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1 2	Elecrochemical Study of the Capacity of Moringa Oleifera to Chelate p-Aminophenol
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7 Abstract

8 We report a sensitive electrochemical voltammetric method for analyzing p- Aminophenol,

9 using a Natural phosphate (NP) modified carbon paste electrode NP-CPE, in Na2SO4,

¹⁰ solution. Operational parameters have been optimized, and the stripping voltammetric

¹¹ performance has been studied using square wave voltammetry. The use of Moringa oleifera as

¹² natural chelating agent gave a significant improvement in the depollution of contaminated

13 water sample.

14

15 Index terms— electroanalysis; modified electrodes; paminophenol; moringa oleifera.

¹⁶ 1 I. Introduction

minophenols are interesting electrochemical materials since, unlike aniline and other substituted anilines [1][2], 17 they have two groups (-NH 2 and OH), which could be oxidized. Therefore, they could show electrochemical 18 behavior resembling anilines and/or phenols [3][4][5]. An important factor would be the relative position of the 19 amino and hydroxyl group in the aromatic ring. Accordingly, the reported electrochemical properties of the 20 three positional isomers (ortho, meta and para) are strongly different. P-Aminophenol (p-AP) is a well-known 21 compound which, in its simple form, or derivative [6], has been used as redox agent in photography. In neutral 22 media, it is oxidized to complex oligometric devise that could be used in enzymatic assays [7]. konopelnik et al. 23 [8] have studied the oxidation of P-aminophenol (P-AP) in aqueous solution on SnO electrodes. According with 24 25 these authors, only the amino group of m-aminophenol undergoes oxidation while the hydroxyl group remains 26 unchanged. Common laboratory-based analytical methods for determining para-aminophenol compounds such as primarily gas and liquid chromatography (HP LC) [9][10][11][12][13], UV-vis spectrophotometry [14][15] and 27 spectrofluorimetry [16] have been reported. The use of enzyme-link ed immunosorbent assay (ELISA) has been 28 studied [17]. However, some sample pretreatment involving separation, extraction and/or adsorption is generally 29 necessary, and this can also be timeconsuming and complex. Electrochemical methods, such as differential pulse 30 polarography (DP P), anodic stripping voltammetry (ASV) and differential pulse voltammetry (DPV), have been 31 widely applied for the determination of pharmaceuticals, dyes, insecticides and pesticides [18][19][20]. In recent 32 years, chemically modified electrodes (CMEs) were used for the voltammetric quantification of various organic 33 and inorganic species after their open circuit accumulation [21][22] Much of the work in this field was directed to 34 exploit the chemical reactivity of the modifier towards a target analyte for electroanalytical purpose. Multitudes 35 36 of modifying agents were used either as coatings on solid electrode surfaces or dispersed within a conductive 37 matrix. It is noteworthy that this last approach is well suited when using electronically insulating modifiers 38 requiring a direct contact to an electronically conducting substrate as used in connection with electrochemistry. 39 The application of silicates and related mineral materials in electrochemistry is rather recent and was directed to combine their intrinsic properties to selected electrochemical reactions in order to improve the response of the 40 electrode. Modified electrodes are being used frequently in the voltammetric determination of organic compounds 41 because of their efficiency, the selectivity that can be obtained by varying the modifier and the sensitivity which is 42 equivalent to that reached in anodic and cathodic stripping. In doing so, zeolite and silica-modified electrodes were 43 prepared, characterized and applied (sometimes tentatively) in various fields including for example electroanalysis 44

7 A) CHARACTERIZATION OF PREPARED ELECTRODES SURFACES

and sensors, electrocatalysis, photochemistry, thin-film technology, fuel cells, molecular recognition. Kauffmann 45 [23] has reported that a carbon paste electrode (CPE) modified with lipids and proteins (enzymes) have potential 46 application in environmental analysis. Recent works, reported in the literature, have shown several applications 47 and electroanalytical methodologies employing glassy carbon electrode as working electrodes [24][25]. Luz and 48 al. [25] constructed a glassy carbon electrode impregnated with a lithium tetracyanoethylenide (LiTCNE) for 49 the determination of para-aminophenol. The oxidation and reduction of this compound has been carried out on a 50 modified glassy carbon electrode using cyclic and DPV [26][27][28]. This study proposed a new modified carbon 51 paste electrode which has been prepared by the Natural Phosphate (NP) for para-aminophenol detection. It 52 has shown a selective preconcentration and quantization of paraaminophenol by cyclic voltammetry (CV). This 53 study has led to the development of a new modified electrode for the determination of para-aminophenol with 54 improved qualities such as simplicity of electrode preparation, wider linear range, and low detection limit (DL), 55 high selectivity and very good stability of modifier. The procedure is based on the oxidation and reduction of 56 para-aminophenol after it was preconcentrated on a carbon paste electrode modified with the clay, under open 57 circuit conditions. 58 In this work we prepared and characterized the phosphate Natual modified carbon paste electrode, which 59

successfully exploits the favorable mechanical and electrochemical properties of carbon paste electrodes. Also, this study therefore focused on the determination of the effectiveness of the Moringa oleifera in the purification

62 of water contaminated by 4-Aminophenol.

⁶³ 2 II. Experimental a) Reagents

p-Aminophenol and sodium sulfate were of analytical grade and from Aldrich. A natural phosphate (NP) used
 in this work was obtained in the Khouribga region (Morocco). Stock solutions of p-Aminophenol were prepared

by dissolving p-Aminophenol in deionized water. All preparations and dilution of solutions were made deionized

⁶⁷ water. Provisions were made for oxygen removal by bubbling the solution with azotes gas for about 5 min then

the solution was blanketed with azotes gas while the experiment was in progress. For reproducible results, a fresh

69 solution was made for each experiment. Carbon paste was supplied from (Carbon, Lorraine, ref. 9900, French).

70 **3 b**) Electrodes preparation

Firstly, the carbon-paste electrode was prepared according the following procedure [29]. The carbon-paste
electrode was prepared by mixing the graphite powder with paraffin oil used as a binder.

The mixture was grinding in a mortar agate and then a portion of the resulting composite material was housed

in PTFE cylinder. The geometric surface area of the working electrode was 0.1256 cm2. A bare of carbon vitreous
 inserted into carbon paste provided the electrical contact, and then the NP film is electrodeposited onto carbon

paste electrode. The deposit of Phosphate natural on carbon paste electrode surfaces was processed at 20 V. The

⁷⁷ current was maintained by a galvanostat with a function generator.

⁷⁸ 4 c) Prepared electrode characterization

All the electrochemical experiments were performed in a standard one-compartment three electrode cell. The reference electrode was SCE and the counter electrode was platinum. All electrode potentials were referred to this reference electrode. The working electrode was NP modified carbon paste electrode (NP-CPE).

⁸² 5 d) Apparatus

Electrochemical experiments were performed using a voltalab potentiostat (model PGSTAT 100, Eco Chemie
 B.V., Ultrecht, The Netherlands) driven by the general purpose electrochemical systems data processing software
 (voltalab master 4 software)

85 (voltalab master 4 software).

⁸⁶ 6 III. Results and Discussion

⁸⁷ 7 a) Characterization of prepared electrodes surfaces

The surface structure of natural phosphate modified carbon paste surface was observed using scanning electron 88 89 microscopy (Fig. 1). The film layer of NP was formed on the surface of carbon paste electrode; it was not 90 disintegrated or detached from the surface when immersed in the electrolytic solution (0.1M Na2SO4). The 91 morphology of the phosphate rock surface was observed by scanning electron microscopy (Figure 1). The 92 treatment described previously gives compact particle fractions between 100 and 400 µm rich in phosphate. Rock phosphate treaty has the following chemical composition: CaO (54.12%), P 2 O 5 (34.24%), F (3.37%), SiO 93 2 (2.42%), SO3 (2.21%), CO 2 (1.13%), Na 2 O (0.92%), MgO (0.68%), Al 2 O 3 (0.46%), Fe 2 O 3 (0.36%), K 2 94 O (0.04%) and order of several ppm metals. We can see that the shape of the cyclic voltammogram was modified 95 in the presence of NP at CPE surface, suggesting that the carbon paste electrode was effectively modified by 96

97 natural phosphate.

⁹⁸ 8) Electrochemical detection of studied metals

⁹⁹ The experimental conditions have been optimized and the response characteristics determined in a previous work ¹⁰⁰ [29]. The results obtained are:

101 ??p H ? ? 7 ??Pre conce ntra tion time = 13 min.

In order to avoid the strong residual of reduction, the starting potential was fixed at -1V versus SCE. Fig. 3 shows a cyclic voltammograms performed between-1 V and 1 V for NP-CPE, in 0.1M Na2SO4 solution (curve a), and in 0.1M Na 2 SO 4, after exposure NP-CPE to 0.510-3 mmol/L P-Aminophenol for 13 min, in a stirred solution (curve b). The reversible system could be observed at NP-CPE, with cathodic potential value, of -0.3 V and anodic potential value of -0.1 V.

The square wave voltammetry (SWV) corresponding to the determination of P-Aminophenol, was recorded in the supporting electrolyte (curve a) and after, 13 min of accumulation in a solution containing P-Aminophenol (curve b).

The Square wave voltammograms are showed in Figures 4. A well-defined and enhanced peak is observed at NP-CPE, imprinted in P-Aminophenol solution. This peak is attributed to P-Aminophenol oxidation. The scheme 1 shows the proposed mechanism of this oxidation. Since its preferred habitat is dry sandy soil, it tolerates poor soils, such as those in coastal areas [31].

Firstly, the Moringa oleifera was prepared according the following procedure [30]. Moringa oleifera seeds collected for the analysis were shelled off and sun dried to maintain constant weight. The sun-dried seeds were grinded into powdered form using machine. The powdered was added to the solutions containing heavy metals. After 15 min of contact with moringa oleifera, the solutions were purified and analysis in electrochemical sensor.

The SQWV's recorded at prepared electrode, in supporting solution containing p-aminophenol (curve a) and 118 after addition of the moringa oleifera (curve b), are shown in Figure 7. The peak current decreased considerably 119 after moringa treatment. This current density reduction is due to a sharp decline in paminophenol concentrations, 120 121 which suggests that moring has a strong complexing power of paminophenol (Figs. 7 and 8). We not that the 122 solution pH was varied after moring a treatment. It was decreased from 7.2 to 6. Figure 7 and 8 shows the CV and SQW curves recorded, for different concentrations of moringa oleifera (MO), at NP-CPE was increased from 2 123 ml/200 ml (electrolytical solutions) to 13 ml/200 ml (electrolytical solution). Both the anodic and cathodic peak 124 current increases linearly with the concentration of moringa oleifera and the plot of current versus concentration 125 obeys Randles-Sevic equation, which implies that the electrode process is adsorption controlled reaction. It was 126 also observed that the cathodic peak potential shift towards negative values and anodic peak potential shift 127 towards positive side. This kind of shift in Ep in the cathodic and anodic. V en ml moringa The effect of 128 the MO concentration of MO is investigated (Figure ??), this significantly affects the oxidation peak current of 129 p-aminophenol. The peak current decrease greatly with MO concentration. 130

Similarly, the effectiveness of the Moringaenol to chelate p-aminophenol increases considerably with the MO concentration. (Fig. 10) In Figure 11, we present the SEM images, taken to the surface of the modified electrode; we can see that after MO treatment, the surface morphology changed dramatically with the advent of large compact crystals, leaving suggest that MO-p-aminophenol complex is adsorbed on the electrode by forming a continuous film.

¹³⁶ 9 IV. Conclusion

In conclusion, it was possible demonstrating the potentiality of the proposed electrode (NP-CPE) for determining
P-aminophenol. Such a sensor is characterized by a higher sensitivity and reproducibility.

The Moringa oleifera seeds have the ability to retain p-aminophenol. The metal is sequestered by chemical sites naturally present in the moringa matrix. The chelating process is rapid and takes place under normal temperature and pressure. Moringa oleifera is an environmentally-friendly natural complexing most suitable for the treatment of water containing undesirable p-aminophenol concentrations. The removal efficiencies were 60 %

for P-aminophenol. It is an eco-friendly technology that is economically more advantageous than other treatment alternatives. $1 \ 2 \ 3 \ 4$

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Figure 1: Figure 1 :



Figure 2: Figure 2 :



Figure 4: Figure 4 :



Figure 5: Figure 7 :

7



Figure 6: Figure 8 :



Figure 7: Figure 7 : Figure 8 :



Figure 8: Figure 9 : Figure 10 : Figure 11 :



Figure 9:



Figure 10:

9 IV. CONCLUSION

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