Data Article

Experimental data on the degradation of caffeine by photo-electro-fenton using BDD electrodes at pilot plant

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A B S T R A C T

Emerging contaminants (EC) are an imminent risk due to potential toxicity to aquatic ecosystems and human beings. This type of contaminants is found in low concentrations and usually present incomplete or inefficient removal by conventional treatments, which entail its permanence and constant increase. Advanced Oxidation Processes (AOP) are an alternative for the elimination of dangerous and resistant substances in wastewater. So, this research evaluates the caffeine degradation in aqueous solution by AOP, such as: Fenton, Electro-Oxidation (EO) with boron doped diamond (BDD) electrodes, Electro-Fenton (EF) and Photo-Electro-Fenton (PEF). The influences of pH, concentration of the supporting electrolyte and specific electric charge were investigated using a Taguchi's factorial design, which allowed to identify the contribution of each variable in the process. The data obtained in this work can be useful for scaling process and cost analysis because it provide the information at pilot plant scale.

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The data obtained show that PEF process is better method to the caffeine degrade in aqueous solution. Data may be useful for scaling process and cost analysis.

1. Data

This brief data set describes the use of the PEF process for caffeine degradation present in water. Table 1 shows the physical and chemical properties of caffeine, Table 2 shows the experimental factors and assigned values in each level, Table 3 contains the statistical design matrix and the Fig. 1 presents a photography of the reactor.

Table 1
Physicochemical properties of caffeine [1,2].

| Molecular structure |
|---------------------|
| ![Molecular structure](image) |

| Molecular formula | C8H10N4O2 |
|-------------------|-----------|
| Molecular weight (g mol⁻¹) | 194.194 |
| Melting point (°C) | 236.2 |
| Water solubility (mg L⁻¹ at 25 °C) | 21600 |
| pKa (at 25 °C) | 14.0 |
The verification of the anodic oxidation potential (cyclic voltammogram) of caffeine and of the supporting electrolyte is illustrated in Fig. 2, the behavior of the supporting electrolyte, caffeine, salt Mohr, hydrogen peroxide and the mixing of all when the anodic differential pulse technique is applied is shown in Fig. 3, the variation of the caffeine concentration for specific electric charge of 2 and 3 A h L⁻¹ can be seen in Figs. 4 and 5 respectively; alike, the profile of caffeine degradation by

| Table 2 |
| Factors and levels of the experimental design [1,2]. |

| Factors                                    | Levels |
|-------------------------------------------|--------|
|                                           | Low    | High   |
| pH                                        | 2.8    | 3      |
| Concentration of supporting electrolyte (mM) | 17     | 20     |
| Specific electrical load (A h L⁻¹)        | 2      | 3      |

| Table 3 |
| Taguchi design matrix. |

| Specific electrical charge (A h L⁻¹) | pH  | Concentration of supporting electrolyte (Na₂SO₄ in mM) |
|-------------------------------------|-----|-------------------------------------------------------|
| 2                                   | 2.8 | 17                                                    |
| 2                                   | 2.8 | 17                                                    |
| 2                                   | 2.8 | 20                                                    |
| 2                                   | 3   | 20                                                    |
| 2                                   | 3   | 17                                                    |
| 2                                   | 3   | 20                                                    |
| 3                                   | 2.8 | 20                                                    |
| 3                                   | 2.8 | 17                                                    |
| 3                                   | 3   | 17                                                    |
| 3                                   | 3   | 20                                                    |
| 3                                   | 3   | 17                                                    |

**Fig. 1.** Photography of the pilot plant.
Fig. 2. Cyclic voltammogram of caffeine at two concentrations and of the electrolyte support with a scanning speed was 100 mV s\(^{-1}\) and pH = 3.

Fig. 3. Anodic differential pulse technique for each of the chemical species in solution and of the mixture with a scanning speed of 100 mV s\(^{-1}\) and pH = 3.

Fig. 4. Degradation of caffeine (\(C_{\text{AO}} = 30 \text{ mg L}^{-1}\)) by PEF process with specific electric charge \(Q = 2 \text{ (A h L}^{-1})\) at different pH values and concentration of electrolytic support.
Fig. 5. Degradation of caffeine ($C_{A0} = 30 \text{ mg L}^{-1}$) by PEF process with specific electric charge $Q = 3 \text{ (A h L}^{-1})$ at different pH values and concentration of electrolytic support.

Fig. 6. Degradation of caffeine by Photolysis (PO) and oxidative methods: electro-oxidation (EO) and Fenton (F), and combined Photo-Fenton (PF), Electro-Fenton (EF) and Photo-Electro-Fenton (PEF).

Table 4

| TOC$_{initial}$ $(\text{ppm})$ | COD$_{initial}$ $(\text{ppm})$ | pH | [Na$_2$SO$_4$] $(\text{mM})$ | % TOC | % COD |
|-------------------------------|-------------------------------|----|-----------------|------|------|
| 18.45                         | 21.07                         | 3.0 | 17              | 38.43 | 38.46 |
| 18.17                         | 25.66                         | 3.0 | 20              | 56.01 | 77.27 |

Conditions that present better degradation with specific electric charge $Q = 2$.

Conditions that have better degradation with specific electric charge $Q = 3$.
- 15.29 22.69 2.8 17 64.72 64.29
- 14.70 21.07 3.0 17 71.41 76.92
each oxidative process are show in Fig. 6. Also, the variation of the total organic carbon (TOC) and the chemical oxygen demand (COD) under the best conditions are included in Table 4.

2. Experimental design, materials and methods

For the development of the experimental section of this work was used the Taguchi factorial design [3]. This design uses an orthogonal arrangement which recognizes that not all the causative factors of variability can be controlled [1]. From this matrix the best design parameters for the description of the initial stage of the caffeine degradation by PEF were found.

A through analytical techniques such as cyclic voltammetry and differential pulse the oxidation potential (on BDD electrodes) for the chemical species present in solution (Sodium sulfate, Na₂SO₄, hydrogen peroxide, H₂O₂, Caffeine and Fe²⁺ contained in Mohr’s salt) were corroborate with the reported literature. For these measurements, it was used the potentiostat Gamry (galvanostat series G750).

Then tests were performed in reactor with BDD electrodes taking into account the minimum oxidation value of each species, and the production potential of hydroxyl radical. In addition, it was verified the synergy of the oxidative methods (PO, EO, Fenton, FF and PEF) with respect PEF.

On the other hand, the caffeine concentration was quantified using a UV–vis spectrophotometer (SHIMADZU) at a wavelength of 273 nm, which was found with the spectral sweep. The test that showed the best degradation were subjected to two types of analysis more, chemical oxygen demand (COD), which we determined by the open reflux method [4] and mineralization through measurements of total organic carbon (TOC) in a TOC-V CPH SHIMADZU analyzer [5].

Also, the generation of H₂O₂ was verified by UV–vis spectrophotometry at a wavelength of 408 nm. This measurement methodology is based on the photoelectric quantification of the color intensity of the H₂O₂ solutions treated with titanium sulfate reagent, which produced a yellow color in the reaction, this color is due to the formation of per titanic acid (H₂TiO₄). For this experimental process, 1 mL of sample and 1 mL of titanium sulfate reagent were added to 10 mL graduated balloon and completed with distilled water up to its capacity. After of 5 min, the measurement of the solution in the spectrophotometer was performed [6].

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Transparency document. Supplementary material

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