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VOLTAMETRIC CHARACTERIZATION OF HYDRAZIDE MACROCYCLIC COMPOUNDS

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Introduction

Hydrazide macrocyclic compounds represent an important class of organic and coordination compounds that combine the structural characteristics of hydrazides with the unique geometry of macrocyclic systems. These compounds have attracted considerable interest in medicinal chemistry, supramolecular chemistry, catalysis, and materials science due to their strong binding affinity, structural versatility, and diverse biological activities [1]. The main objective of this study was to investigate the electrochemical properties of two hydrazide macrocyclic compounds (achyM4 and achyM6) and their interactions with metal cations (Ag^+ and Cu^{2+}) using voltammetric techniques and Job's method of continuous variations [2].

Materials and methods

The investigated hydrazide macrocyclic compounds were prepared by the reaction of pyridine-2,6-dicarbohydrazine with selected dialdehydes (**Scheme 1**). Voltammetric measurements were performed using a three-electrode electrochemical cell. The glassy carbon electrode was used as the working electrode, Ag/AgCl electrode was used as the reference electrode, and a platinum wire served as the counter electrode. Electrochemical measurements were carried out in a tris-buffer (pH = 7). The applied potential range was from −1.0 V to 1.0 V, and the total volume of the measurement solution was 15 cm^3. Spectrophotometric measurements were performed in quartz cuvettes using dimethyl sulfoxide (DMSO) as the solvent. Absorbance spectra were recorded in the wavelength range from 200 nm to 1100 nm.

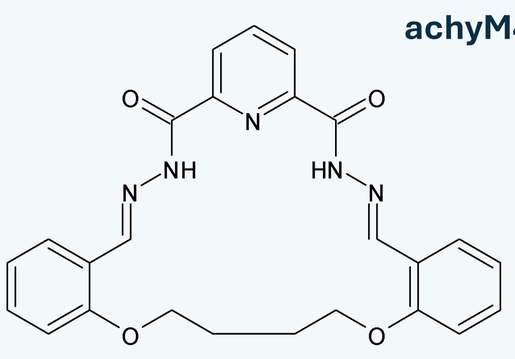
Conclusions

The cyclic voltammetry results showed that copper nitrate and silver nitrate were electroactive. Both macrocyclic ligands were electroactive, and a single irreversible oxidation peak at approximately 0.9 V was observed for each compound. Interactions between the investigated metal cations and the macrocyclic ligands were confirmed by shifts in the oxidation peaks of the cations in solutions containing both the metal ions and the macrocyclic ligands. The metal-to-ligand stoichiometric ratio was estimated using Job's method of continuous variations, revealing a 1:1 stoichiometry for all investigated metal-ligand interactions.

References

[1] P. Xin, L. Zhang, P. Su, J.-L. Hou, Z.-T. Li, Chem. Commun. 51 (2015) 4819.
[2] R. C. Feitosa et al., *Pharmaceutics* 15 (2023) 1285.

Results



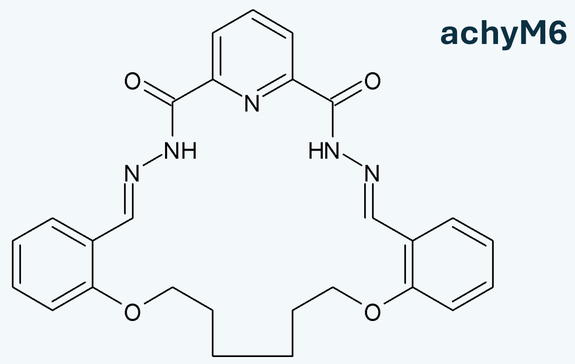


Fig 1. Chemical structures of hydrazide macrocyclic compounds achyM4 and achyM6.

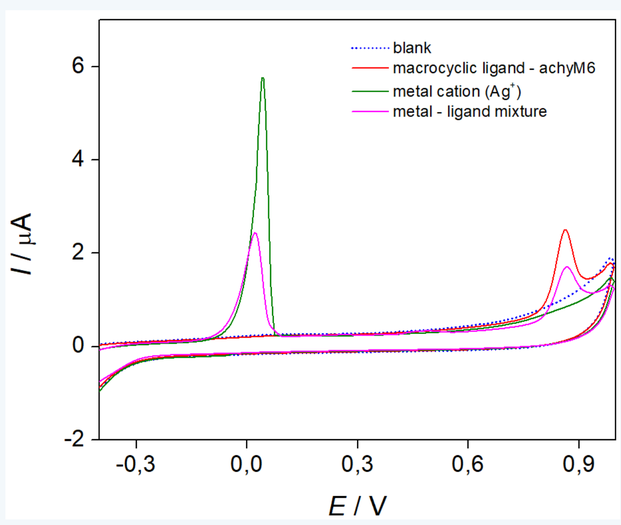


Fig 2. Cyclic voltammograms of blank solution, achyM4 and achyM6 in tris-buffer (pH = 7). Scan rate 100 mV s^{-1}.

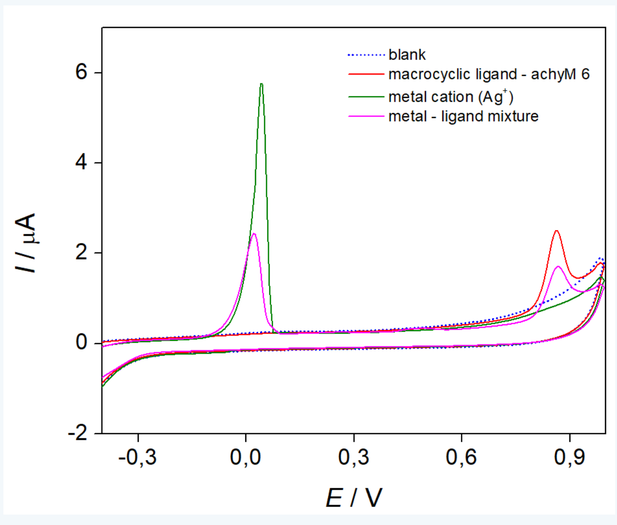


Fig 3. Cyclic voltammograms of blank solution, Ag nitrate, achyM6, and Ag-achyM6 reaction mixture at a 1:1 molar ratio, in tris-buffer (pH = 7). Scan rate 100 mV s^{-1}.

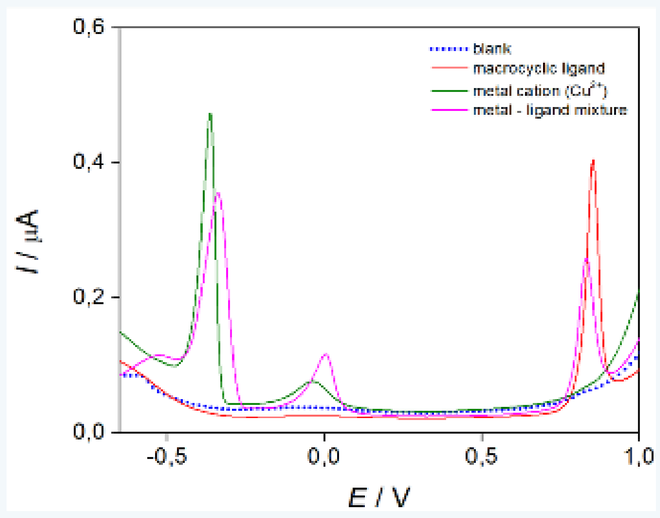


Fig 4. Differential pulse voltammograms of blank solution, Cu(II) nitrate, achyM4, and Cu(II)–achyM4 reaction mixture at a 1:1 molar ratio. Scan rate 5 mV s^{-1}.

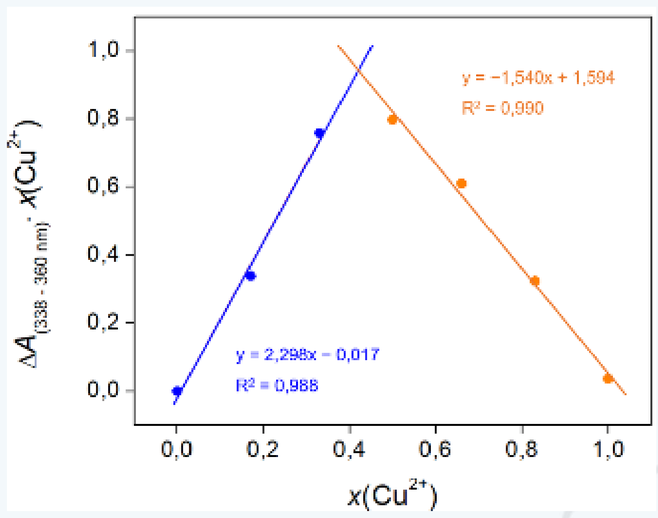
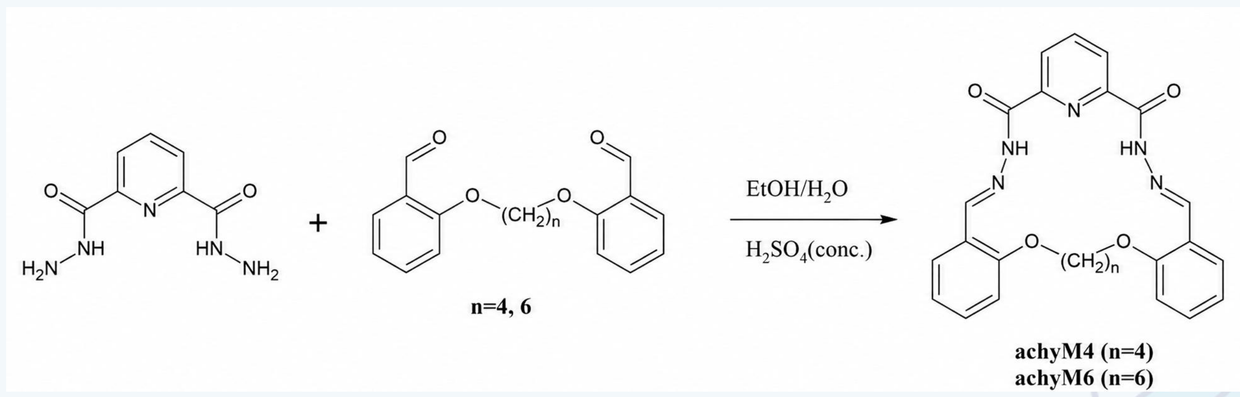


Fig 5. Determination of the Cu^{2+} ion-to-macrocyclic ligand (achyM4) ratio in the complex using Job's method.



Scheme 1. Synthetic strategy for the preparation of hydrazide macrocyclic compounds.