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By A. Zaroual, S. El Qouatli, A. Bellouchou, A. Guenbour, R. Najih & A. Chtaini
University Sultan Moulay Slimane

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An Electrochemical Sensor for the Detection of P-Anisidine through Electrochemistry

A. Zaroual [α], S. El Qouatli [σ], A. Bellouchou [ρ], A.Guenbour [ω], R. Najih [¥] & A.Chtaini [§]

Abstract- In this study, a sensitive electrochemical voltammetry method for the analysis of p-anisidine (pA) using a carbon paste electrochemical (CPE) modified with a porous material, such as natural phosphate (NP), was proposed. p-anisidine strongly adsorbed on a electrode surface and prepared electrode NP-CPE provides easy methods for electrochemical quantitative electro-p-anisidine. Operational parameters have been optimized, and performance voltammetric stripping was investigated by cyclic voltammetry (CV). The current intensity peaks are very linear, with good sensitivity NP- CPE.

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I. Introduction

Para-anisidine (p-anisidine), is the most toxic [1] of the three isomers of anisidine and causes damage to the blood when ingested orally, by inhalation or skin contact. If heated strongly it can release highly toxic fumes of oxides Trogen lev- [2-4]. Melting and boiling point 243°C P-anisidine (PA) is colorless crystal with point 57.2 it is an important intermediate for the synthesis of dyes, medicine and fragrances, pigments, and other chemical compounds [5-7].

P-Anisidine reacts with secondary oxidation products such as aldehydes and ketones in the fats and oils to form products which absorb at 350 nm of light wavelength; therefore, it is used as an official method for detection them by the American Oil Company Chemistsy [8-9]. It is particularly good at detecting unsaturated aldehydes, which are those most likely to generations at unacceptable flavors, making it especially useful in food quality testing [10]. Traditional preparation of p-Anisidine uses iron powder or sodium sulfide as reducing agent [11], which reproduces a large quantity of waste and results in the problem of serious environmental pollution. [12] In this article, we describe the electrochemical analysis of p-anisidine on a modified clay carbon paste electrode. The electrochemical characterization of adsorbed p-anisidine was assessed using cyclic voltammetric (CV) and square wave voltammetry (SQW).

II. Experimental

a) Reagents

Potassium nitrate was dissolved into Bidistilled deionized water (BDW) to form 1mg.L^{-1} stock solutions. Working standards for calibration were prepared by diluting the primary stock solution with BDW. Carbon paste was supplied from (Carbon, Lorraine, ref. 9900, French). All chemicals were of analytical grade and used without further purification.

b) Electrodes preparation

Firstly, the carbon-paste electrode was prepared according the following procedure [13]. 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 0.1256cm^2. A bare of carbon vitreous inserted into carbon paste provided the electrical contact, and then the Phosphate natural film is electrodeposited onto carbon paste electrode. The deposit of Phosphate natural on carbon paste electrode surfaces was processed at 20 V. The current was maintained by a galvanostat with a function generator.

c) 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).

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 natural phosphate modified carbon paste electrode (NP-CPE).

III. Results and Discussion

a) Characterization of prepared electrodes

The surface structure of NP- CPE surface was observed using scanning electron microscopy (Fig. 1). The film layer of NP was formed on the surface of

Author α ρ ω: Laboratoire des Matériaux, nanotechnologies et Environnement, Faculté des Sciences de Rabat . e-mail: s.elqouatlu@yahoo.fr
Author σ ¥ § : Equipe d'Electrochimie Moléculaire et Matériaux Inorganiques, Faculté des Sciences et Techniques de Béni Mellal
e-mail: a.chtaini@usms.ma

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carbon paste electrode; it was not disintegrated or detached from the surface when immersed in the electrolytic solution (0.1M Na_2SO_4). The treatment described previously gives compact particle fractions between 100 and 400μm rich in phosphate. Natural phosphate treaty has the following chemical composition: CaO (54.12%), P_2O_5 (34.24%), F (3.37%), SiO_2 (2.42%), SO_3 (2.21%), CO_2 (1.13%), Na_2O (0.92 %), MgO (0.68%), Al_2O_3 (0.46%), Fe_2O_3 (0.36%), K_2O (0.04%) and order of several ppm metals.

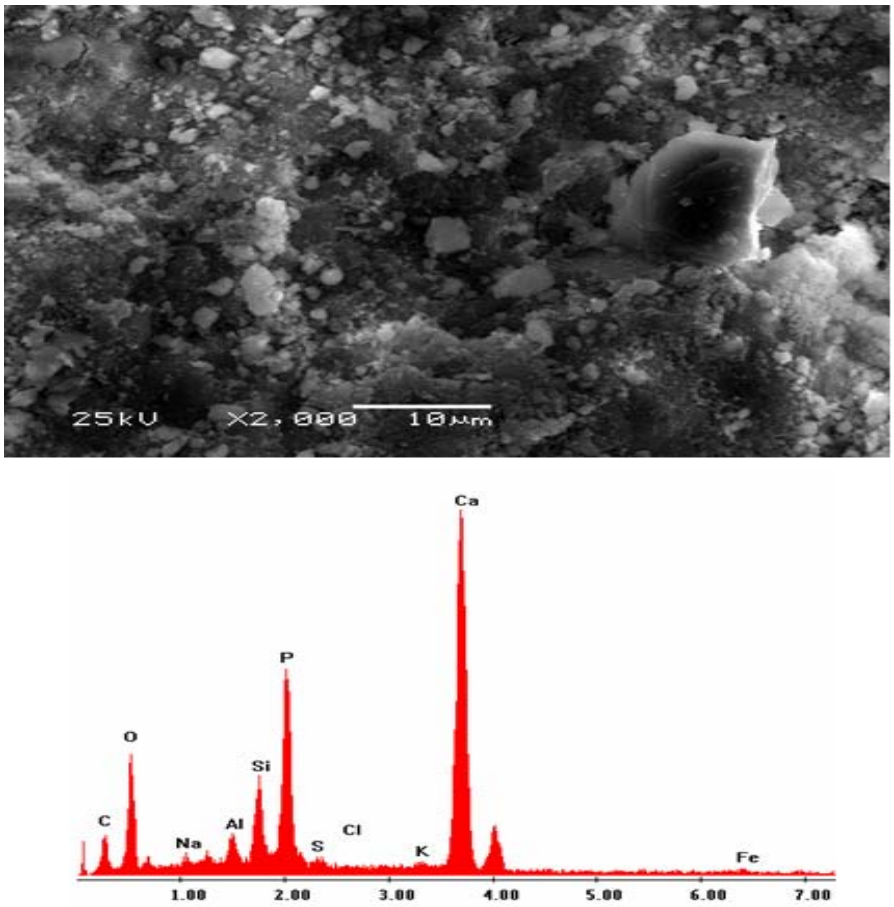

Figure 1 : Scanning electron micrograph of NP-CPE

b) Voltammetric characteristic of p-anisidine

The determination of p-anisidine at the NP-CPE was performed by using cyclic (CV) and square wave voltammetry (SQW). The results are presented in Figures 2 and 3 show the CV and the SQW measurements, respectively. Two oxidation peaks were observed at NP-CPE towards the positive sweep direction, the first one around −0. 2 V and the second at approximately 0.15 V versus SCE, scanning in the negative sense brings up a cathodic peak at -0.4V. Fig. 3 shows the square wave voltammograms obtained in 0.1 mol L^{-1} Na_2SO_4 for unmodified and modified carbon paste electrode. When the NP-CPE was dipped into the accumulation medium containing p-anisidine followed by square wave voltammetry two well defined peaks appeared.

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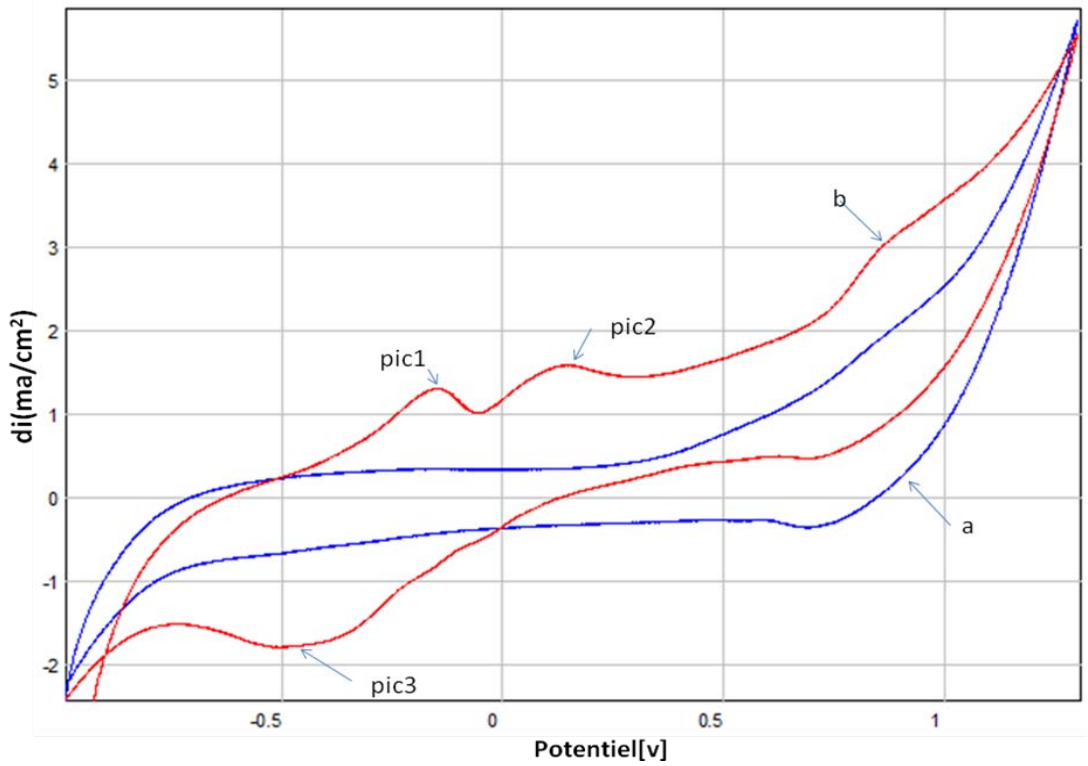

Figure 2 : Cyclic voltammetry recorded at NP-CPE in 0.1 M Na_2SO_4 solution
a - without p-anisidine b - with p-anisidine , pH = 7, accumulated time = 10min

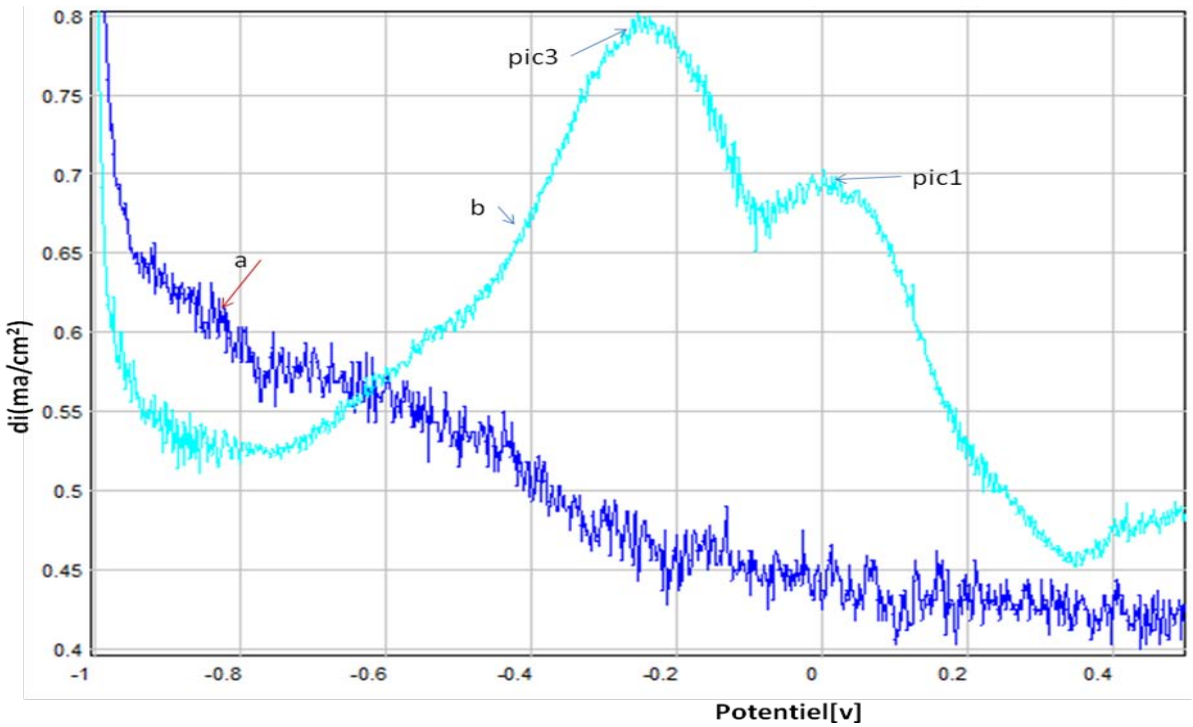

Figure 3 : Square wave voltammetry recorded at NP-CPE in 0.1 M Na_2SO_4 solution
a - without p-anisidine, b - with p-anisidine, pH = 7, accumulated time = 10min

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c) Calibration graph

Under these selected conditions, the peak current increased linearly with the p-anisidine concentration in solution using a preconcentration time of 10 min (Figs. 4 and 5). The linear dynamic range was comprised between 0.8 and 2.4 mM in terms of the relationship between p-anisidine concentration and the oxidation peak current (Fig. 6). . The relationship can be described in the following linear regression equation in the mentioned concentration range:

$IP1 = 0{,}105[P\text{-}A] + 0{,}438 \quad R^2 = 0{,}983$

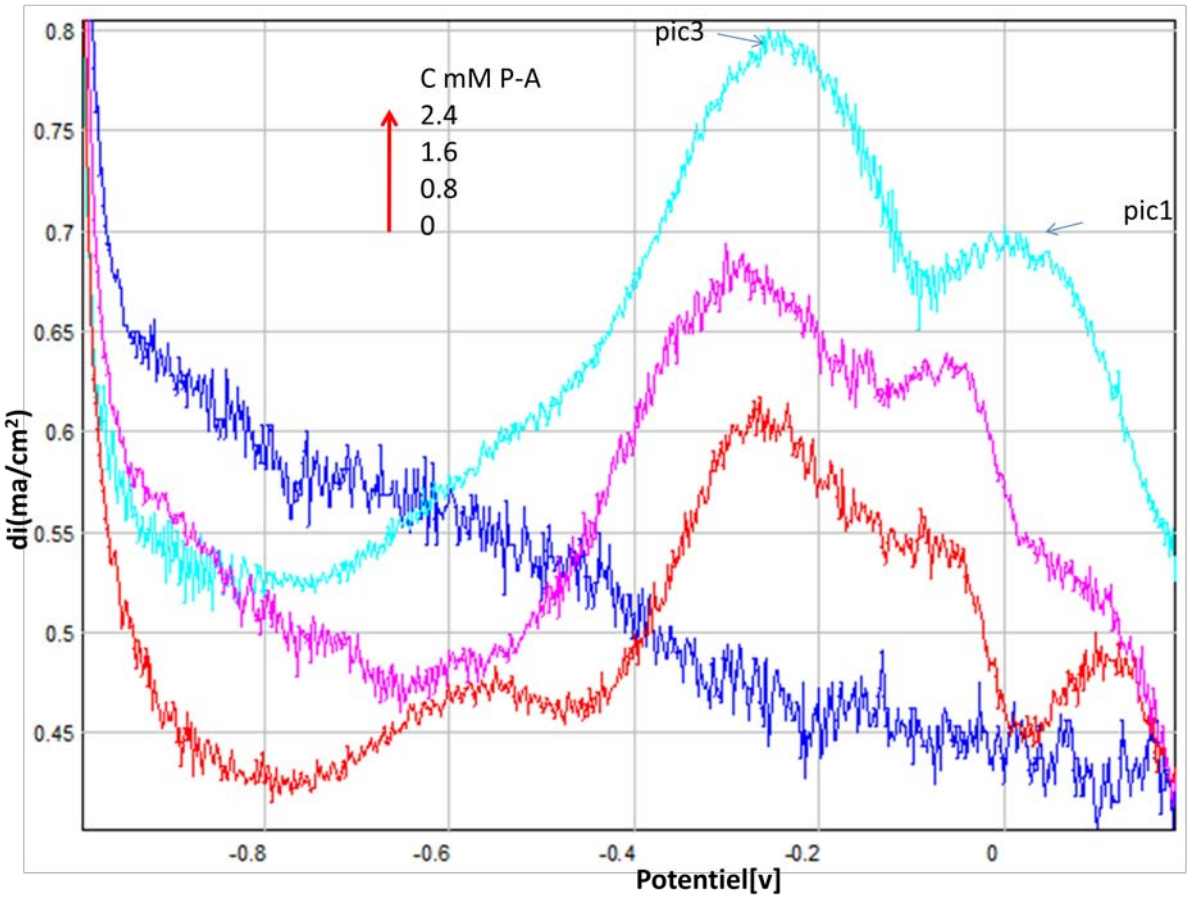

Figure 4 : Square wave voltammograms at different concentration of p-anisidine recorded at NP-CPE in 0.1M Na_2SO_4,

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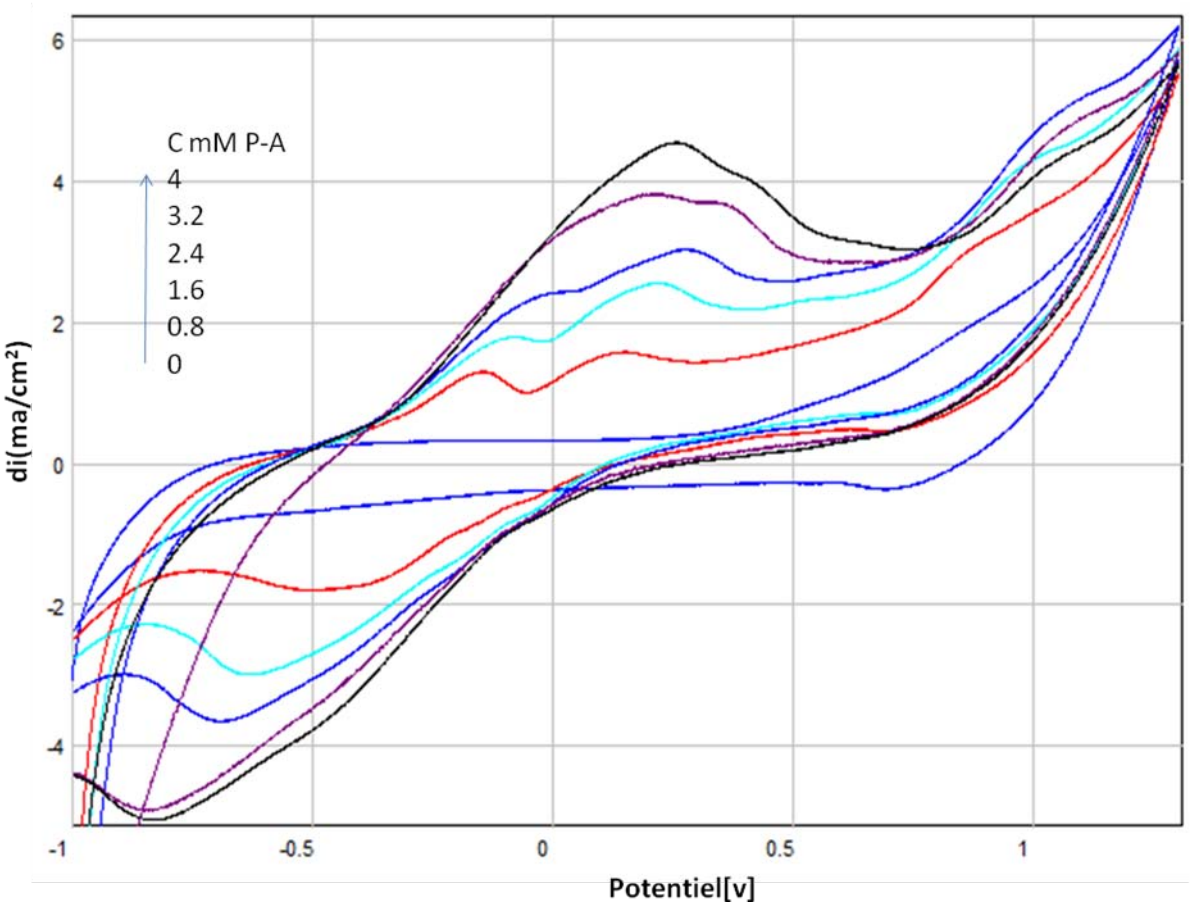

Figure 5 : CV's recorded at NP-CPE, in different concentration of p-anisidine, the scan rate and 100 mV / s

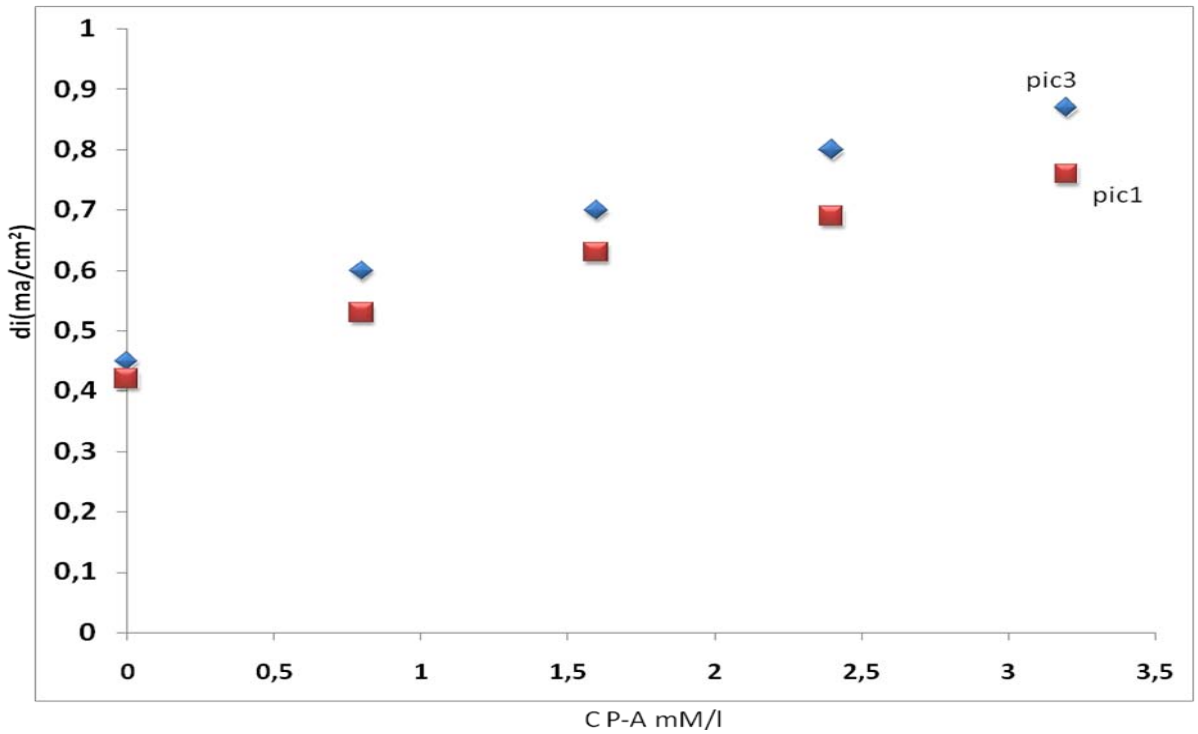

Figure 5 : Plot of peaks area versus added concentration of p-A

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IV. Conclusion

A new chemically modified carbon paste electrode has been developed with natural phosphte for the determination of p-anisidine at trace levels by square wave and cyclic voltammetry. The electrode offers attractive properties such as simplicity of electrode preparation. Also, there is no leaching of the electrode because of the low solubility of the NP in aqueous solution.

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