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Asha Meena
Assistant Professor, Department of Chemistry, S.D. Govt. College Beawar, Ajmer, Rajasthan, India

Corresponding Author:
Asha Meena
Assistant Professor, Department of Chemistry, S. D. Govt. College Beawar, Ajmer, Rajasthan, India

Characterization and antibacterial screening of copper surfactants

Asha Meena

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Abstract

In the applied areas of research and many industrial processes, the complexes of copper metallic soaps with some aromatic ligands are found very useful, in studying the biocidal activities of above complexes in non-aqueous solvents. These complexes have been analyzed for their characteristics in non-aqueous media and have been derived from copper (II) groundnut soaps with substituted 2-amino-6-ethoxy benzothiazoles. The synthesized complexes were characterized by elemental analysis, melting point determination and their IR, NMR and ESR spectral studies have also been done to understand structural aspects. In the present work, all these copper (II) soap complexes were screened for their antibacterial activity against Gram-positive bacteria *Staphylococcus aureus*. The chosen solvent in dilution is benzene, which results in affecting the aggregation of complex molecules and has the tendency to interact with complex molecules. Zone of inhibition have been measured in mm at various concentrations. The detailed study of aforesaid complexes clearly indicates that the synthesized complexes were found to possess appreciable bactericidal properties.

Keywords: Biocidal activities, non-aqueous solvents, *staphylococcus aureus*, substituted benzothiazole, concentration

Introduction

Metal ions play important roles in biological processes. A characteristic of metals is that they easily lose electrons to form positively charged ions which tend to be soluble in biological fluids. It is in this cationic form that metals play their role in biology. The attraction of these opposing charges leads to a general tendency for metal ions to bind and interact with biological molecules [1-2]. Substituted benzothiazoles as ligands affect the environment of the complex in such a way that their lipophilicity increases which is a major factor in designing a drug [3-4]. In this paper we have explored the overview of important application of metal soap complexes in pharmaceutical industry. Metal complexes as antimicrobial agents enhanced upon coordination with a suitable metal ion can often be the efficacy of the various organic therapeutic agents [5-6]. The pharmacological activity of metal complexes is highly dependent on the nature of the metal ions and the donor sequence of the ligands because different ligands exhibit different biological properties. There is a real perceived need for the discovery of new compounds endowed with antimicrobial activities [7-8]. The newly prepared compounds should be more effective and possibly act through a distinct mechanism from those of well-known classes of antimicrobial agents to which many clinically relevant pathogens are now resistant [17].

Materials and Methods

Synthesis of copper soap complexes

Direct Metathesis process was used to prepare Copper (II) soaps whereas substituted benzothiazoles were prepared by thiocyanation method. Due to the occurring of thiocyanation as well as para position to the amino group, the method needs para occupied anilines. The copper (II) soap complexes were prepared by reacting ethanolic solution of ligand (substituted benzothiazole) with copper (II) soap in 1:1 molar ratio. In 25-30 ml of ethyl alcohol, 0.001 moles of ligand molecule was dissolved and in 10-15 ml of benzene, 0.001 moles of copper (II) soap derived from groundnut oil was dissolved and then ethanolic solution of ligand was added in it. After the reaction, with constant stirring, the mixture was fluxed for about two hours. The solid precipitate which is separated on cooling was filtered, washed with hot distilled water and ethyl alcohol and dried in vaccum [10-11]. The dried complexes were then purified and recrystallized with hot benzene twice. These complexes are solids and dark green or greenish brown in colour, which are soluble in various solvents such as benzene, ether and methanol-benzene mixture but are insoluble in water, all the copper soap complexes are quite stable.

Instrumentation and characterization

Chemical structures of these copper (II) groundnut-soap complexes were confirmed by IR, NMR and ESR techniques. The Infra-red spectra of complexes derived from groundnut oil were obtained from Dept. of Chemistry, S.P.C. Govt. College, and Ajmer in the range of 4000-600 cm^{-1} on a ABB Horizon MB 3000 series spectrophotometer instrument. The ESR spectra of copper (II) soap complexes were recorded at liquid nitrogen temperature (LNT) from SAIF, IIT, POWAI, Mumbai using TCNE (g=2.00277) as a internal standard. ^1H NMR spectra of these complexes were recorded at CDRI, SAIF, and Lucknow using C_6D_6 as reference.

Results and Discussion

Infra-red spectra

The absorption bands correspond to asymmetric and symmetric stretching of methylene ($-CH_2$) group observed at 2924 and 2854 cm^{-1}. In the spectra of carboxylate ion (COO^-) two bands observed such as a strong asymmetrical stretching band in the range 1551-1558 cm^{-1} and a weaker symmetrical stretching band near 1373 cm^{-1}. Also>C=O stretching bands were observed at 1744 cm^{-1}. Out of plane C–H bending vibration appeared at 825-872 cm^{-1}.

Table 1: IR spectral data of copper (II) groundnut-soap 2-amino-6-ethoxy benzothiazole complex

Absorption Bands	CGB (cm^{-1})
IR absorption bands corresponding to soap moiety	
$-CH_3$ and $-CH_2$, C–H antisymmetric stretching (ν_{as})	2924
$-CH_3$ and $-CH_2$, C–H symmetric stretching (ν_s)	2854
>C=O stretching	1744
COO^-, C–O antisymmetric stretching	1551
$-CH_3$, C–H symmetric bending (δ_s) (scissoring)	1450-1458
COO^-, C–O symmetric stretching	-
$-CH_2$, C–H bending (δ) (twisting and wagging)	1227
=C–H, out of plane bending of C–H (strong)	872
$-CH_3$, C–H rocking	1111-1119
$-CH_2$, C–H rocking	725
Cu–O stretching	602
IR absorption bands corresponding to ligand moiety	
asymmetric $-NH_2$, N–H stretching	3742
symmetric $-NH_2$, N–H stretching	3564
C–N stretching	2361
C=S stretching	1119-1227
unconjugated C–N stretching	941
C–H deformation due to benzene	679

A broad band near 3742 cm^{-1} and 3564 cm^{-1} was observed corresponding to N–H symmetric and asymmetric stretching of amide groups in copper groundnut-soap 2-amino-6-ethoxy benzothiazole complex. Also unconjugated C–N stretching band was observed at 1057-1065 cm^{-1}. An absorption band at nearly 1273 cm^{-1} attributed to N–C=S stretching. It is confirmed that the complexation was done between copper surfactant with 2-amino-6-ethoxy benzothiazole ligand via nitrogen atom (Figure 1), on the basis of the above observations.

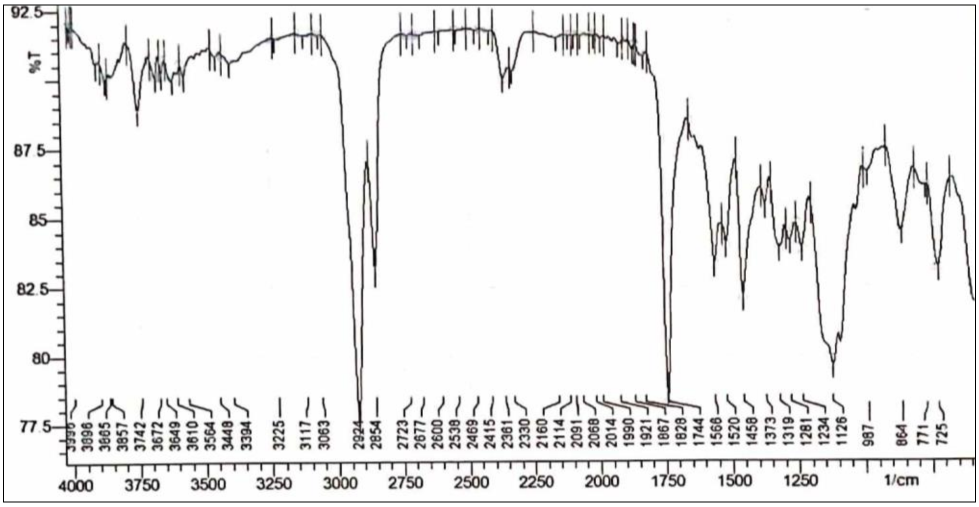

Fig 1: IR spectra of copper (II) groundnut-soap 2-amino-6-ethoxy benzothiazole complex

Nuclear magnetic resonance spectra: Figure 2 shows NMR spectra of copper (II) groundnut-soap 2-amino-6-ethoxy benzothiazole complex (CGB $_e$). Aliphatic $-CH_3$ and $-CH_2$ proton attached to $-CH_2R$ group show signal at nearly 0.957 δ and 1.290 δ respectively. The signal observed at 2.10 δ corresponds to $-CH_2$ proton attached to –C=C– group.

Table 2: ^1H NMR spectral data of copper (II) groundnut-soap 2-amino-6-ethoxy benzothiazole complex

Peak/Signal	Copper (II) groundnut, 2-amino-6-ethoxy benzothiazole complex (δ)
$-CH_3-CH_2R$	0.910
$-CH_2-CH_2R$	1.296
$-CH_2$–C=C–	2.109
–C=C–H (vinylic proton)	5.490
–C=C–CH_2–C=C–	2.485
$-NH_2$ (broadened peak)	4.00 - 4.30
Tautomeric $-NH_2$ (Weak Signal)	7.160

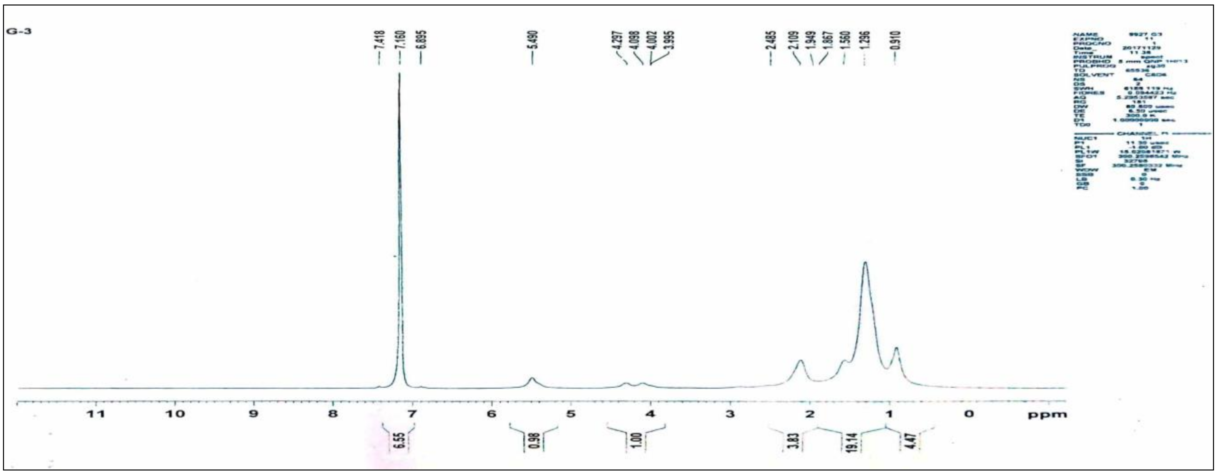

Fig 2: NMR spectra of copper (II) groundnut-soap 2-amino-6-ethoxy benzothiazole complex

Electric magnetic resonance spectra

The ESR spectra of Cu (II) provide information about the extent of the delocalization of unpaired electron. The ESR spectra of the copper (II) soap complexes derived from groundnut oil were recorded on X-band at modulation frequency of 100 KHz in the solid state at room temperature. TCNE (g = 2.00277) was used as the field marker and G_{ia}, g⊥, g_{ov}, G values have been calculated. ESR spectra of the complexes revealed two g values (G_{ia} and g⊥) shown in Table 3.

Table 3: ESR spectral data of copper (II) groundnut-soap 2-amino-6-ethoxy benzothiazole complex

Complex	$g_{\parallel}$	$g_{\perp}$	g_{av}	G	$H_{\parallel}$	$H_{\perp}$
CGB	2.0634	2.0108	2.0283	5.8704	2426	2491

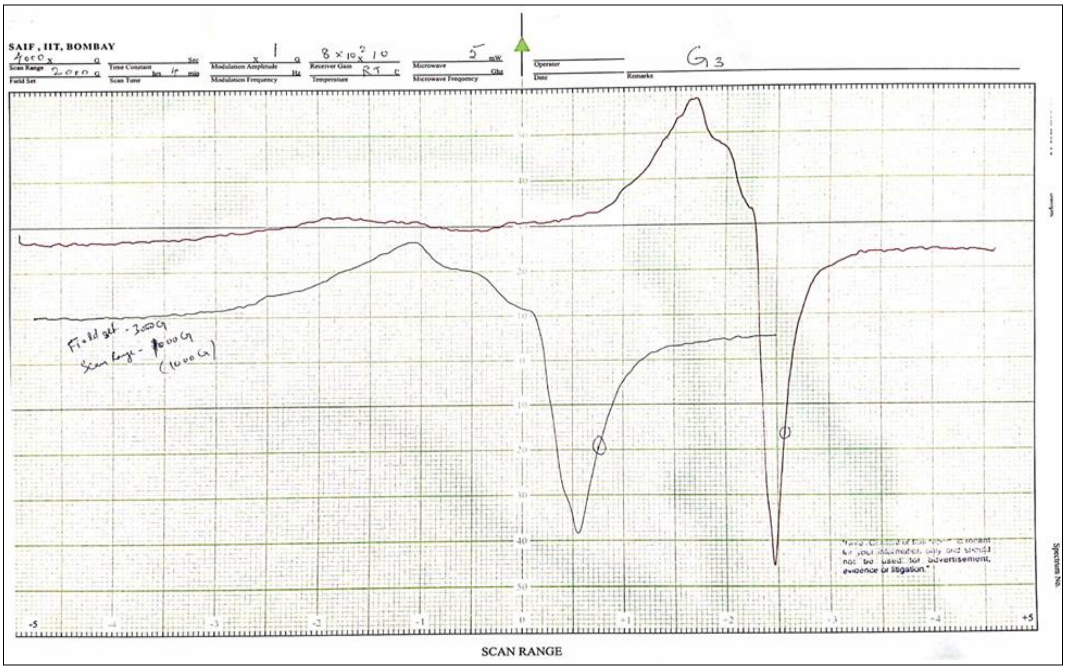

Fig 3: Scan range

Processing of samples

All the copper (II) soap complexes were screened for their antibacterial activity against *Staphylococcus aureus*. These complexes were tested at different concentrations after 24 and 48 hours incubation times and zone of inhibition have been measured in mm. The complexes were dissolved in hot benzene. Stock solutions of tested compounds were prepared in benzene to a final concentration of 50mg/ml. Two different dilutions C_1 (50 mg/ml) and C_2 (25 mg/ml) were made in hot benzene to get antimicrobial activity of each compound against *Staphylococcus aureus*. Streptomycin was used as positive control (300mcg/ml concentration).

Mueller-Hinton agar medium was used for antimicrobial activity of complexes on two different concentrations by disc/well diffusion susceptibility testing. Fresh culture of *Staphylococcus aureus* strain ATCC-25923 was inoculated in peptone water and kept for incubation for 30 minutes at 37 °C. Inoculum size of bacteria was adjusted using McFarland Turbidity standard as reference. The bacterial suspensions were compared to 0.5 McFarland turbidity standard. Microbial culture was swabbed onto the Mueller-Hinton agar surface through sterile cotton swab sticks. After proper marking of plates, 50µl extracts from different dilutions prepared was loaded into the respective wells. The swabbed *Staphylococcus aureus* plates were kept for incubation at 37 °C for 24-48 hours [12].

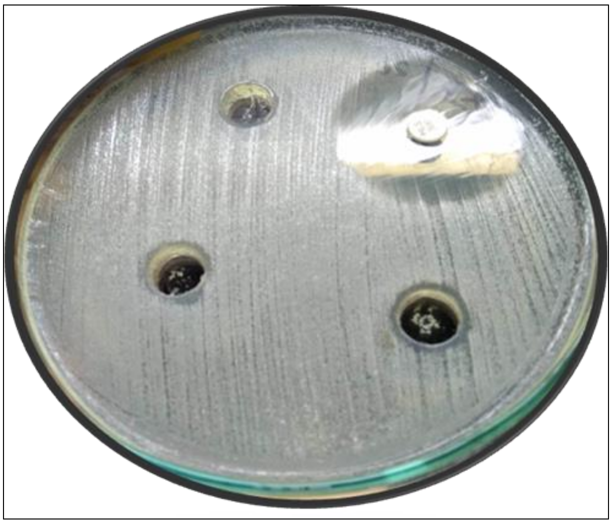

Figure 4:- Antimicrobial activity against *Staphylococcus aureus* of copper (II) groundnut-soap 2-amino-6-ethoxy benzothiazole complex

Table 4 shows the biological activities of copper (II) soap complexes determined by screening against bacteria at 5×10^4 ppm and 2.5×10^4 ppm. The copper (II) groundnut-soap complexes with ligand like substituted benzothiazole were screened for their antibacterial activity against *S. aureus.* These compounds were tested at different concentrations and zone of inhibition have been measured in mm. The antibacterial activity results are summarized in Table 1 respectively.

Table 4: Zone of inhibition of two different concentrations of copper (II) groundnut-soap 2-amino-6-ethoxy benzothiazole complex against *Staphylococcus aureus*

Compound	PC	C_1 (5×10^4 ppm)		C_2 (2.5×10^4 ppm)		NC
		24 hrs	48 hrs	24 hrs	48 hrs	
CGB	32 mm	12 mm	12 mm	10 mm	10 mm	NZI

The enhanced activity of newly bio-based copper (II) soap complexes can be explained on the basis of chelate formation, the presence of donor atoms such as nitrogen, sulphur and the structural compatibility with molecular nature of the toxic moiety. Due to partially sharing of its positive charge with the donor ligands and π- electron delocalization over the whole chelate ring, polarity of the central metal ion reduces mainly, in the complex. Consequently helping the penetration of the bacterial cell membranes and restricting further growth of the micro-organisms, such chelation could enhance the lipophilic character of the central metal atom. It has been attributed to the fact that the donor atom such as N and S introduced into the complex through the ligand also play an important role in enhancing the capability of synthesized Biocidal molecule on the basis of above results [13].

ANOVA test: The results of ANOVA for the Biocidal activities for copper (II) soap complexes on two different concentrations are shown in Table 3 and 4. The predicted R^2 are in good agreement and closer to 1 [14]. The value of R^2 confirms that the experimental data are well satisfactory. All the tests were performed in triplicate and the standard deviation has been calculated. The result is statistically significant, by the standards of the study, due to $F. > p$ [15].

Table 5: Descriptive statistics bacterial data for copper (II) groundnut-soap 2-amino-6-ethoxy benzothiazole complex against *Staphylococcus aureus*

Compd.	Conc. (ppm)	Count	Sum	Average	Variance	Std. Deviation	Coefficient variance	Std. Error
CGB	5×10^4	3	27.0	9.000	0.1600	0.4000	0.0444	0.2309
	2.5×10^4	3	30.3	10.100	0.1300	0.3605	0.0356	0.2082

Conclusion
The present research work may attempt to review the bactericidal activities for copper (II) groundnut-soap 2-amino-6-ethoxy benzothiazole complex in the current work with new research findings. These investigations show relevant physicochemical mechanisms of newly bio-based copper (II) soap complexes from natural edible oil such as groundnut. These pharmacologically interesting copper soap complexes could be a suitable strategy to develop novel therapeutic tools for the medical treatment. The present research work provides valuable information to the pharmaceutical, agrochemical, and other industries to synthesis some degradable, safe, and useful molecules for various applications.

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Author's Contribution
Not available

Conflict of Interest
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