Synthesis and characterization of copper sulfide

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ABSTRACT :
In this paper, the copper sulfide (CuS) nanomaterial was prepared by a hydrothermal process, using copper chloride and Thiourea. The copper sulfide (CuS) nanostructure and particles are invested. The nanostructure and morphologies of the copper sulfide (CuS) material was characterized by using powder X-ray diffractometer, Field Effect Scanning Electron Microscopy, Energy Dispersive X-ray analysis and Fourier Transform Infra-Red Spectrometer respectively.The XRD patterns revealed the formation of hexagonal structure of covellite CuS and crystal size was determined by using Debye Scherrer formula was found to be 17.11 nm in the Nano range.
Keywords: Hydrothermal, copper sulfide, XRD, FESEM, crystal size.

I.INTRODUCTION

Today the High performance electrical energy storage devices are more required in our life. The CuS has attracted great attention, because of its optical and electrical properties. Copper sulfide is an excellent material for optoelectronic applications. Copper sulfides are demonstrated the potential for applications in an extensive collection of energy sectors, including catalysts, solar cells and gas sensors. Copper sulfide serve as an important base material as absorber coating and is broadly used in photo voltaic and photo detectors applications due to its exclusiveadjacent solar controllingfeatures. In this paper, we have successfully synthesized CuS nanoparticles using hydrothermal method and examined various properties and it go under a number of characterization techniques.

II.EXPERIMENTAL SECTION

Copper chloride and Thiourea were analytical grade and used as received. All chemicals were used without further purification. All the solution were prepared in DI Water.

Preparation of CuS:

In this work, the CuS was prepared by hydrothermal method. In this method, 2 mmol of copper chloride($CuCl_2.2H_2O$) was dissolved by 60 ml deionised water, immediately pale green colour solution was formed in which 5 mmol of Thiourea (CH_4N_2S) was added under stirring 600^0C for 1 hour to achieve homogenous mixing. The solution was transferred into a Teflon-lined stainless steel autoclave with the volume of 50 ml.

The autoclave was heated at 150^0C for 24 hours. After that the autoclave was brought to room temperature naturally. () The black colour precipitate was recovered carefully, washed with DI water in several times. The sample was dried at room temperature. The sample was put into the silica crucible for annealing at 400^0C for 1 hour and then retrieve from the furnace after 24 hours and cool at room temperature and grind well to get fine powder for further characterization.

III.CHARACTERIZATION

The morphology of the products were studied by Field Emission Scanning Electron Microscopy (FESEM, Carl zeiss microscopy pvt.ltd, UK) with an X-Ray energy dispersive spectrometer (EDAX). The phase and crystallographic structure were identified by X-Ray Diffractometer (XRD, Panalytical, Netherland and x'pert^3 powder) analysis was performed by using a Cu Kα radiation. Diffraction patterns were recorded from 5^o to 80^o and Fourier Transform Infrared Spectrometer (FT-IR).

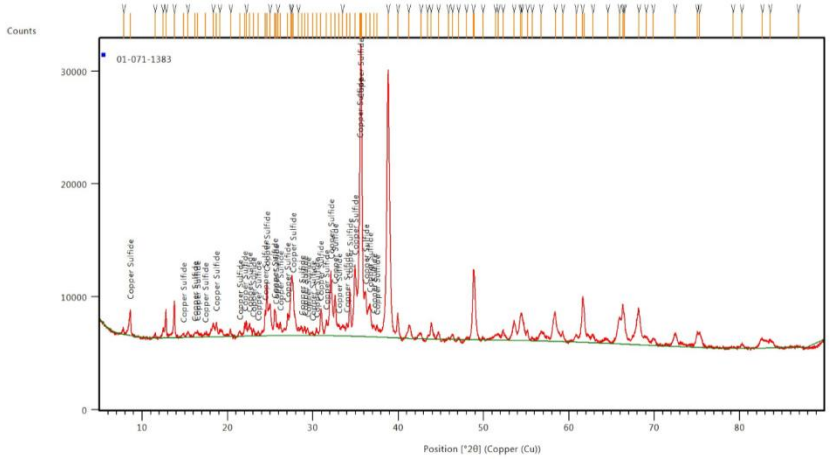
IV.RESULTS & DISCUSSION

Structural Analysis

The structural analysis was performed by using an X-Ray diffractometer. The X-ray generator was operated at 45kV and 30mA. The scanning regions of the diffraction angle (2θ) where 5-89° and Cu kα radiation were used to collect the spectrum. The step interval was kept 0.013° with a scan rate at 48.1°/sec. The diffraction patterns showed four broad peaks that could be indexed to the hexagonal covellite crystalline phase of CuS with characteristic (1 01), (1 02), (1 03) and (00 6) and in good agreement with the standard data for CuS (JCPDS Card No. 06-0464). Average particle size can be calculated from the XRD pattern using the well-known Debye – scherrer formula given in the equation 1.

$$D = \frac{K\lambda}{\beta \cos\theta} \qquad (1)$$

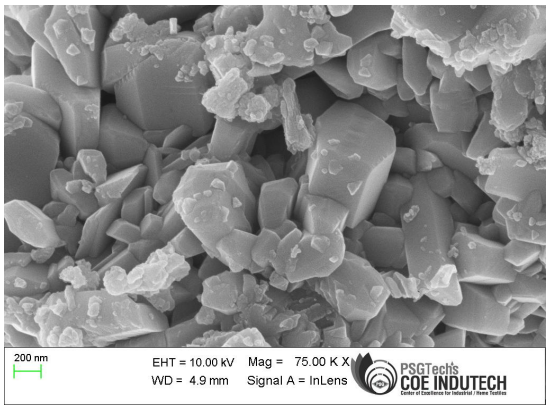
Where, D is the crystalline size, k is the shape factor, β is the Full Width Half Maximum (FWHM) of the highest peak in radiation, λ is the wavelength of the incident X-ray beam used for measurement. Then the calculated particle size D is 17.11 nm as shown in fig. 1. It was proved that pure phase nano-copper sulfide was synthesized in the experiment; no other diffraction peaks were observed, indicating that the material was of high purity.



Fig(1)X-Ray Diffraction for CuS

Surface Morphology Analysis

Field emission scanning electron microscopy (FESEM) was used to study the morphology of nano-copper sulfide. Figure 2a, b shows the FESEM images of copper sulfide at different magnifications. Fig 2a reveals that low magnification, copper sulphide consists of nano plate like structure with uniformly arranged and contain tiny porous were observed. Fig.2b shows the microscopic morphology of copper sulfide at a higher magnification; it can be seen from the figure that there are some particle deposits on the nano plate like shaped paricles with less cracks and some agglomeration. This is clearly revealed that CuS nano particles are irregular shape with the hexagonal aggregated structure was distributed. These results conclude that the surface morphology strongly influenced to CuS nano particles.



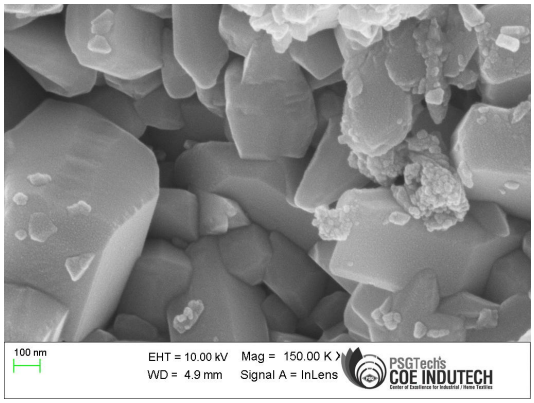


Fig 2a. Sem image for 200 nm, b. Sem image for 100 nm

Elemental Analysis

EDAX analysis detects X-ray emitted from the sample during bombardment by the FESEM electron beam and characterizes the composition of the analysed volume. The maps shows lateral distribution of chemical elements and compositional profiles across the surface, it confirms the presence of copper (Cu), sulphur (S) andoxygen (O) in the sample as shown in below.

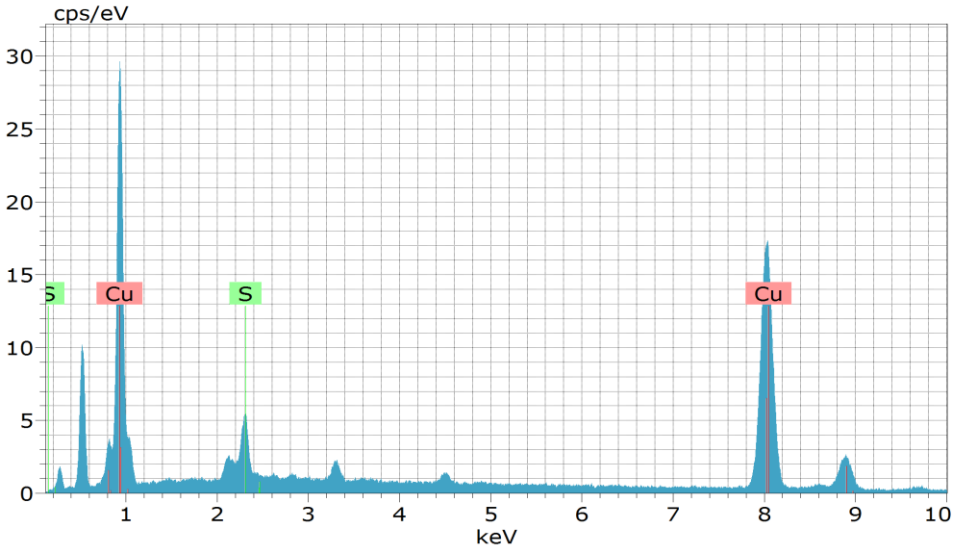


Fig (3) EDAX analysis result

FTIR analysis:

The sample was exposed to the IR radiation in the pallet form the pallet is made by mixing a very small amount of the sample into potassium bromide (kBr) and the mixer is grinded properly for uniform distribution of the sample into the kBr base.

Finally this grinded mixture is pressed in a hydraulic press applying a pressure of 7 to 8 tons. The spectrum is acquired in the range 4000-600 cm^{-1} and resolution of $2cm^{-1}$.

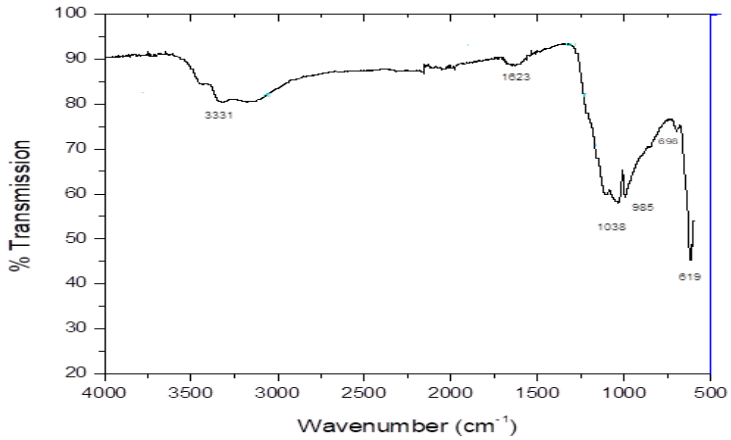


Fig (4) FT-IR result analysis

The characteristic band at 3331 cm^{-1} corresponds to the vibration mode of water indicating the presence of small amount of water absorbed on the sample. The band occurring at 1038 cm^-1 is due to the OH bending of water.

The absorption band located at 985 cm^{-1} is due to asymmetric stretching of carbonyl (C=O) group .The peaks at 619 signify the existence of Cu-O bond.

CONCLUSION

The copper sulfide CuS Nanocrystals are prepared by hydrothermal method by using copper chloride for a copper source and Thiourea for a sulfur source at 150^0C for 24 hours reaction time without adding any surfactant. The structure of the sample has been studied by using XRD image and the morphology was studied by using FESEM images. The chemical elements are analyzed by using EDAX and FT-IR.

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