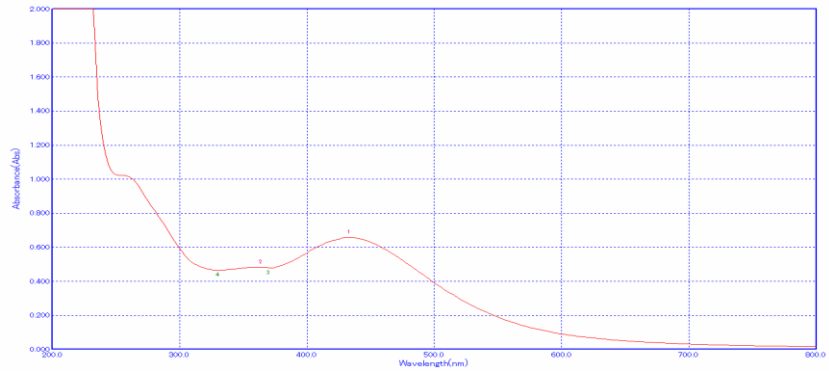
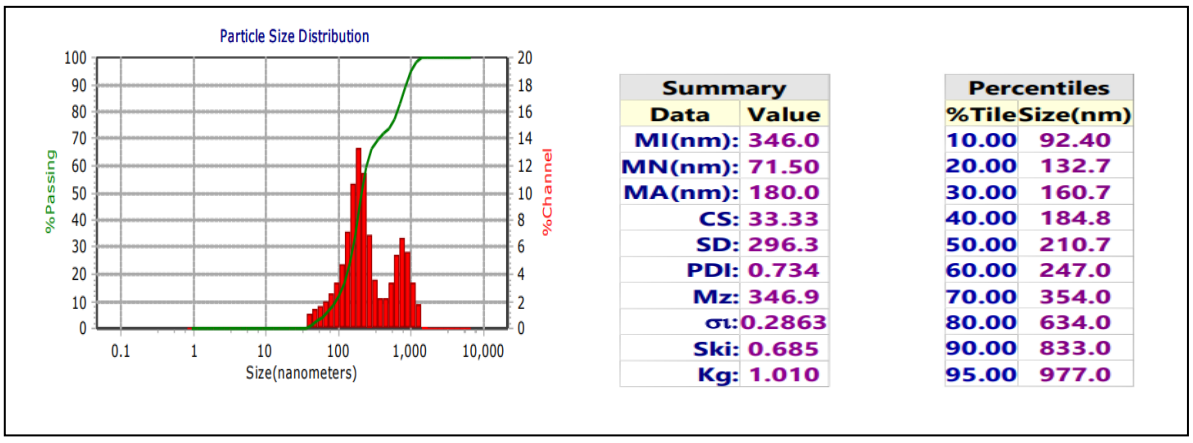


Figure 1: UV spectrum of Ag-NPs

Zeta potential sizer

The Zeta potential of Silver Nanoparticles is -25.1mv. The PDI value is 0.731, it indicates good monodispersed and capping of Silver Nanoparticles by negatively charged groups. The particles size of silver nanoparticles was in the range of 36nm to 180nm. The mean value of distribution is 211nm.



Summary	
Data	Value
MI(nm):	346.0
MN(nm):	71.50
MA(nm):	180.0
CS:	33.33
SD:	296.3
PDI:	0.734
Mz:	346.9
σι:	0.2863
Ski:	0.685
Kg:	1.010

Percentiles	
%Tile	Size(nm)
10.00	92.40
20.00	132.7
30.00	160.7
40.00	184.8
50.00	210.7
60.00	247.0
70.00	354.0
80.00	634.0
90.00	833.0
95.00	977.0

Figure 2: Zeta potential and particle size analysis

FT-IR Spectroscopy

The Fourier -transform infrared spectroscopy analyses were conducted to identify the potential functional groups of biomolecules in Silver Nanoparticles. The FTIR analysis showed the presence of alcohols, phenols, carboxylic acids, primary amines, aliphatic amines, aromatics, and alkyl halides groups. The Frequency 3442.18cm^{-1} showed the presence of O-H stretch, H-bonded Alcohols and phenols,the2928.11 cm^{-1}and2863.16 cm^{-1}shows the presence of O-H stretch carboxylic acid,2424.28 cm^{-1}shows the presence of CO_2,1628.21 cm^{-1} shows the unsaturated N-H bending amines,1384 cm^{-1} shows O-H bending phenol,1107.86 shows C-N stretch Aliphatic amines, 830.80 cm^{-1} shows the aromatics and 609.06 shows the presence of C-Br stretch Alkyl halides.

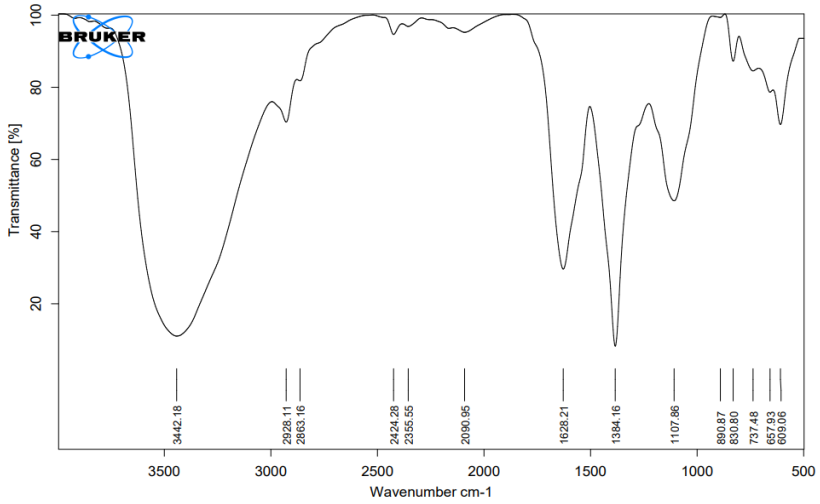


Figure 3: FT-IR Spectrum of Ag-NPs

Transmission Electron Microscopy

The Silver Nanoparticles appears in spherical in shape and they are polycrystalline and polydisperse in nature as shown in figure 3. The size and morphology of the synthesized Silver Nanoparticles were studied by Transmission electron microscopy(Fig3a). The size of the particles was found in the range of 25.80nm to 66.142nm and the average size was 32. 5nm and they were poly dispersed particles.

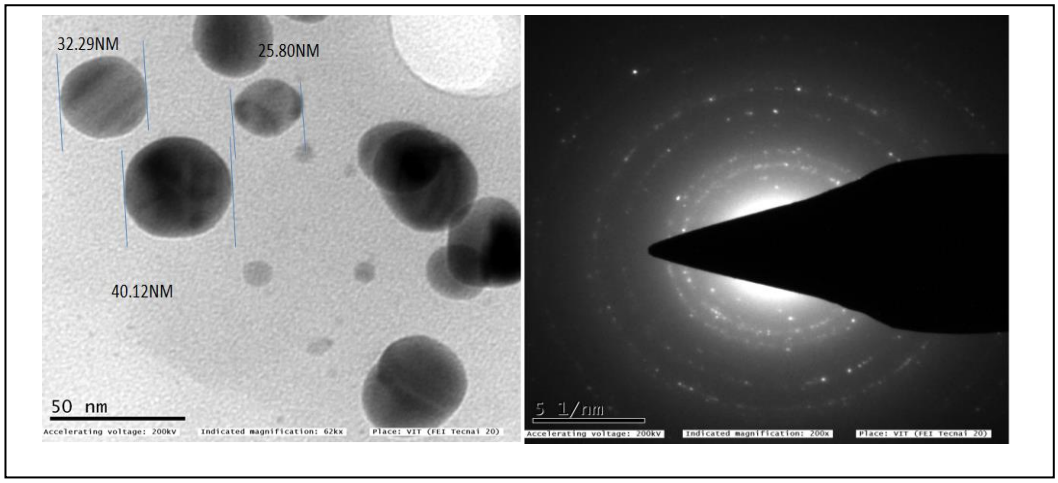


Figure 4: TEM analysis of Ag-NPs

FESEM

Field emission scanning electron microscopy images of produced silver nanoparticles are shown in figure. According to FESEM studies, the nanoparticles have spherical surfaces and larger average sizes than ranging from 20 to 42 nm. According to FESEM images, the nanoparticles are homogenous without any substantial agglomeration.

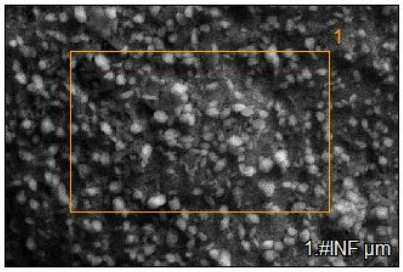


Figure 5: Field emission scanning Electron microscopical study of Ag-NPs

X-ray diffraction studies & EDX

Figure shows the x-ray diffraction patterns of synthesized silver nanoparticles from flower extract. The XRD is use to confirm the crystalline nature of Ag nanoparticles. Debye-Scherrer equation is used to determine the size of crystalline silver nanoparticle.

$D = 0.9\lambda/\beta\cos v$

Where, D-size of nanoparticle, λ- wavelength of x-ray, β- full width at half maxima of the diffraction peak.

XRD spectrum of Ag-NPs revealed diffraction peaks at 36.39˚, 47.50˚, 63.89˚ and 78.11˚ corresponding to (111), (200), (220) and (311) planes and comparable with the standard values planes of silver. The inclusion of phytochemical substances in the produced nanoparticles produced the appearance of few other minor peaks. The presence of the phytocompounds over the Ag particles was validated by x-ray diffraction measurements. Synthesized nanoparticles are not pure Ag nanoparticles. It consists of Ag, Ag_2O. The average size of crystal was approximately 40 nm. EDX spectrum strongly reveals the signal from silver region for the formation of Ag-NPs. Silver was the major constituent element comparable to other elements such as C, O, Al, Si, P, S, Cl, K, Fe, Ni. The conversion of silver nitrate to silver nanoparticles was affirmed by the dense peak of silver. A silver peak possesses thicker peak than the other. This validates the spectrum's depiction of the complete reduction of silver compounds to Ag-NPs.

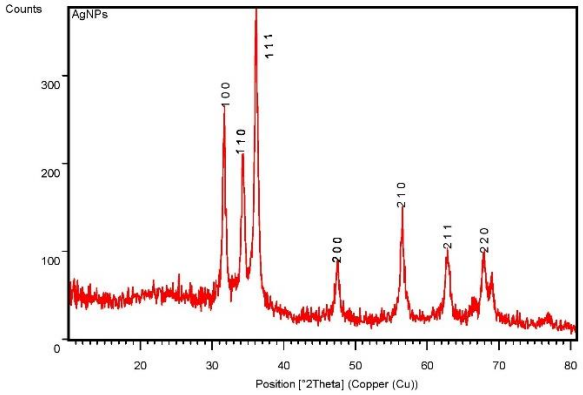


Figure 6: XRD pattern of Ag-NPs

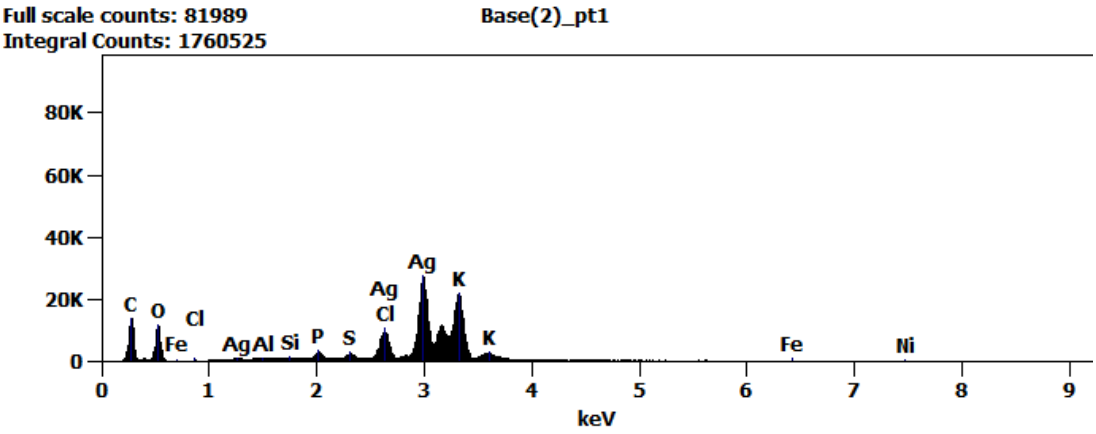


Figure 7: EDX spectrum of Ag-NPs

Conclusion

The current study deals with phytochemical analysis by qualitatively and quantitatively, synthesis and characterization of silver nanoparticle. The preliminary phytochemical study of aqueous extract of Chrysanthemum indicum shows the presence of phytochemicals such as tannin, saponin, flavonoids, steroids, terpenoids, alkaloids, phenols, coumarin and phytochemicals like anthraquinone, emodins and anthocyanins were absent. Quantitative analysis of total phenols, flavonoids, steroids and tannins yields good results. Green synthesis of silver nanoparticles was done using the flower aqueous extract of Chrysanthemum indicum. This method helps the flower extract to serve as a reductant and a stabilizing agent for synthesis of Ag-NPs. The UV-visible spectra peak at 436nm confirmed the reduction of silver ions leading to Ag-NPs synthesis. FT-IR confirmed the presence of bioactive compounds act as a stabilizer for synthesis of Ag-NPs. The stability of nanoparticles is stable in zeta potential analysis. TEM analysis shows that Silver Nanoparticles appears in spherical in shape and they are polycrystalline and polydisperse in nature. FESEM also confirms the synthesized nanoparticles are spherical in shape. XRD confirms the crystalline structure of Ag-NPs along with the confirmation of EDX spectrum.

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