## Abstract

 $Ni_{0.3}Co_{2.7}O_4$  oxide embedded Polypyrrole (Ppy) thin films were successfully synthesized over glassy carbon disk (GC) part of rotating ring disk electrode (RRDE) by two different electropolymerisation route i.e. galvanostatic (GS) and multi-cyclic voltammetry (CV), from a solution containing 0.1 mol L<sup>-1</sup> pyrrole (Py) and 7 g L<sup>-1</sup> of Ni<sub>0.3</sub>Co<sub>2.7</sub>O<sub>4</sub>. The Ppy electrodeposition signal has been studied by varying two parameters i.e. the oxide nanoparticles (NPs) presence and the preparation method. In addition, the obtained hybrid electrode morphology, stability especially in methanol presence, and electrocatalytic activity towards the oxygen reduction reaction (ORR) have been also investigated. The results reveal that the obtained films properties have been affected by the used route and furthermore by the oxide addition. The CV shape was maintained in the NPs presence, however the reversibility behavior was better, since the potential gap between doping/dedoping processes became closer and its corresponding currents are significantly risen. The two synthesis methods lead to hybrids electrodes with pronounced ORR electrocatalytic activity. Thus, the GS obtained material exhibits a weak selectivity regarding electrons exchanged number and the  $H_2O_2$  produced amount was only about 50%. However, these interesting properties were enhanced i.e. mainly 2 electrons were exchanged by producing 96% of H<sub>2</sub>O<sub>2</sub>, when the composite electrode was prepared by CV route. Moreover, this catalyst has good stability, as well as a higher tolerance to methanol for relative long time. Therefore, the CV method can be very useful on conducting polymer/NPs materials synthesis for environmental applications requiring peroxides use.