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Full Paper

Simultaneous Electrochemical Detection of Copper and Chromium in Drinking Water using 3-Pyrazolyl-pyran-2-one Modified Carbon Paste Electrode

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Abstract- The contamination of drinking water with heavy metals such as copper (Cu^{2+}) and chromium (Cr^{3+}) poses significant health and environmental risks, necessitating the development of sensitive and selective detection methods. In this study, a carbon paste electrode (CPE) modified with 3-pyrazolyl-pyran-2-one, a heterocyclic ligand containing nitrogen and oxygen donor atoms, was developed for the simultaneous electrochemical detection of Cu^{2+} and Cr^{3+} ions in drinking water. The interaction between the ligand and metal ions was characterized by Fourier-transform infrared (FTIR) spectroscopy, providing valuable insights into the surface modifications and coordination mechanisms. Electrochemical performance was evaluated using cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS), and square wave voltammetry (SWV), revealing significant improvements in charge transfer efficiency and electrocatalytic response compared to the unmodified electrode. The sensor demonstrated high sensitivity, low detection limits, and excellent selectivity for Cu^{2+} and Cr^{3+} ions, with recovery rates ranging from 95–106% for Cu^{2+} and 92–105% for Cr^{3+}, confirming its accuracy and potential for practical applications in water quality monitoring. These results highlight that surface modification with 3-pyrazolyl-pyran-2-one

substantially enhances interfacial electron transfer, providing a promising and reliable platform for the detection of heavy metal ions in aqueous environments.

Keywords- Electrochemical sensors; Ligand-modified electrodes; Chemically modified electrodes; HMIs; Electrochemical characterization

1. INTRODUCTION

Heavy metals such as copper and chromium are among the most hazardous pollutants threatening environmental and public health [1,2]. Unlike organic pollutants, heavy metal ions (HMIs) are non-biodegradable and tend to bioaccumulate in living organisms, leading to long-term toxic effects. Exposure to Cu(II) and Cr(III), even at trace levels, can result in serious damage to the nervous system, liver, kidneys, and other vital organs [3,4]. Given their persistence and toxicity, it is essential to develop fast, accurate, and cost-effective analytical tools for their monitoring, particularly in drinking water sources .

Although traditional analytical techniques such as atomic absorption spectrometry (AAS) [5], inductively coupled plasma mass spectrometry (ICP-MS) [6], X-ray fluorescence (XRF) [7], and colorimetry [8] offer high sensitivity and selectivity, their dependence on sophisticated instrumentation, high operational costs, and labor-intensive protocols restricts their widespread use in routine analysis .

In contrast, electrochemical methods [9,10] have emerged as powerful alternatives owing to their simplicity, low cost, rapid response, and capability to detect multiple species simultaneously with good sensitivity [11,12]. One of the most promising strategies to enhance electrochemical detection involves modifying the surface of working electrodes. Among various electrode materials, carbon paste electrodes (CPEs) have garnered significant attention due to their tunable surface, good electrical conductivity, chemical inertness, and ease of modification [13]. Notable examples include studies by Junrong Chen [14], Zul Arham [15] and Zichun Yang [16]. Incorporating organic molecules with nitrogen and oxygen donor atoms into the CPE matrix is particularly effective for forming stable coordination complexes with metal ions. This approach can significantly enhance the selectivity and sensitivity of sensors for detecting specific analytes .

In this context, we introduce, for the first time, the modification of a carbon paste electrode with 3-pyrazolyl-pyran-2-one. The selection of this compound as a modifying agent is motivated by its structural features, particularly the presence of adjacent nitrogen and oxygen donor atoms [17], that can serve as bidentate coordination sites. In coordination chemistry, such ligands are known to form stable complexes with transition metal ions such as Cu(II) and Cr(III), often producing distinct electrochemical signatures due to ligand field effects and enhanced electron transfer properties. These electron-rich sites significantly strengthen the interaction with heavy metals, providing a promising platform for sensitive and selective detection. While various organic ligands have been explored for electrochemical sensing, 3-

pyrazolyl-pyran-2-one remains largely unexplored in this context, underscoring the originality of our approach.

In this work, we present the fabrication and application of a modified CPE for the simultaneous detection of Cu(II) and Cr(III) in tap water, a unique feature not yet reported. The system's performance is evaluated through cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS), and square wave voltammetry (SWV), while the electrode surface is characterized using scanning electron microscopy (SEM) and Fourier-transform infrared spectroscopy (FT-IR).

2. EXPERIMENTAL SECTION

2.1. Reagents

Each chemical used was of analytical quality and required no further purification before use. Potassium chloride (KCl), Copper sulfate ($CuSO_4$), Chromium (III) chloride hexahydrate ($CrCl_3$, $6H_2O$), and graphite were obtained from Sigma Aldrich.

2.2. Instruments

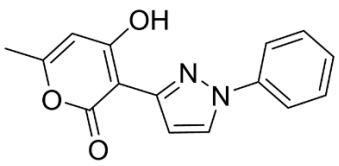
All electrochemical Experiments of this study were performed using a potentiostat OrigaStat 100, equipped with Origamaster5 software. The EIS, CV, and SWV measurements were performed with a 3-electrode system, including CPE and Mol/CPE as working electrodes, a platinum as the counter electrode, and a saturated calomel electrode (SCE) as the reference electrode.

The FEG 450 system in a scanning electron microscope (SEM) was used for morphological examination.

FTIR analysis of ZnO nanoparticles was conducted using a JASCO FT/IR-4600 spectrometer equipped with an ATR accessory. Spectra were recorded over the 4000–600 cm^{-1} range at a resolution of 4 cm^{-1}, accumulating 16 scans.

2.3. 3-Pyrazolyl-pyran-2-one Synthesis

The 3-pyrazolyl-pyran-2-one (Scheme 1) used in this study was synthesized following the procedure described by Madani Ouhaddou [18], this compound was obtained with high purity and used as a modifier for the carbon paste electrode in the simultaneous electrochemical detection of Cu(II) and Cr(III) ions.



Scheme 1. Chemical structure of 3-Pyrazolyl-pyran-2-one

2.4. Preparation of CPE and MCPEs

For the modification of the carbon paste electrode (CPE), 10% of the 3-pyrazolyl-pyran-2-one molecule was mixed with graphite powder. The components were then combined with paraffin oil using a mortar and pestle to form a homogeneous paste. The paste was placed into a syringe tube with a 2 mm internal radius, and a copper wire was inserted to provide electrical contact. An unmodified CPE was prepared using the same process, but without the addition of the 3-pyrazolyl-pyran-2-one molecule.

3. RESULTS AND DISCUSSION

3.1. Surface analysis

Three samples were selected for surface analysis using Scanning Electron Microscopy (SEM) and Fourier-Transform Infrared Spectroscopy (FTIR) to investigate the incorporation of the 3-pyrazolyl-pyran-2-one molecule into graphite and its interaction with metal ions. The samples analyzed include: (A) CPE, (B) CPE modified with the 3-pyrazolyl-pyran-2-one molecule, and (C) CPE modified with the molecule and immersed for 2 hours in a solution containing metal ions. These analyses aim to provide insights into the morphological changes and the chemical interactions at the surface of the modified sensors.

The Meb analysis of the modified CPE, after a 2-hour preconcentration in a solution containing 0.6 µM Cu(II) and 0.7 µM Cr(III) (Figure 1C), shows the formation of small clusters, which are attributed to the complexes formed by the interaction of the 3-pyrazolyl-pyran-2-one molecule with the metal ions. These clusters are notably more prominent than those observed on the modified electrode without the metal ions (Figure 1B). This observation confirms that the electrode was successfully modified with the 3-pyrazolyl-pyran-2-one molecule, allowing it to effectively form complexes with the metal ions on its surface [19].

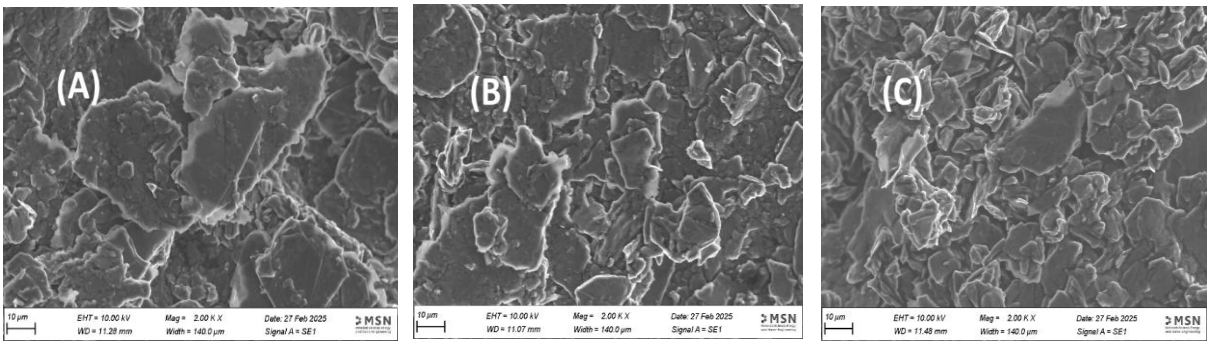


Figure 1. (A) SEM analysis of CPE (B) CPE modified with 3-pyrazolyl-pyran-2-one molecule (C) CPE modified with 3-pyrazolyl-pyran-2-one molecule and immersed for 2 hours in a solution containing Cu(II) and Cr(III)

The FTIR spectrum of the CPE sample (Figure 2A) exhibits a relatively flat baseline, a broad absorption band around 3460 cm^{-1}, which corresponds to O–H stretching vibrations from adsorbed moisture or surface hydroxyl groups. The strong peaks at 2920 and 2860 cm^{-1} are attributed to asymmetric and symmetric C–H stretching of aliphatic groups, suggesting the presence of surface-bound hydrocarbons. The peak near 1455 cm^{-1} arises from C–H bending vibrations, while the band at 943 cm^{-1} relates to C–O stretching mode, possibly from epoxide or hydroxyl groups.

Upon incorporation of the organic molecule into the graphite (Figure 2B), several changes confirm successful molecular integration. A strong and broad absorption band appears around 3129 cm^{-1}, corresponding to O–H and/or N–H stretching vibrations, suggesting the presence of hydroxyl and/or amine groups in the grafted molecule. The distinct peaks at 2251 cm^{-1}, 2105 cm^{-1}, and 1910 cm^{-1} are characteristic of nitrile (C≡N), azo (N=N), or imine (C=N) functional groups, confirming the presence of the pyrazole and pyran rings. The persistence of the 1683 and 1447 cm^{-1} band likely includes contributions from both C=C and C=O stretching. Vibrations around 1300–1000 cm^{-1} correspond to C–O and C–N stretching.

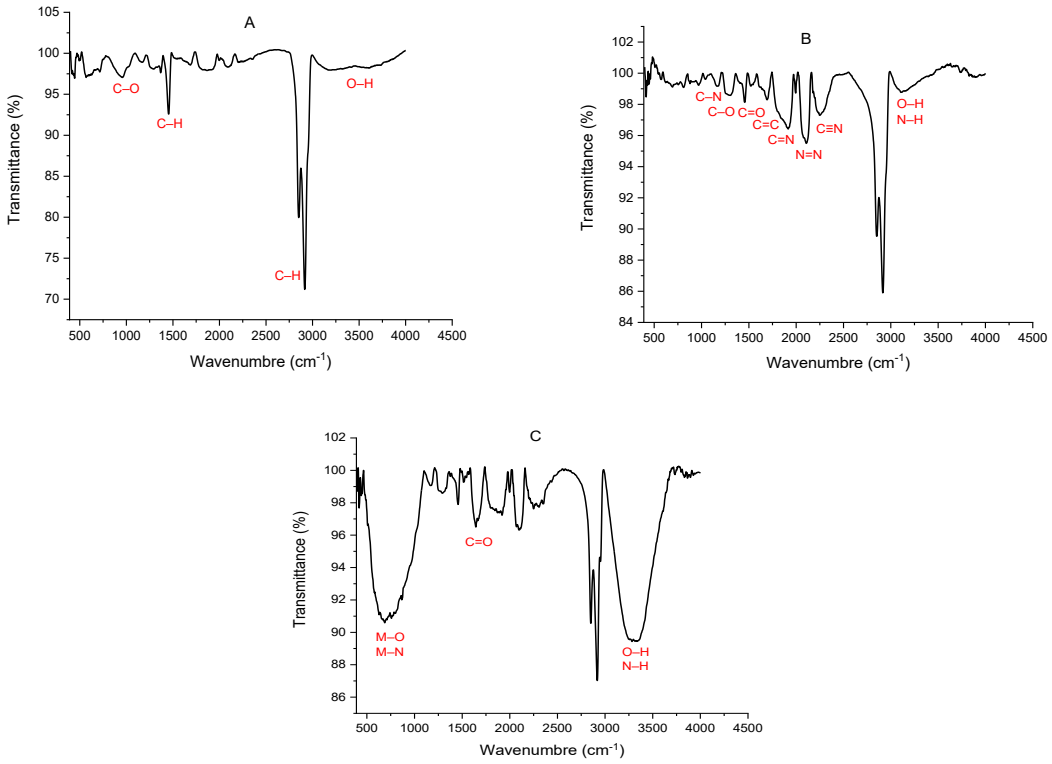


Figure 2. FT-IR analysis of (A) CPE (B) CPE modified with 3-pyrazolyl-pyran-2-one molecule (C) CPE modified with 3-pyrazolyl-pyran-2-one molecule and immersed for 2 hours in a solution containing Cu(II) and Cr(III)

The spectrum Figure 2C, representing the CPE–molecule–metal complex, shows distinct signs of metal coordination. The broad band around 700 cm^{-1}, absent in previous spectra, is

characteristic of M–O or M–N vibrational modes, suggesting direct interaction of the ions with the functional groups of the molecule. Furthermore, the broad band near 3129 cm^{-1} becomes even more intense and widened, indicating potential hydrogen bonding or metal coordination involving O–H/N–H groups [20-23].

3.2. Electrochemical attitude of Cu^{2+} and Cr^{3+} on CPE and Mol /CPE

3.2.1. CV

The interaction between Cu^{2+} and Cr^{3+} ions with the 3-Pyrazolyl-pyran-2-one molecule in a 0.1 M KCl solution (pH 3.0) was investigated using cyclic voltammetry (CV). Figure 3 displays the cyclic voltammograms of both the unmodified CPE and Mol/CPE electrodes in the presence of 0.6 µM Cu^{2+} and 0.7 µM Cr^{3+} at a scan rate of 50 mV/s, within a potential range of -1.5 to +1.5 V. The voltammograms reveal a small oxidation current at the unmodified CPE electrode, whereas the Mol/CPE electrode exhibits pronounced oxidation peaks for both Cu(II) and Cr(III). These results suggest that the donor atoms of the 3-pyrazolyl-pyran-2-one molecule enhance the electrode's conductivity and electrochemical performance, promoting better adsorption of Cu^{2+} and Cr^{3+} ions onto the electrode surface [24]. Therefore, this modified electrode shows great potential as an effective sensor for detecting copper and chromium ions.

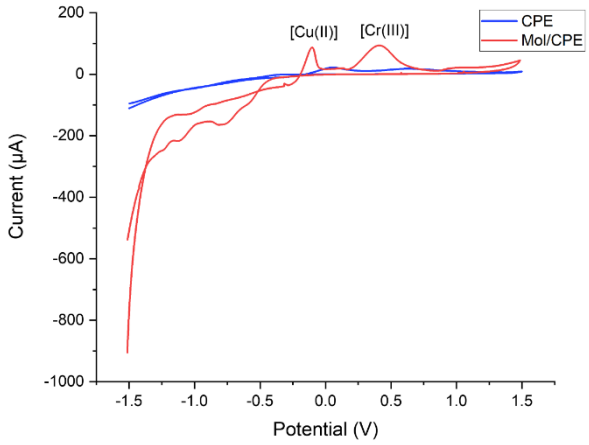


Figure 3. CV of CPE and Mol /CPE in the presence of Cu(II) and Cr(III) in a 0.1 M KCl

3.2.2. EIS

The reaction of Cu^{2+} and Cr^{3+} at the surface of the Mol /CPE electrode was also confirmed by EIS. Figure 4 shows that the curve for the modified electrode is notably smaller compared to the bare CPE, suggesting an enhancement in conductivity due to the surface modification with the 3-pyrazolyl-pyran-2-one molecule, which is consistent with the findings from CV. Additionally, the Nyquist plots display a semi-circular form indicating that the electrochemical reaction of metal ions on the electrode surface is a charge transfer-limited process [25].

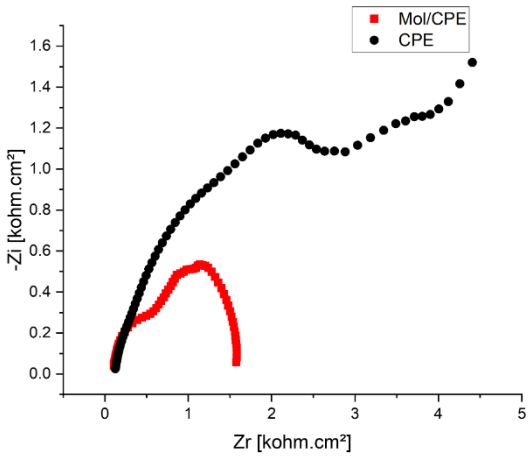


Figure 4. Nyquist plots of CPE and and Mol /CPE in the presence of Cu(II) and Cr(III) in a 0.1 M KCl solution (pH 3.0)

3.3. Optimization of experimental conditions

3.3.1. Scan rate effect

The effect of scan rate on the oxidation peak current (I_{pa}) was evaluated in the range of 20 to 60 mV/s to study the reaction kinetics of metal ions at the surface of the 3-pyrazolyl-pyran-2-one-modified electrode.

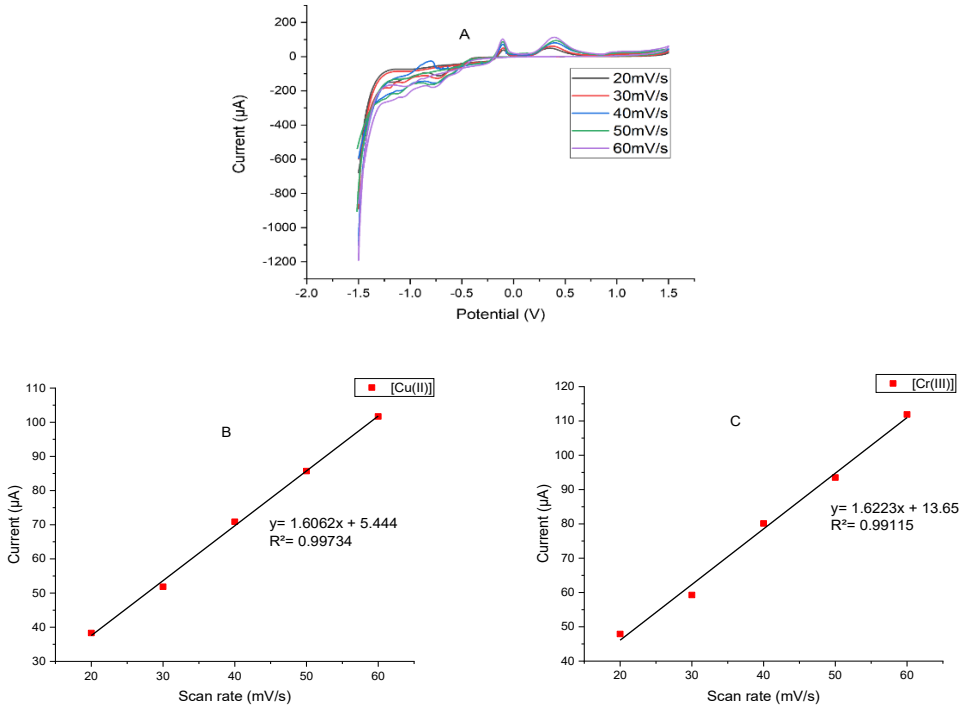


Figure 5. (A) Mol/CPE C. Voltammograms in 0.1 M KCl solution (pH= 3.0) with 0.6 µM of Cu(II) and 0.7 µM of Cr(III) at various SR: 20, 30, 40, 50, 60 mVs^{-1}. (B) Plot of I_{pa} (Cu(II)) *vs* scan rate. (B) Plot of I_{pa} (Cr(III)) *vs* scan rate

As shown in Figure 5-A, the oxidation peak currents for both Cu(II) and Cr(III) increased linearly with the scan rate. The corresponding linear regression equations are presented in Figure 5-B and Figure 5-C: I_{pa}(Cu(II)) = 1.6062x + 5.444 (R^2 = 0.99734) and I_{pa}(Cr(III)) = 1.6223x + 13.65 (R^2 = 0.99115), respectively. These strong correlations indicate that the oxidation processes of both copper and chromium are controlled by an adsorption mechanism at the modified electrode surface [26]. This behavior is in good agreement with the results obtained from Electrochemical Impedance Spectroscopy (EIS).

3.3.2. pH effect

The influence of pH on the electrode response was investigated over a pH range from 2 to 5. As shown in Figure 6, the peak currents for both Cu(II) and Cr(III) initially increased from pH 2 to 3. However, a reduction in current was observed at pH values above 3. This decline is attributed to the hydrolysis of metal ions, which becomes more pronounced at higher pH values [27]. The maximum peak current was recorded at pH 3, identifying it as the optimal condition for the simultaneous identification of Cu^{2+} and Cr^{3+}.

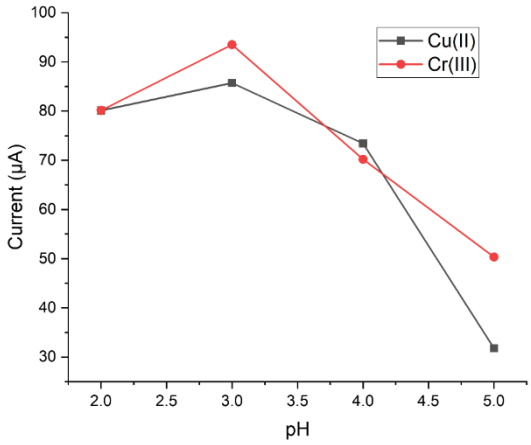


Figure 6. Influence of pH on the peak current of Cu(II) and Cr(III) at Mol/CPE in a 0.1 M KCl solution

3.3.3. Simultaneous Electrochemical Detection of Cu(II) and Cr(III)

The SWV recorded at various concentrations of Cu(II) and Cr(III) (Figure 7-A) displays well-defined peaks for each ion, with the peak currents appearing near −0.1 V for Cu^{2+} and around 0.5 V for Cr^{3+}. Importantly, the peak current intensities increase proportionally with increasing metal ion concentrations, demonstrating a linear correlation (R^2 = 0.99), as illustrated in Figures 7-B and 7-C. The detection limit (LOD=3Sb/S) and quantification limit (LOQ=10Sb/S) were calculated in order to verify the sensitivity of the sensor. Sb is the seven-measurement standard deviation, and S is the calibration plot's slope [28]. The results are presented in Table 1. From these findings, it can be concluded that the Mol/CPE sensor has been effectively assessed for the simultaneous detection of Cu^{2+} and Cr^{3+}.

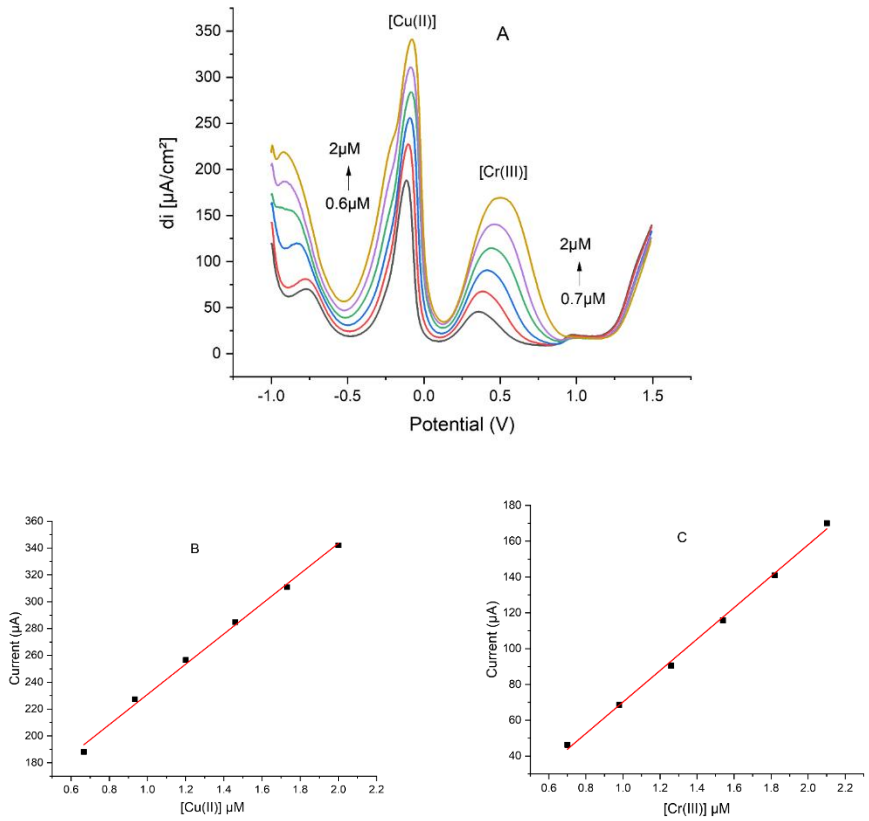


Figure 7. (A) SWV of Mol/CPE for the identification of Cu(II) and Cr(III) in 0.1M KCl solution. (B) Calibration graph corresponding to the oxidation peaks of Cu (II) obtained from different concentrations. (C) Calibration graph corresponding to the oxidation peaks of Cr (III) obtained from different concentrations

Table 1. LOD and LOQ results for Cu(II) and Cr(III) on Mol/CPE

Ion	LOQ µM	LOD µM
Cu(II)	0.43	0.13
Cr(III)	0.36	0.10

The Mol/CPE displays remarkable performance, highlighting its suitability for analytical applications and their competitiveness compared to other sensors presented in Table 2.

3.3.4. Real sample

Figure 8-A presents the square wave voltammograms (SWV) of Mol/CPE at different concentrations of Cu(II) and Cr(III) in drinking water. To determine the concentrations of Cu(II) and Cr(III) in drinking water samples, the regression equations obtained from the SWV data in Figures 8-B and 8-C were used. The results, shown in Tables 3 and 4, demonstrate a strong correlation between the calculated and added concentrations, thereby confirming the

reliability and accuracy of the Mol/CPE electrode for the simultaneous detection of Cu(II) and Cr(III) in drinking water [36].

Table 2. A comparative assessment of analytical performance for the detection of Cu(II) and Cr(III)

Ion	Electrodes	LOD	Reference
Cu^{2+}	Cu^{2+}-IIPs/GCE	5.99 µM	[29]
	PGE/PPY/SSA-Cu	5.42 µM	[30]
	ZnO@rGO	14.9 µM	[31]
	FP-MCPE	0.8 µM	[32]
	This work	0.13 µM	
Cr^{3+}	ZnO@GO	7.05Mm	[31]
	ICP-OES	0.07 mg/L	[33]
	γ-PGA-Au NPs	100 ppb	[34]
	4-MBA-Au@Ag NPs	9 Mm	[35]
	This work	0.10 µM	

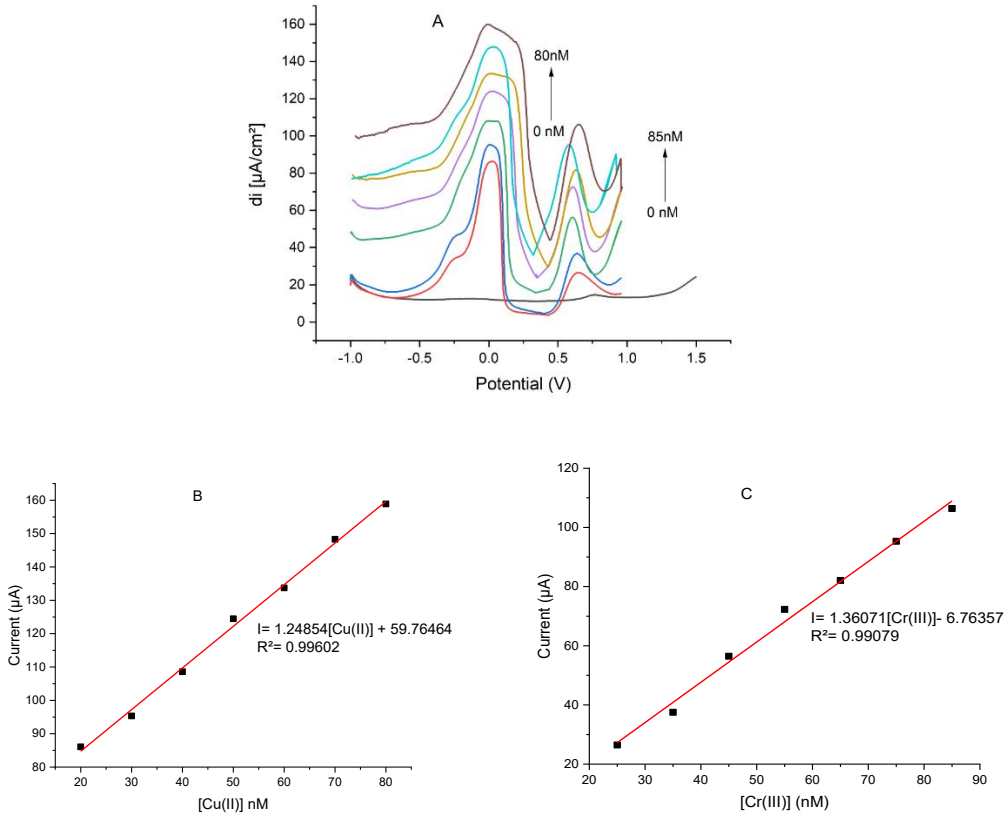


Figure 8. (A) SWV response of the molecule in drinking water at varying Cu(II) and Cr(III) concentrations (B) Calibration curve displaying the peak current of different drinking water Cu(II) concentrations. (C) Calibration curve displaying the peak current of different drinking water Cr(III) concentrations

Table 3. Determination of Cu(II) concentration in drinking water using Mol/CPE electrode

	Added (nM)	Found (nM)	Recovery (%)
1	0	Not detected	
2	20	21.25	106
3	30	28.67	95
4	40	39.28	98
5	50	52	104
6	60	59.43	99
7	70	71.10	101
8	80	79.58	99

Table 4. Determination of Cr(III) concentration in drinking water using Mol/CPE electrode

	Added (nM)	Found (nM)	Recovery (%)
1	0	Not detected	
2	25	24.37	97
3	35	32.50	92
4	45	46.41	103
5	55	58.05	105
6	65	65	100
7	75	75	100
8	80	83	100

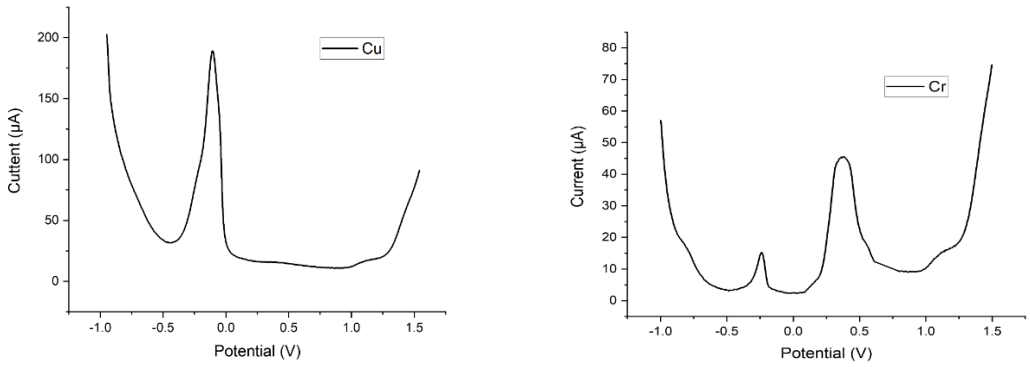


Figure 9. Individual detection of Cu(II) and Cr(III)

3.3.5. Selectivity

A selectivity study was conducted by individually detecting 0.6 μM of Cu(II) and 0.7 μM of Cr(III) (Figure 9). The results showed that the current peaks obtained for each metal in the individual samples were almost identical to those observed during the simultaneous detection,

indicating the electrode's selectivity for the simultaneous detection of copper and chromium [37].

3.3.6. Stability

After three months of storage at room temperature, the stability of the enhanced sensor was evaluated for 0.6 µM Cu(II) and 0.7 µM Cr(III). As shown in Figure 10; the electrode retained 98% and 99% of its original response for copper and chromium, respectively, indicating the superior stability of the electrode [38].

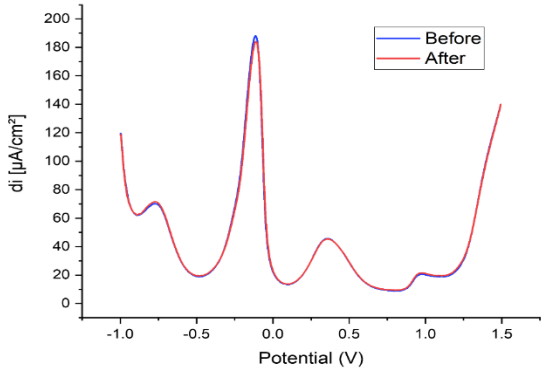


Figure 10. Mol/CPE stability for Cu(II) and Cr(III)

4. CONCLUSION

In this study, the organic molecule 3-Pyrazolyl-pyran-2-one was employed for the simultaneous electrochemical detection of Cu(II) and Cr(III) in drinking water. The incorporation of the molecule into a carbon paste electrode (CPE) and its interaction with Cu^{2+} and Cr^{3+} ions were confirmed through infrared spectroscopy (IR). Electrochemical characterization using cyclic voltammetry (CV), electrochemical impedance spectroscopy (EIS), and square wave voltammetry (SWV) demonstrated a significant enhancement in charge transfer efficiency and electrocatalytic performance compared to the unmodified electrode. The modified electrode exhibited high sensitivity, excellent selectivity, low detection limits, and ease of fabrication, making it a promising sensor for the simultaneous detection of Cu^{2+} and Cr^{3+} ions in complex matrices such as drinking water.

Declarations of interest

The authors declare no conflict of interest in this reported work.

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