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Synthesis, Characterization and Studies on Antimicrobial Properties of Copper Nanocomposite

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Abstract

Copper nanocomposite has been synthesized by simple chemical precipitation method. Firstly, the polymer metal complex was synthesized which was followed by the application of calcination technique. The synthesized polymer metal complex was analyzed by (IR) infra-red spectroscopy and nuclear magnetic resonance (NMR). Thereafter chemical composition and crystallographic structure of nanocomposite was confirmed by XRD measurement, which reveals that crystal system and size of nanoparticles is 19.34. While the scanning electron microscopic (SEM) images were taken for finer details of surface morphology of nanocomposite. These synthesized nanocomposites were also studied for antimicrobial activity.

Keywords:* *Copper nanocomposite; Polymer; SEM; Morphology; NMR

Introduction

Nanocomposites exhibit different optical, mechanical, thermal, magnetic and antibacterial properties. The antimicrobial properties of metal nanoparticles have been of focus during the last decade. Some of the biological properties of nanoparticles of various metals have worked find out by testing their antimicrobial potential. Nanoparticles of Ag, Cu, Zn and Au show a wide spectrum of antimicrobial activity against different bacterial and fungal species [1-7]. The development of polymer based nanocomposite with antimicrobial activity offer interesting possibilities as the polymer matrix can be multiplied in order to fulfil not only specific technological requirement but nanostructures too with shape and size depending on properties that can be absorbed [8]. Various inorganic nanostructures with antibacterial properties have been used in a wide range of matrix [9,10]. Among the polymer nanocomposites investigated so far, those incorporating ligand nanoparticles have been regarded as particularly useful for applications in various fields, including biomedical equipment and devices, water

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treatment, and food processing [11]. Ag has been widely investigated for its antimicrobial activity due to its superior effectiveness and strong cytotoxicity towards a broad range of microorganisms [12]. The great interest for Cu based nanocomposite can be easily perceived by the number of polymer matrix investigated in their preparation, both of synthetic and natural origin [13]. These biocompatibility and biodegradability that can be used in a variety of formulations depending on the envisaged functionality. The present study was carried out to find out the antimicrobial potential and the mechanism of interaction of copper nanocomposites with some selected species of bacteria i.e. *S. haemolyticus, K. pneumonia, B. cereus and E. faecalistaking* the criteria bacterial growth inhibition.

Experimental

All AR grade chemicals were used, namely, aniline, formaldehyde and sodium hydroxide (Central Drug House Pvt. Limited) and hydrochloric acid (Fisher Scientific). Metal ion solutions have been prepared by dissolving suitable amount of in distilled water.

Synthesis of copper nanocomposites

Copper nanocomposites have been synthesized in two steps: Synthesis of polymer metal complex 9.5 ml of aniline in dilute hydrochloric acid was taken in 250 ml beaker to which 10 ml of formaldehyde solution (40%) was added slowly with stirring. The mixture was then stirred well for 15 minutes to form an orange red solution. This solution is fed into aqueous sodium hydroxides solution (1%) and refluxed for 30-45 minutes. The aniline formaldehyde was precipitated out. Thereafter 15 ml of 1N Metal ion solution was added drop by drop. The reaction mixture was continuously stirred for 30 minutes then heated at 450°C for one hour on heating mental. After heating, polymer metal complex has been formed. The excess metal ion and impurities on the solid sample was purified by the washing with distilled water solution.

Synthesis of nanocomposite: Decomposition of polymer metal complex at 900° in muffle furnace for 45 minutes was done in order to get nanocomposite of the complex.

Purification of copper nanoparticles

1st Step: Removal of volatile impurity: Several volatile impurities were separated during decomposition. Thus, nanocomposite becomes free from these impurities.

2nd Step: Removal of metallic impurities: For removal of metallic ions, nanocomposites were kept in 12N hydrochloric acid solution for 24 hours. Then they were centrifuged and washed with distilled water till hydrochloric acid was fully removed.

Antimicrobial activity of nanocomposite

The MIC of nanocomposites was determined following method suggested by Chitwood (1969). The sample was dissolved in DMSO for making different concentrations ranging from 100 µg/ml to 2000 µg/ml.100 µl of each concentration was added to tube containing Muller Hinton broth medium. 100 µl of microbial culture of *Gram positive bacteria (Klebsiella pneumonia, Staphyllococcushaemolyticus*) and *Gram negative bacteria* (*Enterococcus faecalis*, *Bacillus cereus*) with 10-5 CFU was inoculated in each tube with different concentrations of the sample. 200 µg/ml, 500 µg/ml, 1000 µg/ml and 1500 µg/ml concentration was also tested for the zone of inhibition using agar well diffusion method (Sen and Batra, 2012) against the above four microorganisms.

Results and Discussion

Infra-red spectroscopy

The IR spectra of polymer metal complex are given in TABLE 1 and FIG. 1.

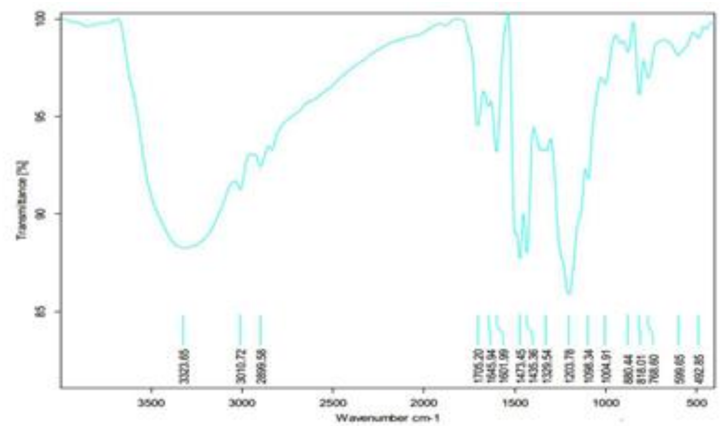

FIG. 1. **IR spectrum of aniline-formaldehyde resin complex.**

TABLE 1. **Prominent absorption band cm^{-1} and functional groups of aniline-formaldehyde resin complex.**

Prominent absorption band cm	Functional group
3323 cm	N-H stretching
3010-2899 cm	C-H stretching
1645-1601 cm	C=C stretching
1309 cm	C-N aromatic stretching

The 1H NMR spectrum of copper doped complex of aniline-formaldehyde polymer metal complex showed a signal at 6.4–7.7 ppm for the aromatic protons of aniline. The methylene groups of the polymer showed signals at 3.6 and 4.2 ppm. All the experiments were carried out at 300 K over a spectral width of 15 ppm. Chemical shifts were referenced to the solvent signal.

X-Ray diffraction-Orthorhombic copper nanocomposite has been determined using XRD technique. Different copper nanostructure size was obtained using wet chemical precipitation method. Other use of XRD technique is to determine the particle size is using Scherrer equation.

$D=K\lambda/(B \cos\theta)$

Where,

D=The mean size of crystallites (nm), B=Full width at half the maximum (FWHM)

K=Crystallite shape factor, θ=bregg angle, λ=X-ray wave length

Applying Scherrer equation to the obtained XRD pattern of copper nanoparticles, crystalline size was found to be 19.34 nm and crystal system was orthorhombic. The Bravais lattice is base centered and 2θ=41.960. The XRD spectra given in FIG. 2.

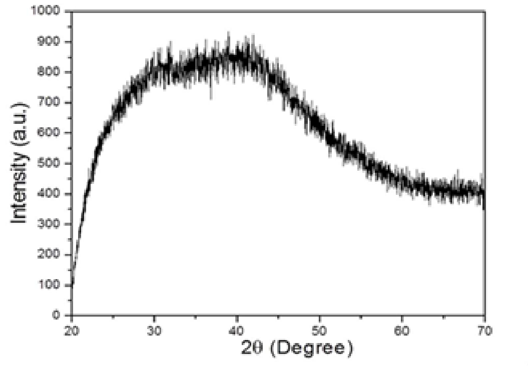

FIG. 2. **XRD spectrum of copper nanocomposite.**

Finer details of the surface morphology of the resulting powder were examined by scanning electron microscope. FIG. 3A shows that spherical pots are present on the surface and bland like are visible Spherical dots were assigned as the metal ion present on the surface of the nanomaterial The spherical metal ion present on the surface of composite material plays an important role in the one dimensional electron transportation. FIG. 3B shows clear picture of the polymer composite.

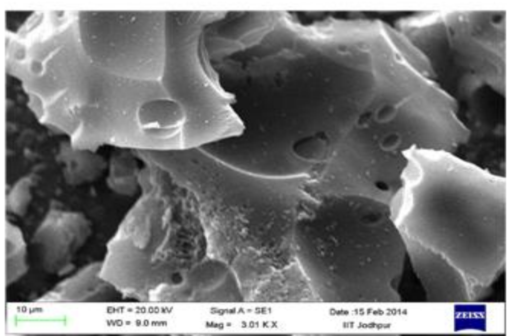

FIG. 3A. **SEM image metal doped polymer nanocomposite in aqueous phase.**

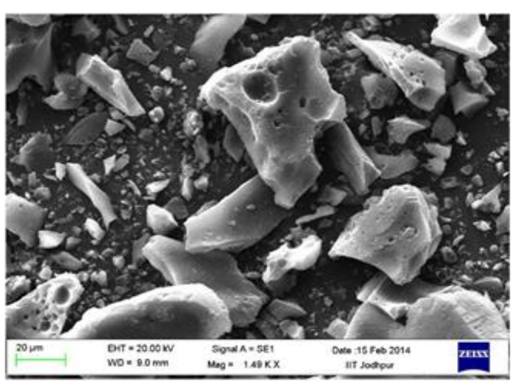

FIG. 3B. **SEM image metal doped polymer nanocomposite in aqueous phase.**

Antimicrobial activity:

The growth of each microorganism under the regime of different concentrations of copper nanocomposite was assessed at optical density of 600 nm (TABLE 2). The OD ranged from 0.04 to 0.50 for *S. haemolyticus*, 1.05 to 1.48 for *K. pneumonia*, 0.22 to 0.91 for *B. cereus* and 0.17 to 1.02 for *E. faecalis* which shows no significant inhibition in the growth of microorganisms. However, growth was reduced with the increased concentration of the compound. Upon testing with well diffusion agar plate method, no zone of inhibition was observed indicating that the compound does not have the antimicrobial activity against the above mentioned four microorganisms (FIG. 4). The antimicrobial activity of copper nano-composite has been reported by Singhal et al. and Pinto et al. The biological activities of chemically synthesized nano-composites depend on the pore size of copper nano-particle which increases with the increase pore size [14-16].

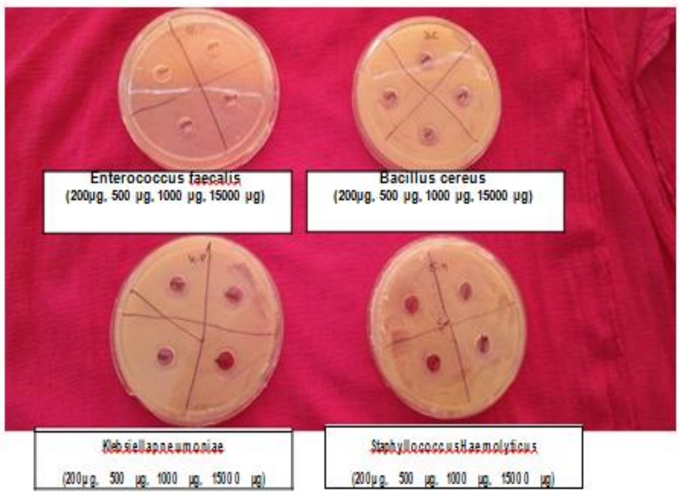

FIG. 4. **Antimicrobial activity of nanocomposite using agar well diffusion method.**

TABLE 2. **Optical density at 600 nm of microbial growth against different concentration of nanocomposites dissolved in DMSO.**

Concentration	*Staphylococcus haemolyticus*	*Klebsiella pneumoniae*	*Bacillus cereus*	*Enterococcus faecalis*
100 µg/ml	0.5	1.48	0.91	1.02
200 µg/ml	0.25	1.48	0.8	0.92
300 µg/ml	0.25	1.44	0.46	0.92
400 µg/ml	0.23	1.43	0.36	0.81
500 µg/ml	0.23	1.42	0.35	0.67
600 µg/ml	0.22	1.41	0.35	0.51
700 µg/ml	0.22	1.15	0.32	0.31
800 µg/ml	0.2	1.14	0.32	0.28
900 µg/ml	0.18	1.1	0.29	0.21
1000 µg/ml	0.16	1.09	0.23	0.2
1500 µg/ml	0.1	1.08	0.23	0.19
2000 µg/ml	0.04	1.05	0.22	0.17

Conclusion

The present paper reports a simple, fast, effective polymer based technique to synthesize copper nanocomposite using thermosetting polymer and copper metal salt as a precursor. Nano composite synthesized by the simple chemical precipitation method. The IR spectroscopy confirmed the formation of polymer composite. XRD analysis proves the nanostructure of the chromium nanoparticles through its crystal analysis and spacing pattern. The antimicrobial activity has also been observed in the present nanocomposite.

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