Effect of piper betle leaf extract in the solution of $CuSo_4$-Structural reduction phenomena

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ABSTRACT: *Among various methods in preparing nanomaterials for various applications, an eco friendly method of green synthesis acquired attention of recent researchers of nanomaterials. Aamong various methods the superficial method (Co precipitation method) was considered to prepare copper oxide nanoparticles from the extract of piper betle leaf. The prepared CuO nanoparticle was characterized by using X-ray diffraction (XRD) technique to determine some structural parameters like particle size, dislocation density and micro strain. And it was found from the structural determination that the average particle size of Cuo nanoparticle lies in nono order.*

Key Words: *CuO nanoparticles; Green synthesis; Piper betle; XRD;*

1. Introuduction

Copper oxide is a P-type semiconductor with the band gap of >1.7 eV. It is widely used for field emission emitter, solar cells, heterogeneous catalysts etc. Although various types are followed for the synthesis of copper oxide nanoparticles, most of the methods are much costly and generate toxic wastes to the environment [1-3]. So recent trends is research is preparation of nanoparticles using plant extract. The presence of bio active functional elements in biomaterials acts as reducing groups in green physics.

Piper betle is an important species of the Piperaceae family; it is an evergreen and perennial creeper, with glossy heart-shaped leaves that are magnificent reservoirs of phenolic compounds with antiproliferative, antimutagenic, antibacterial and antioxidant properties[4-5]. Piper betle has various chemical constituent Chavibetol, Chavibetol acetate, Caryophyllene, Allylpyrocatechol Diacetate, Campene, Chavibetol methyl ether etc. Due to its medicinal application and no previous work done on it by researchers, which was considered to prepare CuoNPs in the present research work with the expectations of enhancement in structural phenomena.

2. Materials

Metallic reagent AR grade Cooper Sulphate (fig 1b) and leaf of piper betle (fig 1a) was used to synthesis of Copper oxide nanoparticles.

Figure 1a : Leaf of piper betle

Figure 1 b : Cooper sulphate powder

3. Synthesis of Copper oxide nanoparticles

CuO nanoparticles were synthesized using the co-precipitation method with the leaf extrat of piper betle . The leaf of piper betle were collected and washed thoroughly with distilled water to make them free from dust particles and surface contamination and it was dried well. Then 4.9396g of copper sulphate ($CuSo_4.5H_2O$-) was mixed with 15ml of piper betle leaf extract in 100ml of double distilled water. The solution was stirred for 4hrs using a magnetic stirrer. The obtained precipitate (fig 2) was filtered using whatman filter paper and collected in a petri dish and dried in the hot air oven for one day at 500^0c.

Figure 2 : Reaction process of Piper Betle

Figure 2 : Reaction process of CuONPs

4. X-Ray Diffraction Analysis

X-ray diffraction method is generally used to identify the structure and to calculate various structural parameters of nanomaterials like lattice parameters, particle size, dislocation density and microstrain etc[6]. So the prepared sample of CuONPs were analysed by X-Ray Powder diffractometer (X'pert pro PANalytical) with monochromatic beam of Cu Kα radiation (1.5406 A□) under the accelerating voltage of 40 kV and a current of 30 mA with a scan rate of 0.01° at room temperature.

5. Results and Discussion

XRD pattern of the synthesized copper oxide nanoparticle is depicted in fig 3with its reference peak. The peak intensities gives peak position and also details about the electron density inside the unit cell. The major peaks obtained for CuONPs, corresponding hkl values and the XRD parameters found from XRD analysis is given in table 1. The obtained values are well coincidence with the JCPDS file No: 771898 of CuO .

Table 1 : XRD parameters of CuO nanoparticles

S. No	2θ	Measured d-spacing (Aº)	FWHM (degree)	hkl	Relative Intensity	Particle size	Micro strain ($x10^{-3}$)	Dislocation density (m^{-2}) 10^{15}
1	13.478	6.56	0.2460	(1 1 1)	34.54	33.983459	1.065	0.8658
2	16.092	5.50	0.2460	(0 2 0)	38.64	34.084086	1.062	0.8607
3	22.399	3.96	0.2460	(2 1 2)	75.13	34.403293	1.052	0.8448
4	30.289	2.95	0.2952	(1 1 5)	25.52	29.136056	1.24	1.1777
5	33.059	2.10	0.2460	(2 3 2)	54.34	35.202192	1.02	0.8069
6	34.083	2.63	0.2952	(0 1 6)	21.57	29.405424	1.23	1.1559
7	35.256	2.54	0.2460	(1 4 1)	100.00	35.405424	1.02	0.7977
8	36.103	2.48	0.2952	(3 2 3)	25.02	29.579642	1.22	1.1491
9	37.344	2.40	0.2952	(0 2 6)	18.19	29.686107	1.21	1.1347
10	40.933	2.20	0.2952	(4 2 0)	16.78	30.018091	1.20	1.1097
				Average	32.0910	32.091021	1.13540	0.99039

Thus, the comparison confirms the presence of CuO phases in the present specimen and found to have cubic structure with lattice constant a= 9.740 A°. The average crystalline size was found to be 32.0910 nm using the following Debye Scherrer formula [7].

$$Dhkl = k \lambda / \beta \cos\theta \quad (1)$$

Where, Dhkl is the grain size, K is a dimensionless shape factor (0.94), λ is the wavelength of the X-ray, β is the line broadening at half the maximum intensity (FWHM), θ is the Bragg's angle. The micro structural parameters such as dislocation density,δ and micro strain(ε) have been calculated using the following relations [8].

$$\text{Dislocation density } (\delta) = 1/ D2 \quad (2)$$

$$\text{Micro Strain } (\varepsilon) = \beta \cos\theta / 4 \quad (3)$$

The dislocation density in the sample which means the average dislocations in a unit volume of the crystalline material was found to be 0.99039x10^{15} m^{-2} and the amount of average deformation due to the applied force (micro strain) was found to be 1.13540x10^{-3}.

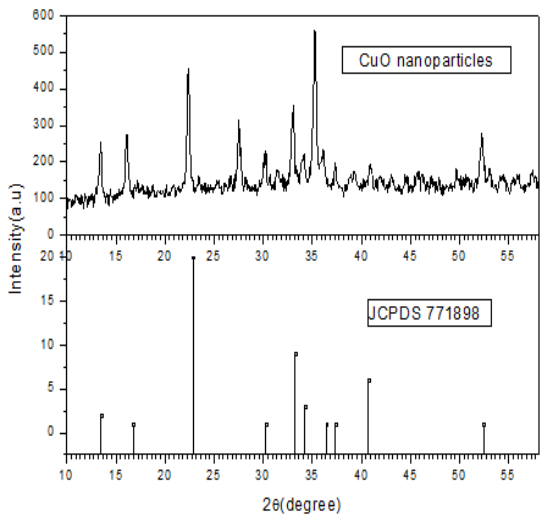

Figure 3 XRD Pattern of CuO nanoparticle

Hall-Williamson plot (figure 4) was also plotted using calculated values of XRD parameters using the relation β cosθ = (0.9 λ/D) + (ηsin)[9].The crystallite size was found to be around 43 nm from this plot . The difference obtained in particle size of the sample by scherrer formula and Hall-Williamson plot may be due to the strain on the nanoparticles[10].

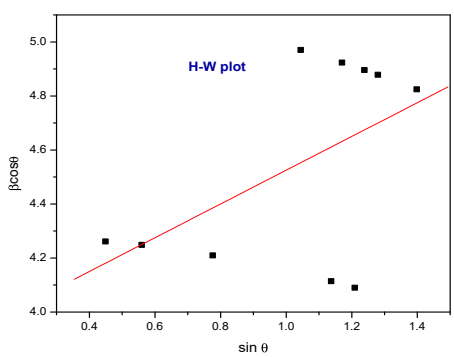

Figure 4: Hall-Williamson plot

Conclusion

The particle size and structure of synthesized nanoparticles were determined from the XRD data. Moreover this bio mediated leaf extract synthesis method represents a considerable improvement in the preparation of CuONPs since it require less time for reaction process and no need of catalytic reducing agent. During the biosynthesis self-assembling of the chemical components of the leaf extracts around the the CuO+ ion is the reason for reduction of CuO+ ion from Coppor sulphate. From the XRD studies it is confirmed that the structure of the synthesized nanoparticles is cubic.

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