



ISSN 0975-413X
CODEN (USA): PCHHAX

Der Pharma Chemica, 2017, 9(14):119-122
(<http://www.derpharmachemica.com/archive.html>)

Spectrophotometric and Cyclic Voltammetric Studies of Iron Complex with NTA Ligand and Investigation of Its Catalase-like Activities

Guluzar Ozbolat^{*}, Abdullah Tuli

Department of Medical Biochemistry, Cukurova University, 01330 Adana, Turkey

ABSTRACT

In this study, iron(III) complex of NTA was synthesized and structurally characterized in its solid state and solution state by IR, UV-vis, elemental analysis and magnetic susceptibility. The catalase activities of complex were investigated. It was showed that the complex has catalase activity. It suggesting that this type of complex may constitute a new and interesting basis for the future search of new and more potential drugs. Electrochemical behavior of ligand and complexes were examined as supporting electrolyte and platinum electrode for cyclic voltammetry. The reductions of free ligand and the complex take place differently.

Keywords: CV, NTA, Fe (III), Ligand

INTRODUCTION

Nitrilotriacetic acid (NTA) belongs to the wide family of so called aminopolycarboxylic acids (APCAs) containing several carboxylate groups bound to one or more nitrogen atoms. NTA was used as a chelating agent. NTA a tetradentate ligand, is known to form close shell ternary complex with Fe(III) [1,2]. The application of the iron(III) - NTA complexes in medicine has been investigated [3]. For example; Fe-NTA-induced ROS play a protective role in human HSCs by regulating Bcl-2 family proteins and mitochondrial membrane potential [4]. One of the most utilized and studied iron(III) chelating agents is nitrilotriacetic acid (NTA) [5]. It is well known from the literature that NTA produces dissolved complexes with iron(III) [6]. The aim of this work is to study the two electrochemical properties and spectrophotometric of iron (III) ions with NTA ligand that may be used in pharmaceutical applications.

MATERIALS AND METHODS

In this study, iron complex of Nitrilotriacetic acid (NTA) was synthesized. The complexes have been characterized by elemental analyses, FT-IR spectra, magnetic susceptibility and H-NMR. Electrochemical behavior of ligand and complexes were examined as supporting electrolyte and platinum electrode for cyclic voltammetry. The infrared spectra of the ligand and complexes were recorded on a Thermo FT-IR spectrophotometer; Smart FITR diamond attenuated total reflection (ATR); 4000-400 cm^{-1}) using KBr pellets. Elemental analysis (C, H, N, S) was carried out by Thermo Scientific Flash 2000. Magnetic susceptibility values were measured at room temperature by Guoy's method. UV-vis spectroscopic measurements were collected on Perkin Elmer Lambda Uv-Vis spectrophotometer with a 10 mm quartz spectrophotometer cell. The electrochemical experiments were performed on a CHI 660D.

SYNTHESIS AND CHARACTERIZATION

Complex was prepared by treating Nitrilotriacetic acid (0.1 mmol, 0.025 g) with $\text{FeCl}_3 \cdot 6\text{H}_2\text{O}$ (0.1 mmol, 0.028 g) in ethanol under solvothermal conditions at 80°C for four days. Brown crystals of complex were harvested. Yield (0.038 g, 96%). The results from the C, H, N elemental analyses are in excellent agreement with the expected chemical formula, Anal. Calc. for $\text{C}_6\text{H}_6\text{FeNO}_6$: C 41.21; H 2.69; N 11.82 %. Found: C 41.12; H 2.73; N 11.74. IR (KBr, cm^{-1}): 3040(s, $\nu(\text{C-H})$), 1730 (s, $\nu(\text{C=O})$), 484 (s, $\nu(\text{Fe-O})$). UV (nm) 244,250,271,320 (For the synthetic route as shown in Figure 1).

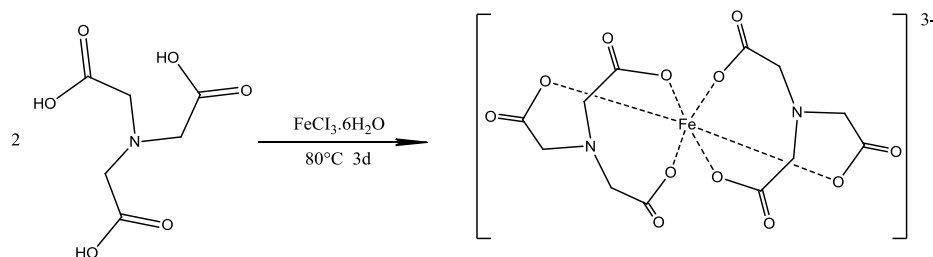


Figure 1: The synthetic route of complex

Cyclic voltammetry

The cyclic voltammetric studies of Fe(III)-NTA complex at platinum electrode. The cyclic voltammogram of the complex, at a scan rate of 100 mVs^{-1} . The current-potential curves for ligand, and NTA-iron(III) complex using are presented in Figures 2-4 respectively. A standard three-electrode assembly was employed: Platinum as working electrode, Ag/AgCl as reference electrode, and platinum wire as counter electrode. The CV of the complex is reversible.

Catalase activity

Catalase activity was measured using Biodiagnostic Kit which is based on the spectrophotometric method described by Aebi. The catalases-like activities of different concentrations of complex was done by reacting with known quantity of H_2O_2 catalase reacts with a known quantity of hydrogen peroxide and the reaction is stopped after 1 min with catalase inhibitor. In the presence of peroxidase, the remaining hydrogen peroxide reacts with 3,5-Dichloro-2-hydroxybenzene sulfonic acid and 4-aminophenazone to form a chromophore with a color intensity inversely proportional to the amount of catalase in the sample. The absorbance was measured at 510 nm.

RESULTS AND DISCUSSION

Structural characterization

Synthesis complex was characterized by the techniques like CHN analysis, FT-IR, UV-Vis and magnetic susceptibility. The results from the C, H, N elemental analyses are in excellent agreement with the expected chemical formula, Anal. Calc. for $\text{C}_6\text{H}_6\text{FeNO}_6$: C 41.21; H 2.69; N 11.82%. Found: C 41.12; H 2.74; N 11.72. The infrared spectrum of the complex recorded the band characteristics of the coordinated ligand. The absence of broad characteristic peak at 3426 cm^{-1} corresponding to O-H stretching vibration confirmed the coordinate nature of Fe-O bond. Formation of Fe-O band was further supported by appearance of bend at 484 cm^{-1} . All these frequencies confirmed the formation of complex. The UV-vis absorption spectrum of Fe(III), ligand and complex were shown in Figure 2, in which three characteristic absorption bands were found. The spectrum of the NTA and its Fe(III) complex were recorded in the pH 6-8 in the range of 200-800 nm. The complex shows λ_{max} at 244-250 nm and 271 nm-326 nm corresponding to $\pi-\pi^*$ and $n-\pi^*$ transition, respectively. Magnetic susceptibility measurements of the complexes were performed at room temperature. The magnetic moment values for the various Fe(III) complexes is in the range 1,73 BM is for octahedral complexes.

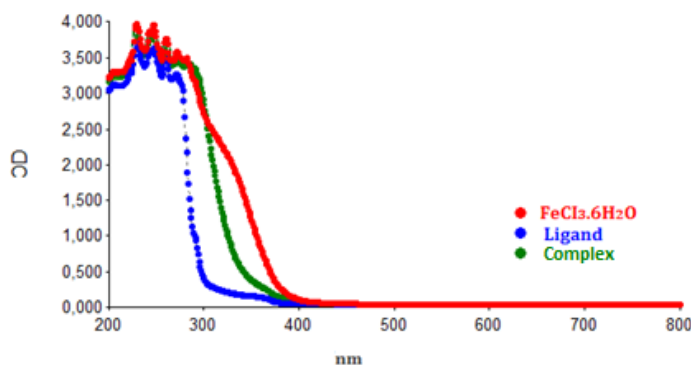


Figure 2: UV-visible wavelength scanning chart

Cyclic voltammetry

Firstly, the complex was synthesized and characterized. Then the electrochemical property of this complex Fe (III)-NTA was investigated by using cyclic voltammetry technique on platinum electrode. The cyclic voltammetric behavior of Fe (III) complex of NTA was studied in at room temperature. The cyclic voltammograms of iron (III)-NTA complex and free NTA ligand were compared first. Then the electrochemical reduction of iron (III) complex of NTA complex was investigated by cyclic voltammetry. The current-potential curves for NTA ligand, iron (III) and NTA-iron (III) complex using are presented in Figure 2. A standard three-electrode assembly was employed: Platinum as working electrode, Ag/AgCl as reference electrode, and platinum wire as counter electrode. The scan rate was 100 mVs^{-1} , and the quoted E values are versus Ag/AgCl. Figure 2 shows the cyclic voltammogram (CVs) of collected at NTA (blue line curve), NTA-Fe (III) (green line curve) and Fe (III) (red line curve) coated electrodes. In contrast to the CV of the green line curve, the redox waves with 0.30 V and were attributed to the reduction/reoxidation of the iron redox center in the NTA-Fe (III) complex.

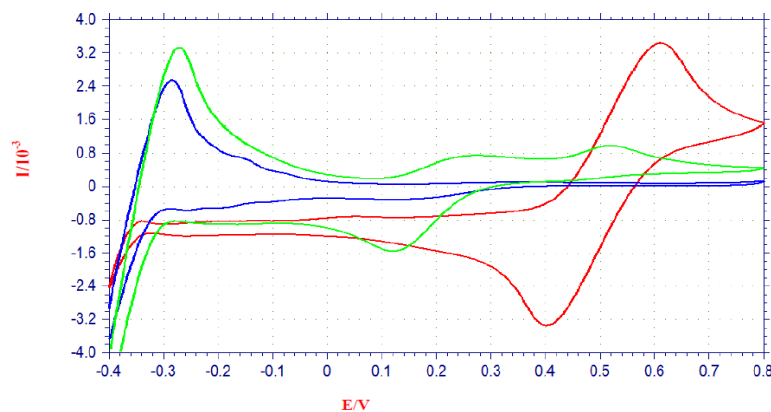


Figure 3: CV of NTA (blue line curve), Fe (III) (red line curve), NTA-Fe (III) complex (green line curve)

The current-potential curves for NTA ligand and its Fe (III) complex are presented in Figure 3. The voltammetric behavior of free ligand is characterized by one reduction peak at a platinum surface (blue green line). The peak potential is about -0.80 V (vs. Ag|Ag+) at a scan rate $\nu=0.1 \text{ Vs}^{-1}$. The peak height corresponds to a one-electron reduction. There appears no oxidation peak in the reverse scan. It is seen that the free ligand reduces irreversibly with one electron transfer on platinum surface. The cathodic peak is for the reduction of NTA ligand to the anion radical. In contrast to free ligand, the Fe (III)-NTA complex undergoes a quasi-reversible process at the platinum surface. The complex gives one electron transfer redox system at -0.6 V and -0.5 (vs. Ag|Ag+) which occurs at 0.1-0.2 V more positive potential than free ligand. These results indicate that the reductions of free ligand and the complex take place differently.

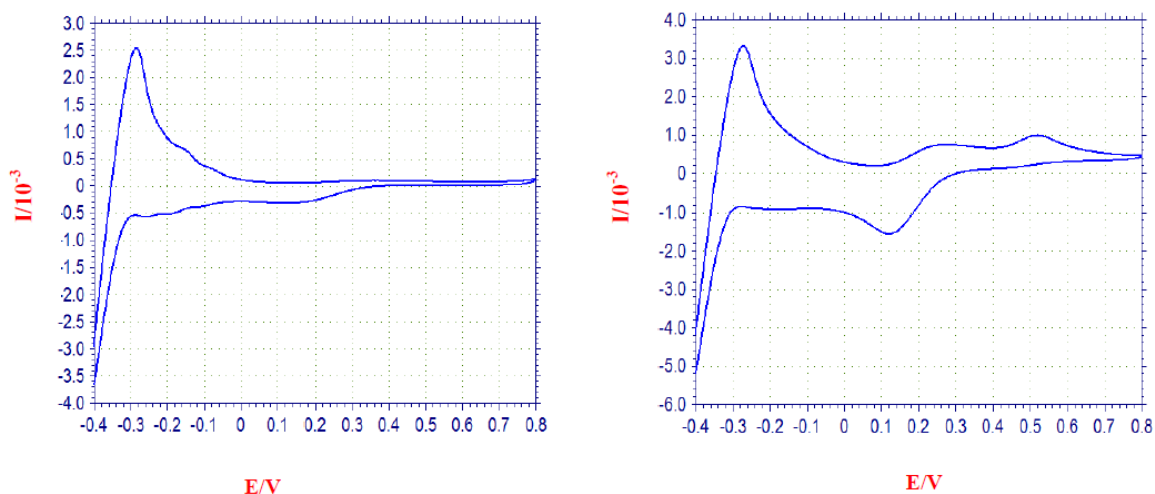


Figure 4: NTA ligand and its Fe (III) complex

Catalase activity

The catalytic activity studies of Fe(III) complex in DMSO towards the disproportionation of hydrogen peroxide were also performed. The studies showed that complex is catalytically active. The study showed that complex is catalytically active. Where at a concentration of 2.0 mM, the activity was equivalent to 510.7 U/L. When the coordinate Fe(III) ion is present in structure, the catalytic reactivity enhances.

CONCLUSION

In this study a Fe (III) complex of NTA was synthesized and structurally characterized. The electrochemical reductions of ligand and Fe(III)-NTA complex were investigated at platinum surface in DMSO medium. The voltammetric behavior of free ligand is characterized by one reduction peak at a platinum surface (blue green line). The peak potential is about -0.80 V (vs. Ag|Ag+) at a scan rate $\nu=0.1 \text{ Vs}^{-1}$. The peak height corresponds to a one-electron reduction. In contrast to free ligand, the Fe (III)-NTA complex undergoes a quasi-reversible process at the platinum surface. The complex gives one electron transfer redox system at -0.6 V and -0.5 (vs. Ag|Ag+) which occurs at 0.1-0.2 V more positive potential than free ligand. These results indicate that the reductions of free ligand and the complex take place differently. As a conclusion the ligand and the complex show different. The free ligand reduces irreversibly when the complex behaves a two-electron quasi-reversible redox system. The peak potential is about 0.075 V and 0.28 V (vs. Ag|Ag+) at a scan rate $\nu=0.1 \text{ Vs}^{-1}$. The complex gives two electron transfers redox system at 0.3 V and 0.5 V (vs. Ag|Ag+). The oxidation peak is also observed in the reverse scan for the complex. The peak potential is about -0.3 V and 0.56 V. These results indicate that the reductions of free ligand and the complex take place differently. As a conclusion the ligand and the complex show different. The complex presented catalase-like activity. It may constitute a new and interesting basis for the future search of new and more potential drugs. These results indicate that may be used of NTA in pharmaceutical applications. Although promising NTA as medicine needs, more *in vitro* and *in vivo* studies are need.

ACKNOWLEDGEMENTS

The financial support of Cukurova University is gratefully acknowledged. Project Number: ‘TDK-2015-3577’.

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