Journal of Environmental Chemical Engineering 4 (2016) 4330–4341

Contents lists available at ScienceDirect

Journal of Environmental Chemical Engineering

journal homepage: www.elsevier.com/locate/jece

Fabrication and performance of magnetite (Fe_3O_4) modified carbon paste electrode for the electrochemical detection of chlorite ions in aqueous medium

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ARTICLE INFO

Article history:
Received 19 August 2016
Received in revised form 25 September 2016
Accepted 30 September 2016
Available online 1 October 2016

Keywords:
ClO_2^-
Fe_3O_4 nanoparticles
Modified CPE
SWV
Voltammetric sensor

ABSTRACT

The magnetite nanoparticles (Fe_3O_4), synthesized by surfactant aided hydrolysis of the stoichiometric amounts of ferrous (Fe^{2+}) and ferric (Fe^{3+}) ions, were dispersed in carbon paste (CP) to fabricate magnetite modified carbon paste electrode (Fe_3O_4/CPE). The field emission scanning electron microscopy (FESEM) was used to evaluate the morphology of the synthesized powder in the pure phase and its distribution in the CP that revealed the well-dispersed Fe_3O_4 nanoparticles in the graphite sheet with a mean size of 10 nm. The microstructure analysis of the synthesized magnetite was performed by high-resolution transmission electron microscopy (HRTEM). The phase purity of the synthesized magnetite was evaluated by x-ray diffraction (XRD) analysis. After initial assessment of charge transport in the fabricated electrode by electrochemical impedance spectroscopy (EIS) that exhibited a substantial decrease of 87% in the charge transfer resistance, the suitability of the Fe_3O_4/CPE was assessed for the detection and determination of chlorite ion (ClO_2^-) in the aqueous medium. The modified CPE loaded with the optimized amount of Fe_3O_4 showed considerably enhanced oxidation current as compared to pure CPE for the oxidation of ClO_2^- and exhibited a near-reversible peak at ~+0.73 V in 0.1 M pH 7 phosphate buffers, at a scan rate of 50 mV/s. The optimum analytical conditions for the nanomolar detection of ClO_2^- by square wave voltammetry (SWV) were established. Likely interferences influencing the detection of ClO_2^- were also investigated. The excellent performance of the fabricated electrode was also established for the real tap and bottled water samples.

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1. Introduction

Among the metal oxide nanoparticles, Fe_3O_4 (magnetite) nanoparticles because of their catalytic properties, high surface area, and low mass transfer resistance have attracted an increased interest in fabrication of electrochemical sensors and biosensors [1–7]. On the other hand, the superior electrocatalytic activity, reproducibility, stability, and the effortless regeneration process of the electrode surface with the marked improvement in sensitivity, the modified carbon paste electrodes (CPEs) have become the most attracting electrode material for the electrochemical detection of various analytes [8–10]. Although, a number of nanosized metal oxides have been investigated however, the reports regarding the use of Fe_3O_4 nanoparticles as the modifiers for the CPE are rare [11–14]. Keeping in mind the catalytic and electrostatic properties of Fe_3O_4, we reasoned that CPE modified with Fe_3O_4 might be a good

Abbreviations: CP, carbon paste; CPE, carbon paste electrode; Magnetite, Fe_3O_4; Chlorite ion, ClO_2^-; FESEM, field emission scanning electron microscope; HRTEM, high resolution transmission electron microscope; XRD, X-ray diffraction; EDX, energy dispersive X-ray spectroscopy; SAED, selected area electron diffraction; EIS, electrochemical impedance spectroscopy; CV, cyclic voltammetry; SWV, square wave voltammetry; KOH, potassium hydroxide; PEEK, polyether ether ketone; SCE, saturated calomel electrode; GC, glassy carbon; Pt, platinum; GCE, glassy carbon electrode; IC, ion chromatography.

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http://dx.doi.org/10.1016/j.jece.2016.09.036
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candidate for electrochemical determination of disinfection by-products.

Due to ever-increasing use of disinfectant agents in water treatment, the detection of disinfectant associated by-products is of great concern to ensure the safety and quality of the drinking water. Being cost effective, efficient, and easy to handle, chlorine and its dioxide are the disinfectant of choice to control taste and odor of the drinking water [15–17]. However, the reduction of chlorine dioxide in water leads to the formation of chlorite ion (ClO_2^-) in drinking water that are hazardous to living beings [18]. It has been reported that the exposure to ClO_2^- cause methemoglobinemia in infants, a condition that results in the abnormal form of hemoglobin besides a number or disorders in adults [19,20]. Therefore, as per recommendations of World Health Organization (WHO) and the US environmental protection agency (EPA), the concentration of ClO_2^- in the in drinking water should not exceed above 0.2 mg/L [21,22].

ClO_2^- could be determined by chromatographic methods, flow injection methods, and capillary electrophoresis [23–27]. However, the expensive instrumentation, associated time-consuming labor, sample pretreatment and preparation, and lack of portability are the drawbacks that overcome the primary advantages of these analytical methods. Additionally, the sophisticated instrumentation associated restricts the use of these tools in the controlled environment only. Electrochemical detection is the most promising alternative approach for the direct determination of ClO_2^- and showed great promise in terms of sensitivity, selectivity, and reproducibility. The differential pulse polarographic and cyclic voltammetric determination of ClO_2^-, based on its reduction or oxidation behavior, at different electrodes such as dropping mercury electrode, tungsten electrode, graphite electrode, gold electrode, and platinized platinum microelectrode has been reported [28–36]. However, ClO_2^- at these electrodes was not determined satisfactorily and the obtained detection limit was quite high. Recently, picket-fence iron porphyrin based RGO nanocomposite (FeTMAPP/RGO) has been investigated for ClO_2^- determination [33]. Although, the method showed a linear detection range of 5.0×10^{-8}–1.2×10^{-4} with a detection limit of 2.4×10^{-8} M for the detection of ClO_2^- however, for continuous and long-term analysis, the elimination of amperometric response generated by other electroactive components in the sample matrix was not ensured.

In this work, we synthesized and characterized the Fe_3O_4 and used it as the modifier for the fabrication of CPE. The synthesis of Fe_3O_4 was carried out by a user friendly approach avoiding harsh and sensitive experimental conditions. The morphology and particle size was controlled by the use of surfactant. The synthesized magnetite after required characterization was dispersed in CP for the fabrication of modified electrodes. The amount of the dispersion was optimized for optimal response. The Fe_3O_4/CPE with the high electrocatalytic activity was used for the detection of ClO_2^- in the aqueous medium. Various parameters were investigated and results were correlated to establish the mechanism of oxidation/detection.

2. Experimental

Sodium chlorite ($NaClO_2$), ferrous chloride ($FeCl_2{\cdot}4H_2O$), ferric chloride ($FeCl_3{\cdot}9H_2O$), and potassium ferricyanide [$K_3Fe(CN)_6$] were obtained from Sigma-Aldrich. Graphite fine powder (99.9% purity) was purchased from Fluka. Paraffin oil (purity >99%) was purchased from Sigma-Aldrich. Aqueous solutions were prepared

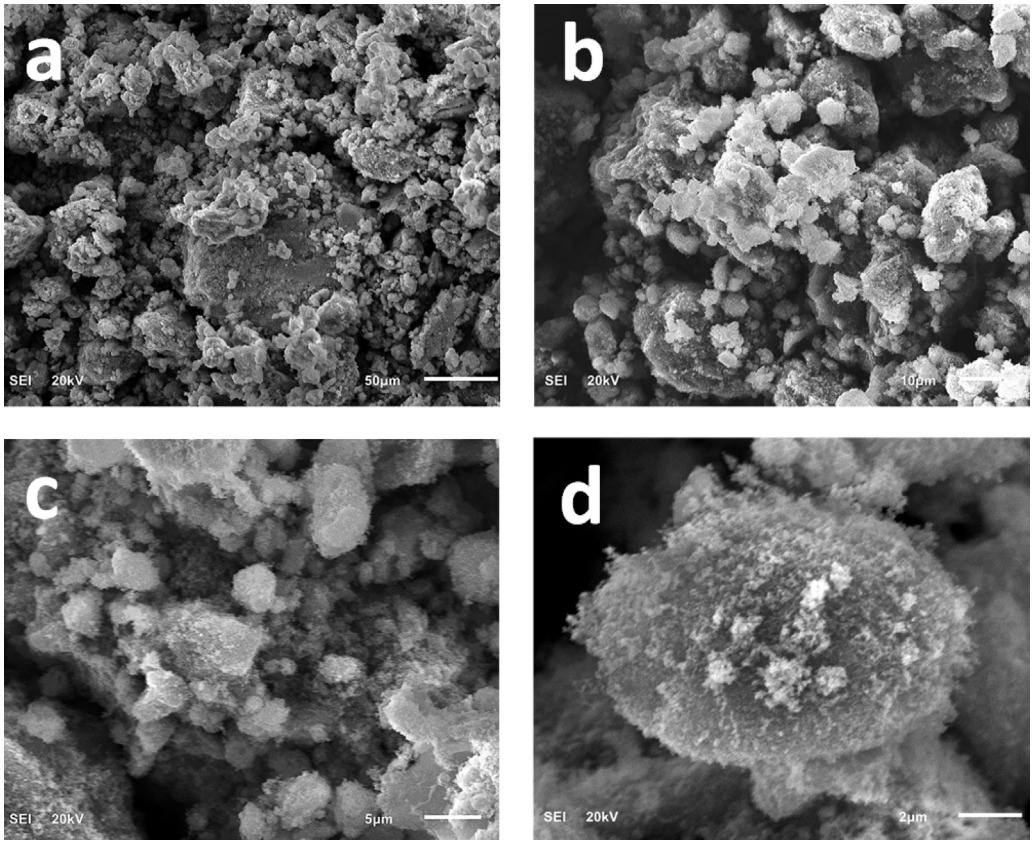

Fig. 1. The FESEM images of the synthesized magnetite (Fe_3O_4) at various resolutions.

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