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Synthesis and Characterization of copper oxide(II) nanoparticles prepared by hydrothermal process

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Abstract

Hydrothermal process was used to prepare CuO nanoparticles. CuO nanoparticles can be prepare without organic solvents, expensive raw materials by a hydrothermal method. XRD diffraction reveals that CuO nanoparticles have a monoclinic structure with particle size 20nm , and AFM analysis showed that the diameter of the Grain size is in a nanometer range. The analysis by FTIR spectra assure that the composition was CuO, and the features of vibrational types of Cu–O were fixed. also the optical properties was analysed with UV–vis showed that CuO nano particles have considerable a blue shift , which have aband gab equal to (4.9 eV) , and this is because the effect of quntum confienment of prepared CuO nano particles.

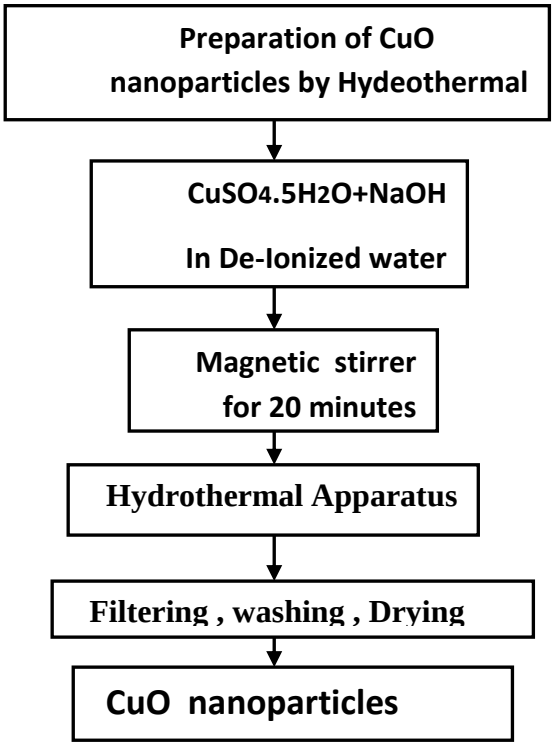
1- Introduction

The nano-materials have attractive at concern, because of different properties. New properties such as physical properties of nano materials, chemical properties, also thermodynamic which is expected to found in such p-type [1,2,3]. It is arising from the a great part of low coordinated atoms at the surface, and the confinement of the electrons to a small volume, respectively [4,5,6]. CuO is a semiconductor with type-p, and have a band gap of [1.8 – 2.5] eV. Copper oxide (II) is one of the most intensified studied metal oxide, because of its applications in many sectors, such as catalysis, solar cells, semiconductors, transistors and gas sensors [7,8,9].

CuO is known as cupric oxide, have attracted attention from scientists , because of its alone physical and chemical features , such as high electron communication features, also a high specific surface area, and high solar absorbency ,All these properties are relative to its diversity of nanostructures [10,4,2]. There are many methods to the prepare CuO nanostructures including hydrothermal, thermal oxidation, electrochemical and solvothermal ,simple hydrolysis , Microwave assisted, and sol-gel methods[11,12,7].

2- Experemental:

Hydrothermal method is a low and simple coast method to produce CuO nanostructures**.** Copper oxide nanoparticles were prepared by hydrothermal process. The preparation scheme started with dissolving 0.1M of Copper sulfatepentahydrate CuSO4.5H2O in distilled water to obtain blue solution , then 0.2 M of sodiuom hydroxide NaOH solution was added by dropwise respectively with vigorous stirring for 20minutes , The pH value of the solution equal to 8.The resultant solution was transferred to the stainless steel (autoclave) , and hydrothermal synthesis was carried out for about 160°C for 8h. The obtained black precipitate was filtered and cleanded with (washing by Eathanol then deionized water several times). The final product is dried at 70 °C for 2 h. fig 1 shows diagram to prepare CuO nanoparticles:



Figuer 1: Shows the diagram to synthesis of CuO nanoparticles by hydrothermal method

3- RESULTS AND DISCUSSIONS

3-1 XRD analysis

The X-ray diffraction was carried out on the prepared CuO nanoparticles with the (Philips pw system) by using(copper) CuKα as a source of radiation which having a wavelength of 1.540 $\mathring{A}$.The XRD shows that the prepared particles was a copper oxide and have a monoclinic structure, fig.2 depicts the XRD of CuO nanoparticles .

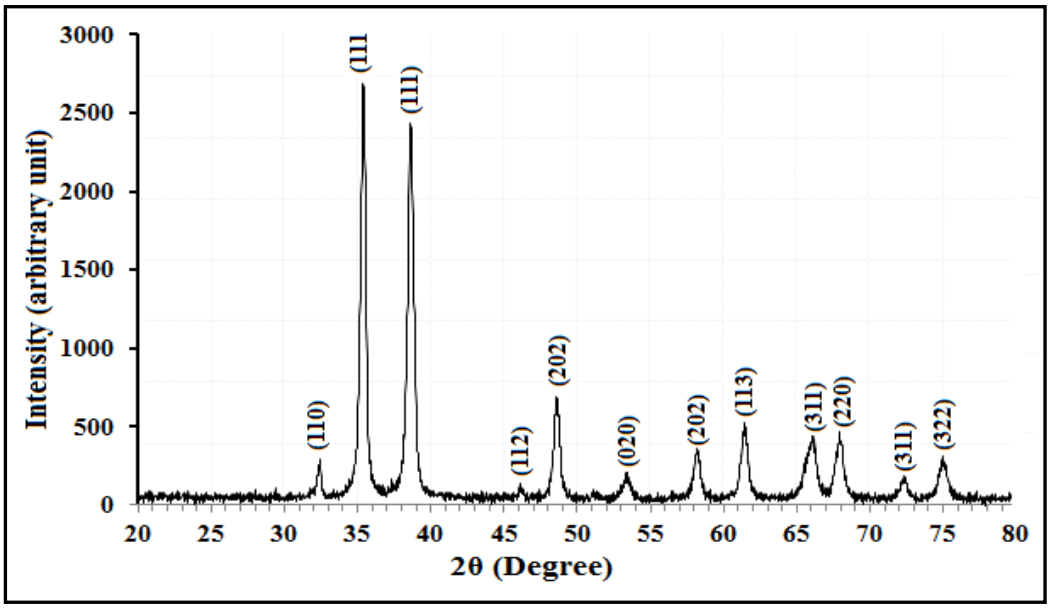


Figure2: XRD pattern of CuO nanoparticles with NaOH=0.2/0.1M.

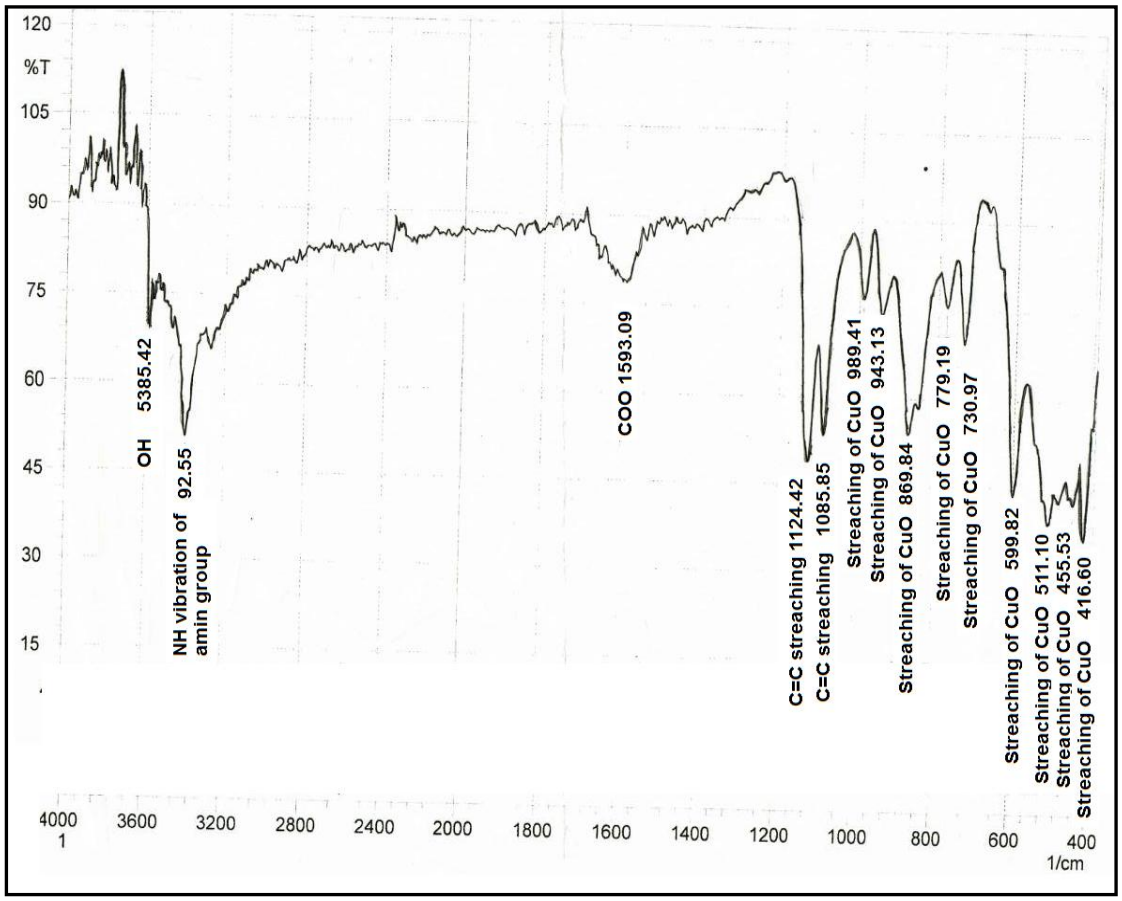
Also the crystallite size was found by using (Debye Scherrer equation **):**

$$d = K\lambda / \beta\cos\theta \quad \text{.....(1)}$$

d: represent the average of particle size (nm) and K: is (shape factor and equal to about 0.9) , λ : is the wavelength in nanometer of the X-ray, β: represent a full width at half maximum (FWHM) of the diffraction angle, and θ represent the Bragg diffraction angle of the 2θ peak**.** The average particle size was estimated to be ~20 nm.

3-2 FT-IR analysis

The FTIR Spectra was analysed by (470infrared –spectrophotometer shimadsu), and the results are shown in fig.2 This technique was used to determine a functional groups of prepare CuO nanoparticles oxide. The strong intensity peak at 3585.42 cm^{-1}assinged to alkyl (O-H) streaching for the deformation of the diethylamine , 3392.25cm^{-1}assigned to (N-H) vibration of amin group, weak band at 1593.09cm^{-1} assigned to carboxylic group (COO) vibration, while the positioned at 1124.24 cm^{-1}revealed the presence of (C=C) streaching for alkyl.The characterstic peak of CuO positioned at 989.41 to 416.60 cm^{-1} [12,8,11].



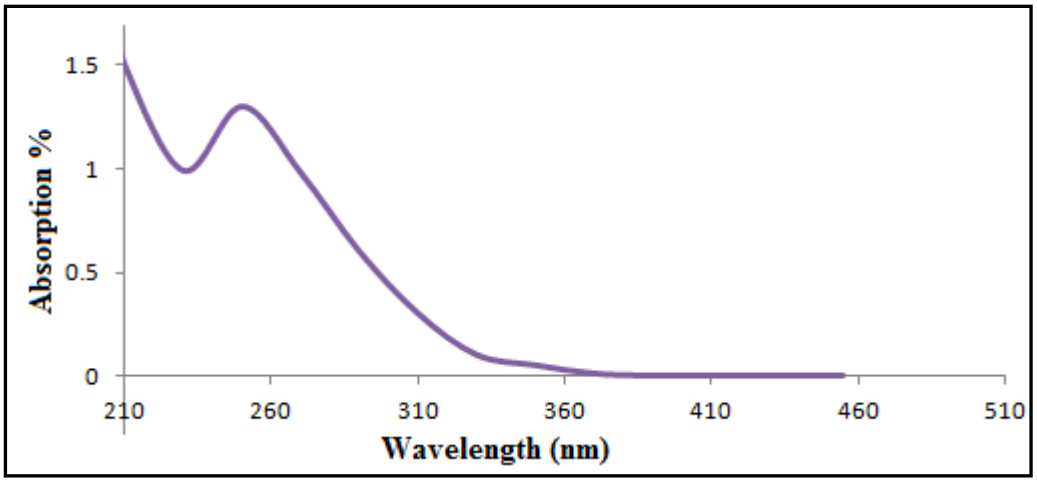
Figuer 3: FTIR analyzing of CuO nano particles.

Table (1): show the FTIR values .

CuO (cm^{-1})	Vibrational modes
3585.42	OH
3392.25	NH vibration ofamine Group
1593.09	COO
1124.42	C=C stretching
1085.85	C=C stretching
989.14	Stretching of CuO
943.13	Stretching of CuO
869.84	Stretching of CuO
779.19	Stretching of CuO
730.97	Stretching of CuO
599.82	Stretching of CuO
511.10	Stretching of CuO
445.53	Stretching of CuO
416.60	Stretching of CuO

3-3 UV- Visible spectroscopy:

Optical properties of the prepared CuO anoparticles are researched by the Spectrum of the optical absorption with the(T90UV Spectrometer System).The absorption spectrum of CuO nanoparticles shows a sharp absorbance onset at 252 nm . Fig.3 shows spectrum of the optical absorption.



Figuer4: shows UV – Absorption spectra of CuO nano particles.

3-4 Determination the(Eg) of synthesizing CuO nanoparticles .

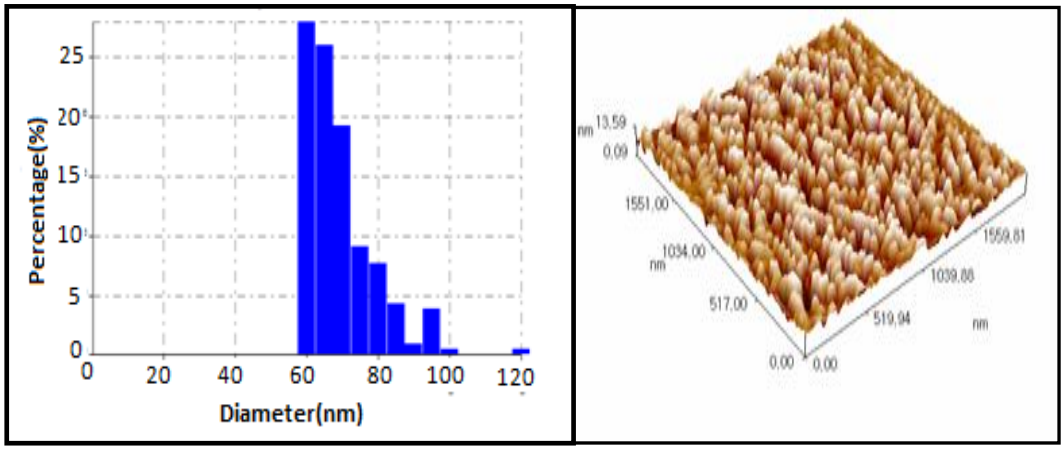
According to UV-V technique , by using the absorbance peak to find the band gap energy of synthesized CuO nano Particles with equation [8,12].

$$E_g = h\upsilon_g = hc/\lambda \quad \ldots\ldots\ldots\ldots\ldots\ldots 2$$

Where Eg represent the optical band gap and (h) is plank's constant (h= 6.63×10^{-34}J.S) and, (υ_g) frequency of the emitted radiation . Using λ =252 nm , The band gap of nano CuO equal to be (4.9) eV, and this mean that the band gap is higher than the value (2.5) eV of bulk CuO ,this is due to the quantum size effect of the synthesized CuO nanoparticles [6,7].

3-4 Atomic Force Microscope:

Atomic force microscope used to study surface of the samples (SPM-AA 3000 USA)model. the average grain size of surface nanoparticles was measured , fig. 3 shows a typical surface AFM images, the average of diameter was (65) nm, and the maximum grain size distribution is located at 60nm. it is clear that the particle size is more bigger than in x-ray . also there is abrigth and dark regions which is explain the difference in the roughness of nanoparticles.



Figuer4: AFM images of CuO nano particles and distribution of particles with

4-**Conclusions**

In our study CuO nano particles were synthesized successfully with a hydrothermal method. The phase structured confirmed by XRD analysis and have a monoclinic phase, also particle size were an (20nm). The blue shift of wavelength was obtained by UV-Visible, and it is equal to 250nm. AFM analysis shows that the average of diameter was 65nm. These particle sizes of obtained CuO nano particles can be used in such application ; anti bacterial applications , sensing gas.

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الخلاصة:

تم استخدام طريقة الهدرجة الحرارية لتحضير جسيمات اوكسيد النحاس النانوية , يمكن تحضير جسيمات النحـــاس النانويـــة بـــدون استخدام المذيبات العضوية او مواد غالية الثمن بأستخدام الهدرجة الحرارية. حيود الاشعة السينية أكد بان جسيمات النحاس النانويـــة ذات تركيب احادي الميل , مع حجم حبيبي 20nm , كما ان التحليل بأستخدام مجهر القوة الذرية اوضح بأن قطر الحجـــم الحبيبـــي هو في ضمن المدى النانوي . التحليل بواسطة طيف (FTIR)اكدت لنا بأن التركيب هو اوكسيد النحاس كما ان خصـــائص نمـــط الاهتزاز لأوكسجين– نحاس تم تأكيده. تم دراسة الخواص البصرية بأستخدام الطيف المرئي للاشعة فوق البنفسجية والتي اوضحت بأن جسيمات النحاس تمتلك انحراف بأتجاه المنطقة المسماة (blue shift) , حيث تمتلك فجوة عاليـــة طاقـــة (4.9eV) , وان هـــذا ربما يعود الى تأثير الحصر الكمي لجسيمات اوكسيد النحاس النانوية.

الكلمات الدالة:عملية مائية ؛ السلف؛ جسيم نانوي CuO ؛ FT-IR ؛ AFM. تقنية الأشعة فوق البنفسجية.