



Synthesis & Characterization of Copper Oxide (Cu₂O) and Copper Sulphide (Cu₂S) Nanoparticles using XRD Analysis

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Abstract:

Nano-materials can be characterized as solids having measurements in the nanometer system. So, nanomaterials structure a unique class of materials that lie in the area that is in the middle of two limits i.e. mass solids and atoms. Solids and atoms have been contemplated and seen very well, anyway nanomaterials show properties that are totally not the same as both of the two. The properties of these materials are described by a particular length scale, normally underneath ~ 100 nm. In the event that the actual size of the material is decreased underneath this length scale, it's all properties like mechanical, strength, warm, optical, attractive, directing and so forth changes and become touchy to its size and shape. The purpose behind the variety in properties with size of the materials originates from very enormous surface-to-volume proportion. As such, nanomaterials have enormous number of particles on their surface when contrasted with those in the inside. Nanoscale metals/metal oxides/metal sulfides show electro-optical and mechanical properties that are not quite the same as their mass partners are promising materials for a few innovative applications, for example, photograph warm transformation, catalysis, sensors and so forth.

In this present paper, an effort is there to explain the utilization of polymer inorganic strong state response course towards the union of copper (Cu), copper oxide (CuO), and copper sulfide (Cu₂S) nano-particles. We endeavored the responses utilizing 1:1, 1:5, 1:10, 1:15 & 1:20 molar proportions of the copper salts, copper acetic acid derivation [Cu(CH₃COO)₂:H₂O], copper chloride (CuCl₂), copper carbonate (CuCO₃), and copper nitrate [Cu(NO₂)₂] and polymer, Polyphenylene sulfide [1/n[C₆H₄S]_n, (PPS)]. The PPS was picked intentionally in light of the fact that it is designing thermoplastic polymer having astounding mechanical and substance opposition properties notwithstanding the polymer being inalienably self-quenching. For these responses, the two reactants were admixed in the molar proportions in an agate pastel-mortar by utilizing CH₃)₂CO. The

resultant admixture was warmed at 2850C (dissolving temperature of PPS) in an alumina pot for 6 hours under a surrounding climate condition. The combined items were portrayed by different physicochemical methods like XRD and TEM analysis. The example relating to warmed admixtures of various [Cu (CH₃COO)₂ H₂O]:PPS proportions obviously shows transcendence of the development of copper sulfide (Cu₂S) stage in equi-molar proportion samples. The samples arranged at other molar proportions show presence of metallic copper with some copper oxide stage present in it. The item integrated from copper nitrate [Cu(NO₂)₂] can frame copper sulfide (Cu₂S), when responded with Polyphenylene sulfide (PPS). The copper carbonate (CuCO₃) surrender copper inadequate construction Cu_{1.96}S though copper chloride (CuCl₂) remains un-reacted for certain hints of Cu_{1.96}S. The future extent of the current work can be expected by examining the viewpoints of novel polymer-inorganic strong state responses for the amalgamation of other inorganic salts and its extension for the expected applications.

Keywords: Nanomaterials, Solids, strength, enormous, contrasted, transformation, PPS, self-quenching, XRD , TEM analysis.

1. Introduction:

The historical backdrop of nanomaterials started following the enormous detonation when nanostructures were shaped in the early shooting stars. Nature later advanced numerous other nanostructures like shells, skeletons and so on Nanoscale smoke particles were shaped during the utilization of fire by early people. The logical story of nanomaterials anyway started a lot later. One of the principal logical reports is the colloidal gold particles combined by Michael Faraday as right on time as 1857 [1]. Nanostructured impetuses have likewise been explored for more than 70 years. By the mid 1940's, accelerated and raged silica nanoparticles were being fabricated and sold in USA and Germany alternative for ultrafine carbon dark for elastic fortifications. As of late, it has been tracked down that the Maya blue paint is a nanostructured half breed material. The cause of its tone and its protection from acids and bio-consumption are as yet not saw yet investigations of real samples from Jaina Island show that the material is made of needle-molded precious stones that structure a super cross section with intercalates of undefined silicate substrate containing considerations of metal nanoparticles[2].

Copper sulfide (Cu₂S) is a significant base material and is broadly utilized in photovoltaic and photograph indicators. As of late, the size and shape controllable amalgamation of Cu₂S is drawing in more consideration than even previously. Additionally, research for understanding the crystallization conduct of such nanocrystals is getting especially significant from the view purpose of acquiring nanomaterials with wanted size and shape[3]. There are different reports about the combination and portrayal of Cu₂S nanoparticles. Composites comprising of directing polymers and nanosized semiconductor particles are logically and innovatively alluring in light of their novel electronic and optical proper. It had been shown that the molecule size can be constrained by changing the convergence of the added substances[4]. Nano crystals through direct restricting with the conductive MWCNTs can prompt phenomenal execution of these nanodevices. Poly vinyl liquor (PVA)/CuS

composite nano-strands were set up utilizing electro-turning and gas-strong response. Utilizing SEM and TEM investigation, it was shown that the normal width of PVA/CuS filaments was ~ 150 - 200 nm and greater part of CuS nanoparticles with a normal breadth of ~ 15 - 25 nm were consolidated in the PVA strands. The XRD investigation and electron diffraction design likewise uncovered the shaping of CuS gem structure in the PVA filaments[5].

Copper oxide has applications as oxygen pressure sensor and as p-type semiconductor material. Nanoparticles of copper oxide with various morphologies have been blended by different procedures, for example, deterioration of metal natural forerunner, basic response in smelling salts arrangement, substance fume nucleation from Copper (II) Acetylacetonate, by basic polyol strategy without surfactant, a surfactant helped aqueous course and so forth [6]. The CuO microflowers made out of nanosheets with tunable size has been manufactured through a basic response in alkali arrangement at 90 - 180 °C. Also, the CuO microflowers showed powerful reactant movement on the deterioration of ammonium perchlorate.

Arrangement of uniform circles of nanocrystallite size CuO particles done by a basic polyol measure utilizing cupric nitrate as an antecedent in ethylene glycol. The crystallite size of CuO was found ~ 11 nm. Single glasslike CuO nanoribbons were combined through a surfactant-helped aqueous course. It was shown that CuO nanoribbons contain significant measures of nanorings and nanoloops. Also, it was emphasized and shown that CuO nanoribbons displayed magnificent detecting execution towards formaldehyde and ethanol fumes with fast reaction and high affectability at low working temperatures. Moreover, the stacking of a modest quantity of Au or Pt nanoparticles was done on the outside of CuO nanoribbons and thus, it can viably improve and functionalize the gas detecting execution of CuO nanoribbons [7].

2 Experimental Details:

2.1 Materials Required for synthesis:

The chemicals that are required for the synthesis of copper oxide and copper sulphide nano composites are, copper chloride $[\text{CuCl}_2 \cdot 2\text{H}_2\text{O}]$, copper carbonate $[(\text{CuCO}_3, \text{Cu}(\text{OH})_2)]$, copper nitrate $[\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}]$ and copper acetate $[\text{Cu}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}]$ solution. Acetone (preferably doubly distilled) and polyphenylene sulphide (PPS) were taken as wetting agent and chalcogen source, respectively.

2.2 Procedure for synthesizing Copper Oxide (Cu₂O) in PPS Matrix

The synthesis of Copper Oxide and Copper Sulphide in PPS polymer matrix was performed by a solid-state reaction method, sequence is shown as below:

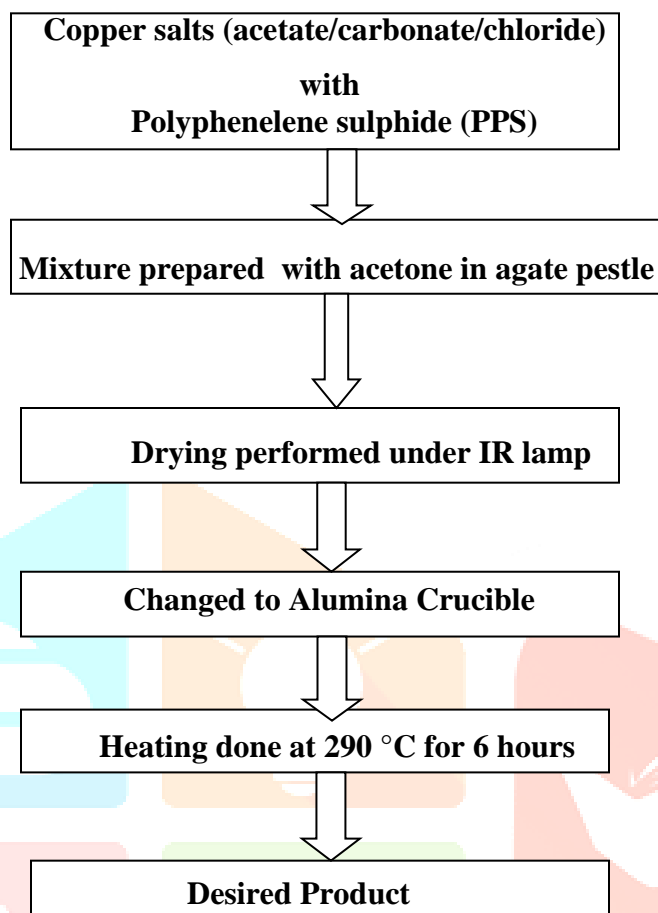


Fig. 1 Flowchart for the Synthesis of Copper Oxide and Copper Sulphide in PPS polymer matrix

For the in-situ preparation of Cu₂O/Cu₂S in polymer network, we endeavored the responses including 1:1, 1:5, 1:10, 1:15, and 1:20 molar proportions of copper salts and PPS. For these responses, the two reactants were admixed in expressed molar proportion in an agate pestle-mortar utilizing (CH₃)₂CO. The resultant admixture, subsequent to drying at room temperature in an alumina cauldron, was exposed to warming at 290⁰C in a stifle heater for six hrs under a suitable climate. The integrated item was additionally cooled to room temperature and afterward truly squashed to powder structure in desired form as required.

2.2 Characterisation of sample prepared using XRD and TEM Analysis Techniques

2.2.1 X-Ray Diffraction (XRD) Analysis:

X-ray diffraction (XRD) is an amazing nondestructive strategy for portraying glasslike materials. It gives data on structures, stages, favored gem directions (texture), and other primary boundaries, for example, normal grain size, crystallinity, strain, and gem surrenders. X-ray diffraction tops are created by helpful impedance of a monochromatic light emission rays dissipated at explicit points from each set of grid planes in an example[8]. The pinnacle forces are dictated by the dispersion of particles inside the grid.

Therefore, the X-ray diffraction design is the finger impression of occasional nuclear courses of action in a given material. X-ray diffraction is presently a typical strategy for the investigation of gem structures and nuclear dividing. X-ray diffraction depends on helpful obstruction of monochromatic X-rays and a glasslike test. These X-rays are created by a cathode ray tube, sifted to deliver monochromatic radiation, collimated to focus, and coordinated toward the sample. The connection of the X rays with the sample produces valuable obstruction (and a diffracted ray) when conditions fulfill Bragg's law $n \lambda = 2d \sin \alpha$

Where, n is a number, λ is the frequency of the X-rays,
 d is the interplanar dividing creating the diffraction, and
 α is the diffraction point.

This law relates the frequency of electromagnetic radiation to the diffraction point and the cross section separating in a translucent example[9]. These diffracted X-rays are then distinguished, handled, and tallied. By filtering the example through a scope of 2α points, all conceivable diffraction bearings of the grid ought to be achieved because of the irregular direction of the powdered material.

3 Results and Discussion

3.1 Using X Ray Diffraction (XRD) Analysis Technique:

The X-ray diffraction considers were performed to discover the development of CuO/Cu₂S in PPS matrix. Figure 2 below shows run of the mill X-ray diffraction example of powder tests comparing to three distinctive molar proportions 1:1, 1:10, and 1:20 of copper acetic acid derivation with PPS. For the correlation reason, X-ray diffraction example of unadulterated PPS arranged under similar experimental conditions is likewise taken[10].

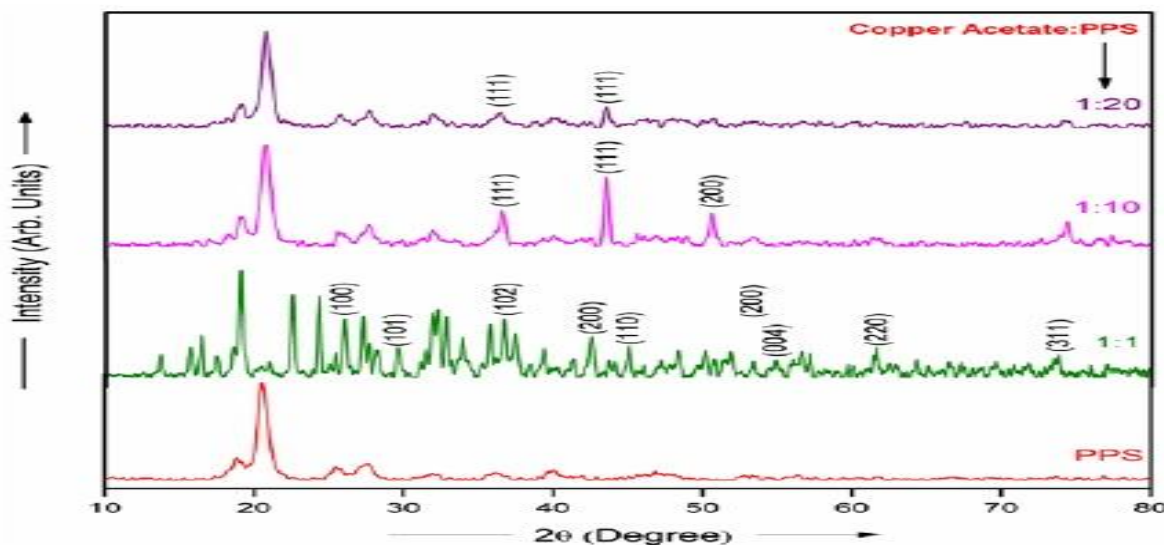


Fig. 2 XRD pattern obtained by taking different molar proportions

Following perceptions have been produced using the XRD investigation of powder samples of copper acetic acid derivation with PPS arranged at various molar proportions,

- a) For every molar proportion, except equi-molar (1:1) proportion, X-ray diffraction design shows generally metallic Cu which can be indexed by significant diffraction tops at $2\theta \sim 43.280$, ~ 50.490 and ~ 74.010 .
- b) The X-ray diffraction design for 1:1 proportion samples uncovers the arrangement of Cu₂S which can be decided by trademark diffraction tops at $2\theta \sim 26.020$, 37.320 , 45.010 , 53.740 , 54.880 and 73.710 . There are less odds of arrangement of monoclinic Cu₂S for other copper acetic acid derivation and PPS molar proportions. Nonetheless, we will in general accept that Cu₂S may have been framed in tiny amount, which is past the location furthest reaches of X-ray diffractometry and subsequently couldn't be seen in XRD.
- c) The presence of significant diffraction top around $2\theta \sim 200$ taking all things together diffraction designs recommends the maintenance of PPS in the samples. This pinnacle turns out to be more articulated as the centralization of PPS is expanded in the response admixture. This maintenance of the polymer network even in the wake of warming at 2850C is positive from the view-point of limited arrangement of Cu/Cu₂S nanocomposites at chose response locales and their expected entanglement in the generally unblemished polymer organization.
- d) XRD designs likewise demonstrate the existence of Cu₂O tops at $2\theta \sim 36.540$, 42.490 and 61.470 . It is fascinating to see that the power of Cu₂O top (at $2\theta \sim 36.540$) increments with expansion in the polymer content in correlation with the Cu top (at $2\theta \sim 43.280$).

Molar ratio	Cu(C ₃ H ₃ O ₂)H ₂ O:PPS heated at 285 °C for 6 hrs								
	Cu ₂ S			Cu			Cu ₂ O		
	Peak at D (Å)	Particle Size (nm)	Mean Size (nm)	Peak At d (Å)	Particle Size (nm)	Mean Size (nm)	Peak At d (Å)	Particle Size (nm)	Mean Size (nm)
1:1	3.0213	21	24	1.8192	23	23	3.6626	27	24
	2.4012	24		----	----		2.4492	27	
	2.0119	26		----	----		2.1252	20	
	3.4241	25		----	----		1.5047	20	
1:5	----	----	----	2.0814	23	21	2.4609	19	23
	----	----		1.8057	21		2.1309	23	
	----	----		1.2763	18		1.5105	17	
	----	----		----	----		1.2865	32	
1:10	----	----	----	2.0777	21	23	2.4557	18	18
	----	----		1.8030	23		----	----	
	----	----		1.2749	24		----	----	
1:15	----	----	----	2.0750	23	23	2.4557	18	18
1:20	----	----	----	2.0786	19	19	2.4661	17	17

Table 1 : For the determination of sample size

From the table 1, it is observed that the level of oxide stage is more in 1:20 molar proportion sample than that of in the 1:5 molar proportion and the sample shows average X-ray diffraction example of powder samples relating to three diverse molar proportions 1:1, 1:10, and 1:20 of copper carbonate with PPS.

The main observations from the readings are tabulated as :

- a) All XRD examples of warmed admixtures of copper carbonate with PPS show power of monoclinic CuO which can be indexed by conspicuous diffraction tops at $2\theta \sim 35.30, 38.70, 53.40, 61.60, 66.10, 68.00, 72.30$ and 75.200
- b) Presence of significant diffraction top at $2\theta \sim 20.0$ on the whole XRD designs propose maintenance of PPS.
- c) XRD design identified with all molar proportion samples likewise uncovers the development of tetragonal $\text{Cu}_{1.96}\text{S}$ sub-stage which can be decided by trademark diffraction tops at $2\theta \sim 27.70, 31.80, 48.80, 53.40, 61.70, 66.00$ and 77.10
- d) Moreover, the XRD designs likewise show existence of cubic Cu_2O tops (for 1:10 molar proportion) at $2\theta \sim 36.20, 42.00$ and 73.50

4. Conclusion:

From the various observations in the present paper, it can be successfully concluded that Nanocomposites comprising nanoparticles of $\text{Cu}_2\text{O}/\text{Cu}_2\text{S}$ have been effectively arranged utilizing a basic, financially savvy, natural agreeable, less perilous polymer inorganic strong state response technique[11]. Also, the XRD investigation infers that solitary copper acetic acid derivation and copper nitrate structure give Copper nanocomposites when responded with PPS. Nonetheless, the copper carbonate yield copper lacking construction $\text{Cu}_{1.96}\text{S}$ though copper chloride remains unreacted for certain hints of $\text{Cu}_{1.96}$. All samples show the presence of nanoparticles implanted in the polymer network[12]. As the polymer content in the samples builds, the quantity of nanoparticles is accessible on a superficial level reductions.

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