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Synthesis and Characterisation of Surfactant Coated Copper Oxide Nanoparticles for Antibacterial Application

J.Jeyasundari^{1*}, P. Vengatesh Priya¹, A.S.Sakthi Athithan¹ and A. Naveena¹

PG & Research Department of Chemistry, NMSSVN College / M.K University, Madurai, Tamilnadu, India^{1*}.

ABSTRACT: The surfactant coated CuO nanoparticles were synthesized by chemical reduction method and coated by cetyltrimethylammonium bromide (CTAB) cationic and sodium lauryl sulfate (SLS) anionic surfactant. The synthesized CuO nanoparticles were characterized by using UV-Visible (UV-Vis) absorption spectroscopy, Fourier Transform Infrared (FTIR) spectroscopy. The average particle size of the synthesized nanoparticles were determined by using X-ray diffraction spectroscopy (XRD) analysis with help of Debye –Scherrer relation. The surface morphology of surfactant coated nanoparticles were examined by Scanning Electron Microscopy (SEM). Antibacterial activity of surfactant coated nanoparticles were tested by disc diffusion method against Gram +ve bacteria (*Staphylococcus aureus*) and Gram -ve bacteria (*E.coli*, *Klebsiella pneumoniae*).

KEY WORDS: CTAB, SLS, Chemical reduction method, UV, FTIR, SEM, XRD

I. INTRODUCTION

Metal oxide nanoparticles has play a vital role in many applications [1]. The divergent properties of metal oxide has emerged from its nano size [2]. The size, shape and morphological character of nanoparticles were strongly modified by capping agent. when the metal oxide nanoparticles were coated by surfactant, which was reduced the size, change the morphology and also gives the stability to the nanoparticles [3]. In general surfactants are classified in to three types. They were cationic, anionic and zwitter ionic. Surfactant contains hydrophobic tail and hydrophilic head. The hydrophobic head was attached on the surface of nanoparticles [4]. Surfactants coating may prevent the aggregation of colloidal suspension [5]. Cu is one of the d block element and its electronic configuration is $3d^94s^2$. Among the d block element, Cu was easily abundant, cheaper also present in many biological molecules [6]. CuO nanoparticles has Specific properties and applications [7]. Chemical reduction method is one of the easiest and time consuming method [8]. In this present work CuO nanoparticles were synthesized by chemical reduction method and coated by cationic and anionic surfactants.

II. EXPERIMENTAL METHODS:

2.1 Materials:

Copper acetate monohydrate $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$, Cetyltrimethylammonium bromide (CTAB), sodium lauryl sulfate (SLS), Sodium borohydrate, (NaBH_4), Ammonia (NH_3) were purchased from sigma Aldrich with 99% purity.

2.2 Preparation of Surfactant Coated Nanoparticles:

The copper oxide nanoparticles were synthesized by chemical reduction method using copper acetate $\text{Cu}(\text{CH}_3\text{COO})_2 \cdot \text{H}_2\text{O}$, surfactant (CTAB & SLS) and NaBH_4 as precursor. About 80 ml of 0.1 N copper acetate added with 20 ml of CTAB 0.001 N solution, heated and stirred. Under constant stirring the reducing agent NaBH_4 was added as dropwise then added NH_3 for solution reach alkaline pH. The color of the solution was turned from green to bluish green and final to the black. After the completion of reaction the solution was allowed to settle for an overnight and the supernatant solution was discarded. The precipitate was filtered, washed with distilled water and it was dried in hot air oven. The dried sample was again calcinated in the muffle furnace. The same procedure was repeated for SLS coated nanoparticles with addition of 0.001 N SLS.



III. RESULTS AND DISCUSSION

3.1 UV-Visible Spectral Analysis:

The formation of CuO nanoparticles were determined by UV-Visible spectroscopy technique. This is the most widely used technique to analyze the optical properties of nanoparticles. The surfactant coated CuO nanoparticles exhibits the strong absorption band at 383 nm and 392 nm for CTAB and SLS coated nanoparticles. The significant sharp absorption peak of CuO nanoparticles indicates the poly dispersed nature of the nanoparticles distribution. The optical absorption peak were explained the surface plasmon resonance of metal and also shifting the initial blue-green turned into red brown. Metal Copper has free electron so it possess the surface plasmon resonance [9].

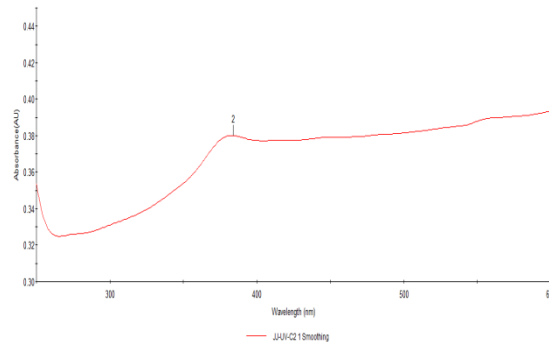


Figure.3.1 UV-Visible spectra of CTAB coated CuO nanoparticles

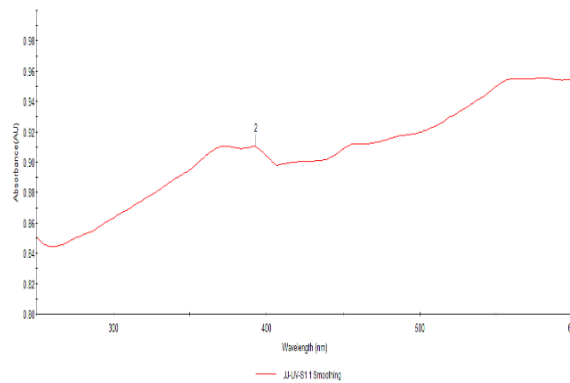


Figure3.2 UV-Visible spectra of SLS coated nanoparticles

3.2 FTIR SPECTROSCOPY ANALYSIS:

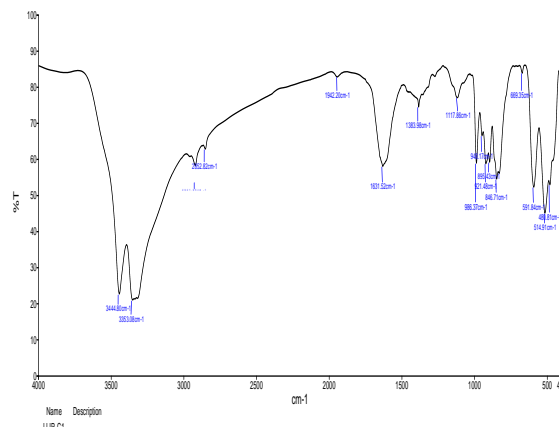


Figure.3.3 FT-IR of CTAB coated CuO nanoparticles

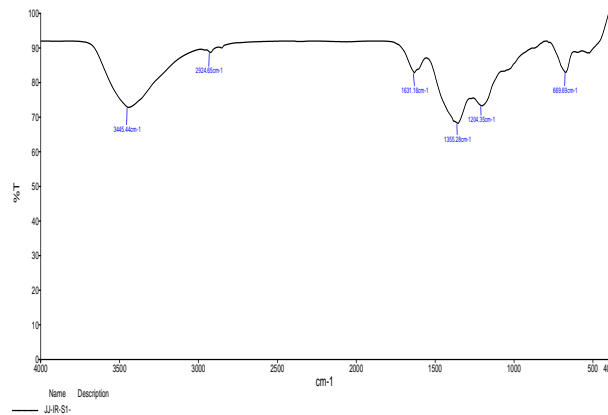


Figure.3.4 FT-IR of SLS coated CuO nanoparticles

The intensity peak observed at 669 cm⁻¹ corresponds to -C-Br stretching, which indicates that the presence of CTAB at the surface of the CuO nanoparticles [4, 10].

Table.3.1. FT-IR peaks comparison of CTAB and SLS coated CuO nanoparticles

CuO nanoparticles		
Assignment	CTAB	SLS
Rocking vibrational mode of H ₂ O molecule	895	-
Stretching frequency of -C-Br	669	-
CuO stretching frequency	591 & 514	669
-C=O stretching frequency	1631	1631
stretching frequency of -CO-NH amide group	1384	-
Stretching frequency of sulfone =S=O	-	1355
-C-O bending vibrations	1116	-
Symmetric and asymmetric stretching for -CH ₂	2921 & 2852	2928
Stretching frequency of -OH	3444	3445
Stretching frequency of -NH	3353	-

FTIR spectroscopy technique is used to identify the surfactant coated CuO nanoparticle. The two strong absorption peaks were absorbed at 514 and 669 cm⁻¹ indicate the formation of CuO nanoparticle, which was associated with the stretching vibration of Cu-O. The intensity peak observed at 3444 and 3353 cm⁻¹ corresponds to the stretching vibration of -OH and -NH. Another peak observed at 1355 cm⁻¹ indicates the stretching frequency of sulfone, which shows the presence of SLS at the surface of the CuO nanoparticles

3.3 X-Ray Diffraction Spectral Analysis:

The crystal structure of surfactant coated nanoparticles were determined by XRD analysis technique. The size of the particles were calculated by using Debye- Scherrer equation. The required equation was

$$D=0.9\lambda / \beta \cos\Theta$$

Where D - particle size

λ - wavelength of the incident x-ray beam

Θ - Bragg's diffraction angle

β - Full width at half maxima (FWHM) of the surfactant coated CuO peak.



The average size of CTAB and SLS coated nanoparticles are found to be 15.01 and 15.17 nm. These peaks were well matched with the JCPDS file no 77-1898. These planes represents that the synthesized surfactant coated CuO nanoparticles were in (Cu₆₄O) tenorite phase and orthorhombic in structure [9].

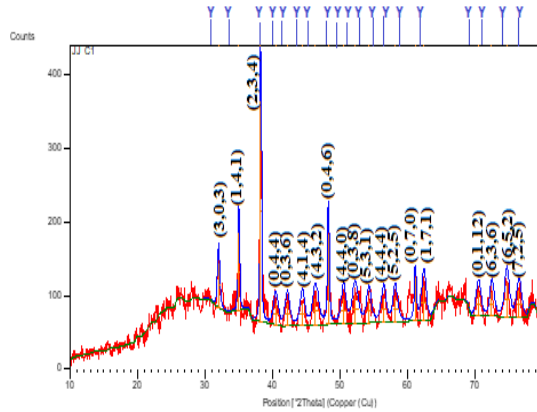


Figure.3.5 XRD pattern of the CTAB coated CuO nanoparticles

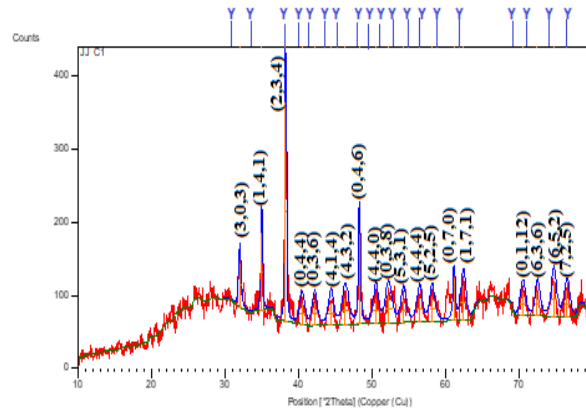


Figure.3.6 XRD pattern of the SLS coated CuO nanoparticles

Table.3.2 XRD peaks comparison for CTAB and SLS coated CuO nanoparticles

Surfactant coated nano particle	Surfactant	2θ Value	Plane	Particle size (nm)	Phase & Structure
CuO	CTAB	38.36	(234)	15.17	(Cu ₆₄ O) tenorite phase, orthorhombic
		48.84	(046)		
		34.63	(141)		
		31.73	(303)		
	SLS	38.24	(234)	15.01	(Cu ₆₄ O) tenorite phase, orthorhombic
		48.23	(046)		
		34.94	(141)		
		31.96	(303)		



3.4 SEM ANALYSIS:

The surface morphology of chemical mediated synthesized CuO nanoparticles was examined by scanning electron microscope. The surface morphology of synthesised CTAB coated CuO nanoparticles are cubic in shape. SLS coated nanopartilces are agglomerated due to its adhesive nature [11].

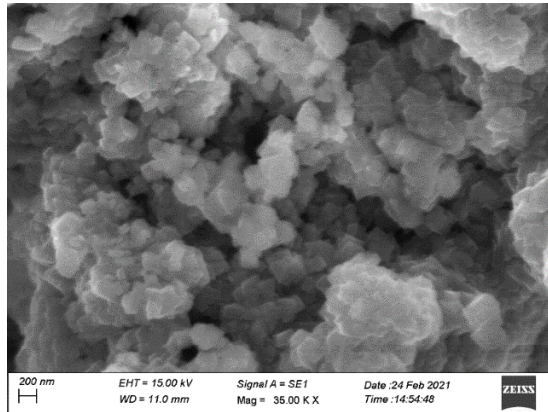


Figure.3.7 SEM image of CTAB coated CuO nanoparticles

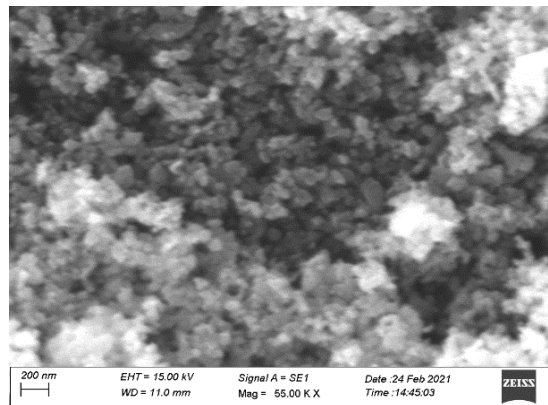


Figure.3.8 SEM image of SLS coated CuO nanoparticles

3.5 ANTIBACTERIAL ACTIVITY:

The antibacterial activity of CuO nanoparticles from cationic and anionic surfactant were tested by disc diffusion method against Gram +ve *Staphylococcus aureus* and Gram -ve *E.coli*, *Klebsiellapneumoniae*. Amikacin were used as the standard antibacterial drug. Both surfactant coated nanoparticles has strongly inhibited the bacterial strains as compare to the standard antibacterial drug. Based on the results SLS coated nanoparticles shows more sensitive to bacterial strains with respect to the CTAB coated nanopartilces

Table.3.3 Antibacterial activity of CTAB coated CuO nanoparticles

SAMPLE	ORGANISMS	ZONE OF INHIBITION		
		A K	100 µl	50µl
CTAB coated CuO nano particles	<i>Staphylococcus</i>	29 mm	12 mm	9mm
	<i>E.coli</i>	18 mm	7m m	6mm



. The variation in the bactericidal effects was due to the small sized nature of the SLS coated CuO nanoparticles as compare to the CTAB. However the small size nanopartilces induce more cell permeability by creating intercellular loss and there by leading to the cell apoptosis [9, 12].

Table.3.4 Antibacterial activity of SLS coated CuO nanoparticles

SAMPLE	ORGANISMS	ZONE OF INHIBITION			
		A K	10 0 µl	50 µl	50 µl
SLS coated CuO nano particles	<i>Staphylococcus</i>	29 mm	6 mm (R)	6m m(R)	6m m(R)
	<i>Klebsiellapneumoniae</i>	22 mm	21 mm	19m m	12m m



Figure.3.9 E. 1. Inhibition Zone of Stephylococcus



Figure.3.10 E. 2. Inhibition Zone of E.coli



Figure.3.11 E. 3. Inhibition Zone of Stephylococcus

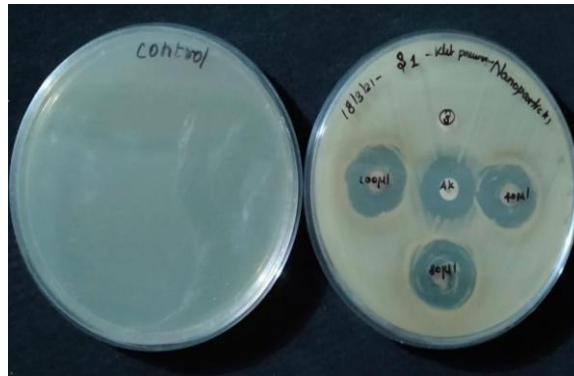


Figure.3.12 E. 4. Inhibition Zone of Klebsiella pneumoniae

IV. CONCLUSION

CuO nanoparticles were synthesized by simple chemical reduction method with CTAB and SLS as surface coating agent. The UV-Visible spectra shows the band at 383 and 392 nm indicating the formation of copper oxide nanoparticles. It was further confirmed by the FTIR which shows the peaks at 591, 514 and 669 cm^{-1} corresponding to Cu-O bond. The particle size was determined by using XRD which was found to be 15.01 and 15.17 nm. The surface morphology and shape of the synthesized CuO nanoparticles was examined by SEM. The synthesized surfactant coated CuO nanoparticles shows good antibacterial activity. Hence from this study, it can be concluded that the antibacterial activity copper oxide nanoparticles were enhanced by the capping agent.

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