RESEARCH PAPER

Antibacterial Activity of CuO - Cellulose Nano rods Depends on Anew Green synthesis (cotton)

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ABSTRACT

In this study CuO nano sheets were prepared using the cellulose extracted from green synthesis (cotton) as a novel me project. Structural properties were examined using X-Ray Diffraction (XRD), Field Emission Scanning Electron Microscopy (FESEM), and Ultra Violet (UV-Vis). The optimum copper oxide peak was at 2 theta 35.44^{o} corresponding to ($\bar{1}11$) while for the cellulose was 22.8^{o} corresponds to (002). FESEM images of CuO nano sheets were relatively homogenous with diameters less than 30 nm. The UV-Vis for CuO-cellulose nano rods was observed at 350-360 nm, which is higher than of pure CuO nano sheet. The energy band gaps were 3.20 eV and 3.30 eV of CuO and CuO-CNR respectively. Finally antimicrobial activities of samples have been investigated against the Gram positive (pneumonia) and gram-negative (pseudomonas). The maximum antibacterial activities against the Gram positive (pneumonia) of CuO nano sheet and of CuO-cellulose nano rods are 16 mm and 19 mm respectively. The maximum antibacterial activities against the Gram negative (pseudomonas) of CuO nano sheet and of CuO- cellulose nano rods are 30 mm and 33 mm.

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INTRODUCTION

Cellulose is natural polymer material a renewable with powerful mechanical properties, wide chemical modulation ability and semi crystalline morphology [1]. CuO nano sheets have been receiving large attention for their possibility usage in optoelectronics, nano device, nano electronics, nano sensors, information stores, and catalysis. through different metal oxide NPs and CuO have enticed special attention because they are the easy member of the family of copper compounds and shows a range of advantageous physical properties such as rising temperature superconductivity, electron correlation influence , and spin dynamics [2].

Crystal structures of CuO have a narrow band gap, giving good photo catalytic and photovoltaic property [3]. Microbial pollution of air, water, and earth due to various kinds of bacteria makes problems in living conditions and is a dangerous problem in health care. Due to the diffusion of antibiotic resistant infections, attention in substitutional antibacterial factors such as small antibiotics, cationic polymers, metal NPs, and antibacterial peptdes have been rising [4].

However, for food packaging applications and biomedical of cellulose based on nanocomposites, contamination from bactera is a large safety challenge from food to water because cellulose does not have antibacterial property[5]. Through last two decades and many researches have outlay their potential to improve antibacterial cellulose nanocomposite films. The efficiency of semiconductor based on antibacterial system such as CuO straight depends on the capability to produce electron–hole couple [6]. As a result,

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the biosynthesized of CuO NPs ability does as a futuristic active antibacterial factor in the biomedical range [7].

MATERIALS AND METHODS

The following materials have been incorporated into this work medical cotton from Iraq / Bagdad purity 100% , Sulfuric acid origin Germany /Scharlau purity 98%,ethanol origin USA/ Sigma Aldrich purity 100% , Sodium hydroxide origin Newzealand/Ajax-Finechem purity 97% , ($Cu(NO3)2.3H2O$) origin Germany purity 99.99% and distil water origin Iraq / Baghdad purity 100%.

Preparation of Cellulose Nano Rods (CNR)

A nano cellulose fibrils are prepared from raw cotton linter that acidic hydrolysis treatment. The method of acidic hydrolysis was applied with minor adaptations. The cottonwas mechanically stirred at a ratio of 2:20 (w/v) of aqueous concentrated sulfuric acid (64%, w/w) with a Teflonc bar dispersing element, at 45 ◦C, for 60 min. The suspension was centrifuged for 15 min at 6000 rpm , and the precipitate was resuspended in distilled water and dialyzed until a pH (6–7) was reached. The process from centrifugation through dialysis was repeated three times.

Preparation copper oxide nano sheets

CuO nanostructures were synthesized by drop casting method using copper nitrate ($Cu(NO3)2.3H2O$). (1) g of copper nitrate dissolved in 50 ml of distilled water and placed on the mechanical stirrer at 90 ° C. Then drops of sodium hydroxide were added to the mixture to change the PH value to reach pH of the mixture to 14. The mixture is left on the stirrer for two hours. The material is filtered and washed with ethanol and distilled water then dried on a slid of glass substrate at 90 °C for 1 h.

Preparation CuO – CNR

In a normal procedure, CuO-cellulose nano rods were synthesized by suspending the CNR in distil water, then mixing with cooper nitrate ($Cu(NO3)2.3H2O$) solution by magnetic stirrer. The weight ratios of ($Cu(NO3)2.3H2O$:CNR) were (1:2)g. After complete mixing, the mixture put on magnetic stirrer at 45 °C for 1 hours. The mixture is placed in the centrifuge for 15 minutes and washed using ethanol and distil water to remove the Residues of sodium hydroxide and additional CNR. After complete washing, the specimen were dried at 100 °C for 2 h to finish the transference of mixture to CuO-cellulose nano rods.

RESULTS AND DISCUSSION

X-ray diffraction

Fig (1) reveal CuO film of eight pronounced diffraction peaks, (31.85°, 35.44°, 38.76°, 48.69°, 58.69°, 611.51°, 67.97°, and 74.97°) corresponding to (110), ($\bar{1}$11), (111), ($\bar{2}$02) , (202) ($\bar{1}$13), (113), and (113) planes of the crystallized structure of CuO respectively. It can be seen that prepared nanocomposites have a pure monoclinic CuO (JCPDS 00-048-1548). Fig (2) show XRD of cellulose film with three pronounced diffraction peaks at 2θ = (16.3°, 22.6°, and 34.7°) corresponds to the (1 0 1), (0 0 2), and (0 4 0) crystallographic planes, respectively. It can be seen that prepared nanocomposites have

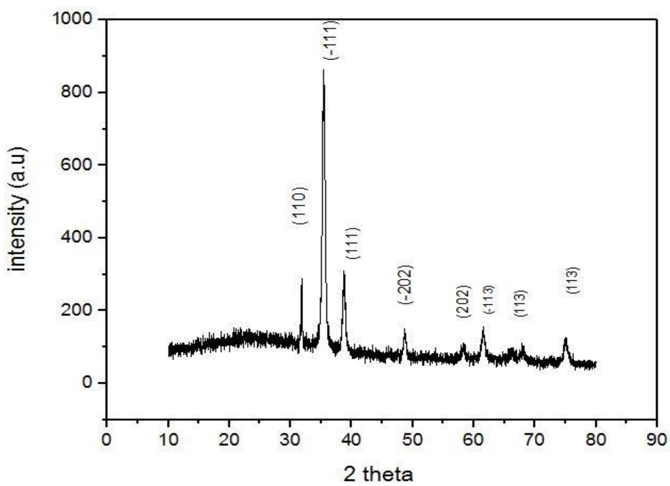

Fig. 1. XRD of CuO nano sheets.

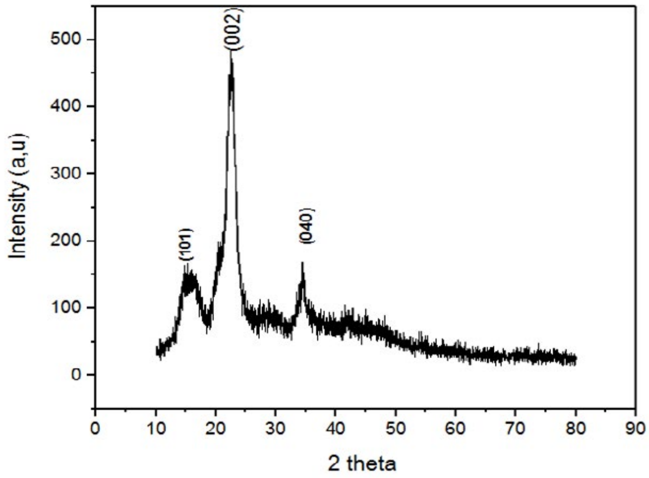

Fig. 2. XRD of pure cellulose nano rods.

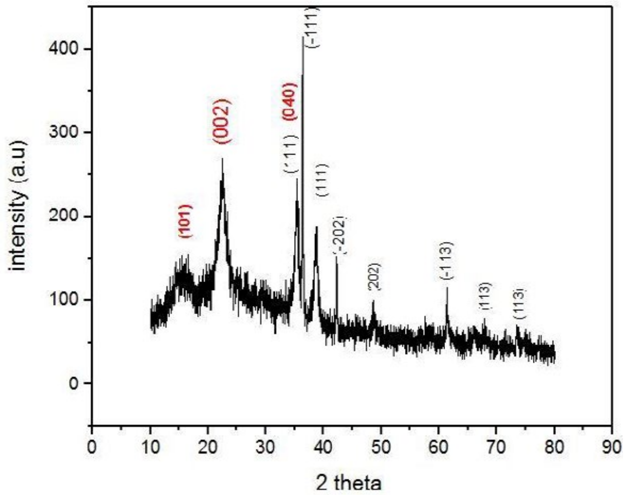

Fig. 3. XRD of CuO- Cellulose nano rods.

cellulose (JCPDS 00-050-0926) Fig (3) show XRD of CuO-cellulose the cellulose pronounced diffraction peaks of 16.3, 22.6, and 34.7 corresponds to the (1 0 1), (0 0 2), and (0 4 0) crystallographic planes and CuO diffraction peaks are (31.85°, 35.44°, 38.76°, 48.69°, 58.69°, 611.51°, 97.97°, and 74.97°) corresponding to (110), $(\bar{1}11)$, (111), $(\bar{2}02)$, (202) $(\bar{1}13)$, (113),and (113) planes. The full width at half-maximum (FWHM) of the peaks corresponding to the samples of CuO , cellulose and CuO- CNC crystals are narrowing peaks form. Crystalline sizes is calculated from Scherrer equation, $Dc = K\lambda/\beta Cos\theta$ [8].

Morphological properties

Fig (4) shows the FESEM images of pure CuO nano sheets, cellulose nano rods and and CuO–cellulose nano rods deposted on glass substrates. (FESEM) is a convenient method to show a close packed morphology of CuO and cellulose, which decrease in grain size with increase cellulose material. In copper oxide it observe the nano sheets forms while in cellulose it observe the nano rods forms. When mixing cellulose with copper oxide and exposing it to 90 ° C, it observe the nano rods more than the nano sheets which disperse in the CuO- cellulose mixture. The treatment of cellulose powder with both CuO and NaOH solutions led to the nucleation and growth of discrete CuO seeds at the cellulose surfaces.

Optical properties

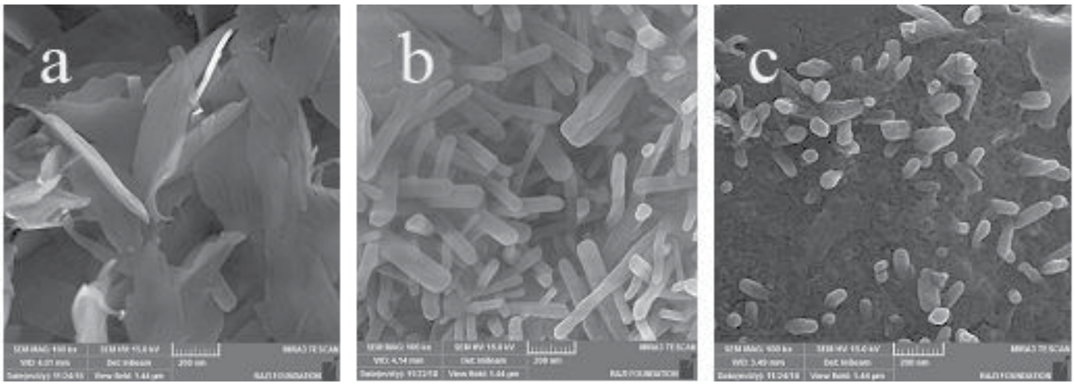

Fig. 4. FESEM images of a) pure CuO nano sheets b) pure cellulose nano rods and c) CuO – cellulose nano rods.

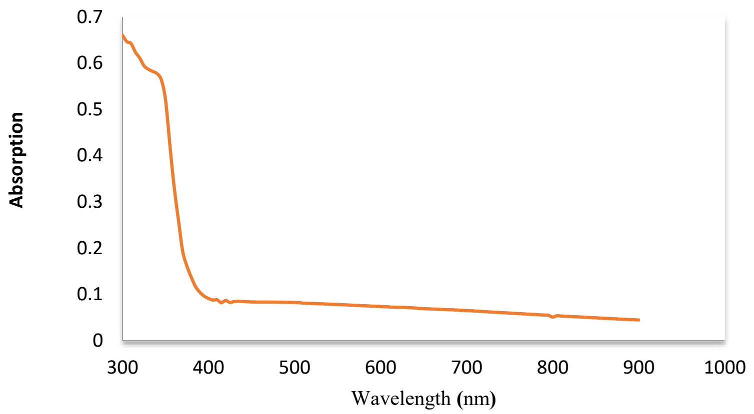

Fig. 5. UV-Vis absorption spectrum of CuO nano sheets.

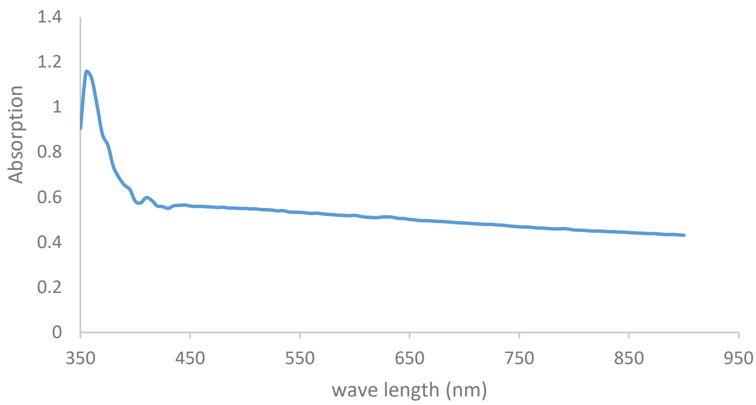

Fig. 6. UV-Vis absorption spectrum of CuO – cellulose nano rods.

UV-Vis absorption spectrum

UV-vis absorption spectrum of CuO nano sheets is shown in Fig. (5). The excitation absorption peak of the sample is 300 nm. The UV-Vis absorption spectrum of CuO-cellulose nano rods is shown in Fig. (6). The absorption peak of the sample is 360 nm. The increase surface area of nanoparticles and their uniform distribution on the cellulose surface might have increase the UV absorption efficiency.

Energy band gap

Fig. (7) show the direct energy gap of CuO nano sheets. The better linear relation is obtained by drawing $(\propto hv)^2$ versus (hυ) the band gap

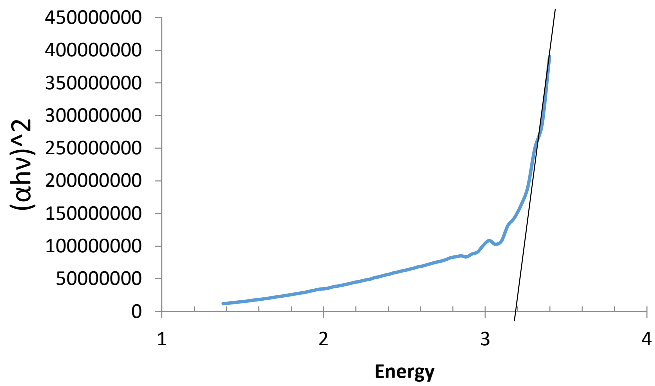

Fig. 7. Energy band gap of CuO nano sheets.

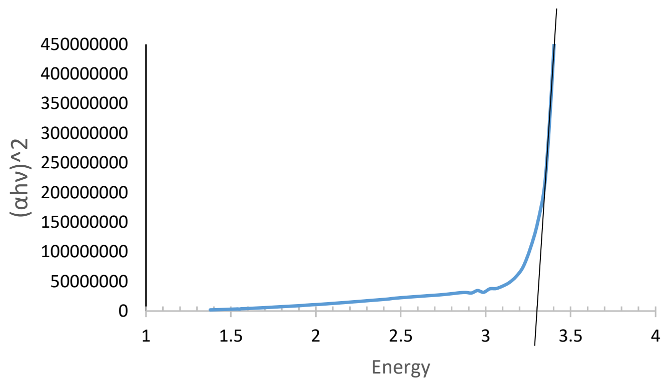

Fig. 8. Energy band gap of CuO – Cellulose nano rods

of the CuO nano sheets is due to a immediate allowed transition. The amount of the energy gap is calculate from the intercept of the direct line at a = 0, which is equal to 3.2 eV. This amount is higher than the of bulk CuO (1.2 eV). The direct band gap of CuO-cellulose nano rods estimated from a a drawing of $(\propto hv)^2$ versus (hυ) is 3.30 eV as shown in Fig (8). energy gap increases with reduceing particle size due to effects of quantum confinement . When photons are incident on the semiconductor material they will be absorbed only when the lower energy of photons is sufficient to excitation an electron from the valence band to conduction band or when the energy of photon equal to the energy gap of the material. The syntheses nanoparticles are very confine and the absorption spectrum of it be more structured because its electronic band structure changes to molecular level with non demise energy spacing. So the material needs more power for electronic transition from valence band to conduction band. Hence the energy gap of CuO nano sheets is more than that of the bulk.

Antibacterial activity

The antibacterial activities were carried out by the disc diffusion method. Antimicrobial activities of samples (CuO nano sheet, and CuO-Cellulose nano rods) have been investigated against the Gram positive (pneumonia) and gram-negative (pseudomonas). The maximum antibacterial activities against the Gram positive (pneumonia) of CuO nano sheet and of CuO- cellulose nano rods are 16 mm and 19 mm respectively. The maximum antibacterial activities against the Gram negative (pseudomonas) of CuO nano sheet and of CuO- cellulose nano rods are 30 mm and 33 mm respectively. The antibiotic Gentamicin(CN) was applied to Gram positive (pneumonia) and found that the killing area of antibiotic (Gentamicin CN) is 17 mm either For pseudomonas, the antibiotic (genemycin) did not kill the bacteria but was discouraged and the area of discouraged was (20 D) mm and the antibiotic (Erythromycin (E)) did not give any effect on gram-negative and gram-positive bacteria indicated an enhancement by composition of CuO- cellulose nano rods. The area

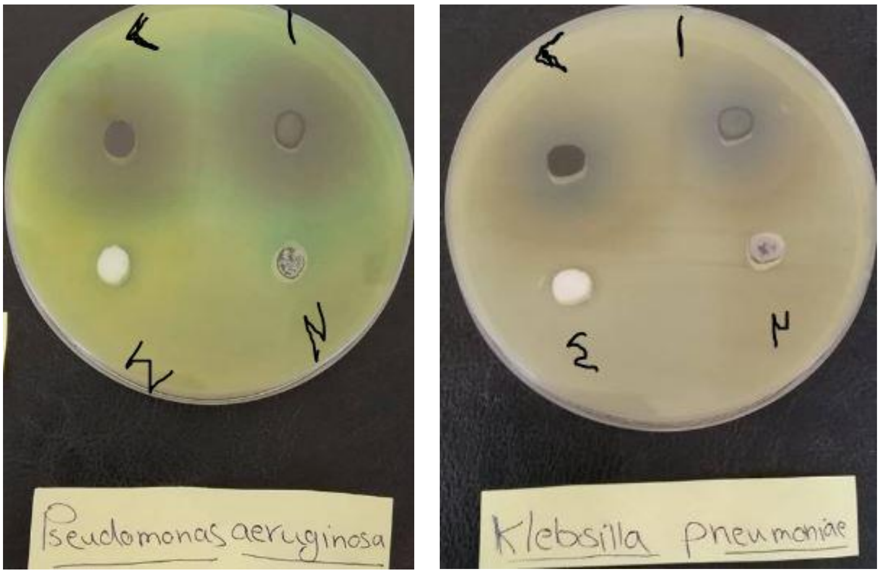

Fig. 9. Optical micrographs of agar plates, showing the variation in the zone of inhibition zone (1) CuO nano sheet (2) CuO-cellulose nano rod and (3) control (4) cellulose nanocomposite for pneumonia and pseudomonas.

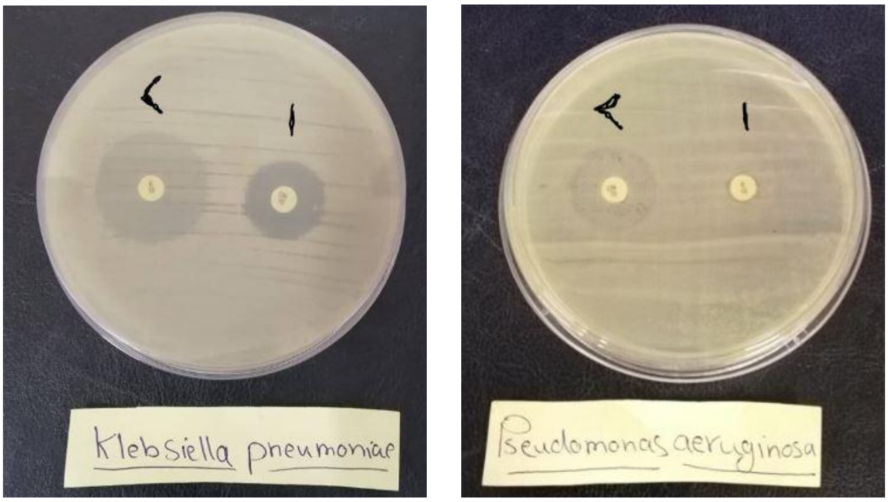

Fig. 10. Optical micrographs of agar plates, showing the variation in the zone of killing and inhibition zone (a (1) Gentamicin a (2) Erythromycin b(1) Erythromycin b (2) Gentamicin

of killing for all types of positive, negative bacteria and antibiotic (Gentamicin) , (Erythromycin (E)) is shown in Table (1) , Fig. (9) and Fig 10.

CONCLUSIONS

The average crystal size of CuO nano sheets have less than 30 nm were successfully synthesized with CNR using a drop casting method. Optimum copper oxide peak was at 35.44 corresponding to ($\bar{1}$11) while for the cellulose was 22.8 corresponds to (002) and the two peaks ($\bar{1}$11), (002) were optimum as the highest peaks of copper Oxide – Cellulose nano rods. In FESEM images the surfaces of cellulose, CuO and CuO-cellulose were relatively homogenous with nano rods and nano sheets and nano rods respectively. These nanoparticle are interlaced with each other, which create an excellent nano rods surface. The UV-Vis for CuO-cellulose nanocomposite synthesized at 100 °C was observed at 350-360 nm, which is higher than of pure CuO nano sheet. The cellulose was crucial for improvement of the nano rods of the CuO surface responsible for an enhanced absorption of UV radiation. The energy band gaps were 3.2 eV

Table 1. The area of killing for all types of positive, negative bacteria and antibiotic (Gentamicin), (Erythromycin (E))

N	compounds	*Klebsiella pneumonia*	*Pseudomons aerugino*
1	CuO	16	30
2	CuO- Cellulose	19	33
3	Control	-	-
4	Cellulose	-	-
5	Antibiotic **Gentamicin(CN)**	17	20 D(discouraged)
6	Antibiotic **Erythromycin** (E)	-	-

and 3.3 eV of CuO and CuO-CNR respectively.

The homogenous dispersion of CuO nano sheets in polymer blend matrix CNR, it can be concluded that the stabilization of CuO nano sheets by cellulose nano rods could help to increase their dispersion in the polymer blend matrix and prevents agglomerations. The maximum antibacterial activities against the Gram positive (pneumonia) of CuO nano sheet and of CuO- cellulose nano rods are 16 mm and 19 mm respectively. The maximum antibacterial activities against the Gram negative (pseudomonas) of CuO nano sheet and of CuO- cellulose nano rods are 30 mm and 33 mm respectively. indicated an enhancement by composition of CuO- cellulose nano rods.

The results caused by the direct reaction between the Nano particles and the surface of the cell membrane of the bacteria. moreover the results reveal a the optimum antibacterial activity by using semiconductor-polymer compositions. This equivelent antibacterial activity is due to the big surface to volume of Nano particle that increases their touch surface with the bacteria. Better results were obtained from certain antibiotics such as gentamicin and erythromycin.

CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this manuscript.

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