



ORIGINAL ARTICLE

# Synthesis and spectroscopic, magnetic and cyclic voltammetric characterization of some metal complexes of methionine: $[(C_5H_{10}NO_2S)_2M^{II}]$ ; $M^{II} = Mn(II), Co(II), Ni(II), Cu(II), Zn(II), Cd(II)$ and $Hg(II)$

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**Abstract** Some metal complexes of DL-methionine were prepared in aqueous medium and characterized by different physico-chemical methods. Methionine forms 1:2 complexes with metal,  $M(II)$ . The general empirical formula of the complexes is proposed as  $[(C_5H_{10}NO_2S)_2M^{II}]$ ; where  $M^{II} = Mn(II), Co(II), Ni(II), Cu(II), Zn(II), Cd(II)$  and  $Hg(II)$ . All the complexes are extremely stable in light and air and optically inactive. Magnetic susceptibility data of the complexes demonstrate that they are high spin paramagnetic complex except  $Zn(II), Cd(II)$  and  $Hg(II)$  complexes. The bonding pattern in the complexes are similar to each other as indicated by electronic absorption spectra and FTIR spectral analysis. The current potential data, peak separation ( $\Delta E$ ) and the peak current ratio ( $i_{pa}/i_{pc}$ ) of the ( $Mn, Cu$  and  $Cd$ ) complexes indicate that the charge transfer processes are irreversible, the systems are diffusion controlled and also adsorptive controlled. The charge transfer rate constant of metals in their complexes are less than those in their metal salts at identical experimental conditions due to the coordination of metal with methionine.

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## 1. Introduction

Methionine is one of the sulphur containing essential amino acids. It is used as nutritional supplement and act as antioxidant in biological system. The transition metals like  $Mn(II), Co(II), Ni(II), Cu(II)$  and  $Zn(II)$  are essential trace elements and used as nutritional supplement. They act as cofactors in various enzyme systems i.e. as metalloenzymes or as enzymatic activators (Moester, 1960; Hugnes, 1981; Berg et al., 2001; Nelson and Cox, 2000).  $Cd$  and  $Hg$  are toxic elements. Methionine is a biological chelating agent may lower the

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degree of toxicity for the formation of chelate with toxic metals (Dwyer and Mellor, 1964). Structure of a biologically important molecule has importance in both the pharmacy and medicine. From the clinical and biopharmacological points like efficacy, less toxicity, side effects in the treatment of different diseases, combination drugs are in great demand now a days. In the present research, the prepared essential trace metal complexes may be a combination of nutritional supplements like metal and essential amino acid; methionine may be a provider of both the micronutrients in biological system. Metal-ligand complexation improves the bioavailability increasing solubility in the gastrointestinal fluid, may lower toxicity, and may give a sustained releases product – required as medication.

The coordinating behavior of amino acid with different metal ions have been examined in our laboratory and reported in detail (Ehsan et al., 1997a,b, 1988, 1999a,b, Haider et al., 1986; Rahman et al., 2007; Ehsan et al., 1996, 1997c). In the current interest we investigated the coordination of DL-methionine with Mn (II), Co (II), Ni (II), Cu (II), Zn (II), Cd (II) and Hg (II) salts in aqueous medium and obtained metal complexes in crystalline form. Their synthesis, isolation and speciation by different analytical methods and cyclic voltammetric studies are demonstrated.

## 2. Experimental

### 2.1. Materials and methods

The chemicals and reagents used in the synthesis were of AR grade (E. Merck, BDH and Aldrich). Elemental analysis of the complexes was done at Tohoku University, Japan, and metal content was determined by AAS (Shimadzu, Japan). Water content in the complexes was determined by using a Karl Fischer Auto Titrator. FTIR spectra were recorded on an Infrared Spectrophotometer (IR-8300, Shimadzu, Japan) in the range 400–4600  $\text{cm}^{-1}$  as KBr pellets. The electronic spectra were recorded on a UV-visible spectrophotometer (1601 PC, Shimadzu, Japan) using nujol mull as the dispersion medium. Magnetic properties were studied at ambient temperature on a Calibrated Magnetic Susceptibility Balance (Magway MSB Mkl, Sherwood Scientific Ltd, Cambridge, England). QSTG analysis was carried out by a temperature control Electric Furnace (Heraeus, FC-222, UK). The crystallinity test for all the compounds were conducted in nujol mull as dispersion medium using ZEISS digital microscope (with CARL ZEISS optical lenses), Germany. Polarimetric analysis was performed using a PC controlled calibrated Polarimeter (LC 341, Perkin Elmer, USA). Cyclic Voltammetric (CV) Studies were conducted with a PC controlled BAS Epsilon electrochemical analyzer, USA. A three-electrode electrochemical cell made of borosilicate glass and Teflon cap was used in this study. The electrodes used are Glassy Carbon Electrode (GCE) as working electrode, Ag/AgCl (Standard KCl) as reference electrode and Pt wire as counter electrode.

### 2.2. Preparation and formulation

The metal complexes were prepared in the aqueous medium. Two general methods (methods A and B) were fixed for the synthesis of the complexes as described below.

*Method A:* About 0.6–1.4 g metal salt was taken in a beaker and dilute ammonia was added. The precipitate of metal hydroxide was filtered and washed thoroughly with deionised water until the precipitate was freed from ammonia. Then the precipitate was taken in a round bottom flask. About 0.74 g methionine was taken and dissolved in 200 mL deionized water. The solution was filtered and mixed with precipitate in the round bottom flask. The mixture was refluxed for about one hour. Then the mixture was filtered with filter paper at the hot condition. The filtrate was taken in a 500 mL beaker and was evaporated to reduce the volume to about 30 mL and was kept overnight for crystallization. The crystals were separated and washed with deionized water and dried at 50 °C in a oven for about 2 h and the product was kept in a desiccator. The methionine complexes of the metals [M = Co(II), Ni(II), Cu(II), Zn(II) and Hg(II)] were prepared following this method.

*Method B:* About 0.74 g of methionine and about 0.265 g of  $\text{Na}_2\text{CO}_3$  were taken in a beaker containing about 200 mL of water and were heated to dissolve the mixture. The pH of the solution was adjusted to about 7.0 and filtered. About 0.30–0.55 g of M(II) salt was dissolved in 50 mL deionized water and was filtered using filter paper. Both the filtrate was mixed together and refluxed for about 1 h. The mixture was filtered in the hot condition in a 500 mL beaker. The solution was then evaporate slowly and reduce the volume to about 30 mL and was kept overnight for crystallization after filtration. The crystals were separated and washed with deionized water and dried at 50 °C in an oven for about 2 h and the product was kept in a desiccator. The manganese and cadmium complex of methionine was prepared using this method.

The formulation of the complexes was done by comparing the experimental and calculated data for elemental analysis. The micro analytical data of C, H, N, S and M in the prepared complexes are tabulated in Table 1.

## 3. Results and discussion

The complexes were characterized by different physico-chemical techniques and some properties were investigated. The compounds are insoluble in most of the common solvents such as water, methanol, ethanol, DMSO etc. but soluble in dilute mineral acids and alkali. The microscopic views of particles of the complexes indicate that the crystalline nature of the complexes is different from the parent ligand and the shapes of the crystals in most of the complexes are irregular.

All the complexes melted with decomposition far below the melting point of the ligand (264 °C) owing to the formation of new compound with metal. The Karl Fischer Analysis, QSTGA and FTIR data of the complexes strongly support that crystalline or chemically coordinated water is absent in the complexes.

## 4. IR spectral analysis

The tentative assignments have been done on the basis of standard references and some published papers (Pavia et al., 1979; Silverstein et al., xxxx; Rao, 1963; Nakamoto, 1978). The characteristic bands of the complexes are listed in Table 2. The pattern of the IR spectra of other complexes are almost similar with each other with some exceptions. Most of the important

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