

SYNTHESIS AND CHARACTERIZATION OF GREEN Cu₂O /PANI NANOCOMPOSITES

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ABSTRACT

Nanotechnology is gaining tremendous impetus in the present century due to its capability of modulating metals into their nanosize. Research in nanotechnology highlights the possibility of green chemistry pathways to produce technologically important nanomaterials. The present study deals with cost effective and environment friendly given synthesis of copper oxide from Benedict's reagent, through the leaf extract of *Tridax procumbens*, as reducing as well as capping agent and also Cu₂O/doped polyaniline (PANI) is prepared by an insitu polymerization method using Cu₂O as the addition agent and H₂O₂ as the oxidizing agent. The products are characterized by UV-Vis, FT-IR, XRD and SEM.

Keywords: Benedict's reagent, Cu₂O /PANI nanocomposites, *Tridax procumbens*.

I. INTRODUCTION

Inorganic materials, such as metals, semiconductors, and metal oxides, have been the subject of intensive research because of their potential applications in electronics, photonics, and catalysis [1-2]. Copper oxide (Cu₂O) is one of the most attractive inorganic materials because of its wide range of potential applications in solar energy conversion, catalysis, sensing, magnetic storage and electrode materials in lithium ion batteries etc. [3-5].

The use of plants for synthesis of nanoparticles is a comparatively new and under-researched technique. Synthesis of metal and metal oxide nanoparticles using plant extracts is very cost effective, so it can be used as an economic and valid alternative for the large-scale production of metal nanoparticles. The *Tridax procumbens* leaves possess biomolecules, such as polysaccharides, reducing sugars, amino acids and vitamins, which could be used as reductants to react with copper ions and as scaffolds to direct the formation of Cu₂O NPs in solution. In addition to inorganic/ inorganic composites, inorganic nano- composites containing conducting polymer have attracted a great deal of interest from researchers because of their unexpected hybrid properties, which are synergistically derived from each component [6]. In this area polyaniline (PANI) is one of the most promising conducting polymers. It is easy to prepare and often exhibits unusual electromagnetic properties combined with moderate electrical conductivity. New inorganic/PANI nanocomposites are triggering increased interest on account of their outstanding microwave absorption properties. Many inorganic/PANI nanocomposites, such as Ni/PANI [7], BaTiO₃/PANI [8], Fe₃O₄/PANI [9], carbonnanotube/PANI [10], andTiO₂/PANI [11]



nanocomposites, have been reported in the literature. In this paper, Cu_2O /PANI nanocomposites are prepared by an in situ polymerization method.

II. MATERIALS AND METHODS

2.1 Materials

All the chemicals including $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$, sodium citrate, sodium carbonate, potassium thiocyanate, potassium ferrocyanide, aniline and H_2O_2 were of analytical grade and were used as received from Merck without further purification.

2.2 Preparation of Benedict's Reagent

Benedict's reagent is prepared by dissolving 10 g of sodium citrate, 3.75 g of sodium carbonate and 6.25 g of potassium thiocyanate in 32mL of hot distilled water. Then it is mixed with 5 mL of 8.38% (w/v) of $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ solution followed by the addition of 0.3 mL of a 5% solution of potassium ferrocyanide and the mixture is stirred well using a mechanical stirrer. The resultant solution is then made up to 50 mL by adding distilled water.

2.3 Preparation of Tridax Procumbens Leaf Extract

About 20g of freshly, taxonomically authenticated healthy leaves of Tridax procumbens were collected, washed thoroughly with distilled water, cut into fine pieces and boiled with 100 mL distilled water in Erlenmeyer flask for 8- 10 min. The extract was cooled to room temperature and filtered through Whatman filter paper (No.42).

2.4 Preparation of Cu_2O Nanoparticles

In a typical experiment, 10 mL of the Tridax procumbens leaf extract was added to 10 mL of a Benedict's solution. After 10 minutes a brick-red precipitate was obtained, indicating the formation of cuprous oxide nanoparticles. The precipitate was then washed with distilled water and dried at room temperature.

2.5 Preparation of Cu_2O /PANI nanocomposite

In the typical experiment, 2.7 g aniline was dissolved into 300 mL of distilled water in a 500 mL beaker and 1.08 g Cu_2O nanoparticles were added into this solution followed by adding 68 mL of 4% H_2O_2 slowly. The mixture is stirred well using a mechanical shaker for 23h at a constant stirring rate. The final product was filtered, washed with distilled water and then dried.

2.6 Characterization

Characterization of the Cu_2O / PANI nano-composite was carried out by different analytical techniques. UV-VIS spectra were measured using a TU-1901 model UV- VIS double beam spectrophotometer (Beijing Purkinje General Instrument Co., Lt, China). FTIR spectra were performed and recorded with a Fourier-transform infrared spectrophotometer Nicolet 870 between 4000 and 400 cm^{-1} , with a resolution of 4 cm^{-1} . The morphologies and compositions were examined by Scanning Electron Microscopy (SEM), using a LEO 1455 VP equipped with energy dispersive. X-Ray Diffraction (XRD) patterns were recorded with a Philips analytical X-ray diffractometer using $\text{CuK}\alpha$ radiation ($\lambda = 1.5406 \text{ \AA}$).

III. RESULTS AND DISCUSSION

The addition of Benedict's solution to the leaf extract containing carbohydrates (glucose) which have aldehyde groups causes the reduction of copper ions resulting in the formation of brick red precipitate of Cu_2O . The chemical reaction may be expressed in the following equation:



Therefore, we took advantage of the ready reactivity of solution with reducing sugars to innovate a facile method for the synthesis of Cu_2O nanoparticles.

The characteristic peaks of PANI at 301 and 334 nm as shown in Fig. 3.1 are assigned to the $\pi - \pi^*$ transition of benzenoid ring which is related to the extent of conjugation between the adjacent phenylene rings in the polymeric chain [12]. The absorption peak at 259 nm in the nanocomposite confirms the presence of copper oxide nanoparticles in $\text{Cu}_2\text{O}/\text{PANI}$ nanocomposite [13].

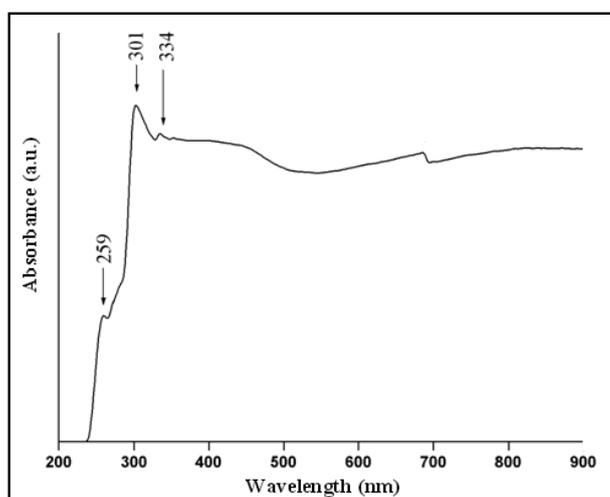


Fig. 3.1 UV-Vis Spectrum of $\text{Cu}_2\text{O}/\text{PANI}$ Nanocomposite

The characteristic FTIR peaks at 1569, 1494, 1297, 1146, 815 and 658 cm^{-1} are shown in Fig. 3.2. The stretching mode of $\text{N}=\text{Q}=\text{N}$ ring, $\text{N}-\text{B}-\text{N}$ ring and $\text{C}-\text{N}$ ($\text{C}_{\text{aromatic}}-\text{N}$) deformation are assigned to the bands at 1569, 1494 and 1297 cm^{-1} respectively (where B refers to benzenoid ring and Q refers to Quinonoid ring). The band at 1146 cm^{-1} is assigned to $\text{C}-\text{N}$ stretching vibration of secondary aromatic amine. The observed band at 815 cm^{-1} attributes to the out-of-plane vibration of $\text{C}-\text{H}$ on 1, 4-disubstituted aromatic rings [14]. The peak at 658 cm^{-1} shows the bending vibrations of the Cu_2O nanoparticles [15].

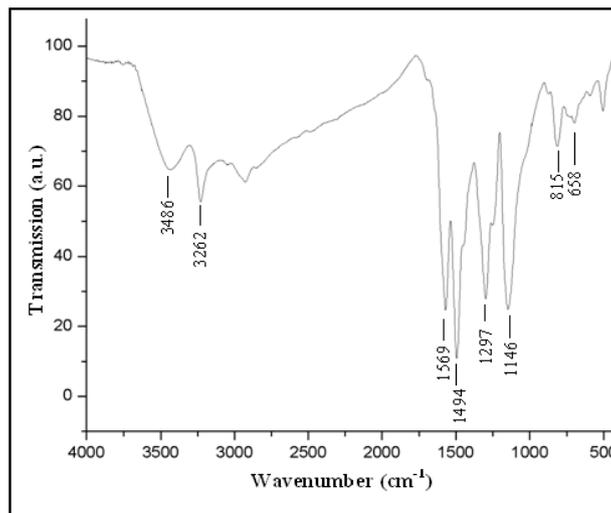


Fig. 3.2 FTIR Spectrum of Cu₂O/PANI Nanocomposite

Fig. 3.3 demonstrates the presence of Cu₂O nanoparticles and PANI in the nanocomposites. The characteristic broad peak between 13.0° and 18.0° as shown in Fig. 3.3 indicates partial crystalline structure of PANI. The peaks at 30.82° and 36.47° corresponding to the crystal planes of (110) and (111) show the presence of Cu₂O in the nanocomposite [16].

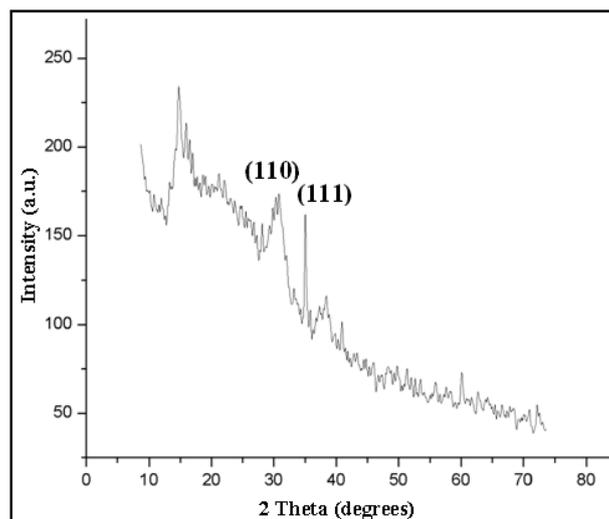


Fig. 3.3 XRD Pattern of Cu₂O/PANI Nanocomposite

In Fig. 3.4 (a&b) the SEM images reveal that the filler particles of Cu₂O nanoparticles were incorporated well in the matrix of PANI as appeared in the light. Further, it shows that many aggregations of agglomerated Cu₂O nanoparticles were well encapsulated in the polymer matrix of cauliflower like structure as appeared in darken.

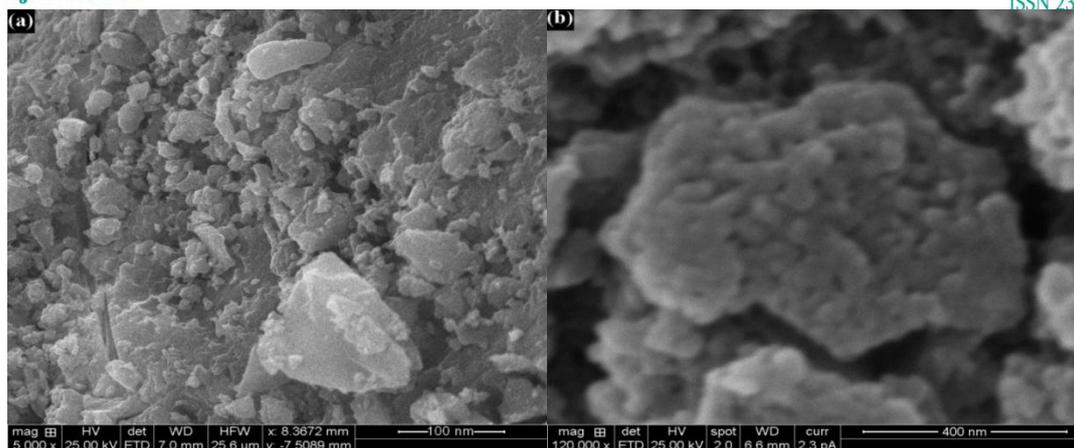


Fig. 3.4 SEM Images of (a) Cu_2O Nanoparticles and (b) $\text{Cu}_2\text{O}/\text{PANI}$ Nanocomposite

IV. CONCLUSION

The “green” route for nanoparticle synthesis is of great interest due to eco-friendliness, economic prospects, feasibility and wide range of applications in nanomedicine, catalysis medicine, nano-optoelectronics, etc. Here, we have reported a simple, eco friendly and low-cost approach for preparation of stable copper oxide nanoparticles by reduction of Benedict’s reagent, with a green method using *Tridax procumbens* leaf extract as the reducing agent. The Cu_2O /PANI nanocomposites were successfully prepared by an in situ polymerization method. The characteristics of the nanocomposites were studied using UV, FTIR, XRD and SEM techniques.

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