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## New Copper(II) and Nickel(II) Complexes with Dimethylglyoxime and Amino-acids: Synthesis, Characterization and Electrochemical Properties

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**Abstract:** Four new Cu(II) and Ni(II) ion complexes were created from the interaction of dimethylglyoxime as primary ligand and methionine or alanine as a secondary ligand, has been investigated using molar conductivity, infrared, UV/vis. The electrochemical behavior of these complexes was determined by cyclic voltammetry. The compounds prepared are solids, insoluble in water, ethanol and methanol, but soluble in dimethylsulfoxide (DMSO) and dimethylformamide (DMF). The molar conductance data confirm that two complexes are not electrolytic. The IR study shows that dimethylglyoxime ligand is coordinated to the metal ion in a bidentate manner with NN donor sites of the oxime function, where the secondary ligand is coordinated by the carboxylate oxygen and the N atom of the amino acid. The electronic spectral data indicated that the synthesized complexes have octahedral or square-planar geometries. An electrochemical application was carried out on these mixed complexes in order to study their redox properties, which indicated an irreversible oxidation corresponding of M to M(I) and of M(I) to M(II).

**Keywords:** Complexes, Dimethylglyoxime, Amino acids, Spectroscopic analysis, Electrochemical application

### Introduction

The chemistry for oxime metal complexes has been investigated actively since the first synthesis of nickel (II) dimethylglyoximate and recognition of the chelate. Copper (II) complexes with dimethylglyoxime are known for their high stability, but their biological activity has been very scarcely studied (Bougherra et al, 2018). Amino acids are classified as promising biomolecules that can be complexed with metals to form stable and active catalysts. Therefore, Amino acid-metal complexes and their derivatives have attracted many researchers in the fields of pharmaceuticals and petrochemical industries (Alrufaydi et al., 2020).

Copper (II) and nickel (II) complexes with amino acids are of significant pharmacological interest because several of them exhibit a broad spectrum of effects, in particular anti-inflammatory, antiulcer, anticonvulsant and even anti-tumor. It has also been established that these complexes often exhibit higher pharmacological activity than that of their free ligands (Adkhis et al., 2022; Wagner et al., 2008; Baran et al., 2009). In this work, we describe the synthesis, spectral characterization and electrochemical application of the new Ni(II) and Cu(II) complexes of dimethylglyoxime as the primary ligand and alanine or methionine as secondary one.

### Experimental

#### Materials and Methods

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All the chemical reagents and solvents used in the synthesis of the complexes were (Fluka products) used without further purification. Melting points were determined on a Stuart scientific SPM3 apparatus fitted with a microscope and are uncorrected. The infrared spectra were recorded in the region 4000–400  $\text{cm}^{-1}$  on a BRUKER TENSOR 27 IR spectrophotometer. Electronic spectra were measured on a thermo Scientific EVOLUTION 220 ultraviolet-visible spectrophotometers (in DMSO solution); measurements were made from 200 to 800 nm. The molar Conductance values were obtained for  $10^{-3}$  M in DMSO solution at 25 °C determined using melting point meter MPM-H2. All electrochemical experiments were carried out using a potentiostat-galvanostat EGG-273 A controlled with Power-suite software. The electrochemical cell was equipped with a modified carbon past disk as the working electrode, a platinum electrode as counter electrode and silver/silver chloride (Ag/AgCl) electrode as the reference electrode. The electrolytic bath consisted of the complexes in DMSO solution ( $10^{-3}$  M) and sodium perchlorate ( $\text{NaClO}_4$   $10^{-1}$  M) was used as a supporting electrolyte to increase the conductivity of the bath. The electrochemical characterization was performed using the cyclic voltammetry (CV) with a scan rate of 50 mV/s.

## Synthesis of Complexes

To an aqueous solution of metal salt (5 mmol) and KI or KSCN (10 mmol) was added dropwise an ethanolic solution of amino acid (5mmol). After stirring and refluxing a hot ethanolic solution of dimethylglyoxime (5 mmol) was added. Then a few drops of NaOH solution were added to keep the pH at 8. The resulting mixture is left stirring under reflux at 70 °C for 3 hours. The precipitate obtained was isolated by filtration and washed with ethanol and diethylether. The solid was dried at room temperature.

## Results and Discussion

The physical properties of the prepared complexes are summarized in Table 1. All the complexes are soluble in DMSO and DMF but insoluble in water, methanol and ethanol. The electrolyte or non-electrolyte nature of the complexes was confirmed by low or high values of molar conductance measured at 25°C ( $10^{-3}$  M) in a DMSO solution.

Table 1. Physical properties of the complexes

| Compound formula                                                        | Yield (%) | Melting point(°C) | Color | $\Lambda(\Omega^1\text{cm}^2\text{mol mol}^{-1})$ | Metal (%) |       |
|-------------------------------------------------------------------------|-----------|-------------------|-------|---------------------------------------------------|-----------|-------|
|                                                                         |           |                   |       |                                                   | Cal       | Found |
| $[\text{Cu}(\text{H}_2\text{dmg})(\text{Meth})(\text{SCN})_2]$ <b>1</b> | 65        | 260               | Blue  | 15.2                                              | 14.35     | 13.98 |
| $[\text{Ni}(\text{H}_2\text{dmg})(\text{Meth})](\text{SCN})_2$ <b>2</b> | 87        | 302               | Red   | 170                                               | 13.4      | 13.58 |
| $[\text{Cu}(\text{H}_2\text{dmg})(\text{Ala})]\text{I}_2$ <b>3</b>      | 56        | 310               | Gray  | 64.5                                              | 12.2      | 12    |
| $[\text{Ni}(\text{H}_2\text{dmg})(\text{Ala})]\text{I}_2$ <b>4</b>      | 47        | 312               | pinck | 11.2                                              | 11.38     | 11.2  |

## Infrared Spectra

The assignments of the more significant IR absorption bands of the free ligands and their metal complexes are shown in Table 2 and Table 3, respectively, and the FT-IR spectrum of  $[\text{Cu}(\text{H}_2\text{dmg})(\text{Meth})(\text{SCN})_2]$  and  $[\text{Ni}(\text{H}_2\text{dmg})(\text{Ala})]\text{I}_2$  are shown in figure 1 and figure 2, respectively. The IR spectrum of dimethylglyoxime shows a band with medium intensity at 1447  $\text{cm}^{-1}$ , which is attributed to (C=N) of the oxime. This band is shifted to higher frequency (1450-1503  $\text{cm}^{-1}$ ) in all the mixed-ligand complexes (Jadhav et al, 2013; Salih, et al, 2009; Chandar et al, 2011).

The strong absorption at 1143  $\text{cm}^{-1}$  due to  $\nu(\text{N-O})$  of the oxime group in the free  $\text{H}_2\text{dmg}$  is shifted towards higher frequency in all the complexes (Osunlaja et al, 2009; Berradj et al, 2021). While the appearance of a band in all the complexes in the region 752- 772  $\text{cm}^{-1}$  is due to the C=N-O deformation vibration of the oxime group, this band is shifted in free ligand at 750  $\text{cm}^{-1}$  (Abane et al, 2019). All these features indicate that the  $\text{H}_2\text{dmg}$  is coordinated to the metal ion through the nitrogen of the oxime.

The  $\nu(\text{NH}_3^+)$  of methionine and alanine is observed in 3146 and 3072  $\text{cm}^{-1}$  respectively. In the complexes,  $\text{NH}_3^+$  gets deprotonated and binds to metal through the neutral  $\text{NH}_2$  group. The IR spectra of complexes show characteristic bands of  $\nu(\text{NH}_2)$  in the region 3100- 3309  $\text{cm}^{-1}$  (Mamun, et al, 2010; Wagner et al, 2004). Whereas the deformation frequency  $\delta(\text{NH}_3^+)$ , which is characteristic for the zwitterions, appear

at about  $1615\text{ cm}^{-1}$  in amino acids, in the metal complexes with methionine,  $\delta(\text{NH}_2)$  is shifted to higher wave numbers, while that of alanine is displaced to the lower wave numbers (Rosu et al., 2005). Hence, it can be concluded that the nitrogen of the amino group is involved in coordination.

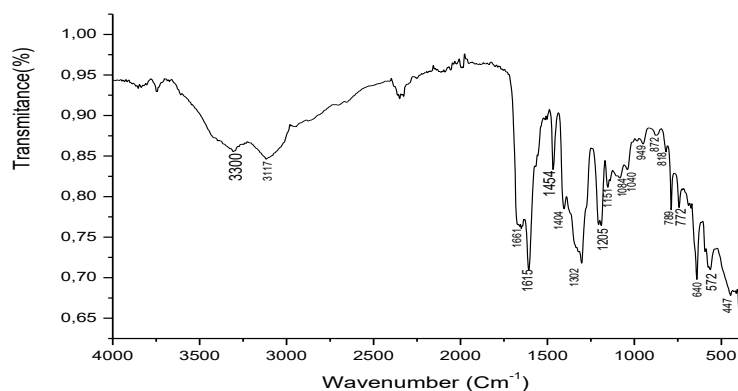
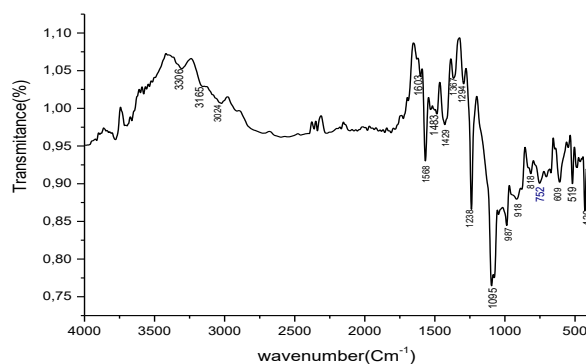
The spectra of amino acids display  $\nu_{\text{as}}(\text{COO}^-)$  and  $\nu_{\text{s}}(\text{COO}^-)$  frequency in the  $1583\text{ cm}^{-1}$  and  $1409\text{ cm}^{-1}$  range, respectively. In the metal complexes,  $\nu_{\text{as}}(\text{COO}^-)$  and  $\nu_{\text{s}}(\text{COO}^-)$  stretching bands are located in the region  $1520\text{--}1616\text{ cm}^{-1}$  and  $1373\text{--}1429\text{ cm}^{-1}$ , respectively (Tidjani-Rahmouni et al., 2014; Adkhis et al., 2000). The infrared spectra of the prepared complexes show weak bands in the range of  $519\text{--}572\text{ cm}^{-1}$  and  $426\text{--}441\text{ cm}^{-1}$ . They are attributed to the  $\nu(\text{M-O})$  and  $\nu(\text{M-N})$  vibration, respectively (Bougherra et al., 2018).

Table 2. Characteristic IR absorption bands ( $\text{cm}^{-1}$ ) of the ligands

| Compound         | $\nu(\text{NH}_3^+)$ | $\delta(\text{NH}_3^+)$ | $\nu_{\text{as}}(\text{COO}^-)$ | $\nu_{\text{s}}(\text{COO}^-)$ | $\nu(\text{CN})$ | $\nu(\text{NO})$ | $\delta(\text{NO})$ |
|------------------|----------------------|-------------------------|---------------------------------|--------------------------------|------------------|------------------|---------------------|
| Dimethylglyoxime |                      |                         |                                 |                                | 1447             | 1143             | 750                 |
| Methionine       | 3146                 | 1615                    | 1583                            | 14109                          |                  |                  |                     |
| Alanine          | 3072                 | 1618                    | 1583                            | 1409                           |                  |                  |                     |

Table 3. Characteristic IR absorption bands ( $\text{cm}^{-1}$ ) of the complexes

| Complex   | $\nu(\text{NH}_2)_{\text{coord}}$ | $\delta(\text{NH}_2)$ | $\nu_{\text{as}}(\text{COO}^-)$ | $\nu_{\text{s}}(\text{COO}^-)$ | $\nu(\text{CN})$ | $\nu(\text{NO})$ | $\delta(\text{NO})$ | $\nu(\text{M-O})$ | $\nu(\text{M-N})$ |
|-----------|-----------------------------------|-----------------------|---------------------------------|--------------------------------|------------------|------------------|---------------------|-------------------|-------------------|
| Complex 1 | 3117-3300                         | 1661                  | 1616                            | 1404                           | 1454             | 1205             | 772                 | 572               | 441               |
| Complex 2 | 3100-3200                         | 1627                  | 1558                            | 1400                           | 1503             | 1238             | 760                 | 544               | 428               |
| Complex 3 | 3309                              | 1581                  | 1520                            | 1373                           | 1450             | 1200             | 752                 | 548               | 426               |
| Complex 4 | 3165-3306                         | 1603                  | 1568                            | 1429                           | 1483             | 1238             | 752                 | 519               | 430               |

Figure 1. Infrared spectrum of  $[\text{Cu}(\text{H}_2\text{dmg})(\text{Meth})(\text{SCN}_2)]$ Figure 2. Infrared spectrum of  $[\text{Ni}(\text{H}_2\text{dmg})(\text{Ala})(\text{I}_2)]$ 

## Electronic Spectra

The electronic spectral data of the ligands and complexes are presented in Table 4 and Table 5 respectively, and the electronic absorption spectrum of  $[\text{Ni}(\text{H}_2\text{dmg})(\text{Meth})](\text{SCN})_2$  is given in Figure 3.

In the UV spectra of the ligands, the bands in the range 250-290 nm can be associated to  $\pi \longrightarrow \pi^*$  intra-ligand transition. In the spectra of the complexes, these bands are shifted toward higher wavelengths, confirming the coordination of the ligands in the metal center. The spectra of complexes show a band at 430 nm for complexes with Copper(II) and at 375 nm for complexes with nickel(II), which is assigned to charge transfer transition.

The electronic spectra of complexes 1 and 3 (complexes with Cu) show one band in the visible domain, respectively at 711 and 520 nm, this band is attributed to the d-d transition characteristic octahedral and square-planar geometry respectively (Solans-Monfort et al., 2011). In the visible range, the spectra of complex 2 exhibit two bands. These transitions are in agreement with a square-planar geometry around the Ni(II) ion (JieShen et al., 2005). Furthermore, the complex 4 shows three bands in the visible. These transitions are in agreement with an octahedral geometry around the Ni(II) ion (Randhır et al., 2000).

Table 4. Electronic spectral data of the ligands

| Compound         | $\lambda$ (nm) | $\nu$ (cm <sup>-1</sup> ) | $\epsilon$ (l mol <sup>-1</sup> cm <sup>-1</sup> ) | Electronic transitions      |
|------------------|----------------|---------------------------|----------------------------------------------------|-----------------------------|
| Dimethylglyoxime | 290            | 34483                     | 100                                                | $\pi \longrightarrow \pi^*$ |
|                  | 270            | 37037                     | 1300                                               | $\pi \longrightarrow \pi^*$ |
| Methionine       | 283            | 35336                     | 20                                                 | $\pi \longrightarrow \pi^*$ |
|                  | 259            | 38610                     | 30                                                 | $\pi \longrightarrow \pi^*$ |
| Alanine          | 276            | 36232                     | 50                                                 | $\pi \longrightarrow \pi^*$ |
|                  | 261            | 38314                     | 43                                                 | $\pi \longrightarrow \pi^*$ |

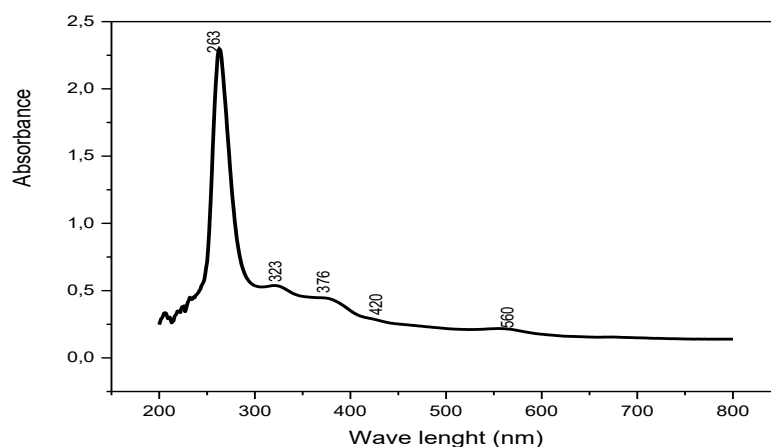
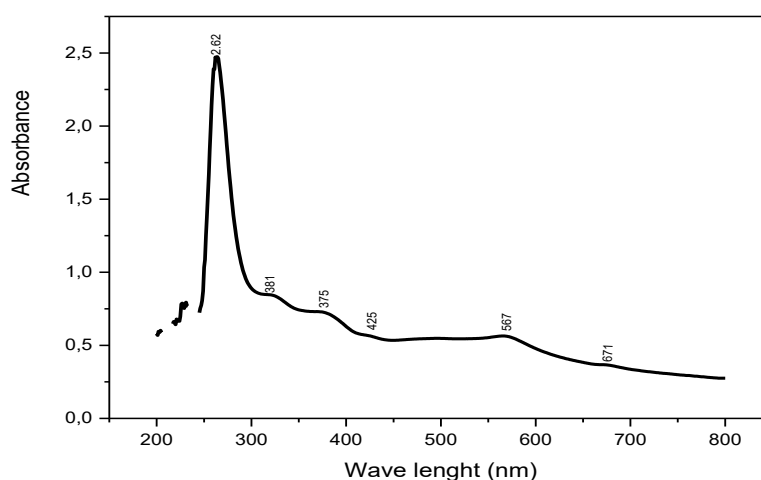

 Figure 3. Electronic absorption spectrum of  $[\text{Ni}(\text{H}_2\text{dmg})(\text{Meth})](\text{SCN})_2$ 

 Figure 4. Electronic absorption spectrum of  $[\text{Ni}(\text{H}_2\text{dmg})(\text{Ala})(\text{I})_2]$

Table 5. Electronic spectral data of the complexes.

| Complex   | $\lambda$ (nm) | $\nu$ (cm <sup>-1</sup> ) | $\epsilon$ (l mol <sup>-1</sup> cm <sup>-1</sup> ) | Electronic Transitions |
|-----------|----------------|---------------------------|----------------------------------------------------|------------------------|
| Complex 1 | 711            | 14065                     | 711                                                | d - d                  |
|           | 430            | 23256                     | 300                                                | charge transfer        |
|           | 289            | 34602                     | 343                                                | $\pi$ - $\pi^*$        |
|           | 275            | 36364                     | 2810                                               | $\pi$ - $\pi^*$        |
|           | 268            | 37313                     | 1850                                               | $\pi$ - $\pi^*$        |
| Complex 2 | 560            | 17857                     | 200                                                | d - d                  |
|           | 420            | 23809                     | 300                                                | d - d                  |
|           | 376            | 26596                     | 400                                                | charge transfer        |
|           | 323            | 30960                     | 500                                                | $\pi$ - $\pi^*$        |
|           | 263            | 38023                     | 2300                                               | $\pi$ - $\pi^*$        |
| Complex 3 | 520            | 19231                     | 1030                                               | d - d                  |
|           | 430            | 23256                     | 800                                                | charge transfer        |
|           | 316            | 31646                     | 400                                                | $\pi$ - $\pi^*$        |
|           | 296            | 33784                     | 4180                                               | $\pi$ - $\pi^*$        |
|           | 273            | 36630                     | 4100                                               | $\pi$ - $\pi^*$        |
| Complex 4 | 671            | 14903                     | 360                                                | d - d                  |
|           | 567            | 17637                     | 570                                                | d - d                  |
|           | 425            | 23529                     | 570                                                | d - d                  |
|           | 375            | 26666                     | 740                                                | charge transfer        |
|           | 321            | 31153                     | 860                                                | $\pi$ - $\pi^*$        |
|           | 264            | 37879                     | 2560                                               | $\pi$ - $\pi^*$        |

### Electrochemical Studies

The electrochemical properties of the ligands and their complexes were investigated by cyclic voltammetry in DMSO solution containing 0.1 M NaClO<sub>4</sub> as supporting electrolyte. All the measurements were carried out in 10<sup>-3</sup> M solution at room temperature in the potential range +1.6 to -1.6 V with a scan rate of 50 mVs<sup>-1</sup>. Electrochemical data of the ligands and the complexes are presented in Table 6 and Table 7, respectively. Typical cyclic voltammogram of [Ni (H<sub>2</sub>dmg)(Ala)<sub>2</sub>] and Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O are shown in Figure 5. Cyclic voltammogram of Cu(NO<sub>3</sub>)<sub>2</sub>·3H<sub>2</sub>O shows an irreversible oxidation process at +0.26 V assigned to the oxidation of Cu<sup>+</sup> to Cu<sup>2+</sup> (Bougherra et al., 2018; Tidjani-Rahmouni et al., 2014).

The cyclic voltammogram of Ni(NO<sub>3</sub>)<sub>2</sub>·6H<sub>2</sub>O shows a quasi-reversible process characterized by the potentials E<sub>pa</sub> = -1.04, E<sub>pc</sub> = -0.92 V, which can be attributed to the reduction of Ni(II) to Ni(0), furthermore, this voltammogram also shows three anodic peaks without cathodic responses. The first and the second peaks observed at E<sub>pa1</sub> = 0.05 and E<sub>pa2</sub> = 0.2 V could be attributed to the successive oxidation of Ni(0) to Ni(I) and the third peak observed at E<sub>pa3</sub> = 0.84 V can be attributed to the oxidation of Ni(I) to Ni(II) (El-Gahami et al., 2004; Ben Mansour, 2014).

By comparing the cyclic voltammograms of complexes to those of the ligands and that of metal salt taken as reference, it is easy to confirm the presence of the metal cation and ligands in the complexes. The cyclic voltammogram of the complexes 1 and 3 (complexes with copper) shows a cathodic peak, without any anodic response, observed at -0.98 V and -1.07V respectively, this peak could be assigned to the reduction of Cu(II) to Cu(0). In addition, these complexes show four peaks anodic without any cathodic response. The first peak located at 0.01 V for complex 1 and at -0.1 V for complex 3 can be attributed to the oxidation of Cu(0) to Cu(I). the second peak at 0.23 V for complex 1 and at 0.13 V for complex 3 could be assigned to the oxidation of Cu(I) to Cu(II).

The peaks anodic at -0.47 V and 1.03 V for complex 1 and at -0.6 V and 0.94 V for complex 3. All these peaks can be attributed to the oxidation of the ligands. The cyclic voltammogram of the complexes 2 and 4 (complexes with nickel) shows a cathodic peak, without any anodic response, observed at -0.66 V and -1.30 V respectively, this peak could be assigned to the reduction of Ni(II) to Ni(0)

In addition, these complexes show three peaks anodic without any cathodic response. The first peak located at -0.24 V for complex 2 and at 0.06 V for complex 4 can be attributed to the oxidation of Ni(0) to Ni(I). the second peak at 0.7 V for complex 2 and at 0.62 V for complex 4 could be assigned to the oxidation of Ni(I) to Ni(II).

However, the peaks anodic at -0.5 V for complex 2 and 1.24 V for complex 4 can be attributed to the oxidation of the ligands.

Table 6. Electrochemical data of the ligands

| Compound                                             | E <sub>pa</sub> (V) | E <sub>pc</sub> (V) | ΔE(Mv) |
|------------------------------------------------------|---------------------|---------------------|--------|
| Cu(NO <sub>3</sub> ) <sub>2</sub> .3H <sub>2</sub> O | -                   | 0.26                | -      |
|                                                      | -1.04               | -0.92               | 120    |
| Ni(NO <sub>3</sub> ) <sub>2</sub> .6H <sub>2</sub> O | 0.05                | -                   | -      |
|                                                      | 0.2                 | -                   | -      |
|                                                      | 0.84                | -                   | -      |
| Dimethylglyoxime                                     | -0.7                | -0.56               | 140    |
| Methionine                                           | -                   | 1.24                | -      |
|                                                      | -0.82               | -                   | -      |
| Alanine                                              | -0.38               | -0.66               | 280    |

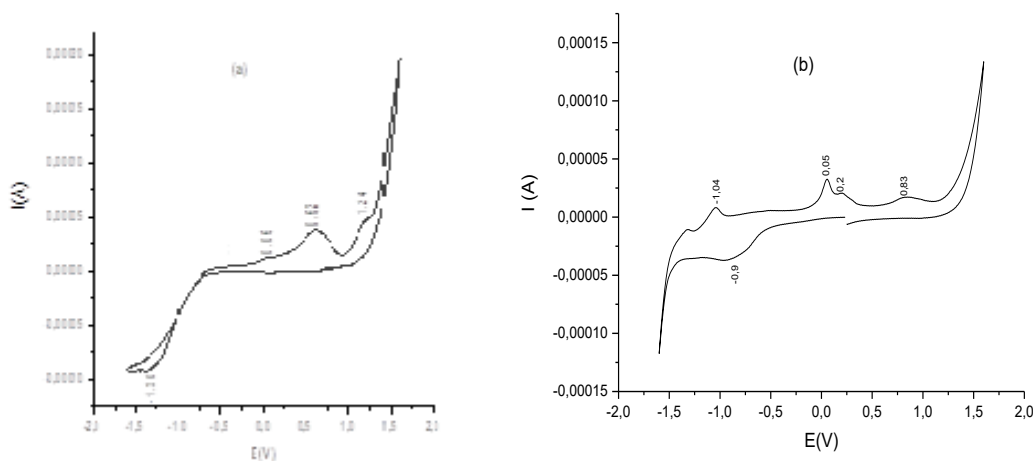

 Figure 5. (a) Cyclic voltammogram of [Ni(H<sub>2</sub>dmg)(Ala)<sub>2</sub> I<sub>2</sub>] and (b) of Ni(NO<sub>3</sub>)<sub>2</sub>.6H<sub>2</sub>O

Table 7. Electrochemical data of the complexes

| Complex   | E <sub>pa</sub> (V) | E <sub>pc</sub> (V) |
|-----------|---------------------|---------------------|
| Complex 1 | -                   | -0.98               |
|           | -0.47               | -                   |
|           | 0.01                | -                   |
|           | 0.23                | -                   |
|           | 1.03                | -                   |
| Complex 3 | -                   | -1.07               |
|           | -0.6                | -                   |
|           | -0.1                | -                   |
|           | 0.13                | -                   |
|           | 0.94                | -                   |
| Complex 2 | -                   | -0.66               |
|           | -0.50               | -                   |
|           | -0.24               | -                   |
|           | 0.70                | -                   |
|           | -                   | -1.30               |
| Complex 4 | 0.06                | -                   |
|           | 0.62                | -                   |
|           | 1.24                | -                   |

## Conclusion

Four complexes of (Cu or Ni) with dimethylglyoxime and amino acids (methionine or alanine) were synthesized and characterized by different physico-chemical and spectroscopic techniques. Conductimetric analysis shows

that two complexes are non-electrolytes. The IR study reveals that all the ligands are bidentate. The electron absorption spectra can be concluded that the investigated mixed-ligand complexes have an octahedral or square-planar geometries. The electrochemical behavior of complexes was determined by cyclic voltammetry, which shows an irreversible redox process corresponding of M(II) to M(0) and irreversible oxidation of M(0) to M(I) and M(I) to M(II).

## Scientific Ethics Declaration

\* The authors declare that the scientific ethical and legal responsibility of this article published in EPSTEM Journal belongs to the authors.

## Conflict of Interest

\* The authors declare that they have no conflicts of interest

## Acknowledgements or Notes

\* This article was presented as a poster presentation at the International Conference on Basic Sciences, Engineering and Technology ([www.icbaset.net](http://www.icbaset.net)) held in Trabzon/Türkiye on May 01-04, 2025.

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