REST Journal on Emerging trends in Modelling and Manufacturing
Vol: 5(2), 2019
REST Publisher
ISSN: 2455-4537

Website: www.restpublisher.com/journals/jemm

Effect of Nyctanthes Arbon- Tristis leaf extract in the properties of CuO Nanoparticles

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Abstract

In recent years, green synthesis of nanoparticles has received considerable attention due to the growing need to develop clean and nontoxic chemicals, low-cost approaches, eco - friendly solvents and renewable materials. In the current study, the green synthesis of copper oxide nanoparticles was attained by a solvothermal method. Copper oxide NPs were characterized by X-ray Diffraction (XRD), UV-Visible Spectroscopy (UV-Vis).

Keywords: Nyctanthes Arbon-Tristis, Green synthesis, copper oxide Nanoparticles, XRD, UV-Vis.

1. Introduction

Nanomaterials are cornerstones of nanoscience and nanotechnology. Nanostructure science and technology is a broad and interdisciplinary area of research and development activity that has been growing explosively worldwide in the past few years. It has the potential for revolutionizing the ways in which materials and products are created and the range and nature of functionalities that can be accessed. It is already having significant commercial impacts, which will assuredly increase in the future [3].

Copper oxide nanoparticles: Copper oxide (CuO), having variety of chemo physical properties, is recently attractive in many fields such as energy conversion, optoelectronic devices, and catalyst. Compared with bulk material, the advanced properties of CuO nanostructures have been demonstrated; however, the fact that these materials cannot yet be produced in large scale is an obstacle to realize the potential applications of this material. CuO, categorized into transition metal oxide group, is a p-type, narrow bandgap semiconductor. It has many interesting characteristics: super thermal conductivity, photovoltaic properties, high stability, and antimicrobial activity.

Green synthesis of nanoparticles: Green synthesis is required to avoid the production of unwanted or harmful by-products through the build-up of reliable, sustainable, and eco-friendly synthesis procedures. The use of ideal solvent systems and natural resources is essential to achieve this goal. Green synthesis of metallic nanoparticles has been adopted to accommodate various biological materials. Among the available green methods of synthesis for metal/metal oxide nanoparticles, utilization of plant extracts is a rather simple and easy process to produce nanoparticles at large scale relative to bacteria and/or fungi mediated synthesis. These products are known collectively as biogenic nanoparticles.

Plant leaf extract-based mechanism: For nanoparticle synthesis mediated by plant leaf extract, the extract is mixed with metal precursor solutions at different reaction conditions. The parameters determining the conditions of the plant leaf extract (such as types of phytochemicals, phytochemical concentration, metal salt concentration, pH, and temperature) are admitted to control the rate of nanoparticle formation as well as their yield and stability. The phytochemicals present in plant leaf extracts have uncanny potential to reduce metal ions in a much shorter time as compared to fungi and bacteria, which demands the longer incubation time. Therefore, plant leaf extracts are considered to be an excellent and benign source for metal as well as metal oxide nanoparticle synthesis. Additionally, plant leaf extract plays a dual role by acting as both reducing and stabilizing agents in nanoparticles synthesis process to facilitate nanoparticles synthesis. The composition of the plant leaf extract is also an important factor in nanoparticle synthesis, for example different plants comprise varying concentration levels of phytochemicals [7].

2. Materials and Methods

Synthesis of Copper Oxide nanoparticles:

Copper oxide nanoparticles have been synthesized by a variety of methods. In the present study we have attempted to prepare copper oxide nanoparticles using solvothermal method mediating green.

Solvothermal method: In solvothermal process the chemical reaction performed in a fixed vessel for example, bomb or autoclave, where solvents are conveyed to temperatures well above their boiling points. In solvothermal method dissolves precursor and reducing agent in a beaker then place in an autoclave after the formation of nanoparticles characterize it. Solvothermal is a versatile, convenient and straight forward method using only a polar, oxygen-containing solvent and by applying heat but synthesis in closed vessels, so temperature-pressure-volume considerations are critical [6].

Figure 1: Schematic representation of solvothermal process of synthesis of nanomaterials

Reagents and chemicals: Copper acetate, urea and ethylene glycol were obtained from nearby chemical store of Nagercoil, Kanyakumari district. Nyctanthes Arbon-Tristis leaves were collected from my garden at Ramanputhoor, Nagercoil.

Preparation of extracts: A 6g of Nyctanthes Arbon-Tristis was thoroughly washed two or more times with distilled water to remove dust particles and then chopped finely and crushed using a mortar and pestle. The extract obtained was filtered through a filter paper. The plain filtrate becomes useful for the synthesis of CuO nanoparticles [8].

Preparation of pure copper oxide nanomaterials: In the present study, pure Copper oxide nanoparticles has been prepared using Solvothermal method. 10g of Copper (11) acetate monohydrate is completely dissolved in 50 ml of Ethylene glycol and 9 grams of Urea is mixed with 50 ml of Ethylene glycol. The precursors are mixed together and heated in microwave oven for nearly half an hour to obtain pure copper oxide nanoparticles. Then separate this nanoparticle using what man filter paper and wash the resultant sample using distilled water repeatedly to remove the by-products, dry the sample for few days again its washed using acetone to remove the impurities. The samples were filtered and allowed to dry. The prepared samples were subjected to various studies.

Addition of leaf extract: The Nyctanthes Arbon-Tristis extract was added to the Pure Copper oxide and mixed CuO samples were prepared by solvothermal method. Dopants of different concentration in 3ml and 6ml of Nyctanthes Arbor-Tristis leaves extract were prepared. The so prepared solution was allowed to settle, after a minute the settled precipitate was washed with distilled water and was allowed to dry for a week. After a week, the dried sample was again washed with acetone to remove impurities. The samples were filtered and allowed to dry. The prepared samples were subjected to various studies.

3. Results and discussion

The results obtained in the present study are reported and discussed here. All the prepared samples are in powder form whereas pure CuO is pure black color and Nyctanthes Arbor-Tristis dispersion changes the color from pure black to dark brownish colored samples. The photographs of the three samples are shown in the figure. The prepared samples are pure CuO, 3ml Nyctanthes Arbor –tristis mixed CuO and 6ml Nyctanthes Arbor-Tristis mixed CuO.

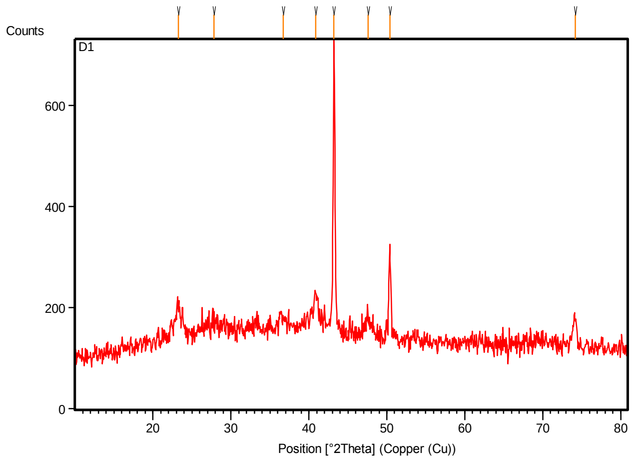
Fig 3.1: pure CuO nanoparticle Fig3.2:3ml Nyctanthes Arbor-tristis mixed CuO; Fig 3.3: 6ml Nyctanthes Arbor –tristis mixed CuO

Structural properties:

XRD ANALYSIS:

X-ray powder diffraction (XRD) is a rapid analytical technique primarily used for phase identification of a nanoparticle material and can provide information on unit cell - dimensions. The analyzed material is finely ground, homogenized, and average bulk composition is determined. X-ray diffraction technique for the investigation of the fine structure of the matter. It has widely used for the determination of crystallinity. Crystal structure and lattice constants of prepared copper nanoparticle. The structure analysis of the prepared CuO nanoparticle was done by X- ray diffraction. The prepared copper oxide powders were characterized by X-ray power diffraction meters using CuKλ radiation (wave length λ=0.15406nm). X-ray diffraction pattern is recorded for the as prepared and copper oxide nanopowders prepared from solvothermal method.

In x-ray diffraction, the Scherer formula that relates the size of sub-micrometer particles, or crystallites, in a solid to the broadening of a peak in a diffraction pattern. It is used in the determination of size of particles of crystals in the form of powder. CuO nanoparticles exhibited Cubic crystal structure. The peak intensities are in agreement with the JCPDS. All diffraction peaks correspond to the characteristic face centered cubic (FCC) copper lines.



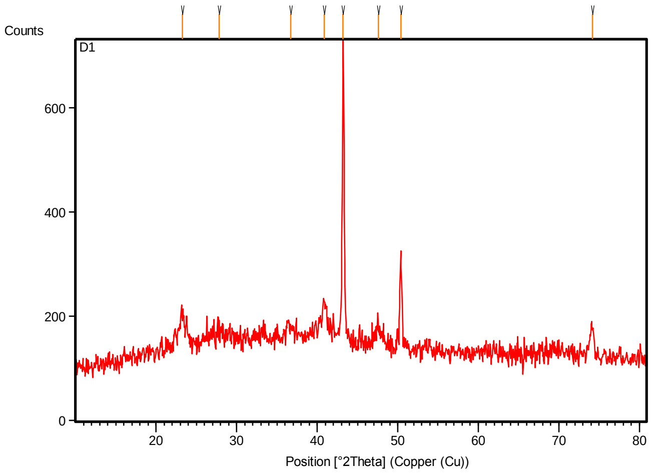


Fig 3.4: XRD spectrum for pure CuO nanoparticle; Fig 3.5: XRD spectrum for 3ml Nyctanthes Arbor –tristis mixed CuO nanoparticle

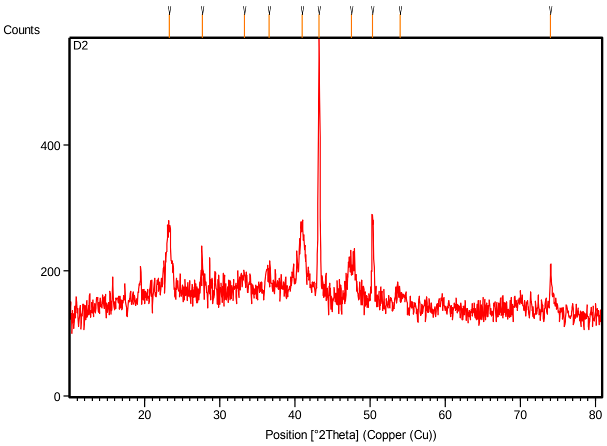


Figure 3.6: XRD spectrum for 6ml Nyctanthes Arbor –tristis mixed CuO nanoparticle

Crystalline size: A crystallite is a domain of solid – state matter that has a same structure as a single crystal. If the individual crystallites are oriented randomly, a poly crystalline structure is involved and if they are oriented in a particular plain, a single crystalline structure is formed. The crystalline size of the film is calculated using Debye Scherer formula by measuring FWHM from the diffraction pattern. The smallest diffraction domain in a specimen is known as a crystallite. If the crystallite size is in nanometers, it will be broadening the XRD peak due to incomplete destructive interference at angle near the Bragg's angle. The average size of CuO nanoparticles is calculated using the formula, $D = k\lambda/\beta cos\theta$

Where, D is the average crystallite size of the particles, K is Debye-Scherer's constant (0.94), λ is the wavelength of the cuK α-radiation (1.5406 Å), β is the full width half maximum (FWHM) of the peak, θ is the Bragg`s angle.

Samples	Crystalline size
Pure CuO	19.7616 nm
3ml Nyctanthes Arbor Tristis	19.3164 nm
6ml Nyctanthes Arbor Tristis	21.6248 nm

Table 1: Crystalline size of samples

Lattice parameter:

Lattice parameter refers to the constant distance between units in a crystal lattice. The unit cell parameters are determined for the cubic structure using the following relation connecting the inter planar distance 'd' and miller indices (h k l) for a=b=c. $$\frac{1}{d^2} = \frac{h^2 + k^2 + l^2}{a^2}$$

Using the above relation, the unit cell parameters of Cuo nanopowders are calculated for the intense peak as a=b=c=4.1849Å. The obtained lattice parameters are compared with standard JCPDS values as a=b=c=4.245Å. The required hkl values are taken from the JCPDS card. It is observed that unit cell parameters matched well with the standard values and tabulated.

Samples	**Lattice parameter, Å a=b=c**		
	Calculated values (a=b=c)	Standard JCPDS value	Structure
Pure CuO	4.1842	4.245	Cubic
3ml Nyctanthes Arbor Tristis mixed CuO	4.1858		
6ml Nyctanthes Arbor Tristis mixed CuO	4.1865		

Table 2: Lattice parameter of CuO nanoparticles

Unit cell volume:

The simplest repeating unit in a crystal is called a unit cell. Unit cell volume can be calculated using lattice parameters. The required (hkl) parameters are taken from JCPDS card. It is observed that the unit cell parameters are nearly in match with the standard values. The unit cell volume of the cubic fluorite structured of CuO nanomaterials are calculated using the formula, $\mathbf{V=a^3\ (Å)^3}$

It is observed that unit cell volume matched well with the standard values and tabulated.

Samples	**Unit cell volume** $(Å)^3$		
	Calculated values	Standard JCPDS value	Structure
Pure CuO	73.255	76.465	cubic
3ml Nyctanthes Arbor Tristis mixed CuO	73.339		
6ml Nyctanthes Arbor Tristis mixed CuO	73.375		

Table 3: Unit cell volume of CuO nanoparticles

Optical properties:

ULTRAVIOLET-VISIBLE ANALYSIS:

Ultraviolet-visible spectroscopy or ultraviolet-visible spectrophotometers refers to absorption spectroscopy or reflectance spectroscopy in the ultraviolet-visible spectral region. This means it uses light in the visible and adjacent (near-UV and near-infrared [NIR]) ranges. The absorption or reflectance in the visible range directly affects the perceived color of the chemicals involved. This technique is complementary to fluorescence spectroscopy, in that fluorescence deals with transitions from the excited state to the excited state. This technique is based on the absorption of light in the ultra-violet (10-420nm) and visible (420-700nm) regions by a powder sample. From UV spectrum, the absorbance in each sample is studied.

Energy gap determination: Band gap denotes the energy separation between the valence and conduction bands. It can be either a direct band gap or an indirect band gap. A direct band gap means that the minimum energy of the conduction band lies directly above the maximum energy of the valance band. In indirect ban gap, direct transition across the band gap does not conserve momentum and hence forbidden. The optical band gap is determined using Tauc's relation, $\alpha h\upsilon=A(h\upsilon-E_g)^n$
Where, 'A' is a parameter that depends on transition probability,
E_g is band gap energy and n is an index with value ½ and 2 for indirect and direct allowed transitions respectively. The best linear fit was obtained for n=2 which show that there are indirect allowed transition between valence and conduction band. The band gap values are estimated by the straight part of plot between $(\alpha h\upsilon)^2$ and Energy (hυ) eV.

Samples	**Energy Band Gap**
Pure Copper oxide	4.03ev
1:3 ratio Nyctanthes Arbor-Tristis mixed CuO	4.32ev
1:6 ratio Nyctanthes Arbor-Tristis mixed Cu0	4.75ev

Table 4: Energy band gap for various samples

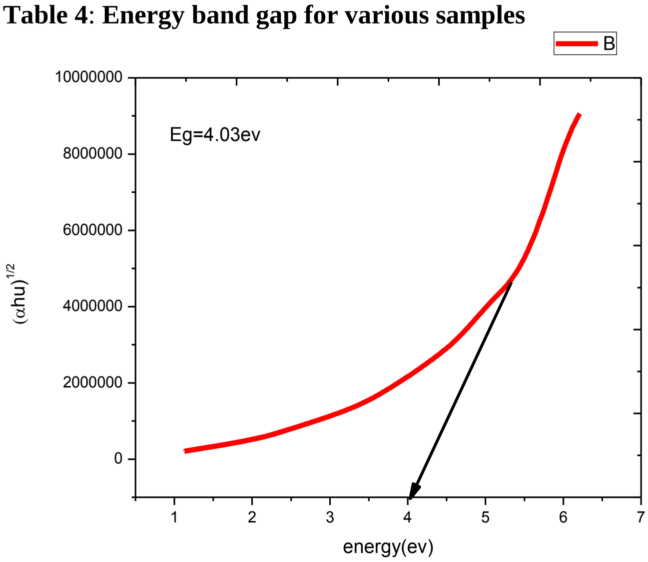
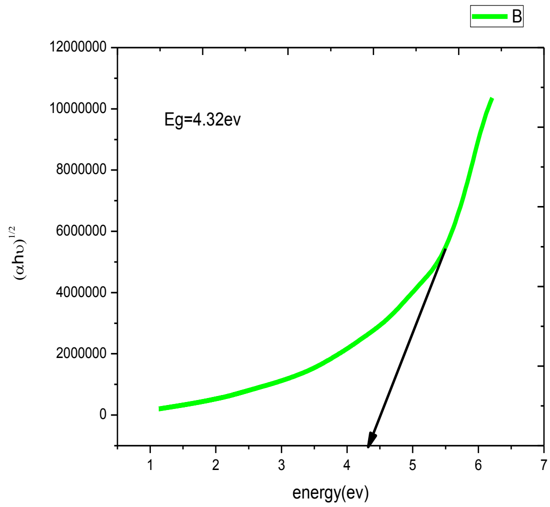


Figure 3.7: Tauc plot of pure CuO nanoparticle



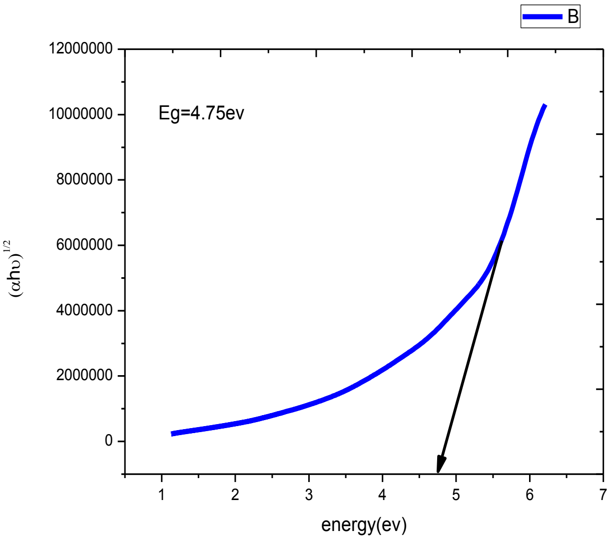


Fig 3.8: Taucplot of 3ml Nyctanthes Arbor-Tristis mixed CuO nanoparticle; Fig 3.9: Taucplot of 6ml Nyctanthes Arbor-Tristis mixed CuO nanoparticle

The absorbance of the pure copper oxide nanomaterial measured in the energy band gap is 4.03eV and the absorbance of the 3ml and 6ml Nyctanthes Arbor-Tristis mixed CuO nanoparticles energy band gaps are 4.32eV and 4.75eV respectively. The optical band energies were calculated using Tauc plots. The results show that the energy band gaps of CuO nanoparticles increases with increasing in concentration. A larger bandgap means that more energy is required to excite an electron from the valance band to the conduction band.

5. Summary and conclusion

Copper and copper oxide nanoparticles have attracted considerable attention because copper is one of the most important in modern technologies and is readily available. There is increasing interest on copper nanoparticles due to their optical, catalytic, mechanical and electrical properties. Copper oxide is widely used in the field catalysis, superconductors and ceramics as a kind of important inorganic materials. Green synthesized CuO nanoparticles have being used as a photo catalyst, enhanced the rates of textile dye decolourisation, anti-microbial agent and better anti-cancer compound with minimal side effects. The present study focused on the synthesis of copper oxide nano particles by green synthesis method. The prepared samples were subjected to structural and optical analysis. The powdered XRD studies revealed that the size of the nano crystals were about 19-21 nm. The optical band gap observed for pure CuO and Nyctanthes Arbor Tristis mixed CuO is comparable with that reported in the literature. The energy band gap values were studied using UV-Vis-NIR spectrometer and the values are obtained as 4-5eV. The energy bandgap increases with decreasing particle size. These type of wide bandgap materials brings the electronic transition energy to the range of the energy of visible light, and they are the key component used to make green and blue LED's and lasers and also in certain radio frequency applications, notably military radars

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