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Spectral and antimicrobial studies on novel ligand and its co-ordination polymers

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Abstract A novel ligand 2,5-bis(4-methoxyphenylcarbamoyl)terephthalicacid (BMPCT) was prepared from 1,2,4,5-benzenetetracarboxylicdianhydride (pyromellitic dianhydride – PMDA) and 4-methoxyaniline (p-anisidine). Various coordination polymers of BMPCT were also prepared using Mn(II), Fe(II), Co(II), Ni(II), Cu(II), and Zn(II) metal salts. All compounds were analyzed by physicochemical, thermogravimetric and spectroscopic techniques. Antimicrobial activity was carried out using the agar-plate method against various strains of bacteria (*Staphylococcus aureus, Bacillus subtillis, Pseudomonas aerugionsa,* and *Escherichia coli*) and spores of fungi (*Aspergillus niger* and *Aspergillus flavous*). The results showed significantly higher antimicrobial activity of coordination polymers compared to the ligand.
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1. Introduction

Co-ordination polymers, often referred to as metal-organic frameworks (MOFs), are currently attracting a high level of attention in inorganic chemistry. Elegant structural chemistry has been undertaken on co-ordination polymers for many years. The recent upsurge of interest in this MOF has resulted largely because some are sufficiently robust for the structure to be maintained. This MOF architecture has witnessed a tremendous growth because of their intriguing structures and potential properties. Much research is being devoted to the preparation of a polymeric chain by the formation of metallic chelates. Some success has been achieved in the preparation of coordination polymers from bi-functional ligand. MOFs derived from bisamic acid have received less attention, excluding bisamic acid based on maleic anhydride (Patel and Panchal, 2005; Dave and Patel, 2011), amic acid based on phthalic anhydride (Tamaki et al., 2003; Ueda and Nakayama, 1996; Yan et al., 2006).

More particularly, multicarboxylate material shows good building blocks in the design of MOFs with desired topologies owing to their rich coordination modes. Therefore, the dianhydride of 1,2,4,5-benzenetetracarboxylic acid (pyromellitic dianhydride) has been selected for further work. It is also used extensively as an important monomer in the preparation of a variety of polymers. Moreover, it is useful in the preparation of high performance coatings that have been widely employed in many fields because of its excellent thermal, oxidative stability and excellent mechanical properties (Ramkumar and

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Ramakrishnan, 2008; Masahiro et al., 2007; Matsuura et al., 2001). This may offer the compound, both metal gripping potentiality and good biological efficacy due to anhydride moiety. The MOF based on bisamic acid of pyromellitic dianhydride has not attracted any attention. Hence, the initial work in this direction has been reported by us recently (Patel et al., 2011; Patel and Patel, 2012). This prompted us to extend our work by using other auxiliary ligand such as 2,5-bis (4-methoxyphenylcarbamoyl)terephthalicacid (BMPCT). Considering the special biological activity along with its properties, we focused our work on this ligand by complexation using Mn(II), Fe(II), Co(II), Ni(II), Cu(II) and Zn(II) metal ions. In this connection, we are trying to make some coordination polymer based on a different dianhydride. We have tried a lot to generate a crystal for single crystal X ray analysis but we did not succeed.

2. Experimental

2.1. Materials and methods

All the chemicals used, including 1,2,4,5-benzene tetra carboxylic dianhydride and 4-methoxyaniline, were of analytical grade and were purchased from local markets. They were purified by standard methods prior to use (Armarego and Chai, 2009). Nutrient agar and potato dextrose agar were purchased from Hi-media Chemicals, India. Metal(II) salts of Mn(II), Fe(II), Co(II), Ni(II), Cu(II) and Zn(II) were used in their hydrated form.

The elemental analyses (C, H, N) were performed with a model Elemental Vario EL C, H, N elemental analyzer. The infrared spectra (FT-IR) were obtained from KBr pellets in the range of 4000–400 cm^{-1} with a Perkin Elmer spectrum GX spectrophotometer (FT-IR) instrument. The ^{1}H NMR and ^{13}C NMR Attached Proton Test (APT) spectra were recorded on a Bruker (400 MHz) instrument using DMSO-d_6 as solvent as well as an internal reference standard. Diffuse electronic spectra were recorded on a Beckman DK-2A spectrophotometer using MgO as a reference. Magnetic moments were determined by the Gouy method with mercury tetra thiocyanato-cobaltate(II), [HgCo(NCS)4] as calibrant ($\chi g = 1644 \times 10^{-6}$ cgs units at 20 °C), by Citizen Balance (at room temperature). The diamagnetic correction was made using Pascal's constant (Patel et al., 2011). The thermogravimetric analysis studies were carried out with a model Perkin Elmer thermogravimetry analyzer at a heating rate of 10 °C min^{-1} in air. The metal contents of the complexes were analyzed by EDTA titration after decomposing the organic matter with $HClO_4$, H_2SO_4 and HNO_3 (1:1.5:2.5) mixture (Jeffery et al., 1989). The melting point of BMPCT was checked by the standard open capillary method and is uncorrected.

1,2,4,5-Benzenetetracarboxylicdianhydride and 4-methoxyaniline were used as starting materials for the synthesis of novel ligand 2,5-bis(4-methoxyphenylcarbamoyl) terephthalicacid (BMPCT) by a method reported in the literature (Awad et al., 1977). While coordination polymers of BMPCT were synthesized by a subsequently reported method (Shah et al., 2008). The outline of synthesis of BMPCT and its coordination polymers are shown in Schemes 1 and 2. The physicochemical parameters of ligand and its coordination polymers are summarized in Table 1.

2.2. Preparation of 2,5-bis(4-methoxyphenylcarbamoyl)terephthalicacid (BMPCT)

BMPCT was prepared by the reported method (Patel and Patel, 2011). According to this method, a mixture of 1,2,4,5-benzenetetracarboxylicdianhydride (pyromellitic dianhydride-PMDA) (m.p. 397–400 °C) of Fluka Analytical-Japan (21.81 g, 0.1 mol) and 4-methoxyaniline (p-anisidine) (25.514 g, 0.2 mol) was refluxed at 60 °C for 1 h with occasional stirring. Thus obtained precipitate was then filtered and washed. Lastly it was air dried. The yield was of approx 65% and its melting point was 252–280 °C (uncorrected).

2.3. Preparation of coordination polymers

All coordination polymers were synthesized by using an equimolar amount of ligand and metal salt. The yields of all coordination polymers were almost quantitative. The synthetic route for the coordination polymer is shown in Scheme 2.

A warm clear solution of ligand (0.01 mol) in dimethyl sulphoxide-DMSO neutralized with 0.1 M sodium hydroxide solution and pH about 7–8 was maintained. A pasty mass was observed. It was diluted with water to make the solution clear and then added to a solution of metal salts (0.01 mol) with constant stirring. pH of the reaction mixture was adjusted to about 4–5. The coordination polymers thus separated out in the form of a suspension were digested on a water bath for 1 h and eventually filtered, washed and then dried in air at room temperature. The coordination polymers are insoluble in common organic solvents like methanol, ethanol, chloroform, acetone, benzene.

2.4. Antimicrobial activities

The antimicrobial activity was assayed by the Cup plate agar diffusion method (Chandra et al., 2009) by measuring inhibition zones in mm. In vitro antimicrobial activity of all synthesized compounds and standard drugs has been evaluated against four strains of bacteria which include two Gram +ve bacteria such as *Staphylococcus aureus, Bacillus megaterium* and two Gram −ve bacteria such as *Escherichia coli, Proteus vulgaris* and one fungus *Aspergillus niger*.

The antibacterial activity was compared with standard drugs viz. Amoxycillin, Ampicillin, Ciprofloxacin, Erythromycin and antifungal activity was compared with standard drug viz. Griseofulvin.

2.4.1. Antibacterial activity

The purified products were screened for their antibacterial activity by using the cup-plate agar diffusion method. The nutrient agar broth prepared by the usual method, was inoculated aseptically with 0.5 mL of 24 h old subculture of *S. aureus, B. megaterium, P. vulgaris,* and *E. coli* in separate conical flasks at 40–50 °C and mixed well by gentle shaking. About 25 mL of the contents of the flask was poured and evenly spread in petridish (90 mm in diameter) and allowed to set for 2 h. The cups (10 mm in diameter) were formed by the help of borer in agar medium and filled with 0.04 mL (40 pg/mL) solution of sample in DMF.

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