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## Bio-Synthesis and Characterization of Copper Molybdate Nanoparticles

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### ABSTRACT

In the present work CuMoO<sub>4</sub> and P/CuMoO<sub>4</sub> nanoparticles were synthesized by simple precipitation method. The synthesized molybdate nanoparticles were characterized by Fourier transform infrared spectroscopy (FT-IR), X-ray diffraction (XRD), scanning electron microscopy (SEM) and UV-visible diffuse reflectance spectroscopy (UV-vis DRS). XRD results show that the average crystalline size of CuMoO<sub>4</sub> and P/CuMoO<sub>4</sub> nanoparticles were found to be 62.9 nm and 59.7 nm respectively. SEM results showed that CuMoO<sub>4</sub> nanoparticles composed of sphere like structure and P/CuMoO<sub>4</sub> with agglomeration. Four absorption bands at 721, 828, 898 and 967 cm<sup>-1</sup> were observed in FT-IR spectrum. The sharp adsorption peak at 270-350 nm was observed in UV-vis DRS technique. The antimicrobial activities of synthesized nanoparticles were observed against various pathogens.

### 1. Introduction

Metal molybdate with formula MMoO<sub>4</sub> have attracted much attention due to their interesting size and shape dependent optical, magnetic and electronic properties [1-6]. Transition metal molybdate have many applications, such as in gas sensors, optical fibers, humidity sensors, pigments, catalytic and biological activity. Copper molybdate (CuMoO<sub>4</sub>) is one of the important inorganic materials that have high potential application in various fields such as catalysts, humidity sensor, photo anodes, laser hosts, microwave application and optical fibers. Also it is considered to be a potential candidate as a visible light photocatalyst for organic contaminants decomposition. Recently, many studies have been reported on the preparation and characterization of copper molybdate such as precipitation [7], hydrothermal [8], flame spray method [9]. However, these methods usually require special equipment, high temperature, templates or substrate and usually result in impurities. The toxic chemicals, non-polar solvent and synthetic additives or capping agent immersing from the chemical reaction are often potentially dangerous to the environment.

The plant with the antioxidant or reducing properties is usually responsible for the reduction of metal compounds into their respective nanoparticles. According to the researchers, the poly phenol components present in the plant extract are responsible for the reduction of metal ions, whereas water soluble heterocyclic components stabilize the formed nanoparticles. The synthesized nanoparticles have shown considerable stability and it was found to have potential for bio-application.

Researchers studied photocatalytic degradation of 4-chloro phenol by copper molybdate doped titanium oxide and the structural and optical properties of molybdate were also reported in various work [10-14]. The synthesis of nano-size CuMoO<sub>4</sub> crystals includes co-precipitation, polymeric precursor method, modified citrate complex technique, hydrothermal method, molten salt method and spray pyrolysis [15-20]. Hence developing of reliable biosynthetic, an environment friendly approach have been added much importance because of its eco-friendly products, biocompatibility and economic viability in the long run and also to avoid adverse effects during their environmental application. In this work, we adopted a precipitation route for the synthesis of CuMoO<sub>4</sub> pure phase with uniform particle size distribution and plant extract as capping agent. CuMoO<sub>4</sub> nanoparticles were explored with respect to their prospective anti-malarial application.

### 2. Experimental Methods

#### 2.1 Materials

AR grade of sodium molybdate, copper acetate and urea were used as received from Merck. FT-IR spectra of the sample were measured in the KBr pellet in the range of 400 cm<sup>-1</sup> to 4000 cm<sup>-1</sup> using Shimadzu spectrometer. UV-visible spectra of the sample were measured in UV-2400 PC SERIES having range 200-900 nm. XRD analysis was carried on atomic X-ray diffraction (1.54 Å) with a scanning speed of 10°/min. SEM images were taken with a HITACHI Model S-3000H instrument.

#### 2.2 Preparation of Plant Extract

The collected *Phyllanthus amarus* leaves were washed thrice with tap water and twice with distilled water to remove the adhering salts and other associated contaminants. 10 g of leaf was taken and boiled with 100 mL of sterile distilled water at 100 °C for half an hour. The boiled extract was filtered through Whatman No.1 and was stored in refrigerator at 4 °C for further studies [21].

#### 2.3 Preparation of CuMoO<sub>4</sub> Nanoparticles

Copper molybdate nano particle were prepared by simple precipitation method. 1:0.5 molar ratio of copper acetate and sodium molybdate were dissolved in 40 mL distilled water in two glass beaker and 0.5 g of urea were dissolved in 10 mL of distilled water. The above solution was mixed well and stirring for 30 minutes. Again the same procedure was repeated and then 5 mL of plant extract was added and it was stirred for 2 hours to form a homogeneous precipitate. After filtering the precipitate washed with distilled water and dried at 80 °C for 5 hours. The obtained nanoparticle was calcinated at 550 °C for 4 hours to get final product [22].

### 3. Results and Discussion

#### 3.1 UV-Visible Diffuse Reflectance Spectra Analysis

The optical absorption properties of CuMoO<sub>4</sub> were characterized by UV-vis absorption spectroscopy (DRS-mode). The UV-visible DRS absorption spectrum of synthesized CuMoO<sub>4</sub> nanoparticles is shown in Fig. 1. The sharp adsorption steep in the region of 270-350 nm which is the characteristic peak of CuMoO<sub>4</sub> nanoparticles. This result strongly confirms the small crystal size of the product particles, which could be ascribed to the strong quantum confinement of the excitonic transition of the nano-structures.

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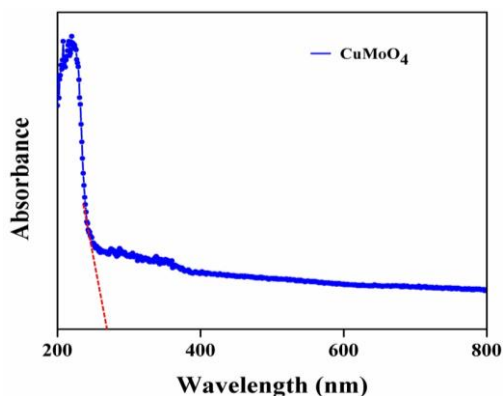


Fig. 1 UV-visible DRS spectra of CuMoO<sub>4</sub>

### 3.2 X-Ray Diffraction (XRD) Analysis

The crystalline phase and the crystalline nature of the synthesized CuMoO<sub>4</sub> were analyzed using XRD which is shown in Fig. 2. The diffraction peaks of CuMoO<sub>4</sub> match well with the standard card of CuMoO<sub>4</sub> (JCPDS card No. 36-0405). The characteristic 2θ regions of 15.85, 19.50, 24.91, 26.78, 29.89, 32.87, 33.34, 34.53, 35.69, 41.19, 44.53, 47.15 and 52.56° were corresponds to the reflections of planes of CuMoO<sub>4</sub> (020), (110), (031), (111), (121), (200), (002), (221), (122) (151), (202), (241) and (212) respectively. The XRD pattern of the P/CuMoO<sub>4</sub> has shown the high intense diffraction peak consists of characteristic peak of both CuMoO<sub>4</sub> as well as the diffraction peak of plant which clearly suggests that formation of P/CuMoO<sub>4</sub>. The strong and sharp diffraction peaks of P/CuMoO<sub>4</sub> indicated the good crystallinity of the nanoparticles. The average crystallite size of the CuMoO<sub>4</sub> and P/CuMoO<sub>4</sub> nanoparticles were determined by the full width at half maximum (FWHM) of the X-ray diffraction peak using Debye Scherer's equation [23],  $D = K\lambda / \beta \cos\theta$ , where  $D$  is the crystallite,  $\lambda$  is the X-ray wavelength,  $\beta$  is the Full Width at Half Maximum (FWHM) of the diffraction peak,  $\theta$  is the diffraction angle and  $k$  is the Scherer's constant of the order unity. The crystallite sizes were calculated for the reflection of the plane CuMoO<sub>4</sub> and P/CuMoO<sub>4</sub> nanoparticles was 62.9 nm and 59.7 nm respectively.

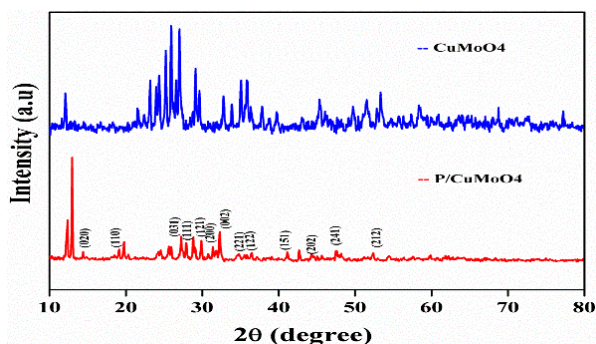


Fig. 2 XRD pattern of CuMoO<sub>4</sub> and PCuMoO<sub>4</sub>

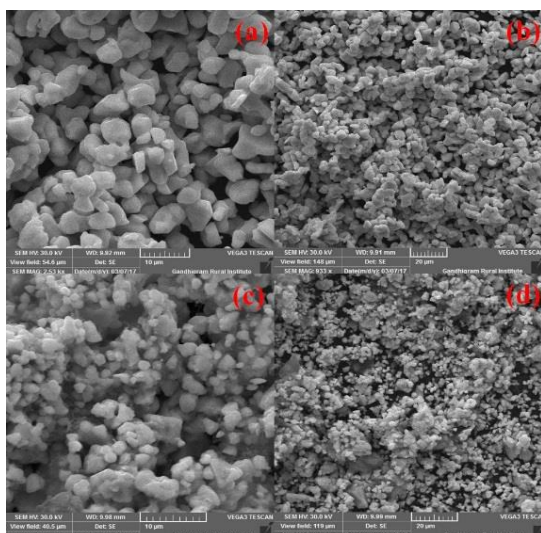


Fig. 3 SEM image of (a, b) CuMoO<sub>4</sub> and (c, d) PCuMoO<sub>4</sub>

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### 3.3 SEM Analysis

The morphology of synthesized CuMoO<sub>4</sub> and P/CuMoO<sub>4</sub> was studied by scanning electron microscopy. The SEM micrographs of the pristine CuMoO<sub>4</sub> given in Fig. 3(a) indicated that the sphere like particles and Fig. 3(b) which closely attached to each other particles the CuMoO<sub>4</sub> surface was very smooth and shining. The SEM micrographs of P/CuMoO<sub>4</sub> showed that there is no agglomeration of CuMoO<sub>4</sub> nanoparticle in the plant matrix and it was uniformly distributed throughout the plant matrix which is shown in Figs. 3c and d.

### 3.4 FT-IR Spectroscopy

To confirm the structural information of CuMoO<sub>4</sub> and P/CuMoO<sub>4</sub> Nps was further evidenced by FT-IR analysis which is shown in Fig. 4. FT-IR spectra of P/CuMoO<sub>4</sub> showed absorption peaks at 500-650 cm<sup>-1</sup> which is the characteristic peak of Cu-O bond. However, in the FT-IR spectrum of the synthesized CuMoO<sub>4</sub> Nps four absorption bands (at 721, 828, 898, 967 cm<sup>-1</sup>) are observed. The band at 1634 cm<sup>-1</sup> was attributed to the metal bonded stretching vibration of C-O groups. The absorption band around 3430 cm<sup>-1</sup> which corresponds to the O-H stretching vibration indicating the presence of hydroxyl groups [24, 25]. No other absorption band except the above mentioned was detected in the observed FT-IR spectrum.

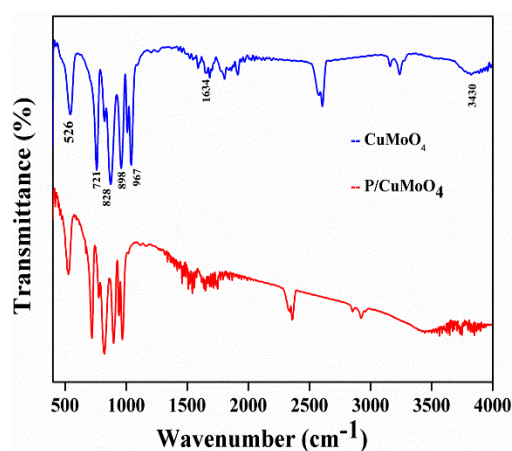


Fig. 4 FT-IR spectra of CuMoO<sub>4</sub> and P/CuMoO<sub>4</sub>

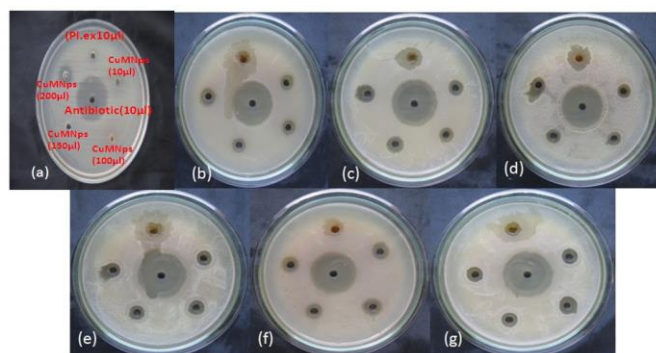


Fig. 5 Zone inhibition of different pathogens a) *Bacillus subtilis*, b) *Staphylococcus aureus*, c) *Streptococcus faecalis*, d) *Candida albicans*, e) *Klebsiella pneumoniae*, f) *Pseudomonas aeruginosa* and g) *E. coli* by CuMoO<sub>4</sub> NPs synthesized at 1 mM concentration

### 3.5 Antimicrobial Activity of Green Synthesized Copper Molybdate Nanoparticles

Due to higher simplicity and eco friendliness, the plant mediated synthesis of nanoparticles gains much importance in this modern era. The presence of phytochemicals in the plants have tendency to reduce the heavy metals from their higher oxidation state to zero oxidation state. For the production of nanoparticles, the electron rich phytochemicals in plants acts as the building block for conversion of metallic precursor, there by replacing the toxic reducing agents [26, 27] to design engineered nanomaterials in medicine, energy, electronics, etc., the green eco-friendly technique is used. The mechanism between the nanoparticles and the microorganism takes place by the electrostatic interaction of positively charged nanoparticles with that of the negatively charged microorganisms. As copper molybdate nanoparticles are having greater surface area and exclusive size, it has an easier tendency to reach the nuclear content of bacteria [28]. Agar diffusion method was adopted for

the determination of the antimicrobial activity using different pathogens such as the gram positive (*Staphylococcus aureus*, *Streptococcus sp* and *Bacillus subtilis*) bacterial colonies, gram positive bacterial (*E. coli*, *Klebsiella pneumoniae*, *Pseudomonas aeruginosa*) colonies and fungus (*Candida albicans*). Nutrient broth was prepared freshly and was incubated. The culture, the plant extracts, the synthesized copper molybdate nanoparticles were loaded using a sterile rod and the plates were incubated for 1 day. After incubation period the zone of inhibition was measured and given in Fig. 5. The maximum zone of inhibition was measured for the bacterial strain *Streptococcus faecalis* followed by the strain *staphylococcus aureus*. The synthesized molybdate nanoparticle comprises activity towards the pathogens can be used as a drug in a thriving approach.

**Table 1** ZOI of pathogen by copper molybdate nanoparticle

Organisms	Concentration of extract and zone inhibition(mm)	
	PCuMoO <sub>4</sub> (100 µL)	CuMoO <sub>4</sub> (100 µL)
<i>Bacillus subtilis</i>	19	18
<i>Staphylococcus aureus</i>	22	21
<i>Streptococcus faecalis</i>	24	23
<i>Candida albicans</i>	18	25
<i>Klebsiella pneumoniae</i>	21	23
<i>Pseudomonas aeruginosa</i>	17	20
<i>E. coli</i>	19	21

#### 4. Conclusion

The copper molybdate nanoparticles were successfully synthesized by using *Phyllanthus amarus* plant which provides cost effective, easy and proficient way for synthesis of CuMoO<sub>4</sub> nanoparticles. The functional group present in the leaf extract has been confirmed by FT-IR. The polyphenols of leaf extract were mainly responsible for the reduction of copper molybdate metal ion into nanoparticles. The synthesized copper molybdate nanoparticles were analyzed using FT-IR, UV-DRS, XRD and SEM. Copper molybdate nanoparticle were effectively utilized for the antimicrobial study. The maximum ZOI was found to be more in gram positive bacteria when compared to gram-negative bacteria. The *Phyllanthus amarus* plant may be effectively utilized for the production of copper molybdate nanoparticle with economically for many pharmaceutical applications.

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