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Synthesis, Characterization and Electrochemical Behaviour of Metal Oxide Nanoparticles from Cressa Cretica Whole Plant

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Abstract:-- Copper Oxide nanoparticles were synthesized by using cressa cretica whole plant extact. The synthesized metal oxide nanoparticles were characterized by using FTIR,SEM,EDAX,XRD and AFM techniques and found to be in the nm range. Cyclic Voltammetric studies exhibit good adherent behaviour on electrode surface and good electroactivity of copper oxide nanoparticles.

Keywords:- *SEM, EDAX, AFM ,Cyclic Voltammetry.*

1. INTRODUCTION

Nanotechnology and Nanoparticles based product and application are increased now a days due to the biological effectiveness. However, it is well known that inorganic nanomaterials are good antimicrobial agents. Current research in bactericidal nanomaterials has opened a new area in pharmaceutical industries[17].Among the various nanoparticles, metal nanoparticles assume special importance because they are easier and cheaper to synthesize and are the most promising in applications[11].

Nanotechnology can be defined as the manipulation of matter through certain chemical and/or physical processes to create materials with specific properties, which can be used in particular applications . A nanoparticle can be defined as a microscopic particle that has at least one dimension less than 100 nanometers in size [15]. Unlike bulk materials, they have unique optical, thermal, electrical, chemical, and physical properties [10] and hence they find a variety of applications in the areas of medicine, chemistry, environment, energy, agriculture, information, and communication, heavy industry, and consumer goods. Conventional nanoparticle synthesis methods like attrition and paralysis have drawbacks such as defective surface formation, low production rate, high cost of manufacturing, and large energy requirement[7].Chemical synthesis methods (e.g., chemical reduction, sol gel technique, etc.) involve the usage of toxic chemicals, formation of hazardous byproducts, and contamination from precursor chemicals [9].

C. cretica L., belonging to the family Convolvulaceae, is a perennial plant with a lifecycle that continues in the summer period when the salt marsh area drains. *C. cretica* is a thermocosmopolitan halophilous species. *C. cretica* usually grows in sandy or muddy saline habitats along the sea coast along with the species*Suaeda maritima, Salicornia europaea, Salsola soda, Limonium vulgares*ubsp. *Serotinum*, and *Crypsis aculeate*[4]. Variation in *Cressa* has been handled in two ways: extreme lumping into the single species *C. cretica*, or extreme splitting of every morphological variant into 19 species[3][13][16]. Those in the New World represent *C. nudicaulis* and *C.* truxillensis[8][14][1]. The two in the Old World, however, are still being placed in a single species, *C. cretica*[2][5].Old World plants are considered one species even though those in Europe, Africa, and Asia are morphologically and geographically distinct from those in Australia. In

this article, a comprehensive account of morphology, phytochemistry, ehthnomedicinal uses, and pharmacological activities are included in view of many recent findings of the importance on this plant.

2. EXPERIMENTAL METHODS

2.1. Preparation of plant sample for experimental studies

The collected whole plants were cut into small fragments and shade dried until the fracture is uniform and smooth. The dried plant materials were granulated or powdered by using a blender and sieved to get uniform particles by using sieve No. 60. The final uniform powder of the plant was used for various experimental studies.

2.2. Preparation of copper oxide nanoparticles

In a typical reaction mixture, 0.1M copper chloride dehydrate solution was prepared in 100ml of distilled water. 10 ml aqueous solution of copper chloride dehydrate $CuCl_2$. $2H_2O$ was treated with 10ml of plant extract and stirred magnetically at room temperature,until the light blue colour changed to light green colour.Then the mixture is heated at 80^0 c for 2 minutes. Afterwards the mixture was treated with 1M sodium hydroxide in drop by drop. As soon as the sodium hydroxide comes in contact copper ions spontaneous change the green mixture to brown precipitate, indicating the formation of water soluble mono dispersed copper oxide nanoparticles. The brown ppt was then taken out and washed repeatedly with deionized water to remove the impurities for the final product.Then a brown powder was obtained after drying at room temperature.

3. CHARACTERIZATION

The electrochemical analyzer, CH Instruments Electrochemical Workstation model 650C was employed for various electrochemical studies The FT-IR spectra were recorded using a Nicolet iS5 instrument. The surface morphology of the nanoparticles was done using Carl Zeiss EVO 18 SEM operating at 15 KV using normal incidence. EDAX measurements were carried out by Quantax 200 with X-Flash-Bruker. XRD measurements were made by Panalytical X'Pert Powder X'Celerator Diffractometer, measurement range: 10 to 80 degree in 2θ and particle size was calculated using Scherrer's equation.

4. RESULT AND DISCUSSION

The characterization results of the synthesized CuO nano particles are described below by various techniques.The results obtained are discussed in detail as follows.

4.1. FTIR Studies:

The IR spectrum was taken using a Nicolet iS5 FT-IR instrument operating at a resolution of 4000-400cm^{-1} in the percent transmittance mode. Generally all the metals and its oxide, give the FT-IR peaks at lower wave number ranging from 400 to 800cm^{-1}.

The FTIR spectrum of CuO nanoparticles synthesized using plant extract was shown in the Fig.1.The broad peak located at 3433.94 cm^{-1} can be assigned to the O–H stretching vibrations, indicating the presence of hydroxyl groups. Few less intense peaks centered at 2494.13 and 2359.82cm^{-1} are probably due to presence of aliphatic asymmetric C-H stretching vibration and O-H stretching in carboxylic acid respectively.The peak at 1439.95 cm^{-1} corresponds to the -C=N- stretching vibrations. The band at 1158.14cm^{-1} corresponds to the presence of water molecule. The peak at 880.31 cm^{-1} was due to the formation of Cu ion. The characteristic peak appeared at 669.98cm^{-1} could be attributed to the metal oxygen (Cu-O) bond. The FTIR analysis of CuNPs suggested that they might surround by the any of these organic molecules such as poly phenols, alkaloids and terpenoids.The chemical constituents present in plant leaves extract such as Flavonoids, alkaloids and fatty acids are responsible for the reduction of copper ions to copper nanoparticles due to their capping and reducing capacity [6].

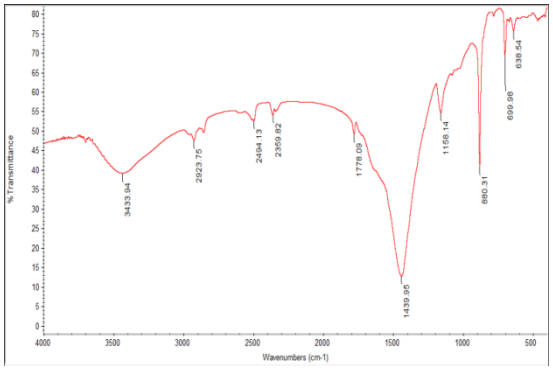

Fig: 1 FT-IR Spectrum of CuO nanoparticles

4.2. XRD Analysis:

The X-ray diffraction pattern of CuO nanoparticles synthesized using plant extract was

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shown in Fig.2. The spectrum of CuO nanoparticles exhibits sharp peaks at 2θ equal to 16.96^0, 29.96^0, 31.66^0, 32.31^0, 35.13^0, 37.94^0, 41.51^0 ,45.04^0 , 55.20^0 and 71.76^0. These peaks are identified to originate from {111}, {122}, {103}, {113}, {340},{321},{410},{420}and {521} planes of the CuO phase respectively. It shows the monoclinic structure and the positions of peaks are in good agreement with the reported values given JCPDS NO. (48-1548). The average crystallite size of CuO nanoparticles is found to be 40.17nm.

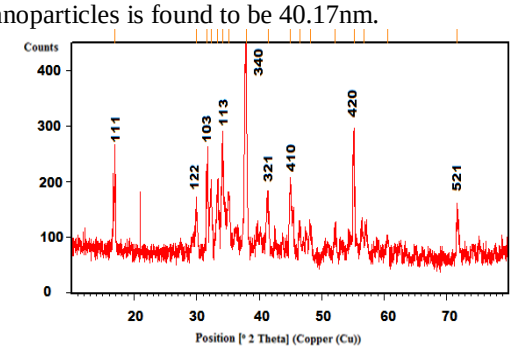

Fig:2 XRD Spectrum of CuO nanoparticles

4.3 SEM Analysis:

Scanning Electron Microscopy was employed to analyze the morphology and the growth features of the as prepared nanoparticles. Fig.3 represents the SEM image of CuO nanoparticles synthesized using plant extract. This picture substantiates the *bundle of rod* shape to the CuO nanoparticles with *granular* nature. From SEM images the crystallite size of CuO nanoparticles synthesized using plant extract was found to be in the nanometer range.

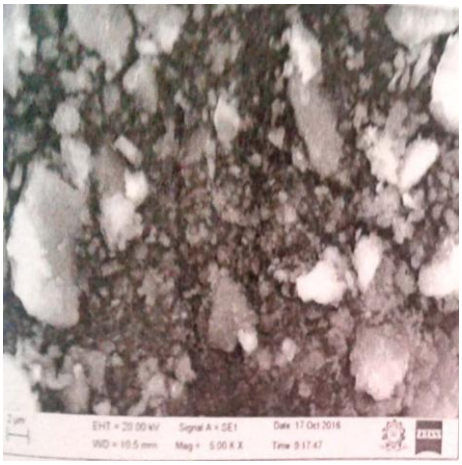

Fig:3 SEM image of CuO nanoparticles

4.4 Energy Dispersive X-ray Analysis:

The elemental composition of the CuO nanoparticles was carried out by EDAX spectroscopy. Fig.4 shows the EDAX spectrum of Copper oxide nanoparticles synthesized using plant extract. Copper oxide nanoparticles were found to have atomic percentage 79.42 of Cu, 20.48 of O, as

shown in Table.1 This confirmed the presence of Cu and O.

Table.1: Atomic composition of CuO nanoparticles synthesized using plant extract

Element	Unn. C [wt. %]	Norm. C [wt.%]	Atom.C [at.%]	Comp. C	Error(3sigma) [wt.%]
Copper Oxygen	35.72 33.74	51.43 48.57	50.00 50.00	79.42 20.48	3.00 12.54
Total	60.27	100.00	100.00		

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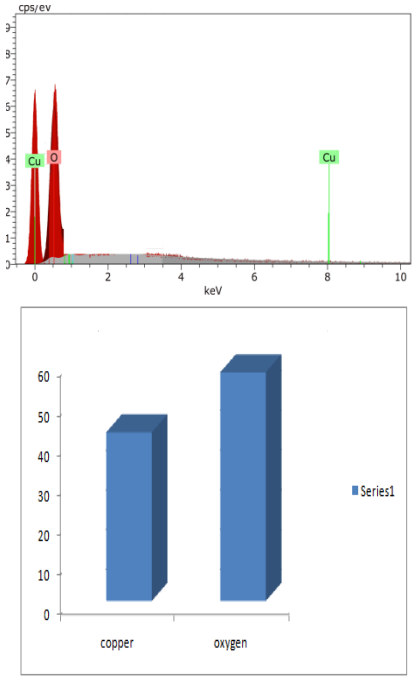


Fig:4 EDAX spectrum of CuO nanoparticles

4.5 AFM ANALYSIS

The shape and particle size of synthesized CuO nanoparticle using plant extract are studied by AFM analysis. AFM spectra were recorded for the CuO nanoparticles deposited on glass plate.Fig:5 shows the AFM image synthesized CuO nanoparticle using plant extract with a scanning area to 0m to 3.13 µm,we found sperical shape distributed over the surface[12].These particles are between 0mY 3.13µm in length and size is in the range of 20-50nm.

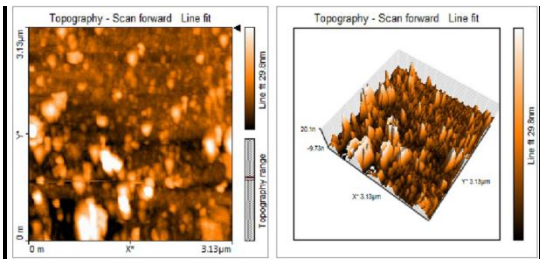
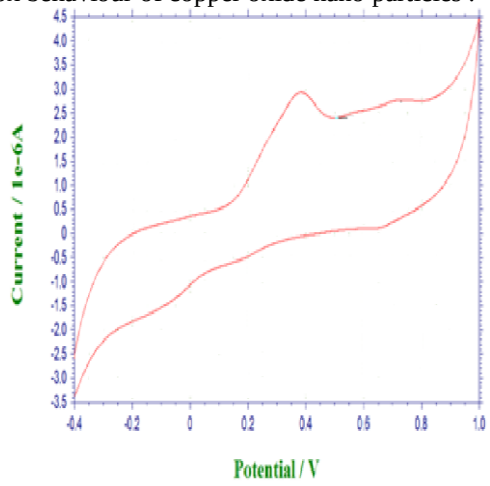


Fig:5 AFM spectrum of CuO nanoparticles

4.6 Cyclic Voltammetry

Cyclic voltammetric behaviour of the nano oxides are recorded.The potential window is between -0.8 to 1 V on GCE at 0.05v/s.Cyclic voltammetric behaviour of CuO showed two oxidation peak(Fig:6) at 0.6679V and 0.3304V which is due to the presence of CuO.

Cyclic voltammetric behaviour of nano copper oxide showed one reduction peak (Fig:6) at 0.6662V is observed.Cyclic voltammetric behaviour of CuO nanoparticle at different scan rates are shown in (Fig:7). These facts revealed that the voltammetric redox behaviour of copper oxide nano particles .



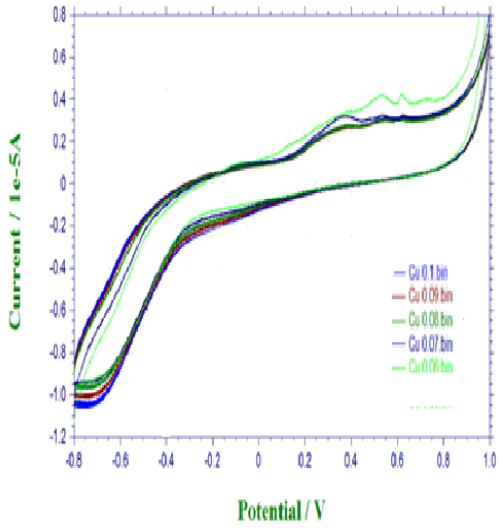


Figure:6 Cyclic Voltammogram of CuO nanoparticles Figure:7 Cyclic Voltammogram of CuO nanoparticles at different scan rates

CONCLUSION

Cressa cretica belongs to the family Convolvulaceae is known Uppu marikozhunthu in tamil. The present investigation was carried out to determine the

nanoparticles synthesis. FTIR, XRD, SEM, EDAX, AFM and CV analysis proved that the prepared CuO nanoparticle is in the nm range.FT-IR spectral results revealed that the presence of Cu-O bond of nano oxide.XRD behaviour also suggested oxide nano particles are in the nano scale range.The surface morphology of the synthesized oxide nano particles exhibited different structures. The crystallite size of the synthesized nano Cu-O is determined to be nm range.EDAX spectroscopy confirmed the presence of Cu and O metal oxides.

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