

ELECTROCATALYTIC REDUCTION OF NITRITE AT POLYPYRROLE  
/CELLULOSIC ELECTRODE FROM POTABLE WATERABDERRAZAK.HAMAM<sup>1,2,\*</sup>, DEHBIA.OUKIL<sup>1</sup>, LAID.MAKHLOUFI<sup>1</sup><sup>1</sup>LECVE Laboratory, Chemical Engineering Department, Faculty of Technology, University of Bejaia, 06000, Bejaia, Algeria.<sup>2</sup>Welding and NDT Research Center CSC-Algeria.\*Corresponding author: e-mail: [hamam.abderrazak@yahoo.com](mailto:hamam.abderrazak@yahoo.com)**Abstract**

Nitrite species are widely involved in environmental chemistry and public health; its role was recognized long time ago [1]. Although, naturally occurring concentrations of nitrite are usually of no health significance, wastes from fertilizers or intentional additions of nitrite for corrosion control are potential sources of contamination [2,3]. pyrrole is one of most important organic conducting polymers, which is found in wide applications in analytical chemistry.

In this work, we report the simple chemical polymerization process with Iron (III) chloride anhydrous (FeCl<sub>3</sub>) as an oxydant. To obtain thin film of polypyrrole (PPy) deposited onto the electrode of the cellulosic. The morphology of the surface of the PPy/cellulosic films were studied by the cyclic voltammetry (CV), electrochemical impedance spectroscopy (SIE), Fourier-transform infrared spectroscopy (FTIR), profilometer apparatus, scanning microscope (SEM) copled with energy dispersive X-ray spectrometry (EDX) and X-ray diffraction (XRD). Then, from electrolysis experiments, their activity and selectivity for the nitrite electroreduction reaction will be evaluated under potentiostatic conditions.

**Keywords:** cellulosic substrate, pyrrole, nitrite reduction, Electrocatalyst.**1. Introduction**

Nitrite is also considered as a pollutant in the environment and there are many studies on sensing [4,13] or reducing it to less harmful/more useful chemicals [5–11]. Reduction of nitrite is not only beneficial for ammonia production but thus also helps reducing pollution. There are several reports on electrocatalytic reduction of nitrite to ammonia using enzymes [5], metals [4,7] or metal complexes [6,10–12] as the electrocatalysts. A few of those works involved the use of metal complexes with inorganic [6,10] or organic [8,9,11,14] macromolecules which is mainly inspired by the enzymatic system in nature. Some organic molecules [8,9,11,14] have been used for electrocatalytic reduction of nitrite to ammonia where nitrite reduction occurred at –0.8 V or below. There are still some practical issues arising from these systems e.g. some complexities in the electrocatalyst preparation, significant overpotentials are required or some unwanted products are formed (especially NH<sub>2</sub>OH) [6,8,9,11,14]. Conducting polymers (CPs) and their derivatives have been reported to electrochemically produce ammonia from various reactants such as nitrogen [15,16], nitrate or nitrite [5,6,10]. Often the electrocatalysts were prepared by forming metal complex with the CP [6,10]. In our laboratory, we have developed methods to incorporate a second electroactive component into CPs [17–18].

In the present study, we report the detailed analysis of nitrite reduction on Cu-Ppy/cellulosic electrodes investigated by cycling voltammetry (CV), galvanostatic and potentiostatic electrolyses performed under variable operating conditions (i.e.

different current were employde, different concentration of the nitrite solution) with the use of various analytical techniques.

## 2. Materials and methods

### 2.1. Materials

Pyrrole, Iron (III) chloride anhydrous ( $\text{FeCl}_3$ ), Ammonium persulfate ( $\text{H}_8\text{N}_2\text{O}_8\text{S}_2$ ), sodium chloride ( $\text{NaCl}$ ), chlorhydric acid ( $\text{HCl}$ ), copper (II) chloride ( $\text{CuCl}_2$ ), Sodium nitrate ( $\text{NaNO}_2$ ), sulfuric acid ( $\text{H}_2\text{SO}_4$ ) and cellulosic substrate.

### 2.2. Preparation of the synthesized PPy/cellulosic electrode

Deposition of pyrrole on cellulosic electrode has been carried out by oxidation of pyrrole in the presnce of cellulosic. Following the method described in the previous reports [19–20]. Briefly, the fabric samples (1.02g cellulosic) were immersed in  $\text{NaCl}$ , pyrrole and  $\text{HCl}$  ( $\text{PH}=1$ ) solution and kept at  $25\text{ }^\circ\text{C}$  for 15 min, followed by adding oxidant solution dropwise to initiate polymerization. the coated fabrics were then washed with water and dried at ambient temperature. The concentration of oxidant was 0.1M. Under variable operating conditions (i.e. different oxydant, different concentration of the Pyrole and different duration of the experiment). **Tab1,tab2,tab3**

**Table 1.** Influence of the oxydant on PPy/cellulosic electrodes synthesized

Electrode	Electrolyte	oxydant (s)
PPy/cellulosic	sodium chloride ( $\text{PH}=1$ )	Iron (III) chloride anhydrous
PPy/cellulosic	sodium chloride ( $\text{PH}=1$ )	Ammonium persulfate

**Table 2.** Influence of the concentration the Pyrole on PPy/cellulosic electrodes synthesized

Electrode	Electrolyte	concentration of the Pyrole (s)
PPy/cellulosic	sodium chloride ( $\text{PH}=1$ )	0.1M
PPy/cellulosic	sodium chloride ( $\text{PH}=1$ )	0.05M
PPy/cellulosic	sodium chloride ( $\text{PH}=1$ )	0.2M

**Table 3.** Influence of the Duration on PPy/cellulosic electrodes synthesized

Electrode	Electrolyte	Duration (min)	E ( $\mu\text{m}$ )
PPy/cellulosic	sodium chloride ( $\text{PH}=1$ )	15	35 $\mu\text{m}$
PPy/cellulosic	sodium chloride ( $\text{PH}=1$ )	120	95 $\mu\text{m}$
PPy/cellulosic	sodium chloride ( $\text{PH}=1$ )	180	73 $\mu\text{m}$

## 3. Results and discussion

3.1. Electrochemical reactivity and stability of the PPy/cellulosic electrode was tested by Cyclic voltamperometry (CV) and technique electrochemical impedance spectroscopy (SIE) experiments .

### 3.2. Electroreduction behavior of $\text{NO}_2^-$ on the CuO-PPy/cellulosic electrode

Electroreduction of nitrate was performed in 200 ml one-compartment electrolytic cell using a potentiostat/galvanostat (PGS-HH9). A three-electrode was used with a CuO-PPy/cellulosic working electrode with a  $2 \text{ cm}^2$  exposed area, platinum wire counter electrode and a saturated calomel electrode (SCE) was used as the reference electrode. Cyclic voltammetry (CV) experiments were conducted using 0.5 M sodium chloride (NaCl), solution as a supporting electrolyte. Sodium nitrite ( $\text{NaNO}_2$ ) solutions were prepared just prior to use (PH=7). The CV experiments were run with a potential scan rate of  $500 \text{ mV s}^{-1}$ .

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