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Green Synthesis of Copper Nanoparticles using Cassia Auriculata Leaves Extract

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Abstract : Knowing the morphology of Copper nanoparticles was confirmed by Field Emission Scanning Electron microscopy (FE-SEM). Herein, we are reporting a novel biological approach for the formation of copper nanoparticles using *leave*. Copper sulphate was made to reduce with aqueous solution of Cassia auriculata leave extracts. X-Ray diffraction (XRD) pattern reveals the formation of Copper nanoparticles, which shows crystallinity. Field emission scanning electron microscopy (FE-SEM) suggested particles size and spherical shape in the range of 38.1-43.5 nm. UV absorption studies of copper naoparticles showed that have absorbance in the range 488.5 to 514.3 nm and FT-IR studies of copper nanoparticles were showed that the stretching frequency of finger print region at 509.21 cm^{-1} for Cu-O bond.

Keywords: Cassia auriculata leaves, Copper nanoparticles, UV-Vis, FT-IR, XRD, FE-SEM.

Introduction

Avaram (*Cassia auriculata Linn*), family Caesalpiniaceae, is also known as Avaram tree, The leaves are alternate, stipulate, paripinnate compound, very numerous, closely placed, rachis 8.8-12.5 cm long, narrowly furrowed, slender, pubescent with an erect linear gland between the leaflets of each pair. Leaflets are very shortly stalked 2-2.5 cm long 1-1.3 cm broad, slightly overlapping, oval oblong, obtuse at both ends, mucronate, glabrous or minutely downy, dull green, paler beneath, stipules very large, reniform-round, produced at base on side of next petiole into a filliform point and persistent. Plants have great potential uses, especially as traditional medicine and pharmcopoeial drugs. A large proportion of the world population depends on traditional medicine because of the scarcity and high costs of orthodox medicine [1]. Medicinal plants have provided the modern medicine with numerous plant-derived therapeutic agents [2]. Many plants contain a variety of phyto pharmaceuticals, which have found very important applications in the fields of agriculture, human and veterinary medicine. Natural products play a dominant role in the development of novel drug leads for the treatment and prevention of diseases [3]. It is one of the principle constituents of 'Avaarai panchaga chooranam'- an Indian herbal formulation used in the treatment of diabetes to control the blood sugar level [4]. The plant has been reported to possess antipyretic [5], hepatoprotective [6], antidiabetic, antiperoxidative and antihyperglyceamic [7], microbicidal [8] and antihyperlipidaemic activities [9]. The flowers are used to treat urinary discharges, nocturnal emissions, diabetes and throat irritation [10]. They are one of the constituent of polyherbal formulation 'Diasulin' in the concentration range of 40 mg/dl which is proven to have antidiabetic activity [11- 12].

Experimental Methods

a) Materials

Cassia auriculata leaves (collected from the Jameen Nathampatti village, Rajapalayam, virudhunagar district) and double distilled water used for the preparation of aqueous extract.

b) Methods [13-14]

i) Preparation of Cassia Auriculata Leaves Extract in Aqueous Medium

Cassia auriculata leaves were cleaned thoroughly and then sun dried for 5 – 7 days. Dried leaves were grinded to fine powder. The aqueous extract was prepared by refluxing 30gm of leave powder and 300ml of distilled water by using soxhlet apparatus at 100^0C for 5 hours. Then, the extracts were collected in an airtight bottle and were kept in deep freezer for further use.

ii) Synthesis of Copper Nanoparticles from Leave Extract

The 10 ml of *Cassia auriculata* leaves extract was added to 40 ml aqueous solution of 1 mM copper sulphate in a conical flask for reduction into Cu+ ions and kept for incubation (darkroom) at room temperature. Here the filtrate acts as reducing and stabilizing agent for 1mM of $CuSO_4$. Suitable controls (40ml distilled water + 10 ml Plant extract) in another test tube were maintained throughout the experiments. Reduction of copper sulphate to copper ions was identified by the color change from light yellow color of the extract to dark brown color as given in Figure- 2a & b. A control setup was also maintained without adding copper sulphate to the plant extract. The formation of Copper nanoparicles was further confirmed by spectral analysis.

Figure – 2(a)

Figure – 2(b)

Figure-2(a) & 2(b): Photographs showing, a) Cassia auriculata leaves extract b) Color changes after adding leaf extract to $CuSo_4$ solution.

iii) Characterization of Copper Nanoparticles

a) Ultraviolet Visible Spectrophotometer (UV-DRS)

The UV-DRS spectrum was recorded in Shimadzu UV-2400PC series. Absorption spectra of copper nanoparticles formed in the reaction media have absorbance peak in the range 488.5 to 514.3 nm (**Figure-3**).

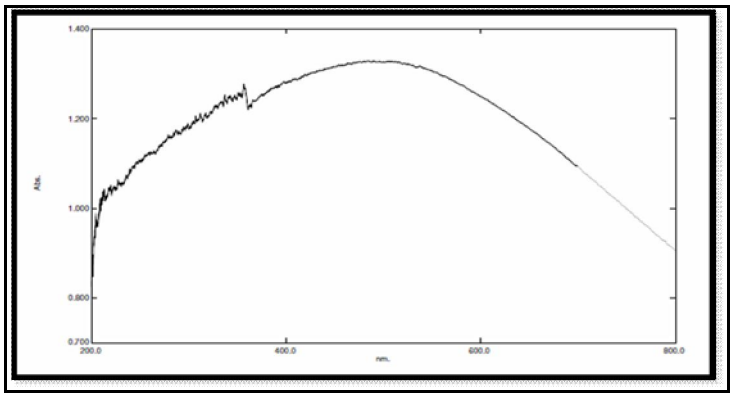

Figure-3: UV-Visible spectrum of Copper Nanoparticles

b) Fourier Transform - Infrared Spectroscopy (FT-IR)

The FT-IR spectrum was recorded in Shimadzu FT-IR spectrometers 7600, Measurement of the samples were performed in transmission mode. In order to identity the binding groups of the plant extract with copper, FT-IR spectra of Cassia auriculata and copper nanoparticles were recorded. On comparing the IR stretching frequency of the functional groups present in Cassia auriculata **(Figure-4)** and copper nanoparticles **(Figure-5)** it was revealed that copper binds to oxygen moiety indicated by M-O stretching at 509.21 cm^{-1}.

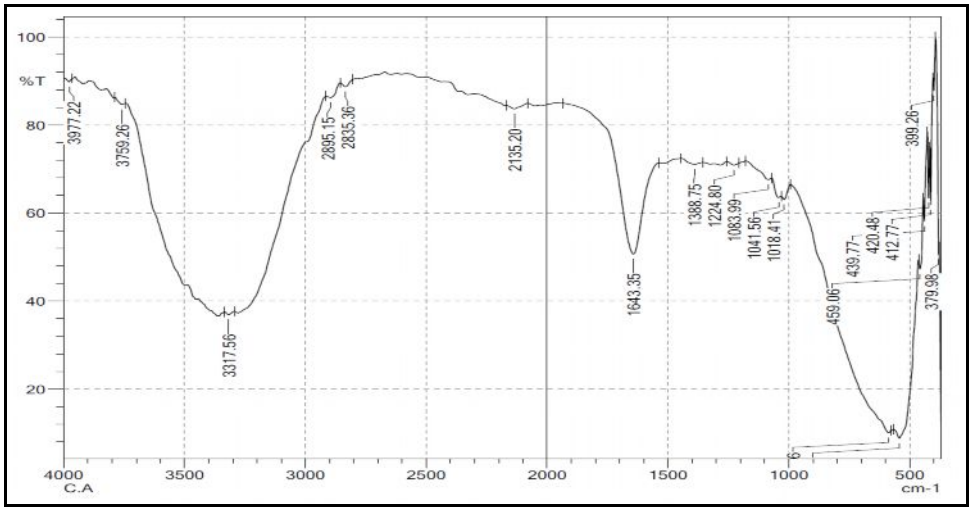

Figure-4: FT-IR Spectrum of Cassia Auriculata

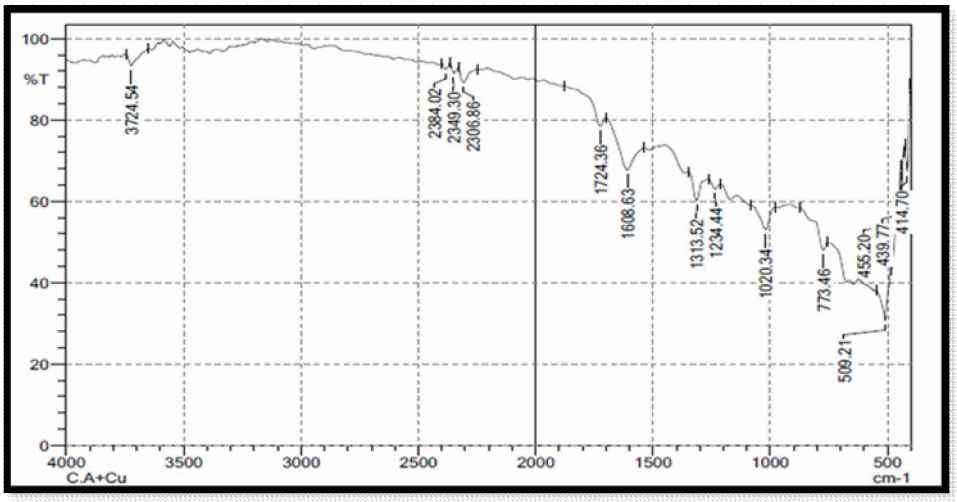

Figure-5: FT-IR Spectrum of Copper Nanoparticles

c) X-Ray Diffraction (XRD)

For the XRD (BRUKER ECO D8 ADVANCE) spectral analysis, prepared particles in solution was purified by centrifugation at 5000 rpm for 20 minutes. An XRD spectrum was recorded at Kalasalingam University and the spectra and their values were given in (**Figure-6**). The dried mixture of copper nanoparticles collected for the formation of Cu nanoparticles by X- pert pro X-ray diffractometer operated at a voltage of 40 kV and a current of 20 mA with Cu Kα radiation in θ-2θ configuration. Morphology of the interplannar distance spacing was calculated using Bragg's equation.

$n\lambda = 2d \sin \theta$

In the XRD curves of copper nanoparticle1s intense and sharp peaks were observed at 24^0, 31.9^0, 42^0, 52.4^0, 58^0 and 63.2^0.

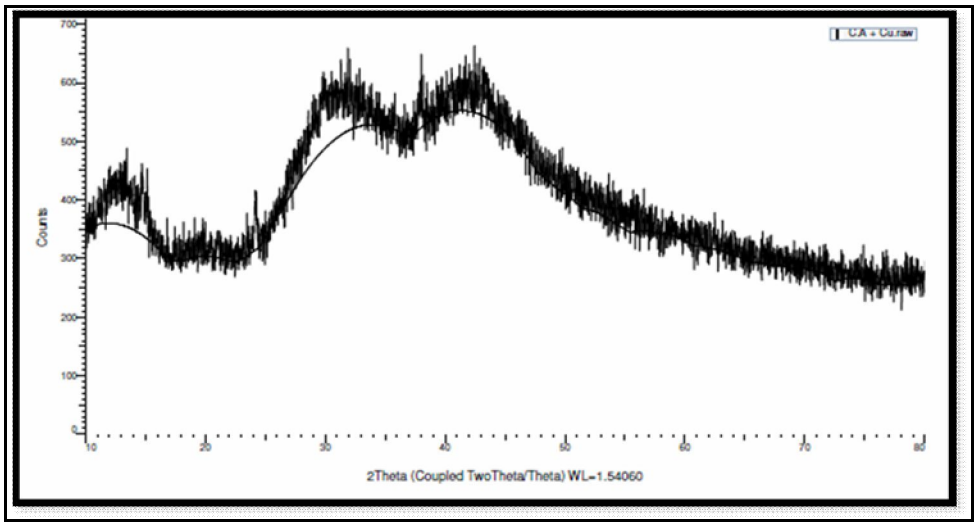

Figure-6: XRD Spectrum of Copper Nanoparticles

d) Field Emission Scanning Electron Microscope (FE-SEM)

The copper nanoparticles sizes were determined using Field Emission Scanning Electron Microscope. The FE-SEM images of copper nanoparticles at 50°C. The formation of copper nanoparticles as well as their morphological dimensions in the FE-SEM study demonstrated that the average size was from 38.1 -43.5 nm with interparticle distance, whereas the shapes were uniformed spherical (**Figure-7)**. However, further observation with high magnification reveals that these Cu nanoclusters were assembled by smaller nanoparticles, which exhibit good uniformity and the average diameter is about 38.1 nm.

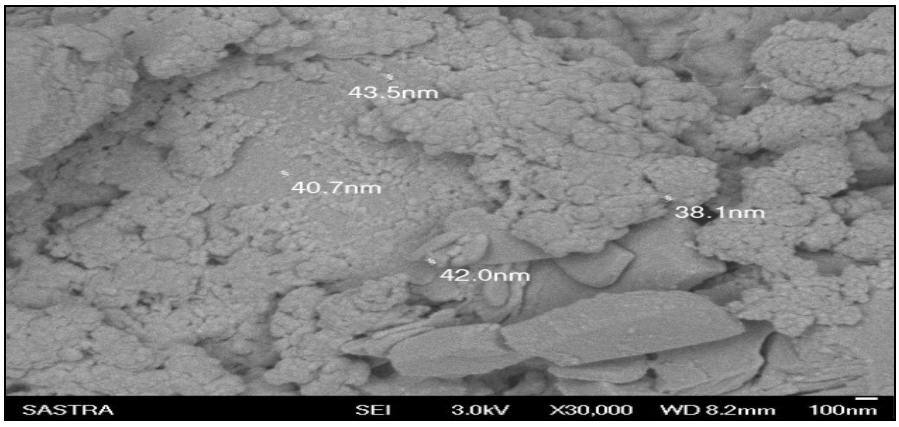

Figure-7: FE-SEM images of Copper Nanoparticles

Result and Discussion

Green synthesis of copper nanoparticles using Cassia auriculata leaves extract was first identified by color changes of the extract from light yellow to dark brown color. Reduction Cu^{+} ion into copper nanoparticles during exposure to the leaves extract was observed as color changes (as given in **Figure-2a&b)**. Secondly copper nanoparticles were confirmed by the spectral studies like UV-Visible Spectroscopy, FT-IR Spectroscopy, X-Ray Diffraction and Field Emission Scanning Electron Microscopy.

a) Ultraviolet Visible Spectrophotometer Analysis (UV-DRS)

It is generally analysed that UV-Visible Spectroscopy could be used to examine formation of copper nanoparticles. Absorption spectra of copper nanoparticles formed in the reaction media have shown the absorbance peak in the range 488.5 to 514.3 nm.

b) Fourier Transform - Infrared Spectroscopy Analysis (FT-IR)

On comparing the FT-IR spectra of Cassia auriculata leaves extract (**Figure-4)** and the copper nanoparticles (**Figure-5)** revealed the following observation. The FT-IR spectra of Cassia Auriculata leave extract showed a broad band at 3317 cm^{-1} indicating the presence of bonded – OH group. Stretching at 1643 cm^{-1} indicated the presence of >C=O group. The FT-IR spectra of copper nanoparticles showed the following stretching frequencies **(i)** at 1608 cm^{-1} (due to carbonyl stretching **(ii)** 509.21 cm^{-1} (due to formation of Cu-O bond).

On comparing the IR spectra of both the extract and its assisted copper nanoparticles formation revealed the following observation. During the formation of copper nanoparticles assisted by Cassia Auriculata, the O-H bonds present in the extract (observed by 3317 cm^{-1}) diminishes that showed that the O-H group oxygen may binds to copper in copper nanoparticles.>C=O stretching frequency at 1643 cm^{-1} of the extract shifted to 1608 cm^{-1} in copper nanoparticles that showed that this may enter into the binding with copper in copper nanoparticles. M-O stretching at 509.21 cm^{-1} also confirms the existence of Cu-O bond. Hence these observations indicated the formation of copper nanoparticles binding strongly to the oxygen atom present in the phytoconstituents of Cassia Auriculata extract.

c) X-Ray Diffraction (XRD)

Copper nanostructure was confirmed by the characteristic peaks observed in the XRD pattern. The analysis was carried out 2θ value ranging from 10^0 to 80^0, with step size 0.020. All diffraction peaks correspond to the characteristic face centered cubic observed at 2θ angle at 24^0, 31.9^0, 42^0, 52.4^0, 58^0 and 63.2^0 respectively. Morphology of the interplannar distance spacing was calculated using Bragg's equation **($n\lambda = 2dSin\theta$)**.

d) Field Emission Scanning Electron Microscope (FE-SEM)

The formation of copper nanoparticles as well as their morphological dimensions in the FE-SEM observation showed that the average size was from 38.1-43.5 nm with interparticle distance, whereas the shapes were uniformed spherical (as given in **Figure-7**). However, further observation with high magnification reveals that these Cu nanoclusters are assembled by smaller nanoparticles, which exhibit good uniformity and the average diameter is about 38.1 nm.

Conclusion

Green synthesis of copper nanoparticles using Cassia auriculata leaves extract revealed the following observation. The formation of copper nanoparticles was confirmed by color changes from light yellow color to dark brown color for Cassia auriculata leaves extract and the characterized UV-Vis, FT-IR spectral studies and their structure space and size by XRD and FE-SEM analysis. UV absorption studies of copper naoparticles showed that have absorbance in the range 488.5 to 514.3 nm and FT-IR studies of copper nanoparticles were showed that the stretching frequency of finger print region at 509.21 cm^{-1} for Cu-O bond. Copper nanostructure was confirmed by the characteristic peaks observed in the XRD pattern. All diffraction peaks correspond to the characteristic face centered cubic observed at 2θ angle at 24^0, 31.9^0, 42^0, 52.4^0, 58^0 and 63.2^0 respectively. Morphology of the interplannar distance spacing was calculated using Bragg's equation **($n\lambda = 2dSin\theta$)**. FE-

SEM observation with high magnification reveals that these Cu nanoclusters were assembled by smaller nanoparticles, which exhibit good uniformity and the average diameter was about 38.1 nm.

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