

RESEARCH PAPER

## Electrooxidation of Phenylhydrazine on Nanostructured Co-Mo Alloy Electrodes in Alkaline Medium

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### ARTICLE INFO

#### Article History:

Received 10 January 2024

Accepted 12 March 2024

Published 15 April 2024

#### Keywords:

Electrocatalytic properties

Electrochemical impedance spectroscopy

Electronic effect

Nanostructured Co-Mo alloys

Phenylhydrazine

electrooxidation

### ABSTRACT

In this work, we investigated the electrocatalytic activity of electrodeposited cobalt and CoMo alloy electrodes towards the oxidation of phenylhydrazine in 1 M sodium hydroxide aqueous solution. A previously proposed nontoxic tartrate electrolyte was employed to electrodeposit alloys. Electrochemical methods such as cyclic voltammetry (CV), chronoamperometry (CA), and electrochemical impedance spectroscopy (EIS) were used to study the electrooxidation of phenylhydrazine. When compared to pure cobalt electrodes, the voltammetric data for cobalt-molybdenum alloy electrodes indicated a lower peak potential and a higher peak current density. According to the EIS results, the polarization resistance of the Co-Mo alloy electrodes was much lower compared to the pure Co in 0.1 M phenylhydrazine basic solution. The CA results demonstrated that Co-Mo electrodes had greater stability than cobalt electrode. The Co-25 at % Mo and Co-33 at % Mo electrodes had higher catalytic activity among other synthesized electrodes for phenylhydrazine oxidation in an alkaline medium, the former being the best electrocatalysts for the phenylhydrazine electrooxidation.

### How to cite this article

Abdolmaleki M. Electrooxidation of Phenylhydrazine on Nanostructured Co-Mo Alloy Electrodes in Alkaline Medium. *Nanochem Res*, 2024; 9(2):138-145. DOI: 10.22036/ncr.2024.02.005

### INTRODUCTION

Phenylhydrazine is used for heat stabilizers, agricultural chemicals, explosives, dyes, textiles, and pharmaceuticals. Similar to hydrazine, it is a strong reducing agent for many transition metals [1, 2]. Therefore, it can enter the environment mainly through its construction process or its use in the chemical industry. Like hydrazine, it is toxic and carcinogenic and can cause serious damage to the liver, kidneys, and central nervous system in humans [1, 3]. Therefore, reliable and sensitive analytical methods are needed to determine this substance in different samples. Several methods for determining phenylhydrazine have been reported in the literature [4, 5]. These techniques, which include chemiluminescence [8], chromatography [9-11], and spectrophotometry [6, 7], are highly time-consuming, expensive, and demand pretreatment procedures. Due to their high sensitivity, increased

simplicity, dependable selectivity, quick response time, and low cost, electroanalytical techniques appear to be the most popular method [12-14]. For the oxidation of phenylhydrazine, graphite electrodes, bare glassy carbon, and carbon paste electrodes have all been widely employed as electrode materials [15-18]. Additionally, since phenylhydrazine oxidation is kinetically slow, the aforementioned electrodes require a high overpotential. [19]. Therefore, to overcome this problem, modified electrodes are required. To this end, transition metal complexes and noble metals have been utilized as electrocatalysts for phenylhydrazine oxidation [20]. However, the main disadvantage associated with the use of these materials is its high cost. Consequently, there is a growing interest in studying and synthesizing non-noble and low-cost metals and their alloys for the oxidation of phenylhydrazine [21].

Fuel cells can employ phenylhydrazine, such

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as hydrazine, as an ideal fuel because it does not exhaust environmentally harmful substances like  $\text{CO}_2$  and its oxidation does not poison catalysts [22, 23]. In the previous work, hydrazine oxidation was completely performed on cobalt-molybdenum alloy electrodes [24]. In this context, for the first time, the electrooxidation of phenylhydrazine in an alkaline medium was investigated by various electrochemical techniques on these electrodes and compared with the pure cobalt electrode. The novelty of this work lies in the simplicity of the preparation method of the Co-Mo electrodes, its low cost, and low overvoltage of phenylhydrazine oxidation on them compared to the electrodes

discussed above.

## EXPERIMENTAL

Using reagent-grade chemicals and triple-distilled water, all solutions were prepared. Electrodeposition was applied to create electroactive Co-Mo coatings on a Cu substrate. In this work, a tartrate plating bath created in our earlier studies was applied [24].

Electrochemical experiments of the cyclic voltammetry (CV), chronoamperometry (CA), as well as electrochemical impedance spectroscopy (EIS) were performed in the 1 M NaOH + 0.1 M phenylhydrazine solution at 25 °C by the use of

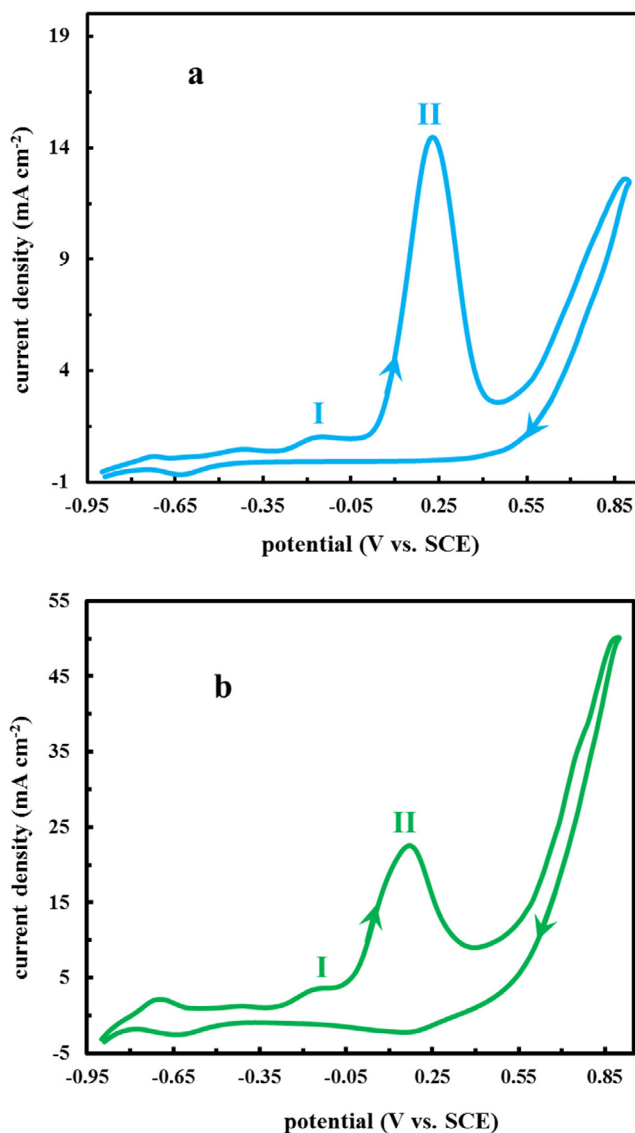
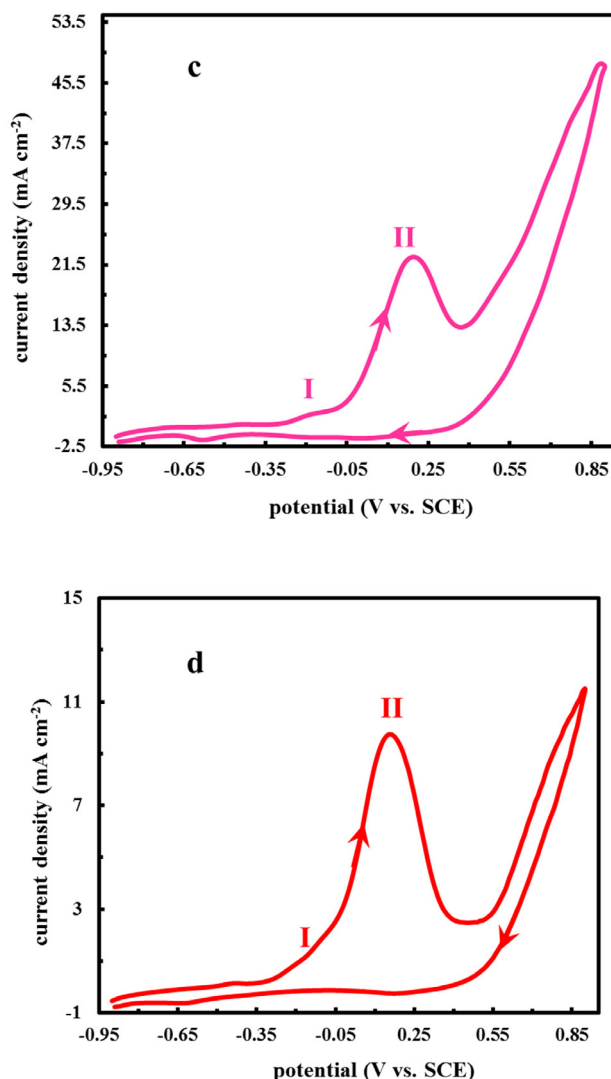


Fig. 1. CVs of 0.1 M phenylhydrazine on pure cobalt electrodes (a), CoMo-1 electrode (b), CoMo-2 electrode (c), and CoMo-3 electrode (d) in a 1 M NaOH solution and at a scan rate of  $20 \text{ mV s}^{-1}$  and  $T=25 \text{ }^\circ\text{C}$ .



Continued Fig. 1. CVs of 0.1 M phenylhydrazine on pure cobalt electrodes (a), CoMo-1 electrode (b), CoMo-2 electrode (c), and CoMo-3 electrode (d) in a 1 M NaOH solution and at a scan rate of  $20 \text{ mV s}^{-1}$  and  $T=25 \text{ }^\circ\text{C}$ .

a vertex potentiostat/galvanostat (Ivium model) outfitted with IviumSoft software.

A platinum sheet ( $20 \text{ cm}^2$ ) was employed as the counter electrode, and a saturated calomel electrode (SCE) served as the reference electrode. The working electrodes were Co and Co-Mo alloy coatings, each with a geometric area of  $1 \text{ cm}^2$ . The CV test was performed within the potential region of  $-0.95$  to  $+0.85 \text{ V}$  at a scan rate of  $20 \text{ mV s}^{-1}$ . The EIS data ( $100 \text{ kHz}$ - $0.01 \text{ Hz}$ ) were both evaluated and fitted by employing ZView (II) software.

## RESULTS AND DISCUSSION

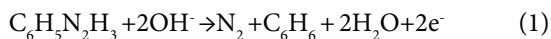
### Cyclic voltammetry

The surface morphology and structure

of cobalt-molybdenum alloy coatings were thoroughly investigated in the previous work [24]. Fig. 1 illustrates curves (a)-(d), the typical cyclic voltammetric (CV) responses of pure Co, CoMo-1, CoMo-2, and CoMo-3 catalysts in 1 M NaOH + 0.1 M phenylhydrazine solution, respectively, at a scan rate of  $20 \text{ mV s}^{-1}$ .

The peak (I), which was mentioned in the earlier work, may be attributed to the conversion of  $\text{Co}(\text{OH})_2$  and  $\text{CoOOH}$  in an alkaline medium [24-28]. When phenylhydrazine is added, a second anodic peak (II) at a greater positive potential is observed, indicating that the electrooxidation of phenylhydrazine occurs following a total oxidation of  $\text{Co}(\text{OH})_2$  to  $\text{CoOOH}$  [24-28].

In general, the phenylhydrazine electrooxidation in alkaline media can occur through a two-electron process represented by the following equation [29]:



The results show that phenylhydrazine electrooxidation occurs only in the anodic scan [30]. It is quite clear that the onset potential of phenylhydrazine oxidation on cobalt-molybdenum alloy electrodes is less than that of pure cobalt. The change of the electron density in d-orbitals and the synergistic effect of interaction between Co and Mo are two reasons for this phenomenon [31, 32]. Additionally, except for the CoMo-3 electrode, the anodic peak current density on these alloy electrodes is much higher compared to pure cobalt. The rise in surface area and surface roughness is attributed to this high current density [33-35].

It can be seen from Fig. 2 that intrinsic electro-catalytic activity and the current density of phenylhydrazine oxidation on the CoMo-1 electrode are higher than those of the other electrodes. In addition, by comparing the CV curves of phenylhydrazine oxidation with hydrazine (previous work [24]), it is evident that the overvoltage of phenylhydrazine oxidation on Co-Mo alloy electrodes is lower than that of hydrazine.

Therefore, these catalysts can be proposed as anode electrodes in the direct phenylhydrazine fuel cells.

#### EIS test

Fig. 3 displays the Nyquist diagram and equivalent electrical circuit for phenylhydrazine electrooxidation at the synthesized electrode at 0.1 V vs. SCE. In the Nyquist diagram, the semicircle at low frequencies is always followed by the semicircle at high frequencies. In Nyquist plots with this feature, porous electrodes are frequently discussed [36-38]. The standard version with two in-phase elements (CPEs),  $R_s$ ,  $R_p$ , and  $R_{ct}$ , respectively, stand in for the solution, pore, and charge transfer resistances. The symbols representing the electrode/solution interface (CPEp) and porous electrode (CPEdl), respectively, are constant phase elements (CPEs).

Table 1 displays the roughness factor ( $R_f$ ) values for the synthesized electrodes and equivalent-circuit characteristics derived from fitting the experimental results.

These values can be calculated by the use of the relation  $R_f = A_{\text{real}}/A_{\text{geometric}}$ . The real surface area for the electrodes is  $A_{\text{real}} = (\text{average double-layer capacitance } (C_{\text{dl}}) \text{ of test electrode}) / (C_{\text{dl}} \text{ of a smooth metal surface}) = 20 \mu\text{F cm}^{-2}$  [39]. The capacitance parameter  $T_{\text{dl}}$  of synthesized electrodes is related to

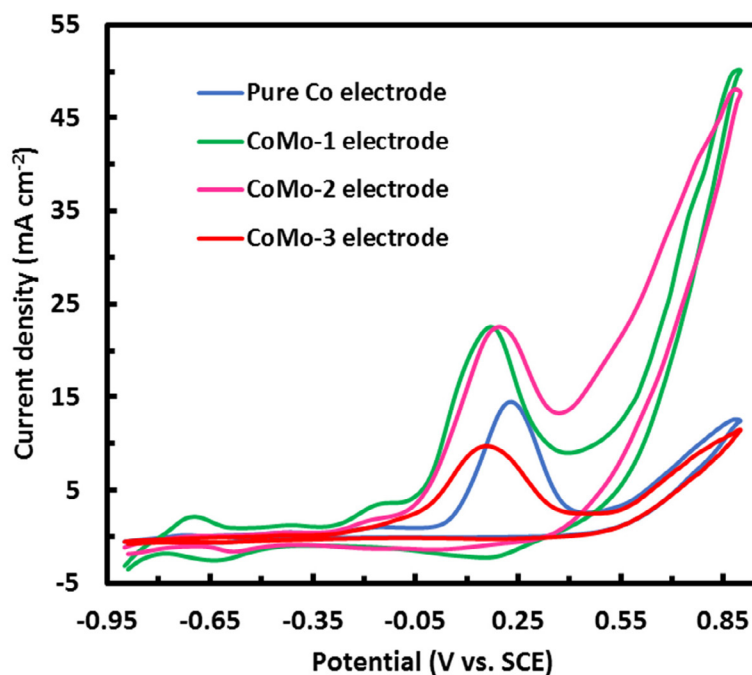


Fig. 2. Comparison of cyclic voltammograms of synthesized electrodes in 1 M NaOH+ 0.1 M phenylhydrazine aqueous solutions at a sweep rate of 20 mV s<sup>-1</sup> and T=25 °C.

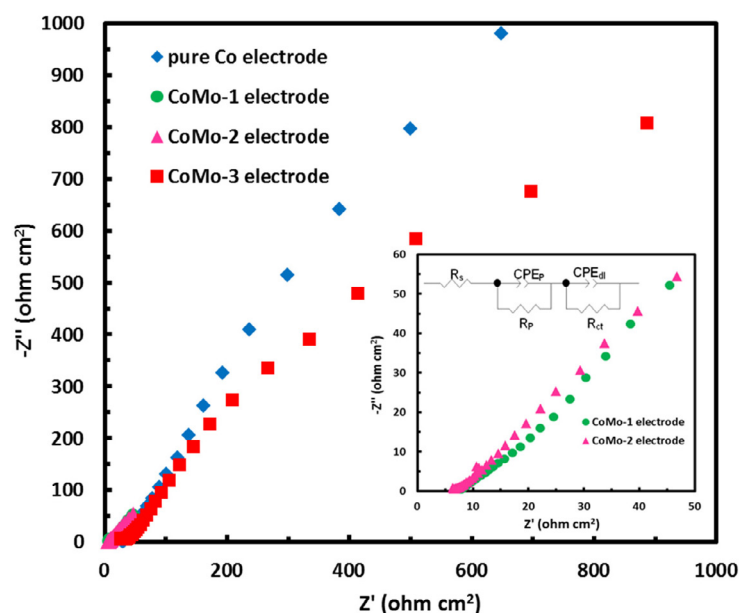


Fig. 3. Experimental Nyquist plots and equivalent electrical circuits for phenylhydrazine electrooxidation on bare Co and Co-Mo alloy electrodes in 1 M NaOH + 0.1 M phenylhydrazine aqueous solution at a potential of 0.1 V vs. SCE and T=25 °C.

Table 1. Equivalent circuit characteristics were determined from Fig. 3 for the electrooxidation of 0.1 M phenylhydrazine on various Co-Mo alloy electrodes in a 1 M sodium hydroxide solution.

Element	Co	CoMo-1	CoMo-2	CoMo-3
$R_s / \text{ohm cm}^2$	40	2.207	5	30
$T_p / \text{ohm}^{-1} \text{cm}^2 \text{s}^\Phi$	0.0049018	0.03617	0.030861	0.0013618
$R_p / \text{ohm cm}^2$	34.33	5.18	9.716	26.83
$\Phi_p$	0.94	0.44	0.57	0.63
$T_{dl} / \text{ohm}^{-1} \text{cm}^2 \text{s}^\Phi$	0.0015506	0.0032383	0.0028617	0.0012332
$R_{ct} / \text{ohm cm}^2$	3427	459.90	1180	2466
$\Phi_{dl}$	0.91	0.80	0.83	0.85
$C_{dl} / \mu\text{F cm}^2$	1249.62	1268.48	1492.18	768.70
$A_{\text{real}} / \text{cm}^2$	62.48	63.42	74.61	38.43
$R_f$	62.48	88.78	74.61	38.43
Error (%)	2.29	3.71	3.88	2.79

the  $C_{dl}$  by the following relation [40]:

$$C_{dl} = \{T_{dl} / [(R_s + R_p)^{-1} + R_{ct}^{-1}]^{(1-\Phi)}\}^{1/\Phi} \quad (2)$$

The phase shift, which is regarded as a measure of surface homogeneity in equation (1), is denoted by the symbol  $\Phi$ . When compared to cobalt electrodes, Co-Mo electrodes have the lowest values for charge transfer resistance ( $R_{ct}$ ) and CPE- $\Phi$ . The nanostructured CoMo-1 electrode thus displayed the lowest  $R_{ct}$ , 459.90-ohm  $\text{cm}^2$ . This indicates that among all synthesized electrodes, the Co-25at.%Mo coating has the maximum activity towards

phenylhydrazine oxidation. EIS measurements are consistent with the results of CVs.

The anodic current density of the CoMo-1 and CoMo-2 electrodes in 1 M NaOH + 0.1 M phenylhydrazine is two times greater than that of the pure Co electrode, according to the CVs shown in Figs. 1 and 2. However, based on the findings of the EIS, the true surface area ratio of the CoMo-1 and CoMo-2 electrodes to the Co electrode is estimated to be 1.02 and 1.19, respectively. Therefore, it can be claimed that a major part of the enhanced activity of these catalysts for phenylhydrazine electrooxidation is due to the improvement in the

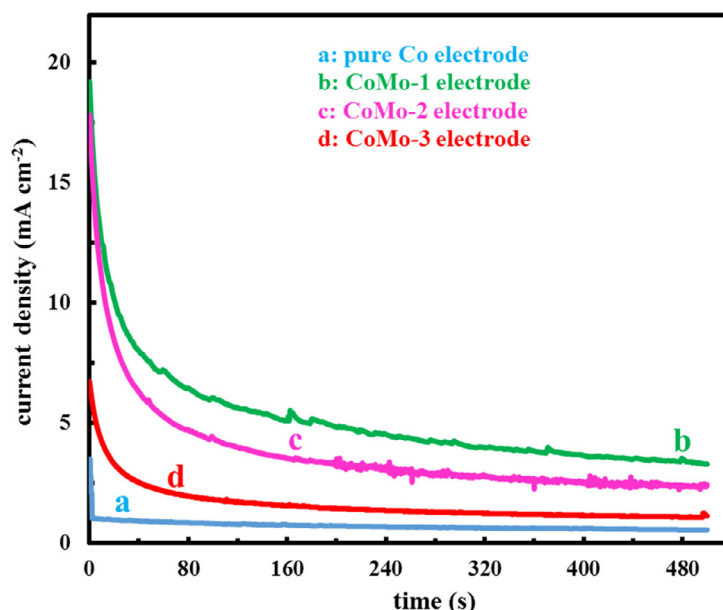


Fig. 4. Chronoamperograms of the synthesized electrodes in 1 M NaOH+ 0.1 M phenylhydrazine aqueous solutions at a potential 0.1 V vs. SCE and T=25 °C.

electronic properties of the catalysts.

#### Chronoamperometry test

Fig. 4 displays the chronoamperometric curves of the electrodes recorded at 0.1 V vs. SCE in 1 M NaOH + 0.1 M phenylhydrazine. Over time, the current resulting from phenylhydrazine electrooxidation decreases slightly and remains constant. The current drop for Co-Mo alloy electrodes is lower than that observed for the pure cobalt electrode, which indicates a decrease in adsorbed species and less poisoning of these electrodes. Furthermore, it can be seen that the current densities of Co-Mo electrodes were higher compared to the pure cobalt electrode; thus, the CoMo-1 electrode has higher activity and represents a promising candidate for the electrocatalytic oxidation of phenylhydrazine in an alkaline medium. These results support the previously described CVs and EIS measurements.

#### CONCLUSIONS

In the current work, cobalt-molybdenum nanostructured alloy electrodes were used for the oxidation of phenylhydrazine in an alkaline medium. The results of the CV test indicated less peak onset potential of phenylhydrazine oxidation for Co-Mo alloy electrodes compared to the pure cobalt. Moreover, except for the CoMo-3

electrode, the current density of the oxidation peak of phenylhydrazine on other alloy electrodes is higher than that of the pure cobalt. Further, the results of chronoamperometry demonstrated that the Co-Mo alloy electrodes have higher stability and more catalytic activity for the oxidation of phenylhydrazine in an alkaline medium than pure cobalt. The EIS measurements confirmed that alloying Co with Mo results in the decreasing of the  $R_{ct}$  values. The results of electrochemical impedance spectroscopy measurement are also in good agreement with the results of cyclic voltammetry and chronoamperometry.

#### ACKNOWLEDGMENTS

The authors would like to acknowledge the financial support of the Office of Vice-Chancellor in the Charge of Research of Sayyed Jamaledin Asadabadi University.

#### CONFLICT OF INTEREST

The authors declare that they have no conflicts of interest.

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