

Synthesis and Characterization of Gold Nanoparticles (Au-NPs) using aqueous extract of Lemongrass

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Abstract— *Biological method was used to synthesize gold nanoparticles (Au-NPs) using H₂AuCl₄, double distilled water (D.D.W), and lemongrass extract. The change in the color of the solution from yellow to red, after aqueous lemongrass extract was added, indicated the formation of Au-NPs. Different techniques were used to characterize the prepared Au-NPs. The X-Ray diffraction (XRD), scanning electron microscopy (SEM), and transmission electron microscopy were used to analyse the structural properties of the Au-NPs. The crystallite size of Au-NPs was found to be 12nm using the Debye–Scherrer’s formula. TEM analysis was used to examine the triangular and spherical shaped Au-NPs, and the average particle size was found to be 13 nm. The optical properties of the nanoparticles were analysed using the photoluminescence emission (PL) spectroscopy and UV–visible absorption spectroscopy. An absorption peak at 543 nm was obtained from the analysis. The Fourier Transform Infrared (FTIR) spectroscopy was used to investigate the functional groups in the leaves of the lemongrass that are responsible for reducing the gold ions to Au-NPs, and stabilizing the nanoparticles.*

Keywords— *Gold nanoparticles, XRD, TEM, FTIR and Optical properties*

I. INTRODUCTION

Nanotechnology is an important and emerging field of science that deals with the development of synthetic and biological techniques for producing nanoparticles. It has gained considerable attentions of researchers around the globe in recent times due to the extensive applications of nanoparticles [1, 2]. The applications of nanoparticles for various purposes, such as biomedical, pharmaceutical, catalysis, drug delivery, and antimicrobial purposes, are increasing rapidly due to the enhanced properties of NPs [3]. Nanoparticles have unique physical and chemical properties due to their small sizes, and this enables them to be used for various novel purposes [4]. Nanoparticles are applied in different areas such as health care, tissue engineering, gene/drug delivery, food industry, optics, mechanics, space industry, and optical devices [5, 6]. The most important nanoparticles are the metallic nanoparticles because they have remarkable antibacterial properties as a result of their large surface area to volume ratio. Researchers are becoming very much interested in the antimicrobial effect of metallic nanoparticles because of the increasing development of resistance by microbes against antibiotics [7, 8]. Silver, gold, platinum nanoparticles are some of the metallic nanoparticles which have gained considerable attention in recent times due to their unique properties. Several studies have been carried out on gold nanoparticles due to their exceptional properties that are dependent on their size and shape, as well as their various potential medicinal applications. Gold nanoparticles (Au-NPs) have unique physical and chemical properties that make them suitable materials for production of novel biological and chemical sensors [9-11]. Au-NPs are the most important nanoparticles due to their biocompatibility and medicinal applications in the treatment of arthritis and cancer [12]. Gold nanoparticles are extensively used for numerous biomedical applications such as bioimaging, biosensor, photothermal treatment and targeted drug delivery [13]. Several methods, such as chemical, physical and biological methods, have been developed for synthesising different shapes and sizes of Au-NPs [14]. Also, plant tissues and microorganisms such as fungi and bacteria have been used to synthesise Au-NPs by biological technique. Plant extracts are useful in producing Au-NPs because they can be used to produce huge amount of Au-NPs with minimal effects on the environment. Also, they are non-hazardous reducing and stabilizing agents. The biosynthesis of Au-NPs with aqueous extract of lemongrass leaves and characterization of the synthesised nanoparticles is presented in this study.

II. EXPERIMENTAL DETAILS

A. Synthesis of plant extract

250 g of lemongrass, which was collected from India, washed with double distilled water (D.D.W) and left to dry for 10 days. The dried leaves was cut into small pieces with a mill. The lemongrass was added to 500 ml of double distilled water (D.D.W) and boiled for 30 min at a temperature of 100 °C. The mixture was separated into an aqueous extract, which was refrigerated at 6°C for further use in the preparation of Au-NPs.

B. Preparation of Au-NPs

In this research, Au-NPs were produced by biological technique using lemongrass extract. 100 ml of double distilled water was used to dissolve 1mM of H₂AuCl₄, and the solution was stirred continuously for 20 min with magnetic stirrer at

140 rpm. Thereafter, 10 ml of lemongrass extract was added to 20 ml of HAuCl₄ solution, which was continuously stirred until the color of the solution changed to red.

III. CHARACTERIZATION

X-ray diffraction (Rigaku Miniflex) with radiations of Cu- K α ($\lambda=1.5406 \text{ \AA}$) was used to examine the crystalline structure of the Au-NPs. This was done with a current of 15 mA and voltage of 30 kV at 2θ , which ranged from 30° to 80° . Fourier transformed infrared spectroscopy were recorded on a spectrophotometer. The sample on the circular disk used for the measurement by Fourier transformed infrared (FTIR) was prepared by drying the Au-NPs for 4 hours at 60°C and mixing the dried Au-NPs with KBr. Several bonding vibrational frequencies was shown by the FTIR spectrum. The optical properties of Au-NPs were examined with the aid of UV-Visible Absorption Spectrophotometer (Perkin Elmer Lambda-35) and Photoluminescence Emission Spectrophotometer within the range of 400-800 nm. The constituents and microstructure of the Au-NPs were examined with energy dispersive X-ray spectroscopy (EDAX) and the SEM (JEOL, Japan). The images of Au-NPs obtained from the transmission electron microscope (TEM; JEOL, JEM-2100) were used to examine the surface structure of the nanoparticles.

IV. RESULTS AND DISCUSSION

A. X-Ray Diffraction

The XRD patterns were used to analyze the crystalline structure of the biosynthesized nanoparticle. The XRD patterns show that the Au-NPs were formed by the reduction of gold ions (Au^{3+}) to metallic gold (Au^0) [15]. The XRD patterns of the Au-NPs produced with the leaf extract is shown in Fig. 1. The patterns show that Au-NPs have the crystalline gold fcc structure. The diffraction peaks obtained at $2\theta= 38.4^\circ, 44.5^\circ, 65.3^\circ,$ and 77.6° were (111), (200), (220), and (311) respectively, and they are similar to those reported for the standard gold metal (JCPDS-card no.04.0784) [16]. These four intense peaks correspond with the Bragg's reflections of gold obtained using the diffraction pattern [17]. The crystallite size of the Au-NPs was found to be 12 nm using the Debye-Scherrer equation, which is given below [18-20].

$$D = k\lambda / \beta \cos \theta \quad (2)$$

Where, D is the average crystallite size, $k = 0.9$ (constant), λ is the wavelength of X-ray radiation (Cu K $\alpha=1.5406 \text{ \AA}$), β is the full width at half-maximum (FWHM), and θ is the Bragg angle 2θ .

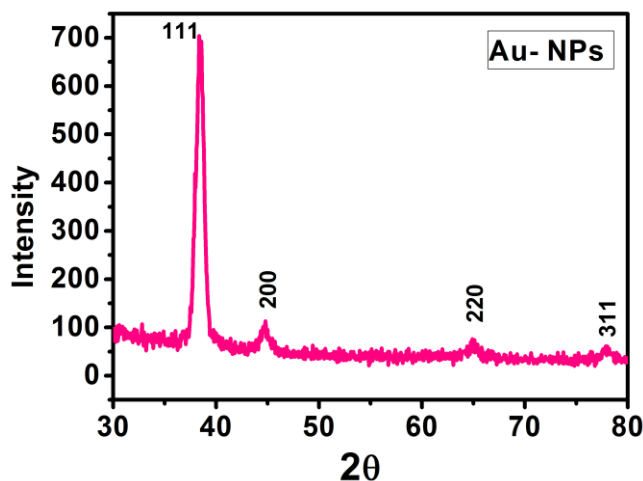


Fig. 1 XRD Pattern of Au-NPs

B. Scanning electron microscopy (SEM)

The SEM device was used to analyse the surface morphology of the Au-NPs. Thin layers of the Au-NPs used for the analysis was prepared by drying a minute quantity of the particle on a copper grid coated with carbon for 15 minutes under a mercury lamp. The different sizes of spherical and cubic shaped Au-NPs which were observed are shown below in Fig. 2 [21]. The large size of some of the nanoparticles in this SEM image are due to the build-up of the small nanoparticles. The EDAX analysis, which is shown in Fig. 2, was used to examine the metallic gold. The elemental constituents of the synthesized particles were examined by EDAX to include ions of chlorine (Cl), sodium (Na), silicon (Si), oxygen (O), Magnesium (Mg), Carbon (C), Calcium (Ca), Potassium (K) and gold (Au). The traces of silicon (Si) observed emanated from the glass (SiO_2) that was used in preparing the sample [22].

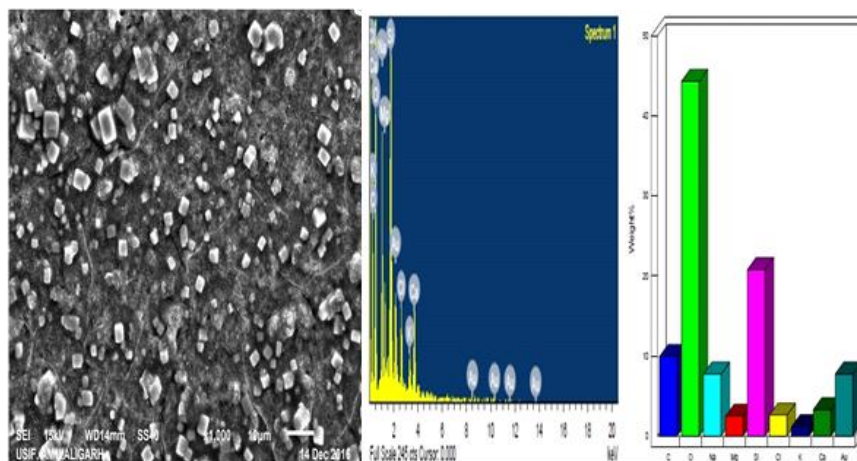


Fig. 2 SEM and EDAX images of Au-NPs

C. Transmittance electron microscopy(SEM)

TEM analysis was carried out for the morphological studies of synthesised Au-NPs. The Au-NPs used for the analysis were prepared by drying minute amount of suspended centrifuged particles on Formvar-coated copper grids. The size and morphology of the Au-NPs as analysed by TEM are shown in Fig. 3. The image shows the presence of spherical and triangular shaped Au-NPs with smooth and uniform particle sizes between 10 nm to 15 nm. An average particle size of 13 nm was obtained using the ImageJ software [23]. The average crystallite size that was calculated by Debye–Scherrer’s formula from the XRD pattern is in accordance to the particle size obtained from TEM analysis. Homogeneous nanoparticles with definite shapes are essential in producing optical, electronic, and medical devices because the optical properties of Au-NPs that make them suitable for such purposes are determined by their shape and size [24].

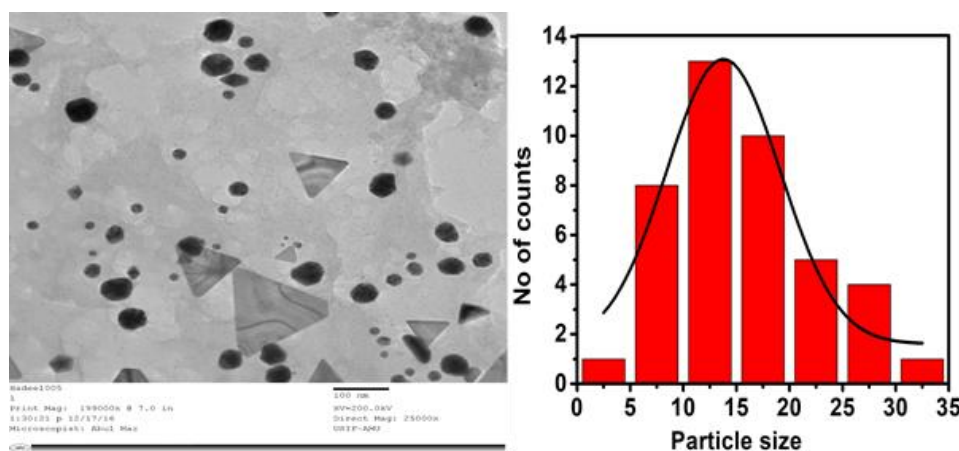


Fig. 3 TEM images of Au-NPs

D. FT-IR spectrophotometer

The FTIR data of Au-NPs which were obtained with the aid of Perkin-Elmer in the range 400–4000 cm^{-1} are shown in Fig 4. FTIR measurement was done to determine the functional groups in the lemongrass leaf that are responsible for both reducing gold ions to Au-NPs and stabilizing the NPs. The FTIR shows the presence of different functional groups, which were identified by their electromagnetic spectra in the IR region. The presence of O-H bond is indicated by a strong and broad band observed at 3,449 cm^{-1} showing the presence of aromatic alcoholic and phenolic compounds [25]. The absorption band at 3040 cm^{-1} indicates the presence of C-H stretching vibrations of alkanes group. The presence of amide group from carbonyl stretch in the plant extract and C-C stretching aromatic ring are indicated by a narrow band at 1680 cm^{-1} and 1480 cm^{-1} respectively [26]. The band at 1219 cm^{-1} indicates the presence of C-N stretching of aliphatic amines, while the weak band at 725 cm^{-1} shows the presence of alkyl halides. Hence, the reduction and stabilization of gold nanoparticles (Au-NPs) were due to the presence of alcoholic, carboxylic, and phenolic compounds in the plant extract [27].

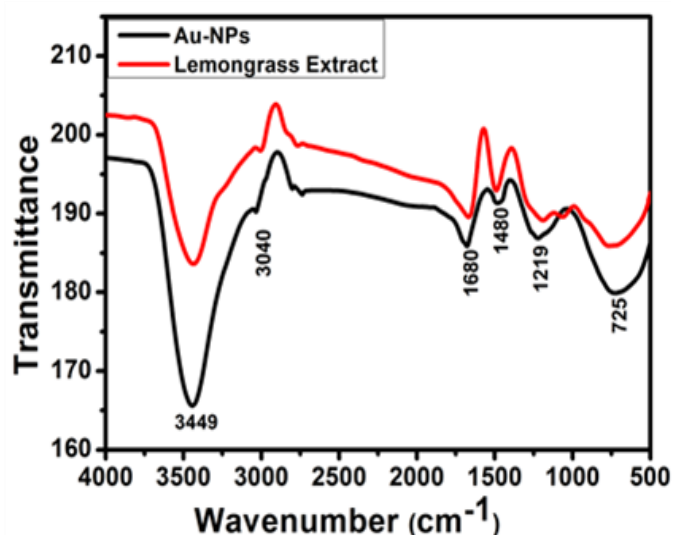


Fig. 4 FT-IR spectra of Au-NPs

E. UV-visible Spectroscopy

The UV-vis spectral analysis was used to observe the synthesised Au-NPs and stability of the NPs in the colloidal solution. The UV-vis spectra of Au-NPs synthesized from lemongrass are shown in Fig 5. The optical properties of metallic nanoparticles are commonly determined by UV-visible absorption spectroscopy because the absorption bands give the precise diameter and aspect ratio of nanoparticles. The optical properties of the Au-NPs were obtained in the range of 400-800 nm. In this range (400-800nm), no absorption peaks were observed for leaf extract but a distinct peak was observed at 543nm for the mixture of leaf extract and HAuCl₄ solutions [28].

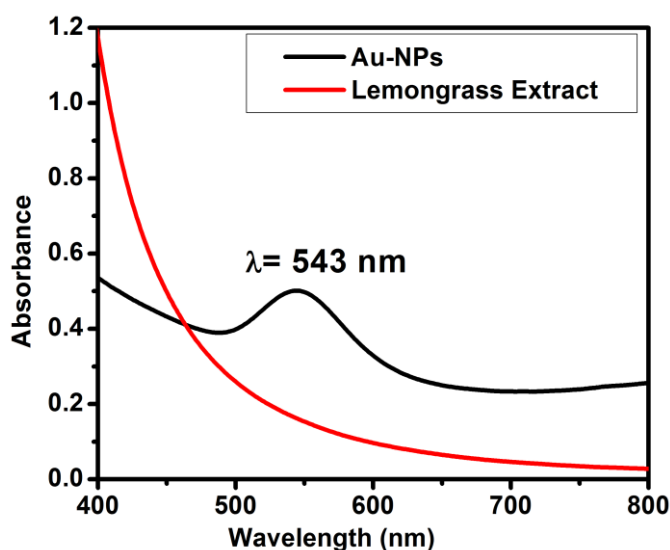


Fig. 5 Absorbance spectra of Au-NPs

F. Photoluminescence spectroscopy

The photoluminescence spectra (PL) show the ability of the NPs to absorb incident energy. Photoluminescence spectrum is used to analyse energy levels, and defects in nanoparticles (NPs), which can be used to obtain their optical and electronic properties [26]. Fig.6 shows the PL spectra of the Au-NPs at room temperature. The electrons in 5d (valence) and 6sp (conduction) orbitals account for optical properties of the Au-NPs. The valence s and d electrons of the each atom consist of a conduction or sp band, and five d bands that are flat and located a few eV below the Fermi level [29]. The photoluminescence Au-NPs were excited with a laser at 405 nm, and emission spectra were observed for both leaf extract and Au-NPs at 519 nm. The green emission Au-NPs is attributable to oxygen vacancies [30].

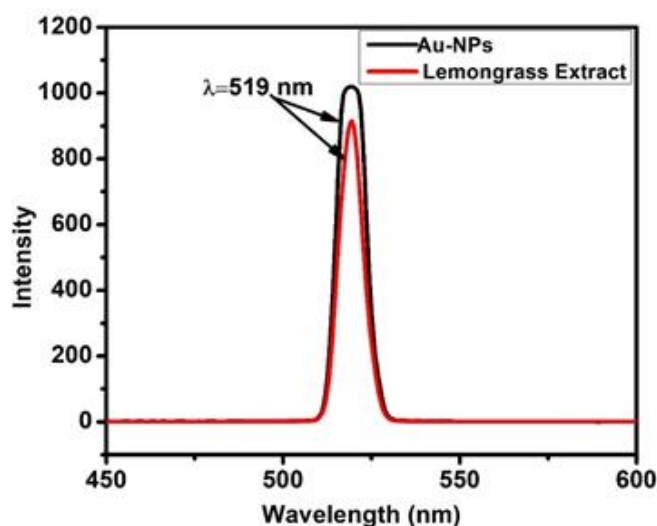


Fig. 6 F. Photoluminescence spectra of Au-NPs

V. CONCLUSIONS

In this study, lemongrass extract was successfully used to biologically synthesize Au-NPs. The XRD data was used to examine the structure of the synthesized Au-NPs. Crystalline size of the Au-NPs was determined to be 12 nm using Debye Scherrer's equation. The shapes of the synthesized Au-NPs were analyzed with the SEM, while the EDAX was used to determine the elemental constituents of the NPs, which was found to contain Au, Cl, Na, Si, Mg, Ca, K, C and O ions. The TEM was used to study the spherical and triangular shapes of the Au-NPs. TEM analysis was used to obtain the average particle size of the NPs, which lies between 10-15 nm. The UV-vis spectroscopy was used to obtain the absorption peak of Au-NPs at 543 nm. Lastly, the functional groups in the plant extract that account for the formation of the gold nanoparticles were determined by the FTIR spectra.

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