Synthesis, Identification and Application studies of 1,1',((1E,1'E)-1,2-phenylene bis(diazene-2,1-diyl))bis(naphthalen-2-ol) complex of Cu(II)

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Abstract

The Schiff base ligands and their corresponding metal complexes have expanded enormously and include a vast area of organometallic compounds and various aspects of bioinorganic chemistry. On the other hand, azo compounds are very important molecules and have attracted much attention in both academic and applied research. The azo compounds and their metal-azo complexes are extremely used in dyes and data storage. New complexes with metal ions Cu(II) and L are synthesized. Elemental and spectroscopic measurements are used to elucidate the structure of the newly prepared metal complex. The azo-dye Schiff's base behaves as a di-negative N_2O_2 tetradentate ligand. The metal complexes exhibited square planar structure. The azo-dye Schiff's base and its complex are characterized by FTIR, DRS and EDAX data.

Introduction

Among the synthetic organic dyes, azo dyes is one of the most representative and large classes. All these dyes contain the azo group -N=N- as part of their molecular commercial dyes belong to these dyes [1-4]. The practical applications of the azo dyes are related to coloring fibers, due to their affinity for wool and silk, photoelectronics, optical storage technology, biological reactions, analytical chemistry, printing systems and food chemistry [4].

In this work, we synthesized 1,1',((1E,1'E)-1,2-phenylene bis(diazene-2,1-diyl))bis(naphthalen-2-ol) shown in Scheme 1 and used it in a Cu(II) complex.

Preparation of azo ligand

All steps of the procedure were performed ice-water bath. To 5 ml of HCl and 20 ml D.I. water, 0.005 mol phenylenediamine was added. 0.005 mol NaNO₂ was also solved in 10 ml D.I. water. The two solutions were added dropwise on stirring. A deep colored solution indicates that diazonium salt is formed. The salt is not stable. Therefore, 0.005 mol β -naphthol was dissolved in NaOH solution. Then, the diazonium salt was added immediately to β -naphthol solution. The obtained brown precipitate was collected and dried. Yield: 80%, mp: 123-125°C. In the next step, this precipitate reacted as an amine, formed the second diazonium salt and coupled with another β -naphthol molecule, see Scheme 1. The final product was again a brown precipitate. Yield: 57%, mp: 205°C.



Scheme 1. Procedure of ligand preparation

Preparation of Cu(II) complex

The Cu(II) complex of this ligand was prepared by dissolving 0.5 mmol Cu(NO3)2 in 15 ml DMF. To the metallic solution was added a solution of 0.5 mmol of the above azo ligand. Changing of the solution color and pH, was attributed to the complex formation. The resulted solution was refluxed for 6h. A black precipitate was obtained on cooling the solution, see Scheme 2. Yield: 67%, mp: $>300^{\circ}$ C.



Scheme 2. Procedure of Cu(II) complex preparation

Results and Discussion

The ligand and its complex was characterized by FTIR, EDX and XRD methods. The results are given below. IR spectrum of azo ligand is shown in Fig. 1. The absorption peak at 3000 cm⁻¹ is attributed to

aromatic C–H bond. The broad peak at 3400 cm⁻¹ is for O–H bond. The C=C is seen at 1625 and 1598 cm⁻¹. The peak at 1512 cm⁻¹ is attributed to N=N stretching vibration [5,6].



Fig. 1 The FTIR spectrum of azo ligand





Fig. 2 The DRS spectrum of complex

The complex is coloured and as is expected the UV absorption falls in visible region, 400-700 nm. The peak in 210 nm is assigned to $\pi \rightarrow \pi^*$. The peak in 370 nm is for azo group.

The EDX spectrum of the complex in Fig. 3 shows Cu, C, N and O atom quantities.

keV	Cnts						Time 30 / 30 00001	•
	2000 2000- 1500 1000- 600- N Ka Cuta				cuKa cuk			
	0-8		5			10		
⇔≫≙∙								
	Quantify Results			Identify Auto Results			Match Results	
Elt		W%	A%	Formula		0x%	HConf 27.00	
C N O Cu Total		76.24 8.97 14.60 0.18 100	80.31 8.11 11.54 0.04 100			0.00 0.00 0.00 0.00 0.00	77.20 10.22 15.34 0.20 11.15	

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