RESEARCH PAPER

A Sensitive Method for Electrochemical Determination of Molybdenum (VI) in Plant Foodstuff Samples Using $Ni_{0.5}Zn_{0.5}Fe_2O_4$ Nanocomposite Modified Carbon Paste Electrode

Abbas Afkhami *, Mahdi Moradi, Atousa Bahiraei and Tayyebeh Madrakian

Faculty of Chemistry, Bu-Ali Sina University, Hamedan, Iran

ARTICLE INFO

ABSTRACT

In the present study, a new chemically modified carbon paste electrode (CPE) is constructed for rapid, accurate, simple, highly sensitive, and selective determination of Mo (VI) using differential pulse voltammetry. The electrode was prepared using magnetic nickel zinc ferrite nanocomposite ($Ni_{0.5}Zn_{0.5}Fe_2O_4$), as the modifier in CPE ($Ni_{0.5}Zn_{0.5}Fe_2O_4$/ CPE). Mo (VI) was determined after preconcentration at the surface of the modified electrode at -0.7 V vs. Ag/AgCl. Under the optimal conditions, the linear dynamic range and limit of detection were 0.005-1.00 and 0.003 μgmL^{-1}, respectively. Ten successive measurements of 0.06 and 0.70 μgmL^{-1} of Mo (VI) ions showed the relative standard deviation of 3.20 and 1.98 %, respectively. The reproducibility and stability of the electrode response were also studied. Investigation of the effects of different cations and anions on the determination of Mo (VI) indicated that the electrode is highly selective. Furthermore, the present method was applied to the determination of Mo (VI) in several plant foodstuff samples with satisfactory results.

How to cite this article

INTRODUCTION

Molybdenum is an essential trace element for animals as well as plants which have an important role in enzymatic redox reactions. It is an essential constituent of enzymes catalyzing the redox reactions, e.g. oxidation of aldehydes, xanthine and other purines, and reduction of nitrates and molecular nitrogen. A deficiency of molybdenum in human diet adversely affects the growth and may lead to neurological disorders and even death. On the other hand, the high concentrations of molybdenum in the body results in severe gastrointestinal irritation and death due to cardiac failure. Excess of molybdenum may also cause teeth vulnerable to caries and disturbances in the metabolism of fats and proteins and bone deformation. Because of the low concentration of this metal in food and biological samples, the development of new methods for quantifying trace metals is required and challenged. Before performing determination of low concentrations of heavy metals in some common procedures, a pre-concentration step is needed [1,2]. Therefore, for the determination of molybdenum in various matrices a variety of analytical methods are developed. Concentrations of molybdenum are commonly measured by various techniques such as spectrophotometry [1, 3-5] and spectrofluorimetry [6], which these methods suffer from low sensitivity values. Some sensitive techniques have also been reported for the determination of molybdenum such as inductively coupled plasma mass spectrometry

* Corresponding Author Email: *afkhami@basu.ac.ir*

[7], inductively coupled plasma atomic emission spectrometry [8], neutron activation analysis [9], flame atomic absorption spectrometry [10], graphite furnace atomic absorption spectrometry [11] and electrothermal atomic absorption spectrometry [12]. However, these methods, in addition to need to the preconcentration and/or separation steps, these techniques are very specialized, bulky, and expensive, making their use for on-site (field) measurements difficult. Most of the sensitive and selective methods recently available such as ICP–AES, ICP–MS, GF–AAS and NAA are too expensive to be used in routine analysis.

Electrochemical methods have several advantages such as far less expensive, more portable, and quick response time than the other techniques [2,13]. Unique properties of this method, such as combining excellent sensitivity, selectivity, precision, and accuracy with low cost of instrumentation and maintenance, have made this technique useful for the trace Mo detection.

One of the most popular electrode materials that has obtained increasing attention among these mercury-free solid electrodes and used for the laboratory preparation of various electrodes, sensors, and detectors over the past five decades, is carbon paste, i.e. a mixture of carbon (graphite) powder and a binder (pasting liquid), that has the advantages of easy preparation, low cost, porous surface, wide potential range (from -1.40 to 1.30 V), low residual current and convenient surface renewal. Chemically modified carbon paste electrodes (CMCPEs) are prepared by the addition of a suitable modifier to carbon paste. The properties of such CMCPEs depends on the properties of the modifier materials used to import selectivity and sensitivity towards the target species [14-16].

In recent years, magnetic nanoparticles (MNPs) have attracted enormous interest because of their unique properties such as large surface area to volume ratios, biocompatibility, non-toxicity and easy surface functionalization. Simple modification of their surface made them attractive for several biomedical applications, nanocarrier for drug delivery and contrast agent in magnetic resonance imaging [17-19].

We used the nanocomposite $Ni_{0.5}Zn_{0.5}Fe_2O_4$ for the removal of several organic dyes [20] and oxyanions from aqueous solutions [21], and also as a modifier for the construction of an electrochemical sensor [16]. In this paper, we

report the construction of a chemically modified CPE for the determination of Mo (VI) ions in different samples after their accumulation on the electrode surface. The magnetic nanocomposite $Ni_{0.5}Zn_{0.5}Fe_2O_4$ was used as the modifier and differential pulse voltammetric method (DPV) was used for the determination of the accumulated ion.

EXPERIMENTAL
Apparatus
A Metrohm model 797 VA Computrac polarograph was used for voltammetric studies. All the reagents used were of analytical grade. Three-electrode cell systems were used to monitor the differential pulse voltammograms. An Ag/AgCl/KCl (3.0 mol L^{-1}) electrode, a platinum wire, and a modified CPE electrode were used as the reference, auxiliary and working electrodes, respectively. A Metrohm Model 827 pH lab (Herisau, Switzerland) pH-meter with a combined glass electrode was used for the pH measurements. Double distilled water (DDW) was used throughout. The prepared electrode was characterized by scanning electron microscopy (SEM) and energy dispersive X-ray spectroscopy (EDS), X-ray diffraction spectroscopy (XRD) and electrochemical impedance spectroscopy (EIS).

Reagents
All chemical reagents were of analytical grade and purchased from Merck (Germany). Double distilled water was used for the preparation of all solutions. Laboratory glassware was kept overnight in a 10 % (v/v) HNO_3 solution and then rinsed with DDW. Stock standard solutions of Mo (VI) (100.0 μg mL^{-1}) was prepared by dissolving 0.012 g of Na_2MoO_4 salt in water and diluting to 50.0 ml in a volumetric flask every day. Among various types of electrolyte solutions tested, the phosphate buffer solution (PBS) was found to yield the best results. A PBS of pH 3.0 (consisting of a mixture of 0.1 mol L^{-1} H_3PO_4 titrated with 0.2 mol L^{-1} NaOH solution to the desired pH) served as a supporting electrolyte solution. For the preparation of conventional carbon paste electrode, graphite powder, with a 10-μm particle size and highly pure paraffin (both were purchased from Merck) were used.

Synthesis of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanocomposite
Ferrite nanoparticles $Ni_{1-x}Zn_xFe_2O_4$ (x = 0.25, 0.50, 0.75, 1) can be prepared according to the chemical co-precipitation route. For synthesis of

$Ni_{0.5}Zn_{0.5}Fe_2O_4$, stoichiometric ratio of the solutions of $NiCl_2.6H_2O$, $ZnCl_2$ and $FeCl_3.6H_2O$, (100 mL of 0.05 mol L^{-1} $NiCl_2.6H_2O$, 100 mL of 0.2 mol L^{-1} $FeCl_3.6H_2O$ and 100 mL of 0.05 mol L^{-1} $ZnCl_2$), were dissolved in DDW with a constant stirring. Then, neutralization of solution was carried out with 1.5 mol L^{-1} sodium hydroxide solution. The reaction temperature was kept at 85 °C for 45 min. The pH of the reaction was kept at 12. The precipitate was thoroughly washed with DDW. The product was dried overnight at 100 °C by an *oven* to remove water contents [16].

Preparation of modified CPE (Ni_{0.5}Zn_{0.5}Fe_2O_4/CPE)

To prepare the modified CPE, 10 % (w/w) $Ni_{0.5}Zn_{0.5}Fe_2O_4$ and 65 % (w/w) graphite powder with 25 % (w/w) paraffin in a mortar and pestle were mixed. The mixture amount of 0.2 g was homogenized in a mortar for 30 min and then the mixed paste was inserted into a plastic needle type capillary tube with 1.5 mm diameter and 5 cm length, using a 0.5-mm diameter copper wire connected to the measurement system.

Recommended Procedure

A 25.0 mL volume of the solution, containing an appropriate concentration of Mo (VI) and 0.1 mol L^{-1} phosphate buffer (pH 3.0) was transferred into a voltammetric cell. The DP parameters including pulse amplitude, pulse time, resting time and voltage step height were 0.19 V, 0.01 s, 10 s and 0.014 V, respectively. The preconcentration step for 90 s at the potential of -0.7 V vs. Ag/AgCl was performed in the stirred solution. The stirring was then stopped and after a resting time of 10 s, to settle the solution and decrease the background current, differential pulse voltammograms were recorded in the positive potential direction. A renewed carbon paste surface was used for each measurement. The surface of electrode was cleaned by a simple polishing of the electrode on a paper. The renewed active surface of the electrode permits to eliminate the irreversible contamination of the surface and to minimize the memory effects, especially in real sample analysis.

Real sample preparation

Lettuce, potato, tea, cucumber, cabbage, carrot and tomato were selected for the analysis. Tomato, potato, lettuce, cucumber, cabbage, and carrot were obtained from Iranian agricultural farms and tea was brought in a tea shop.

For the preparation of samples and the determination of Mo (VI) in plant foodstuffs, about 1.000 g of the dried sample (cucumber, carrot, tomato, potato, tea, lettuce, cabbage) was first ashed for 6 h at 500 °C in a crucible. The samples were cooled and then, the ash was carefully moistened with 5.0 mL of 1:1 concentrated nitric acid: H_2O and then the mixture were heated on a hotplate to near dryness. After dissolution of the residue in 20.0 mL of triply distilled water, the solution was filtered using filter paper (Whatman No. 1) and the filtration was collected into a 50.0 mL volumetric flask and diluted to the mark with triply distilled water. Suitable aliquots of this solution were taken and analyzed for Mo (VI) using the proposed procedure [1].

RESULT AND DISCUSSION

Characterization of nickel zinc ferrite nanocomposite

Fig. 1a shows the SEM image of the synthesized $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanoparticles. As shown in Fig.1a, the average diameter of nanoparticles are around 28 nm. The typical XRD profile of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanocomposites is shown in Fig. 1b and the proposed XRD pattern is accordance with the standard one [22]. The crystallite size from the XRD pattern according to Scherrer equation was obtained around 3.5 nm [16].

The EDS spectrum (Fig. 1c) showed that the $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanocomposites, synthesized in this study, contained Fe, Ni, Zn and O elements.

To investigate the characteristics of the interface properties and differences among the electrochemical performance of CPE and $Ni_{0.5}Zn_{0.5}Fe_2O_4$/ CPE, electrochemical impedance spectroscopy (EIS) was used. As such, the Nyquist plots for 5.0 mmol L^{-1} Fe $(CN)_6^{3-}$/Fe$(CN)_6^{4-}$ in 0.1mol L^{-1} KNO_3 in the frequency ranging from 0.1 Hz–100 kHz showed a significant difference in responses for the electrodes (Fig. 1d). To fitting the obtained impedance data, the Randles circuit was chosen. Dielectric and insulting properties of the electrode/ electrolyte interface affect R_{ct}. The semicircular elements corresponding to the charge transfer resistances (R_{ct}) at the electrode surface with a large diameter were observed for the bare CPE. Additionally, after modification of the electrode, the charge transfer resistance of the electrode surface decreased and the charge transfer rate increased upon using $Ni_{0.5}Zn_{0.5}Fe_2O_4$/CPE. The R_{ct} value for the $Ni_{0.5}Zn_{0.5}Fe_2O_4$/CPE electrode was less than that for CPE. These results indicated that surface
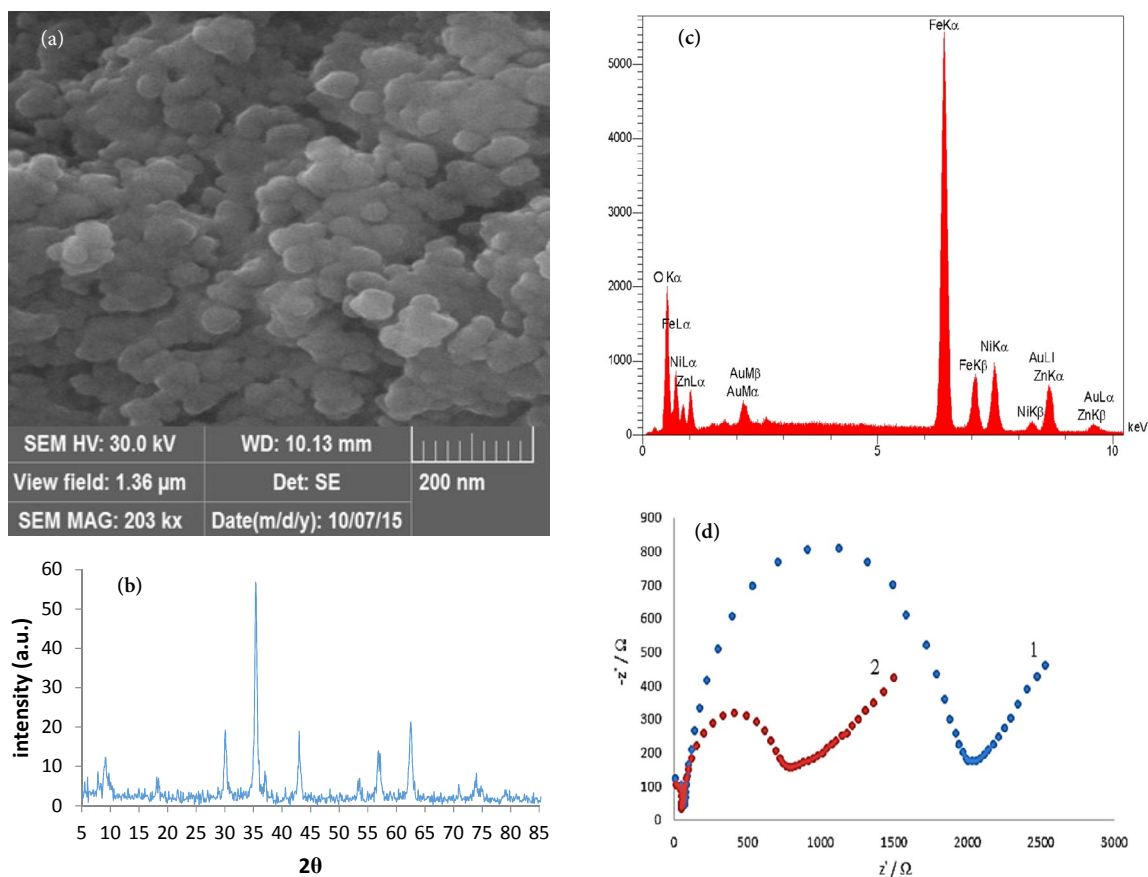
Fig. 1. SEM image for $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanocomposite (a), XRD pattern for $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanocomposite (b), EDS analysis for $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanocomposite (c) and Nyquist plots of 5.0 mmol L^{-1} Fe $(CN)_6^{3-}$/ $Fe(CN)_6^{4-}$containing 0.1 mol L^{-1} KNO_3 solution with unmodified CPE (1) and $Ni_{0.5}Zn_{0.5}Fe_2O_4$ /CPE (2) (d).

modification of the electrode with $Ni_{0.5}Zn_{0.5}Fe_2O_4$ has greatly and successfully improved the conductivity of the CPE surface.

So, the entrapped $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanoparticles show the characteristics of good biocompatibility, large surface area, good dispersing properties and fast electron transfer ability.

Electrochemical behavior of Mo (VI) at the surface of various electrodes

Using DPV technique the electrochemical behavior of Mo (VI) was studied. Fig. 2 shows differential pulse voltammograms (DPVs) for the solutions with $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanocomposite in the presence and absence of target ion using unmodified CPE and CPE modified in the potential range of -1.0 to 1.0 V vs. Ag/AgCl. The DPVs were recorded after a deposition step of 90 s at -0.70 V vs. Ag/AgCl at pH 3.0. As can be seen, in the potential range of -1.0 to 1.0 V vs. Ag/AgCl the voltammograms in the absence of Mo (VI) ion at unmodified CPE (Fig. 2a)

and modified CPE (Fig. 2b) did not show any peaks. However, there was a strong peak current at -0.34 and -0.42 V for unmodified (Fig. 2c) and modified
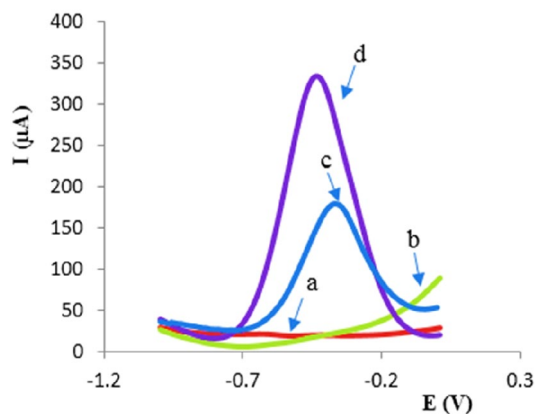

Fig. 2. DPVs of carbon paste electrode in blank (a), $Ni_{0.5}Zn_{0.5}Fe_2O_4$/CPE in blank (b) carbon paste electrode in 0.9 μg mL^{-1} of Mo (VI) solution (c) $Ni_{0.5}Zn_{0.5}Fe_2O_4$/CPE in 0.9 μg mL^{-1} of Mo (VI) solution.

(Fig. 2d) electrodes, respectively, in the presence of 0.9 μg mL^{-1} of the Mo (VI) ion, corresponding to the successive oxidation of molybdenum deposited on the surface of the electrodes. The significant enhancement in current response together with a decrease in peak potential for modified electrode, as clearly seen in Fig. 2, indicates that $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanocomposite, in addition to increasing the microscopic area of the electrode and enhancement of the target ion's current, acts as a catalyst for the oxidation of the deposited molybdenum. On this basis, for the electrochemical determination of Mo (VI) ion, a $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanocomposite modified CPE was prepared and applied successfully.

Effect of parameters

To achieve the optimum experimental conditions, the effect of different variables, including supporting electrolyte, pH, the amount of modifier, deposition potential, deposition time, pulse amplitude, pulse time and voltage step, on the determination of Mo(VI) in a solution containing 0.9 μg mL^{-1} of ion was studied.

Electrode composition

To investigate the effect of the composition of the electrode materials on the voltammetric responses used from DPV. The solution consisted of 0.9 μg mL^{-1} of molybdenum ion in a pH 3.0 phosphate buffer solution. Different electrodes were constructed by fixing the amount of paraffin at 25 % (w/w) and substituting some of the graphite powder by $Ni_{0.5}Zn_{0.5}Fe_2O_4$ magnetic nanocomposite. The results showed that the peak current by increasing the amount of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanocomposite up to 10 % (w/w) increased and then for higher amounts of modifier decreased. The increase in the current by increasing $Ni_{0.5}Zn_{0.5}Fe_2O_4$ on the modified electrode can be related to the larger microscopic area and also the electrocatalytic effect of the nanocomposite on the oxidation of the deposited molybdenum. By increasing the amount of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ the amount of the accumulated molybdenum at the surface of electrode increased and resulted in an increase in anodic current. Therefore, for further studies the electrode composition of 25 % (w/w) paraffin, 65 % (w/w) graphite and 10 % (w/w) $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanocomposite was chosen.

Effect of supporting electrolyte and pH

The influence of various types of supporting electrolytes including phosphate buffer, B–R buffer,

acetate buffer and the KCl solution on peak current was investigated. Among different buffer solutions, the height of the peak reached a maximum and the shape of the curves was better in phosphate buffer solution. Then, the influence of the pH using phosphate buffer solution between 2.0 and 5.5 on the anodic peak current of the Mo (VI) ion was also studied. As Fig. 3a shows, with increasing the pH value, the anodic peak current increased at first up to pH 2.5. From pH 2.5 to 4.5, the peak current was
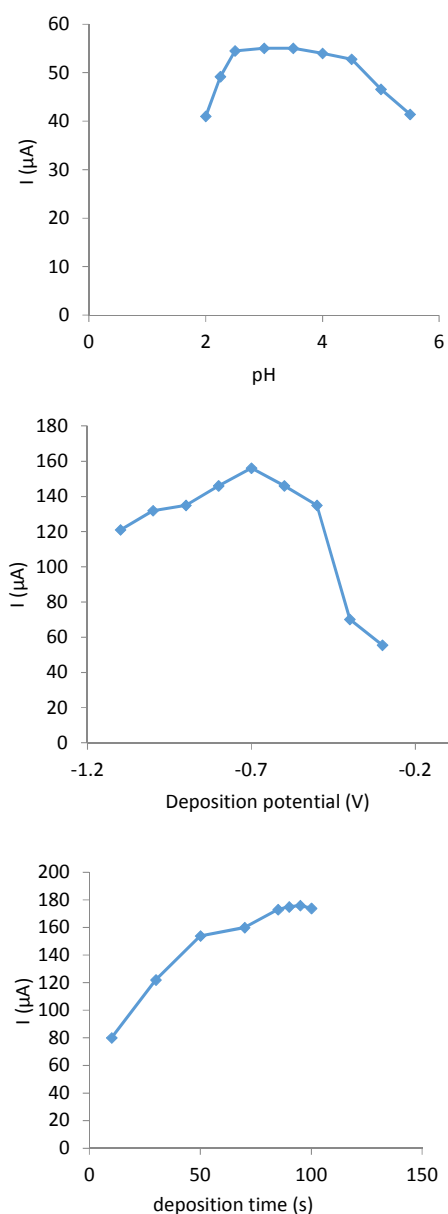
Fig. 3. Effect of pH (a), accumulation potential (b) and accumulation time (c) on the analytical signal of 0.9 μg mL^{-1} of Mo (VI) solution

almost constant and then decreased at higher pHs. At pHs lower than 2.5, the decrease in currents can be due to the dissolution of $Ni_{0.5}Zn_{0.5}Fe_2O_4$ nanocomposite in the solution leading to the instability of the electrode. Therefore, for further investigation the optimal pH of 3.0 was chosen.

Effect of deposition potential and time

It was significant to fix the accumulation potential and accumulation time when adsorption studies were intended. The effect of the accumulation potential on the anodic peak current of the solution of 0.9 μg mL^{-1} of Mo (VI), was examined in the potential range of -0.3 to -1.1 V vs. Ag/AgCl under the above optimum conditions. The effect of deposition potential was performed by recording the voltammograms at different deposition potentials. The deposition time for all the deposition potentials was 50 s. Fig. 3b shows the curve of the current response as a function of deposition potential for the modified electrode. As shown in Fig.3b, the peak current increases with changing the potential from -1.1V to -0.7V and then decreased at potentials more positive than -0.7 V. Therefore, -0.7 V vs. Ag/AgCl was selected as the accumulation potential in the procedure. The dependence of the maximum peak current on the accumulation time for the solution of 0.9 μg mL^{-1} of Mo (VI), in pH 3.0 phosphate buffer solution and deposition potential of -0.7 V, in the range of 10–100 s (Fig. 3c) were also examined. The peak current was proportional to the accumulation time up to 80 s. For longer preconcentration times,

because of the saturation loading of active sites at the electrode surface, the current was found to level off and reached constant values after 80 s. Therefore, a deposition time of 90 s was selected for further works.

Other optimum conditions for the DPV response are related to the instrumental parameters affecting the peak current of the analyte. These parameters were optimized for obtaining maximum signal-to-noise ratio. Optimum values for the studied parameters were chosen as 0.19 V, 10 s, 0.01 s, 0.014 V and 0.5 s, for pulse amplitude, resting time, pulse time, voltage step and voltage step time, respectively.

Analytical parameters

Calibration graph was constructed under the optimum conditions described above using modified electrode. Under the optimized condition, a linear relationship between the oxidation peak current of the molybdenum and the concentration of Mo (VI) was obtained in the range of 0.005–1.00 μg mL^{-1}, following a 90 s accumulation time (Fig. 4). The detection limit, calculated from three times standard deviation of the blank (S_b) divided by the slope of the calibration graph, was 3.00 ng mL^{-1}. S_b was estimated by 10 times determination of the blank signals.

To study the repeatability of the proposed electrode, ten successive measurements of 0.06 and 0.70 μg mL^{-1} of Mo (VI) ions after 90 s accumulation time were analyzed, and showed the relative standard deviation of 3.2 and 1.98 %, respectively.
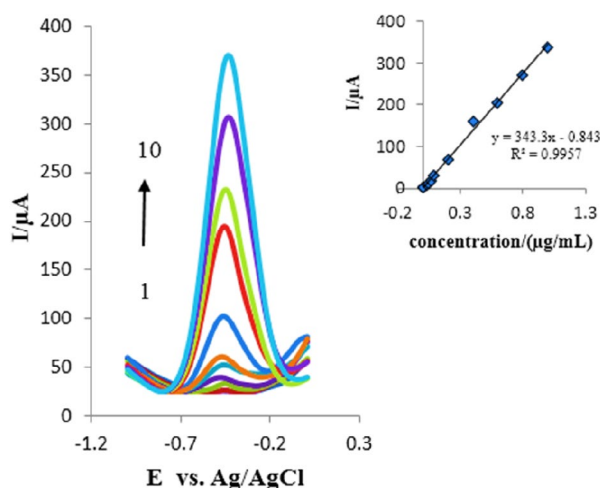

Fig. 4. DPVs at $Ni_{0.5}Zn_{0.5}Fe_2O_4$/CPE in PBS (pH 3.0) containing different concentrations of Mo (VI) (from 1 to 10, the concentrations: 0.008, 0.03, 0.05, 0.07, 0.09, 0.2, 0.4, 0.6, 0.8, 1.0 μg mL^{-1} of Mo (VI)) under the optimized conditions. Inset: Plot of the oxidation peak currents as a function of Mo (VI) concentrations.

The reproducibility of the electrode response was also studied. Five electrodes were prepared from the same batch and were evaluated by performing the determination of 0.9 μg mL^{-1} of Mo (VI) solution. The RSD for the response between electrodes was 3.4 %. The storage stability of the designed sensor was also investigated. The result showed that response sensitivity retained more than 95 % over 6 weeks. After that, the response of the electrode decreased and the noise of the responses increased. The high reproducibility and stability indicated that the modified electrode was suitable for the analysis of real samples.

Interference study

To investigate the selectivity of the prepared electrode for the determination of Mo (VI), the influence of various substances that may potentially interfere with the determination of analyte was studied under the optimum conditions. The effects of various interfering species, accompanied by Mo (VI) in food samples, were studied by the addition of the interfering ion to the solution containing 0.9 μg mL^{-1} of Mo (VI) solution. As seen in Table 1, the maximum tolerable concentrations of foreign species are shown, where the tolerance limit was defined as the concentration of foreign species that produces a change in height of the peak current less than 5%. On the basis of the results, it was found that most of the foreign ions did not interfere with Mo (VI) determination.

Applications and comparison of the proposed method with other methods

To evaluate the applicability of the proposed method, the electrode was used for the determination of Mo (VI) in several plant foodstuff samples. The results are given in Table 2a. The plant foodstuff samples were also analyzed spectrophotometrically [1]. As shown in Table 2a, there is a good agreement

Table 1. Tolerance ratio of interfering ions in the determination of 0.9 μg mL^{-1} of molybdenum

Species	Tolerance limit for Mo (VI)
Na^+, K^+, Al^{3+}, Mn^{2+}, As(III), V(V), SO_4^{2-}, Br^-, Cl^-, SCN^-,	1000
Ca^{2+}	800
NO_3^-, CH_3COO^-	500
Pb^{2+}	100
SO_3^{2-}	50

Table 2a. Results of determination of Mo (VI) in plant food stuff samples

Sample	This work(μg/g)	Cloud point extraction method (μg/g)	t$_{exp}$[a]
Lettuce	0.308±0.041	0.334±0.061	0.76
Potato	5.810±0.116	5.146±0.244	1.96
Cucumber	1.111±0.100	1.123±0.155	0.15
Tomato	2.698±0.131	3.576±0.112	0.29
Cabbage	0.820±0.024	1.0157±0.089	1.83
Tea	0.1731±0.015	0.159±0.009	0.36
Carrot	4.043±0.121	4.294±0.148	1.2

[a] t$_{exp}$ shows the experimental student-t values, (t$_{critical}$=2.31)

Table 2b. Comparison of Some critical points in the present work compared with some previous works for determination of Mo (VI)

Electrode	ligandL	Deposition time	Linear dynamic range (ng/mL)	Detection limit (ng/mL)	Ref
Acetylene black paste electrode	Oxalate	120 s	0.5-960.0	0.19	2
CPE/CTAB	Alizarin violet	10 min	0.5-500	0.04	23
HMDE	AZO compound	20 min	48.0-240.0	14.4	24
HMDE	Oxine	60 s	0.5-150	0.1	25
Lead film electrode	Alizarin S	60 s	0.19- 4.8	0.08	26
HMDE	Cupferron	60 s	0.1-20.0	0.06	27
HMDE	Proton transfer compound	60 s	-	4.0	28
Modified carbon paste electrode By MWCNT	Alizarin violet	120 s	0.038-9.6 And 19.2-768.0	0.009	29
HMDE	morine	1 min	To 70.0	0.45	30
Modified carbon paste electrode by nano composite of $Ni_{0.5}Zn_{0.5}Fe_2O_4$	-	90 s	5.0-1000.0	3.0	This work

between the results obtained by the proposed method with those obtained by spectrophotometry. The obtained results reveal the capability of the method for the successful determination of Mo (VI) in all real samples without considerable error.

In Table 2b, a comparison between the proposed electrode with the previously reported modified electrodes for the determination of Mo (VI) is given. As seen in Table 2b, the linear ranges of this work are so wider than some of other reported electrodes [23-27]. Also, the LOD of the proposed electrode is much better than that for some of the previously reported electrodes [24, 28] and the pre-concentration time is shorter than that for some others [2, 23, 24].

CONCLUSION

In this study, a new modified magnetic nickel zinc ferrite nanocomposite / CPE as an electrochemical sensor for determination of Mo (VI) was presented and its application to the quantification of Mo (VI) ions in different samples was studied. In comparison with unmodified CPE, the prepared electrode had the advantages of high electrical conductivity and high resistance to interferences. In the modified electrode the renewable active surface of the electrode could be easily obtained by a simple polishing of the electrode on a paper, that permits to eliminate the irreversible contamination of the surface and to minimize memory effects, especially in a real sample analysis. The proposed method is simple, sensitive, highly selective, accurate and applicable for the determination of Mo (VI) in real samples.

ACKNOWLEDGEMENT

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