Electrochemical Process of Water Disinfection by Fenton Reaction Using an Iron-Modified Carbon Paste Electrode Catalyst

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Abstract

Natural waters are exposed to significant risks of contamination by antibiotic-resistant bacteria, which pose significant environmental and health risks. Heterogeneous Fenton-type photocatalytic processes can ensure efficient pathogen removal, with advantages in terms of recycling, solid-liquid separation and by-product avoidance. In this study, an iron-modified carbon paste electrode (CPE) was proposed as catalyst. Iron atoms are deposited by electrodeposition of Fe^{2+} ions on the CPE surface. This ensures a more efficient synergistic Fenton-type photocatalytic reaction, generating additional hydroxyl radicals and enabling effective water disinfection. Electrochemical methods including Cyclic Voltammetry, Electrochemical Impedance Spectroscopy, Square Wave Voltammetry and Tafel lines were invested in this work.

Keywords: electrochemical methods; *Escherichia coli*; Fenton reaction; wastewater.

Introduction[•]

Access to reliable and affordable drinking water and sanitation services is an essential prerequisite for human survival. These services are essential to maintain healthy livelihoods and preserve dignity of all people. Rights to water and sanitation are essential to eradicate poverty and build peaceful and prosperous societies. Effective disinfection of water from contamination by pathogenic microorganisms requires efficient and simple ways to implement processes. Current conventional water disinfection process mainly involves chemical and physical techniques, including filtration, flocculation, ozonation, chlorination and ultraviolet (UV) irradiation. Unfortunately, these processes face challenges such

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[•] The abbreviations list is in page 392.

as high cost, complexity, and formation of mutagenic and carcinogenic by-products is unavoidable [1].

Fenton techniques offer an encouraging and promising solution to inhibit resistance of pathogenic microorganisms, due to their powerful oxidative capacity [2]. Fenton reactions produce hydroxyl radicals (•OH) [3]. Studies have shown that •OH free radicals attack bacterial cell membrane and cause irreparable damage to bacteria [4]. In literature, photocatalytic disinfection has been the subject of several research studies that have proven high efficiency in disinfection of several microorganisms, including bacteria, microalgae and parasites [5, 6]. Free radicals act in a non-specific manner and induce a profound alteration on the biofilm's structure [7].

Fenton reactions use metal surfaces that constitute active metal centres supported on semiconductors, they typically use iron-based catalysts, and have been the subject of a significant amount of research [8-11], due to their increased efficiency.

Experimental

Reagents and apparatus

All employed chemicals were of analytical grade and employed without further purification. Solutions were prepared using double-distilled water. Ethanol of 97% purity was sourced from Sigma-Aldrich (USA), while commercial graphite powder was obtained from France (Carbone, Lorraine, ref 9900). Electrochemical measurements were conducted using a VoltaLab potentiostat (model PGSTAT 100, Ecochemie B.V., Utrecht, The Netherlands) controlled by VoltaLab Master 4 software run under Windows 2007. Electrochemical cell configuration comprised carbon paste electrode (CPE), platinum plate and saturated calomel as working, counter and reference electrodes. CPE were fabricated by blending high purity graphite carbon powder with paraffin oil, forming the mixture which was then loaded into the electrode cavity. Electrical connection was facilitated by a carbon rod. Modification of the basic CPE electrode by iron was investigated by electrodeposition in an electrolytic solution containing Fe^{2+} ions, at a potential of approximately 0.5 V, for 5 min.

Results and discussion

Fig. 1 shows cyclic voltammograms (CV) recorded at CPE (curve a) and Fe/CPE (curve b) electrodes surface, respectively.

Modification of basic CPE by electrodeposition of iron manifested by a remarkable change in CV shape, which presents two redox peaks: one in direction of anodic scan towards about -0.5 V; and the other at 0 V in direction of cathodic scan. The two peaks are almost opposite, which means that it is a reversible redox system.

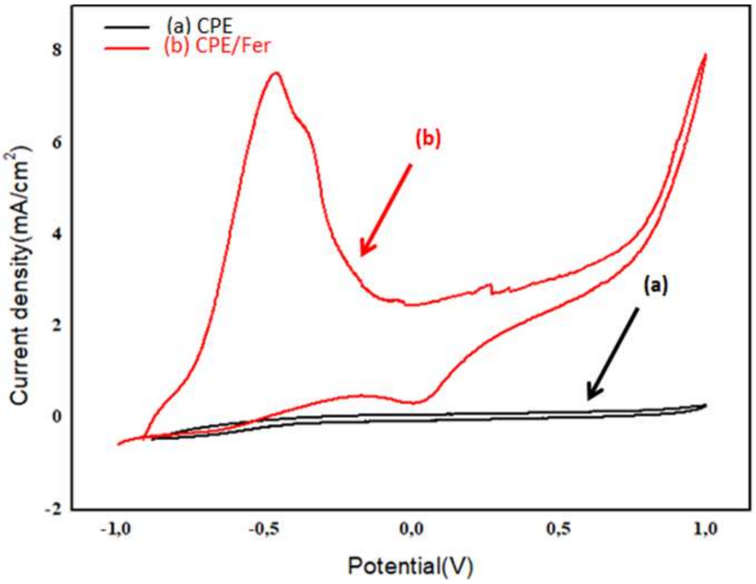

Figure 1: CV recorded in a 0.5 M NaCl medium, at CPE (curve a) and Fe/CPE (curve b), at a scan speed of 100 mV/s.

Fig. 2 illustrates electrochemical impedance diagrams recorded on CPE (curve a) and Fe/CPE (curve b) electrodes surface, in a 0.5 M NaCl electrolytic medium. Both diagrams have the shape of half loops, of which diameter corresponds to electron transfer resistance. This resistance dropped remarkably in the presence of Fe within CPE matrix.

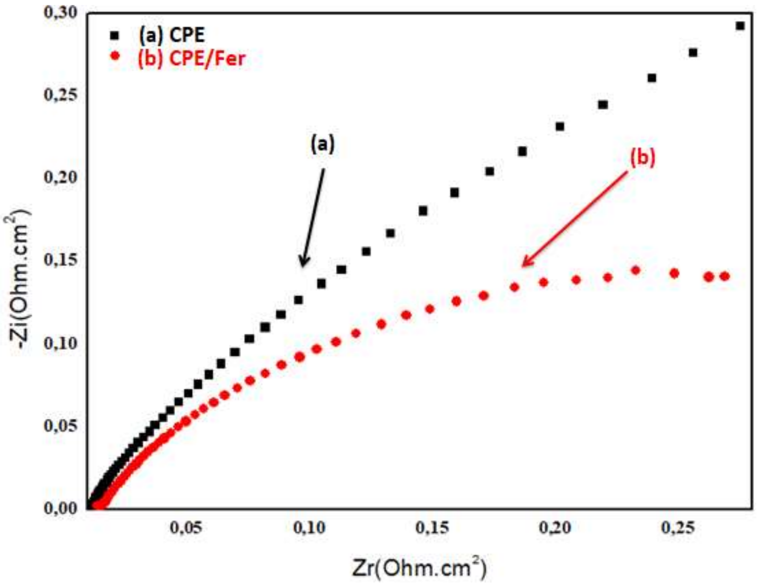

Figure 2: EIS recorded in a 0.5 M NaCl medium, respectively, at CPE (curve a) and Fe/CPE (curve b).

Fig. 3 shows Tafel lines recorded at CPE (curve a) and Fe-CPE surfaces, respectively, prepared by electrodeposition. Both electrodes show diffusion plateaus, indicating mass transport limitations in the reaction. Also, equilibrium potential (E_{eq}) of Fe/CPE is lower than that of plain CPE. The presence of diffusion plateaus on both electrodes suggests that, at higher overpotentials, reaction rate becomes limited by reactants diffusion to the electrode surface rather than by charge transfer kinetics. Lower E_{eq} on Fe/CPE indicates that: iron modification has made the electrode more active for the reaction; electron transfer process requires less energy (overpotential) to occur; and Fe sites likely provide better catalytic activity compared to plain CPE.

Fe modification has likely created active sites on the electrode surface, enhanced its electrocatalytic properties and improved its performance for the specific reaction being studied.

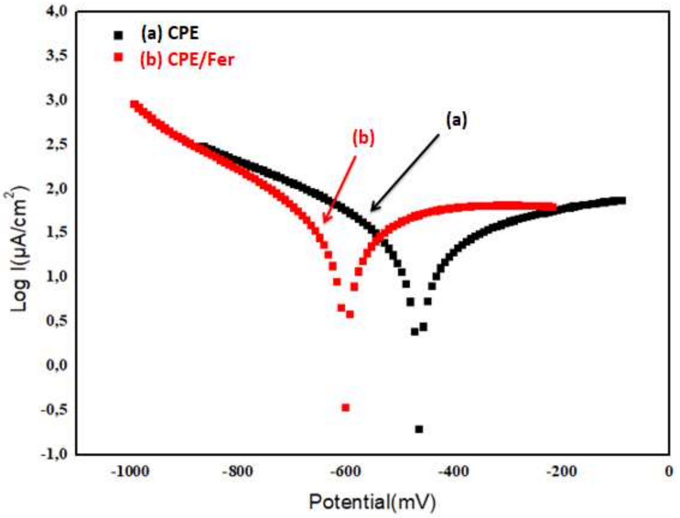

Figure 3: Tafel lines recorded at CPE (curve a) and Fe/CPE (curve b) electrodes surface, in a 0.5 M NaCl 0.5 mol/L solution.

Reduction in hydrogen peroxide at CPE surface manifested by remarkable reduction in cathodic J values, in potential range from -1 to 0 V (Fig. 4).

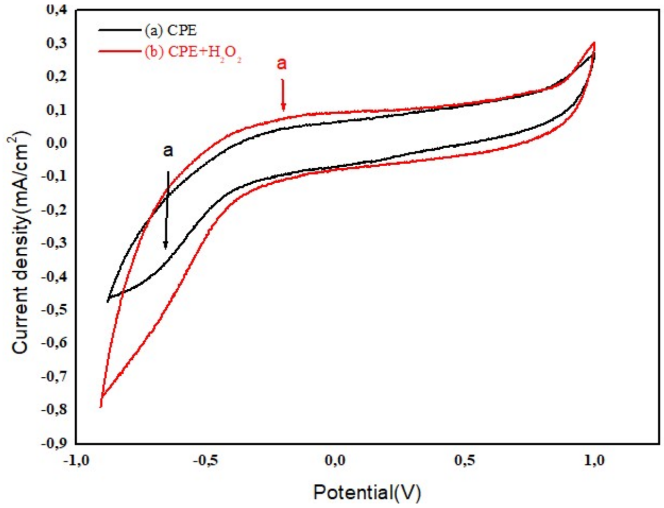

Figure 4: CV recorded at CPE- in blank 0.5 M NaCl (curve a) and with H2O2 (curve b), at a scan speed of 100 mV/s.

Reduction in H_2O_2 on Fe/CPE surface results in disappearance of Fe redox peaks of the CV (Fig. 5).

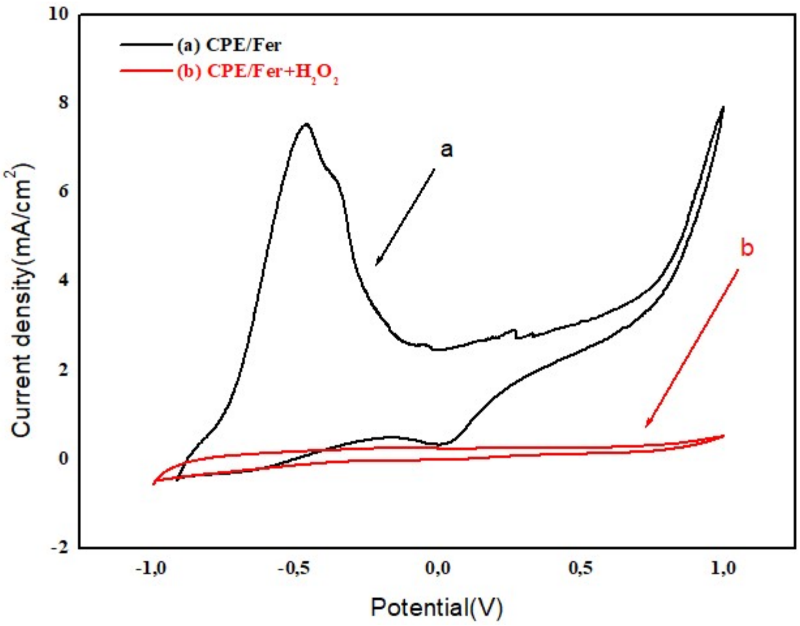

Figure 5: CV recorded at CPE in blank 0.5 M NaCl (curve a) and with H_2O_2 (curve b), at a scan speed of 100 mV/s.

Classic Fenton process involves a reaction between dissolved Fe^{2+} and H_2O_2. Generation of $^{\bullet}OH$ is most effective at the acidic medium [12]:

$$Fe^{2+} + H_2O_2 \rightarrow FeOH^{2+} + {}^{\bullet}OH \text{ (and/or } FeO_2^{+} + H_2O) \qquad (1)$$

Fig. 6 illustrates CV recorded at the basic CPE surface in a 0.1 M NaCl solution with: Fe^{2+} and H_2O_2; H_2O_2; and Fe^{2+}, respectively.

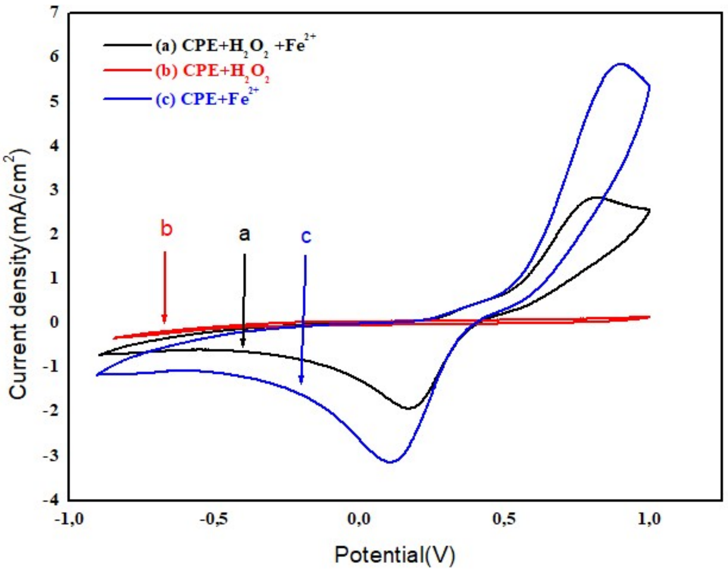

Figure 6: CV recorded at CPE in- 0.5 M NaCl with Fe^{2+} and H_2O_2 (curve a), 0.5 M NaCl with H_2O_2 (curve b), and 0.1 M NaCl with Fe^{2+}(curve c), at a scan speed of 100 mV/s.

The presence of Fe^{2+} ions in the electrolytic medium caused a slight shift in H_20_2 reduction peak from 0.1 to 0.2 V. While Fe oxidation peak kept its position in stable potential, J values decreased (Fig. 7).

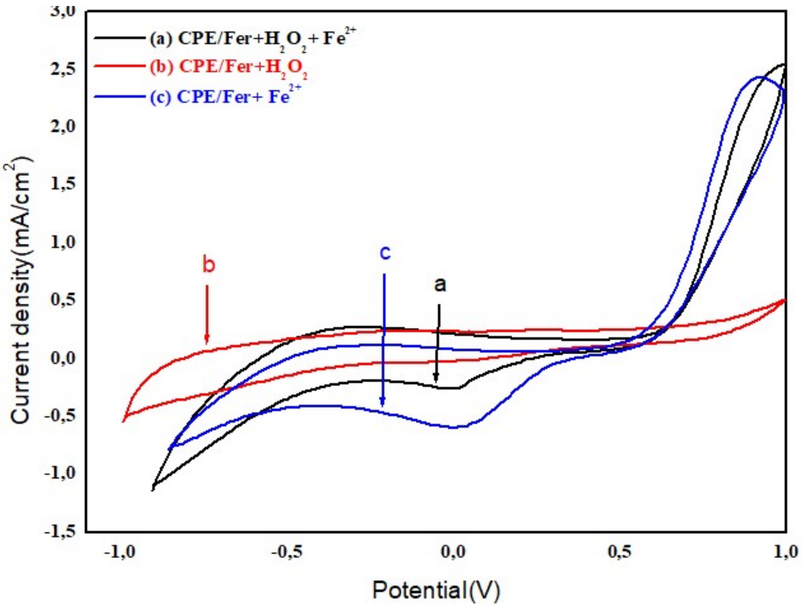

Figure 7: CV recorded at Fe/CPE in 0.5 M NaCl with Fe^{2+} and H_2O_2 (curve a), 0.5 M NaCl with H_2O_2 (curve b), and 0.1 M NaCl with Fe^{2+} (curve c), at a scan speed of 100 mV/s.

On the contrary, at the Fe/CPE surface, reduction of H_2O_2 was activated by Fenton reaction, and it shows on the CV by a sudden drop in current densities in potential range from -1 to -0.5 V (Fig. 8).

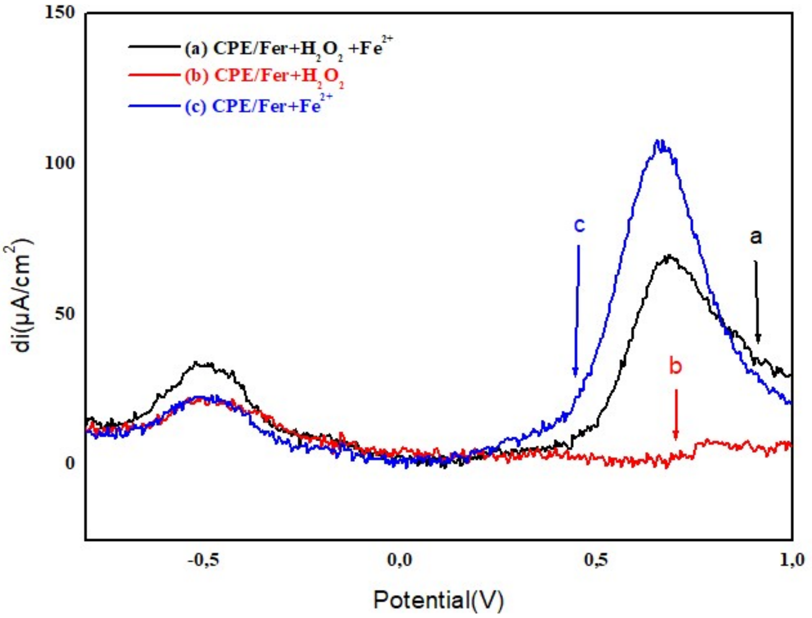

Figure 8: SWV recorded at CPE-Fe in 0.5 M NaCl with Fe^{2+} and H_2O_2 (curve a), 0.5 M NaCl with H_2O_2 (curve b), and 0.1 M NaCl with Fe^{2+} (curve c).

Results of electrochemical parameters deduced from Tafel lines recorded for two CPE and Fe-CV in different media are grouped in Table 1:

Table 1: Electrochemical parameters.

Electrode	**Medium**	**E_{Eq} mV**	**JEq (μA/cm^2)**
CPE	0.5 M NaCl	-605.5	63.35
CPE	0.5 M NaCl + Fe^{2+}	202.4	112
CPE	0.5 M NaCl + H_2O_2	-410	49.57
CPE	0.5 M NaCl + H_2O_2 + Fe^{2+}	-253	41.83
CPE	0.5 M NaCl + bacteria	-672.6	35.62
CPE	0.5 M NaCl + H_2O_2 + Fe^{2+} + bacteria	-876.7	1.04
Fe-CPE	0.5 M NaCl	-597.2	49.22
Fe-CPE	0.5 M NaCl + Fe^{2+}	-527.1	80.55
Fe-CPE	0.5 M NaCl + H_2O_2	-953.4	172.97
Fe-CPE	0.5 M NaCl + H_2O_2 + Fe^{2+}	-631.6	0.09
Fe-CPE	0.5 M NaCl + bacteria	-687.9	91
Fe-CPE	0.5 M NaCl + H_2O_2 + Fe^{2+} + bacteria	-876.7	1.04

Interpretations of obtained results are as follows: for base electrolyte (0.5 M NaCl), E_{eq} values were relatively similar (-605.5 for CPE *vs.* -597.2 mV for Fe-CPE), indicating that iron modification did not significantly alter the electrode's basic electrochemical behaviour in the supporting electrolyte. However, exchange J value was slightly lower for Fe-CPE (49.22 *vs.* 63.35 μA/cm^2), suggesting a modest decrease in electron transfer kinetics.

Concerning the effect of Fe^{2+} addition, a striking difference was observed in E_{eq} values between CPE (202.4 mV) and Fe-CPE (-527.1 mV). This significant shift suggests that Fe-CPE has a different interaction mechanism with Fe^{2+} ions, as it maintained a more reducing character, even in presence of Fe^{2+}. Exchange J values indicated better kinetics for CPE (112 μA/cm^2) compared to Fe-CPE (80.55 μA/cm^2).

For H_2O_2 response, Fe-CPE shows remarkably different behaviour with H_2O_2, much more negative E_{eq} values (-953.4 *vs.* -410 mV for CPE); and significantly higher exchange J value (172.97 *vs.* 49.57 μA/cm^2). This suggests that Fe modification substantially enhanced the electrode's electrocatalytic activity toward H_2O_2 reduction.

On the combined effect of H_2O_2 and Fe^{2+}, an interesting phenomenon occurred: CPE maintained moderate activity (41.83 μA/cm^2) and Fe-CPE showed dramatically reduced J value (0.09 μA/cm^2). This suggests possible competitive interactions or surface blocking effects on Fe-CPE when both species are present.

For bacterial interaction, Fe-CPE demonstrated superior performance with bacteria: similar E_{eq} values (-672.6 *vs.* -687.9 mV) and higher exchange J value for Fe-CPE (91 *vs.* 35.62 μA/cm^2). This indicates better bacterial interaction with Fe/CPE surface.

On complete system Fenton reaction effect (H_2O_2 + Fe^{2+} + bacteria), notably, both electrodes converged to identical E_{eq} (-876.7 mV) and exchange J values

(1.04 μA/cm²). This suggests that, in the complete system, surface modification became less significant, possibly due to complex interactions between all components.

Conclusions

In this work, disinfection power of Fenton reaction using CPE and Fe-CPE was evaluated. The research first focused on complete electrochemical characterization of elaborated catalysts using CV, SWV, EIS and Tafel lines. Results indicate that structural advantages of Fe-CPE provide larger adsorption sites for H_2O_2, thus improving electron transport efficiency and reducing electron transport distance.

Fe-CPE demonstrated enhanced electrocatalytic activity towards H_2O_2 reduction, when H_2O_2 is the sole additive. The presence of Fe^{2+} significantly affected the electrode behaviour, particularly for Fe-CPE, which showed better bacterial interaction. Complex system behaviour suggests possible surface blocking effects or competitive interactions. Fe's modification benefits are most pronounced in single-component systems rather than in complex mixtures.

These results indicate that Fe modification significantly alters the electrode's electrochemical properties, but its benefits are highly dependent on the specific composition of the electrolyte solution.

Authors' contributions

M. Enasraouy: conceptualization; experimental; investigation; original draft preparation and writing; review and editing; data analysis. **M. Oukbab**: conceptualization; investigation; original draft preparation and writing; review and editing; data analysis. **M. Oubaouz**: experimental investigation. **R. Hnini:** review and editing. **N. Ferdous**: data analysis. **R. Najih**: methodology; review and editing. **A. Chtaini**: conceptualization; methodology; original draft preparation and writing; review and editing; supervision.

Abbreviations

CPE: carbon paste electrode
CV: cyclic voltammetry
E_{eq}: equilibrium potential
EIS: electrochemical impedance spectroscopy
Fe/CPE: iron-modified carbon paste electrode
J: current density
J_{eq}: equilibrium current density
NaCl: sodium chloride
SWV: square wave voltammetry

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