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Research Article

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Synthesis and biological activity of o-Cresol-Adipamide-Formaldehyde copolymer resin

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ABSTRACT

A copolymer was synthesized from o-cresol and adipamide using formaldehyde as a cross linking agent at 130 ± 2 ^{0}C in 2M HCl medium. The resin was characterized by elemental analysis, FTIR and NMR spectra. The morphology of the synthesized resin was studied by optical photograph and scanning electron microscopy (SEM). The o-CAF resin was tested for its inhibitory action against pathogenic bacteria and fungi. The resin show potent inhibitory action against bacteria such as Escherichia coli, Klebseilla, Staphylococcus aureus and Pseudomonas aeruginosa and fungi viz. Aspergillus flavus, Aspergillus niger, Pencillium species, Candida albicans, Cryptococcus neoformans and Mucor species. The total cation exchange capacity was measured and effect of pH and metal ion concentration on ion-exchange capacity were studied. The study was carried out over a wide pH range and in media of various ionic strengths. The copolymer resin is soluble in DMF, DMSO, THF, conc. H_2SO_4.

Keywords: Polycondensation; Resins; Antimicrobial screening

INTRODUCTION

A copolymer involving 2, 4-dichlorophenylmethacrylate and vinyl acetate was reported as a significant inhibitor for the growth of microorganism [1]. A series of cyano derivatives of N-alkyl and N-aryl piperazine were synthesized and their antimicrobial activities were evaluated against Gram-positive and Gram-negative strain *S. Aureus, P. Aeruginosa, S. Epidermidis, E. Coli* and antifungal activities against *A. Fumigantus, A. Flavus and A. Niger*. Few of the synthesized derivatives possess potent antibacterial activity and some of the compounds were reported for its cytotoxic activity [2]. Biological evaluation of novel nitrogen containing aniline formaldehyde resin has been studied and the compounds were reported as a potent antifungal and antibacterial agent [3]. Long chain aliphatic esters as well as organic and ferrocene containing Schiff bases were synthesized and reported to have good antitumor, anticancer and antioxidant also a candidate [4]. Poly [(2-hydroxy-4-methoxybenzophenone) ethylene] resin and its polychelates with lanthanides (III) were screened for antibacterial activity and the metal chelated compounds maintain better activity compared to the ligand [5]. A copolymer involving 2, 4-dichlorophenymethacrylate and vinyl acetate was reported as a significant inhibitor for the growth of microorganisms [9]. In an earlier communication [6-12] from this department a number of such copolymers have been reported. However, no work has been carried out on the synthesis, characterization, ion-exchange and biological properties of the copolymer resins from o-cresol, adipamide and formaldehyde.

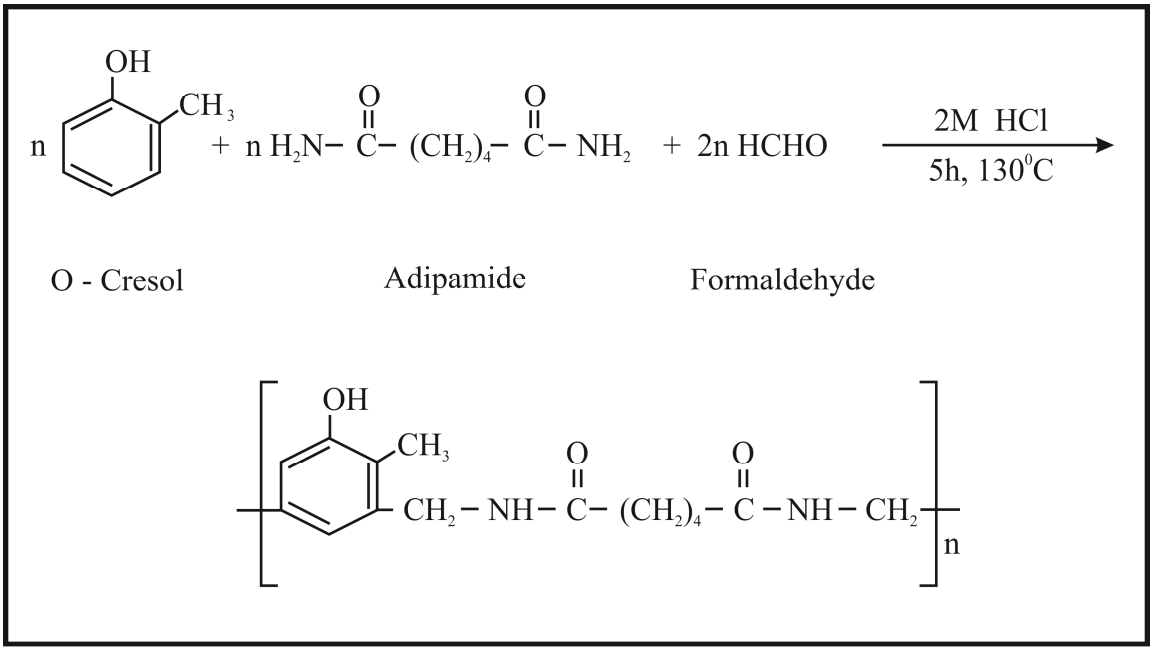
EXPERIMENTAL SECTION

Materials

Adipamide (Merck, India) and o-cresol (SRL, Mumbai) and were purified by rectified spirit, formaldehyde (37 %), metal chlorides and nitrates of selected metals (AR grade, Merck) were used as received. All the other chemicals, solvents and the indicators were analytical grades procured from Qualigens Fine Chemicals, Mumbai, India.

Synthesis of o-CAF copolymer resin

The o-CAF-1 copolymer resin was prepared [16-19] by condensing o-Cresol (0.1mol) and adipamide (0.1mol) with formaldehyde (0.2mol) in the presence of 2M HCl (200ml) as a catalyst at 130 ^{0}C in an oil bath for 5 hrs with occasional shaking to ensure thorough mixing. The solid resinous product obtained was removed immediately from the flask. It was washed with cold water, dried and powdered. The powder was repeatedly washed with hot water to remove unreacted monomers. Then it was extracted with diethyl ether to remove excess of p-cresol-formaldehyde copolymer which might be present along with o-CAF copolymer resin. The purified copolymer resin was finely ground and kept in a vacuum over silica gel. The yield of the copolymer resin was found to be 80%.



Scheme1. Reaction sequence of the synthesis of o-CAF resin

Antimicrobial Screening

Biological assay depends upon a comparison of the inhibition of growth of microorganism by measuring the concentration of the sample to be examined with the known concentration of standard antibiotic. For the antimicrobial analysis the in vitro disc diffusion method has been employed. In this study the ligand and their chelates were tested for their effect on certain human pathogenic bacteria such as Gram-positive (*Aspergillus niger and Candida albicans*).

The nutrient agar medium was boiled and sterilized by autoclaving at 7 kg pressure (121 ^{0}C) for 20 min for the study of antibacterial activity. 20 mL media was poured into the sterilized petri plates and kept at room temperature for a few minutes, and allowed to solidify in plates. It was then incubated for 12 h and inoculated with microorganism using sterile swabs. All of these manipulations were carried out with utmost care under aseptic conditions. The test solution prepared by dissolving the compound in DMSO was filled with the media using a micropipette and incubated at 36 ^{0}C for 48 h. The same procedure was adopted for the antifungal studies in which potato dextrose agar was the medium.

During the course of time, the test solution diffuses and the growth of the inoculated microorganisms such as *Staphylococcus aureus, Escherichia coli, Aspergillus niger,* and *Candida albicans* were found to be affected. The activity developed on the plate was measured by measuring the diameter of the inhibited zone in millimetres. The drug ciprofloxacin was used as the standard for bacteria and nystatin for fungi.

RESULTS AND DISCUSSION

The microbial screening results of o-CAF copolymer ligand show (Table 1) higher activity is due to the donor atoms of the ligand and the π-electrons delocalization. This effect increases the lipophilic character, which favours the permeation through the lipoid layer of the bacterial and fungal membranes. The higher activity may also be due to the presence of - OH and the aromatic ring [10]. It is perceived that the factors such as solubility, conductivity, dipole moment and cell permeability mechanism may be alternative reasons for the increased activity of the metal

complexes [11]. The ligand has good inhibition against the growth of Gram-negative bacteria which induces tumour. Hence the copolymer ligand may possess antitumor activity. The Gram-positive bacteria are both pathogenic and invasive. The copolymer has good inhibition characteristics against the growth of this pathogen. *Aspergillus niger* cause aspergillosis, the growth of the fungus is controlled by the copolymer chelates to some extent. The *Candida albicans* can penetrate into the intestinal walls and cause diseases. From the findings, the growth of *Candida albicans* is inhibited by the addition of o-CAF copolymer resin [14].

Table 1. Antimicrobial activities of o-CAF copolymer resin.

Copolymer	Diameter of zone of inhibition (mm)			
	S. Aureus	***E. Coli***	***A. Niger***	***C. Albicans***
o-CAF	17	18	19	17
Solvent (DMSO)	--	--	--	--

Surface analysis has found great use in understanding the surface features of the materials. The morphology of the reported resin sample was investigated by scanning electron micrographs at different magnification, which is shown in Fig.1 for o-CAF. It gives the information of surface topography and defect in the structure. The resin appeared to be dark drawn in colour. The morphology of polymer resin shows spherulites and fringed model. The spherules are complex polycrystalline formation having as good as smooth surface. This indicates the crystalline nature of o-CAF polymer resin sample. The morphology of resin polymer shows also a fringes model of the crystalline amorphous structure. The extent of crystalline character depends on the acidic nature of the monomer. But the photograph shows the fringed and scatted nature having shallow pits represent the transition between crystalline and amorphous. The resin exhibits more amorphous characters with closed packed surface having deep pits. Thus by SEM micrographs morphology of the resin shows the transition between crystalline and amorphous nature, when compare to the other resin, the o-CAF polymer resin is more amorphous in nature, hence shows higher metal ion exchange capacity.

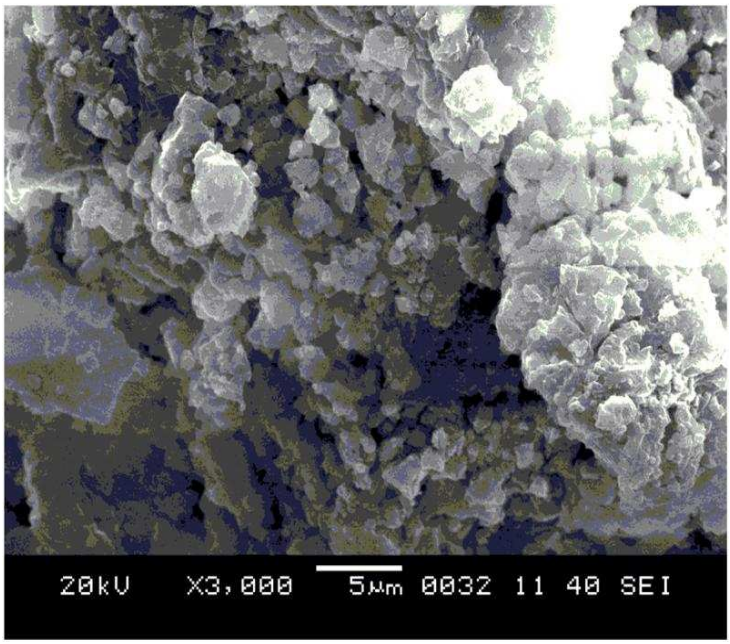


Fig.1. SEM image of o-CAF polymer resin

CONCLUSION

Copolymer resin derived from o-cresol, adipamide and formaldehyde. The copolymer resin shows higher activity against certain bacterial strains such as *Staphylococcus aureus, Escherichia coli*, and fungal strains *Aspergillus niger* and *Candida albicans.*

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REFERENCES

[1] MB Patel, SA Patel, A Ray, RM Patel, *J. Appl. Polym. Sci.*, **2003,** 89, 895.
[2] P Chaudhary, S Nimesh, V Yadav, AK Verma, R Kumar, *Europ. J. Med. Chem.*, **2007**, 42, 471.
[3] S Parveen, T Ahamed, A Malik, N Nishant, *Polym. Adv. Tech*., **2008**, 19, 1779.
[4] H Nawaz, Z Akhtar, S Yameen, HM Siddiqi, B Mirza, A Rifat, *J. Org. Chem*., **2009**, 604, 2198.
[5] MM Patel, MM Kapadia, GP Patel, JD Joshi, *React. Funct. Polym*., **2007**, 67, 746.
[6] SS Rahangdale, WB Gurnule, *Der Pharma Chemica,* **2011,** 3(4), 314-322.
[7] SS Rahangdale, *Arch. Appl. Sci. Res.*, **2012,** (5), 2280-2288.
[8] SS Rahangdale, *J. Chem. Pharm. Res.*, **2012,** 4(10), 4451-4458.
[9] RN Singru, WB Gurnule, *Iran. Polym. J*., **2010**, 19 (3), 1-15.
[10] WB Gurnule, HD Juneja, LJ Paliwal, *React. Funct. Polym*., **2002**, 50, 95-100.
[11] WB Gurnule, DB Patle, *Polym. Bull*., **2011**, 66, 803-820.
[12] VD Mane, NJ Wahane, WB Gurnule, *J. Appl. Polym. Sci*., **2009**, 111, 3039-3049.
[13] N Singh, S Gupta, G Nath, *Central National De La Recherche Scientifique (CAT. INIST)*, **2000**, 14, 484.
[14] GB Bagihalli, SA Patil, PS Badami, *J. Iran. Chem. Soc.*, **2009**, 6 (2), 259.
[15] A Riswan Ahamed, RS Azarudeen D Jeyakumar, AR Burkanudeen, *Int. J. Polym. Mat.*, **2011,** 60, 1-19.
[16] SS Rahangdale, WB Gurnule, *The IUP J. Chem.*,**2012,** 5 (1), 7-17.
[17] SS Rahangdale, WB Gurnule, *Polymer Processing and Characterization*, Apple Academic Press, Inc. USA (April **2012**).
[18] RH Gupta, SS Rahangdale, Gurnule WB, *Bionano Frontier,* **2012,** 5, 423-427.
[19] SS Rahangdale, WB Gurnule, *Chem. Sci. Trans.*, **2012,** 2(1), 287-293.