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The fabrication and characterization of copper phosphate nanoparticles

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Copper acetate has been subjected to phosphoric acid treatment, and copper phosphate nanoparticles have been synthesized by the chemical co-precipitation approach. The method seeks to synthesize copper phosphate in the most uncomplicated manner of dimensions of nanoparticles. Different characterization approaches have been utilized to identify the synthesized material, revealing the information it may provide or the materials for which it is intended. X-ray diffraction, Raman spectroscopy, UV-Visible spectroscopy, SEM and FT-IR have been utilized to analyze the synthesized nano particles. The stretching and bending frequencies of the sample's molecular functional groups have been examined using FT-IR spectra. When using XRD, the size of the copper phosphate nanoparticles has been identified to be 57.74 nm. The resultant product copper (II) phosphate has well-crystallized particles, as clear in the SEM image. The unique electronic excitation states of copper (II) phosphate are detected *via* the ultraviolet spectra. The UV-Vis spectrum notifies the distinct electronic excitation states of copper (II) phosphate. The distribution of copper (II) phosphate nanoparticles is suitable, as demonstrated by the linear regression analysis. The Raman spectra peaks attest to the existence of copper phosphate.

Keywords: Copper Phosphate, XRD, IR, SEM

Recently, there has been a surge in research in the synthesis and manufacture of metal nanoparticles of various forms and dimensions. Phosphate chemistry has attracted a lot of attention, which has generated a great deal of unique materials with structural variety. Copper compounds also have a broad value in terms of its application in the industrial world. Copper phosphate (CuP) has applications as a catalyst, fungicide, fertilizer, corrosion inhibitor, metal protectant, energy storage, biosensing, and in many more fields. Copper compounds are acknowledged for its good antibacterial activity and little toxicity and these simple inorganic compounds have its applications as catalysts and promoters, oxidants, reductants, and have other numerous uses. Metallic copper is utilized significantly in electronics systems owing to its outstanding electrical conductivity and acceptable cost. Phosphate-based materials are preferable due to their strong bonding, flexibility in structural design, and low curing temperatures. This work presents the preparation and analysis of CuP nanoparticles employing current techniques of characterization. CuP was created by S. Ramesh *et al*. by adding hydrazine hydrate after a stoichiometric amount of copper acetate and phosphoric acid were combined. The work below synthesizes CuP without the usage of chemical hydrazine hydrate, which was employed as a reducing agent and catalyst. The acquired results were in compliance with CuP guidelines, so lowering the expense and utilizing less of the hazardous chemical hydrazine hydrate[1-7].

Experimental Section

S. Ramesh *et al*. developed a method of preparation of copper phosphate using stoichiometric amounts of copper acetate and phosphoric acid, followed by addition of hydrazine hydrate[10]. In this study, CuP nanoparticles were created without the inclusion of hydrazine hydrate by reacting copper acetate with phosphoric acid.6.5 g copper acetate in 1 L distilled water and 2.3 g phosphoric acid in 10 mL distilled water were taken. Copper acetate was initially dissolved in water, then followed by addition of dil. Phosphoric acid. The mixture was boiled for an hour to obtain a blue colour, which indicated more presence of hydroxyl groups. Later, it was boiled continuously to obtain a green colour precipitate, washing was done 3 times by distilled water. The precipitate obtained was dried at 130 °C for a period of 6 hours.

Characterisation Methods

To the greatest extent possible, it is imperative to characterize the nanomaterials that have been created. The best method for figuring out the crystallite size of

powder samples is X-ray diffraction. Precise measurement of the peak widening is the fundamental component of this determination process. The sample of powder's crystallite size was ascertained in this inquiry using the XRD line broadening method of particle size measurement. A Perkin Elmer FTIR spectrophotometer was used to observe an infrared spectroscopy (FTIR) spectrum between 4000 and 400 cm^{-1}. FESEM EDS (Field Emission Scanning Electron Microscope) Mapping data of the sample was analysed using Carl Zeiss Model Supra 55 instrument to capture the microstructure image of the materials and understand the morphology of the sample. The absorbance mode was used to acquire the UV spectra in the wavelength span of 190 to 900 nm. Raman spectrum was recorded using Horiba Japan Xplora Plus for chemical and structural characterisation[8-17].

Results and Discussion

X-ray Diffraction

A diffraction technique called X-ray diffraction (XRD) serves to detect the framework of the material and its arrangement of all the integrants. CuP is struck with an X-ray incident as the foundation of the XRD process. Fig. S1 showcases XRD patterns of the prepared Copper (II) Phosphate samples. The samples are crystalline and nanosized, according to XRD investigations. The X-ray line broadening in Fig. S1 reflects the tiny particle composition of the samples. Employing the Scherrer equation, one can assess the size of the generated Copper (II) Phosphate nanoparticles.

$$D=0.9\lambda/\beta\cos \quad \ldots (1)$$

In this case, λ stands for the X-ray wavelength, β for the half width at full maximum, and θ for the diffraction angle. 2θ = 33.8612 θ = 33.8612/2 = 16.9306

$$D = (0.9 * 0.154) / (0.00251) * \cos (16.9306) = 57.74 \text{ nm}$$

The average particle size of CuP is determined as 57.74 nm. Table 1 provides the peak list for the XRD pattern. The dislocation density is the length of dislocation lines per unit volume of a crystal. A great deal of a material's qualities is impacted by the existence of dislocations from the Fig. S2.

Dislocation density is inversely proportional to the particle size. Some dislocations in the sample prevent a dislocation from moving as freely as it would otherwise. Increased hardness is therefore implied by increased dislocation density. Table 1 demonstrates an inversely proportional relationship between particle size and dislocation density.

$$\Delta = 1/D^2$$

where D is the size of the crystallite and δ denotes the dislocation density.

A XRD morphology index (MI) is generated from the FWHM of XRD data using the relationship.

$$\text{M.I.} = \frac{\text{FWHMh}}{\text{FWHMh} + \text{FWHMp}}$$

The morphological index is denoted by M.I., FWHMh, the most prevalent FWHM value found

Table 1 — Intensity, Particle size and dislocation density of XRD

2-Theta (°)	Height (count)	FWHM (deg) (beta)	d(ang.)	Rel. height	Particle size	Dislocation density
5.2687	346.14	0.2086	5.7982	50.6	55.73	$3.219*10^{13}$
18.4362	430.95	0.192	4.80855	63	55.96	$3.192*10^{13}$
23.8782	273.59	0.2442	3.72354	40	56.46	$3.136*10^{13}$
28.3913	11.93	0.8128	3.14107	1.74	56.98	$3.079*10^{13}$
30.3699	123.17	0.2311	2.94078	18.01	57.24	$3.052*10^{13}$
33.8612	317.93	0.3564	2.64513	46.48	57.74	$2.999*10^{13}$
34.1036	543.79	0.2627	2.62688	79.5	57.78	$2.995*10^{13}$
36.7435	154.81	0.1952	2.44398	22.63	58.21	$2.951*10^{13}$
37.8377	243.03	0.1931	2.37578	35.53	58.40	$2.932*10^{13}$
38.9771	263.47	0.2104	2.30892	38.52	58.60	$2.912*10^{13}$
43.6678	102.48	0.2123	2.07115	14.98	59.51	$2.823*10^{13}$
47.6382	52.37	0.9927	1.90738	7.66	60.38	$2.742*10^{13}$
48.9412	70.25	0.2115	1.8596	10.27	60.69	$2.714*10^{13}$
53.5343	161.62	0.2983	1.71038	23.63	61.87	$2.612*10^{13}$
56.7863	142.28	0.2774	1.61991	20.8	62.79	$2.536*10^{13}$
59.7341	181.05	0.1918	1.54681	26.47	63.70	$2.464*10^{13}$
63.1193	85.44	0.4743	1.47176	12.49	64.83	$2.379*10^{13}$

among the peaks, and FWHMp, the proportion of the FWHM of a particular peak, which is used to compute M.I. The findings are displayed in the Table 2 and indicate a clear correlation between the Morphology Index and particle size[18-25].

Scanning Electron Microscopy (SEM)

To create SEM pictures, a focused electron beam is traversed over the sample's surface. Several signals, containing secondary electrons (SE) as well as backscattered electrons (BSE), appear while the substance is interacting with the electron beam. To create SEM images, a focused electron beam is traversed across the sample's surface. Various signals are produced when the material is exposed to the electron beam. Secondary electrons (SE) and backscattered electrons (BSE) are two types of signals that appear in these. The dimensions and size of the generated nanoparticles of Copper Phosphate were examined by making use of scanning electron microscopy. When electrons contact specimen atoms, they produce a wide range of signals which reveal information concerning the contents of the specimen and surface topography. The original image is placed over each of the maps for Cu, P, and O, which are displayed separately in Fig. S3. The focus elements inside the investigated field are Cu, P, and O, as shown in the EDX spectrum (Fig. S4). SEM pictures of Copper (II) Phosphate nanoparticles reveal well-crystalline fragments with layered crystallized morphology. In this specific case, the particle specifications are somewhat enlarged, further the particles are spread uniformly in clusters[26-33].

UV-Visible Spectroscopy

UV spectroscopy makes use of UV radiation across the electromagnetic spectrum. Electronic transitions, in which bonding or outermost electrons are promoted to higher energy levels, are seen in both organic and inorganic species. A spectrum is produced when a substance absorbs light and goes through excitation and de-excitation. This approach is utilized to investigate the electronic transitions that molecules go through within the electromagnetic spectrum. The fierce widespread absorption band at 271 nm (λ_{max}) in the Fig. S5 is assigned to the electronic excitation in phosphate. The not so high absorption band at 829 nm (λ_{max}) is due to the electronic transition in Cu^{2+}. The broad absorbance is associated with octahedral conditions. The optical band gap of CuP nanoparticles found is $\approx$ 3.25 eV [33-38].

Table 2 — Relation between Morphology Index and Particle size

FWHM B (radians)	Particle size	MI (unitless)
0.003641	55.73	0.8968
0.003351	55.96	0.9043
0.004262	56.46	0.8813
0.014186	56.98	0.6905
0.004033	57.24	0.8870
0.00622	57.74	0.8358
0.004585	57.78	0.8735
0.003407	58.21	0.9028
0.00337	58.40	0.9038
0.003672	58.60	0.8960
0.003705	59.51	0.8952
0.017326	60.38	0.6462
0.003691	60.69	0.8956
0.005206	61.87	0.8588
0.004842	62.79	0.8673
0.003348	63.70	0.9043
0.008278	64.83	0.7927
0.031652	69.35	0.5

FTIR Studies

FTIR is a crucial microanalytical approach that can be applied to samples with dimensions as small as 10 μm. The frequencies at which molecular bonds vibrate are distinctive. A molecule can absorb infrared light with a defined frequency that excites a specific chemical vibration if it causes an alteration in the dipole moment of the bond. The nanoparticles of CuP were prepared by reacting Copper acetate and phosphoric acid. The free O-H group has a significant peak in the CuP's FTIR spectra at 3658.10 cm^{-1}as shown in Fig. S6. The bending mode of the O-H group is well-represented by the peak at 966.81 cm^{-1}. The peak, which stands at 1058 cm^{-1} depicts phosphate vibrations. P=O double bond in phosphate is depicted at 1251 and 1149 cm^{-1}. Peaks at 609 cm^{-1} can be credited to the Cu-O stretching bond. Copper nanoparticles have an absorption peak at 556 cm^{-1}, and they exhibit a wide absorption range from 550 to 700 nm as. Fig. S6 FTIR spectrum of CuP [39-46].

Raman Spectrum Studies

A Raman spectrum is displayed as a change in wavelength *versus* intensity. A unique Raman spectrum can be captured between 4000 and 10 cm^{-1}[11]. When light interacts with a sample's molecular vibrations, the Raman effect happens. The Fig. S7 displays the Raman spectra of the produced CuP sample. The phosphate vibrations correspond to the striking peak at 980 cm^{-1}. The peak cluster about 500 $cm^{-1}$is caused by vibrations in Cu-O. The stretching of the O-H bond is shown by the peak at 3469 cm^{-1}.

The spectrum exhibits distinct peaks, which were able to identify as components of CuP by comparing them to vibrations observed in certain references[47-53].

Conclusion

Using the chemical co-precipitation approach, CuP nanoparticles were synthesized. When subjected to XRD analysis, the particle size was revealed as 57.74 nm. The particle size, according to SEM, is in the nano range. The SEM image conveys the particle morphology. SEM images of Copper (II) Phosphate nanoparticles display well-defined crystalline pieces featuring a layered structure and the particles are uniformly distributed. With the FTIR spectrum, the specimen's molecular functional groups stretching and bending frequency bands are investigated. The optical band gap of 3.25 eV was discovered using the UV spectrum which confirms the existence of active particles present in the sample. Characteristic Raman peaks corroborate the existence of CuP. The method used for synthesizing was successful without the use of hydrazine which was proven with the data confirmation.

Supplementary Information

Supplementary information is available in the website http://nopr.niscpr.res.in/handle/123456789/58776.

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Author Contribution

Each author has contributed to the synthesis, analysis, and recommendations of the content. The final manuscript has been read and approved by every author.

Data Availability

Upon reasonable request, data sets created during the current investigation can be obtained from the relevant author.

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