Data Article

Experimental data on the photoelectrochemical oxidation of phenol: Analysis of pH, potential and initial concentration

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ABSTRACT

The data collected in the present work correspond to percentages of phenol degradation by means of photoelectrochemical oxidation (PEC). Also, the information related to the energetic and kinetic performance of this advanced oxidation process (AOPs) is shown. The tests were divided into two stages: 1. Supporting electrolytes tests to determine the electrolyte that presents a better response to photocurrent and 2. Degradation of phenol to obtain the adequate conditions for the elimination of the contaminant. A central rotary composite design with uniform precision at two levels was used to analyze the influence of the initial pH, electrode potential and the initial concentration of substrate. Finally, with all the data obtained, calculation of degradation rates and the electrical energy per order EEO were made.

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1. Data

1.1. Supporting electrolytes tests

Fig. 1a and Fig. 1b show a voltammogram and a chronoamperometry of 0.1 M of Na$_2$SO$_4$ and NaCl as solution electrolytes in the absence and presence of light.

All the data reported for the following tests were obtained using Na$_2$SO$_4$ as supporting electrolyte.

Fig. 2 and Fig. 3 show the voltammetry and chronoamperometry essays of the different concentrations of phenol in presence and absence of 0.1 M of Na$_2$SO$_4$.

1.2. Degradation of phenol

Table 1 shows the degradation percentage of phenol by the photoelectrochemical oxidation along with the conditions used.

With all the data obtained, the analysis of variance shown in Table 2 was made to identify the influence of the variables in the elimination of the pollutant.

The Pareto chart in Fig. 4 evidence the importance of the variables in the elimination of the pollutant.

The residual graph in Fig. 5 shows the change in variability according to the fluctuations of the predicted value of pollutant degradation by the regression model.

In the surface response shown in Fig. 6, the optimal conditions to achieve the highest degradation percentage of the contaminant are observed.
1.3. Kinetics of photolysis, photocatalysis, and electrochemical oxidation in the degradation of phenol

The phenol degradation by Photolysis (PT), Photocatalysis (PC), Electrochemical oxidation (EO), Photocatalysis + Electrochemical oxidation (PC + EO) by separate and photoelectrochemical oxidation (PEC) well fit a zero-order kinetics. The adjustments were plotted in Fig. 7 and the zero rate constants for all the treatments are shown in Table 3.
### Table 1
Phenol degradation percentage and operating conditions.

| Test | pH | $C_0$ [mg/L] | $\phi_a$ [V] | Anodic potential | Degradation (%) | $Q$ [C/L] | $J_{avg}$ (mA/cm²) | Average current density |
|------|----|-------------|-------------|-----------------|-----------------|-----------|-------------------|------------------------|
| 1    | 5  | 10          | 0.80        | 57.14           | 131.7           | 0.088     |                   |                        |
| 2    | 5  | 25          | 0.80        | 34.95           | 116.3           | 0.078     |                   |                        |
| 3    | 5  | 10          | 1.60        | 73.76           | 214.5           | 0.143     |                   |                        |
| 4    | 5  | 25          | 1.60        | 33.11           | 159.7           | 0.106     |                   |                        |
| 5    | 9  | 10          | 0.80        | 31.12           | 125.9           | 0.084     |                   |                        |
| 6    | 9  | 25          | 0.80        | 20.72           | 104.5           | 0.070     |                   |                        |
| 7    | 9  | 10          | 1.60        | 53.94           | 212.7           | 0.142     |                   |                        |
| 8    | 9  | 25          | 1.60        | 23.25           | 198.6           | 0.132     |                   |                        |
| 9    | 3.64 | 17.5        | 1.20        | 74.58           | 154.7           | 0.103     |                   |                        |
| 10   | 7  | 17.5        | 0.53        | 21.44           | 92.5            | 0.062     |                   |                        |
| 11   | 7  | 4.89        | 1.20        | 72.95           | 185.5           | 0.124     |                   |                        |
| 12   | 7  | 17.5        | 1.20        | 31.06           | 155.0           | 0.103     |                   |                        |
| 13   | 7  | 30.1        | 1.20        | 24.55           | 164.9           | 0.110     |                   |                        |
| 14   | 7  | 17.5        | 1.87        | 42.35           | 210.6           | 0.140     |                   |                        |
| 15   | 10.36 | 17.5   | 1.20        | 29.08           | 164.8           | 0.110     |                   |                        |

### Table 2
Analysis of variance for the photoelectrochemical degradation of phenol.

| Source       | Sum of Squares | DF  | Mean Square | F-Ratio | P-Value |
|--------------|----------------|-----|-------------|---------|---------|
| A: pH        | 1570.13        | 1   | 1570.13     | 56.86   | 0.0006  |
| B: Concentration | 2514.85        | 1   | 2514.85     | 91.07   | 0.0002  |
| C: Potential | 415.095        | 1   | 415.095     | 15.03   | 0.0117  |
| AA           | 238.106        | 1   | 238.106     | 8.62    | 0.0324  |
| AB           | 59.1328        | 1   | 59.1328     | 2.14    | 0.2033  |
| AC           | 13.9656        | 1   | 13.9656     | 0.51    | 0.5088  |
| BB           | 162.516        | 1   | 162.516     | 5.88    | 0.0597  |
| BC           | 187.695        | 1   | 187.695     | 6.80    | 0.0478  |
| CC           | 3.73075        | 1   | 3.73075     | 0.14    | 0.7283  |
| Total error  | 138.079        | 5   | 27.6158     |         |         |
| Total (corr.)| 5462.06        | 14  |             |         |         |

![Fig. 4. Standardized Pareto chart for degradation.](image-url)
1.4. Electrical energy per order (EEO)

Table 4 shows the specific energy consumption by separate of the different technologies that are integrated into the photoelectrochemical oxidation technique.

2. Experimental design, materials and methods

2.1. Experimental procedure

The electrochemical cell was elaborated in acrylic, considering the dimensions of the light source and the electrodes. Those were fixed in the cell without affecting its active area (back (SE)
illumination), which allowed carrying out experiments under stable conditions in terms of the geometric variables that may affect the mass transfer in the process. The contaminant solution was stirred during the tests with the aim to decrease the limitations associated with the diffusion phenomena. The scheme can be appreciated in Fig. 8.

All the solutions were prepared with deionized water type II prior to the experiments. An aliquot of the contaminant solution was taken and diluted in the cell until reaching the initial phenol concentration needed. Three values of pH were chosen for the tests (pH 5, 7 and 9) to evaluate the behavior of the Photoelectrochemical process in all the range of the pH scale. The initial pH of the solution was adjusted with $\text{0.5 N of NaOH or H}_2\text{SO}_4$ when it was needed. The LED's were turned on 15 minutes previous to the experiments to stabilize the emission of photons, then the LED’s power was verified to assure the same amount of radiation in all the tests.

The work potentials were supplied by means of the potentiostat/galvanostat shown in Tables 6 and 7 and Fig. 8. For each test, 0.1 M of Na$_2$SO$_4$ was used as supporting electrolyte, 60 ml of solution were prepared for the treatment, and samples of 1.5 ml were taken each 40 minutes during 2 hours. Later, the curve was adjusted following a first order kinetics model for each treatment.

![Graph showing phenol degradation kinetics for different processes](image)

**Table 3**
Rate constants of phenol degradation by the different processes and correlation coefficients.

| Treatment            | Rate constant ($\text{mM} \times \text{min}^{-1}$) | $R^2$     | Phenol degradation (%) |
|----------------------|--------------------------------------------------|-----------|------------------------|
| PEC                  | $5.525 \times 10^{-4}$                           | 0.9991    | 73.76                  |
| PC + EO              | $9.350 \times 10^{-5}$                           | 0.9912    | 11.60                  |
| EO                   | $5.440 \times 10^{-5}$                           | 0.9826    | 6.79                   |
| PC                   | $3.995 \times 10^{-5}$                           | 0.9978    | 4.81                   |
| PT                   | $1.232 \times 10^{-5}$                           | 0.9402    | 1.42                   |

**Table 4**
Energy consumption of the evaluated processes.

| Treatment                  | Cell current ($\text{mA}$) | Cell potential (V) | Nominal LEDs potential ($\text{kW}$) | EEO ($\text{kWh/L}$) |
|----------------------------|-----------------------------|--------------------|-------------------------------------|---------------------|
| Photoelectrochemical Oxidation | 1.70                        | 1.84               | $5.72 \times 10^{-3}$               | 599.8               |
| Photocatalysis + Anodic Oxidation | 0.09                       | 1.60               | $5.72 \times 10^{-3}$               | 11923.8             |
| Anodic Oxidation           | 0.09                        | 1.60               | $5.72 \times 10^{-3}$               | 8367.7              |
| Photocatalysis             | –                           | –                  | $5.72 \times 10^{-3}$               | 3732.6              |
| Photolysis                 | –                           | –                  | $5.72 \times 10^{-3}$               | 32075.6             |
they were diluted 6.66 times (1.5 ml:10 ml) using deionized water type II and the concentration of phenol was determined by the direct photometric method using 4-aminoantipyrine, the absorbance measurements were made at 510 nm as it is described in the ASTM D1783 standard method [1].

The operating variables that were fixed for the development of the experimental design were:

- supporting electrolyte concentration ($0.1 \text{ M} \text{Na}_2\text{SO}_4$)
- reaction volume (60 ml)
- stirring velocity (600 rpm)
- temperature ($28 \pm 2$)$^\circ\text{C}$
- reaction time (2 hours)

The degradation percentage was the response variable, which is calculated as follows.

$$\%\text{ phenol degradation} = \frac{C_0 - C}{C_0} \times 100$$

Where $C_0$ is the initial concentration of phenol and $C$ is the final concentration of the pollutant.

A photolysis test (without a working potential and photo-anodes), an electrochemical oxidation test (in the absence of light), a photocatalysis test (without working potential) and a photocatalysis + anodic oxidation by separate, were also carried out with the aim to obtain the degradation percentages of phenol in each one of the techniques mentioned. The data well fitted a zero-order kinetic that is described in the equation (2).

$$C_0 - C = kt$$

This was made with the aim to determine if there is a synergistic effect of the different processes in the photoelectrochemical oxidation of phenol.

The voltammetric tests were carried out with potential barriers from 0 to 2.2 V (50 mV/s) and the chronoamperometries at a potential of 1.2 V. Through these techniques, the effect of the supporting electrolyte and the concentration of phenol on the current generated in the system in the absence and
presence of light was elucidated. All the measurements reported take as reference the \( \text{Ag/AgCl} \) electrode and were made at pH 5.

2.2. Reagents and equipment

Table 5 and Table 6 show all the reagents, materials and equipment used to carry out this work. All the chemical compounds were used as received without further purification. NOMAD (Canadian research group) donated the \( \text{FTO/TiO}_2 \) – nanoparticles electrodes employed. The elaboration methodology is described in detail in [2]. The active area of each electrode was \( 2.5 \text{ cm} \times 2.5 \text{ cm} \) (thickness 3 mm) and the measurements of the platinum electrode used were \( 1.1 \text{ cm} \times 1.65 \text{ cm} \) (sheet) and \( 1.8 \text{ cm} \times 0.1 \text{ cm} \) (wire).

2.3. Experimental design

Through a central rotary composite design with uniform precision at two levels, the data for the photoelectrochemical oxidation of phenol was obtained. All the tests were in function of the initial pH, the initial concentration of contaminant and potential applied. In the development of this work, 20 experimental runs were made (8 factorial points, 6 axial points and 1 central point replicated by 5 times). The tests were carried out in a random order with the aim to avoid systematic errors. For the analysis of the data, some statistics software such as Statgraphics Centurion XV and Minitab 17 were used. Table 7 summarizes the operating levels of the experimental design.

2.4. Electrical energy per order (EEO)

With the aim to calculate the energy consumption of the treatment evaluated, the figure of merit proposed by the international union of pure and applied chemistry (IUPAC) was used. The equation that best fits a zero-order kinetics in advanced oxidation processes is presented as follows:

### Table 5
Reagents and materials used in the tests.

| Material (Purity)                       | Brand             | Application |
|-----------------------------------------|-------------------|-------------|
| Sulfuric acid (95–98%)                  | Fisher Scientific | Tests       |
| Deionized water type II                 |                   | Tests       |
| FTQ/TiO\(_2\) - np electrodes           | Fabricated        | Tests       |
| Platinum electrodes                     | Fabricated        | Tests       |
| Sodium chloride (≥ 99.8%)               | Sigma - Aldrich   | Tests       |
| Phenol crystals                         | Carlo Erba        | Tests       |
| Sodium Hydroxide (98.3%)                | Agenquímicos      | Tests       |
| Sodium sulfate anhydrous (99.8%)        | Agenquímicos      | Tests       |
| 4 – Aminoantipyrin (98%)                | Panreac           | Direct photometric method |
| Ammonium chloride (99.9%)               | Fisher Scientific | Direct photometric method |
| Potassium hexacyanoferrate (98%)        | Panreac           | Direct photometric method |
| Ammonium hydroxide (28.89%)             | Fisher Scientific | Direct photometric method |

### Table 6
List of equipment and instruments.

| Equipment (Model)                     | Brand             |
|---------------------------------------|-------------------|
| Analytical Balance (AS 220/C/2)       | Radwag            |
| Ag/AgCl Reference electrode (930–15) | Gamry             |
| UVA LEDs 3W 25 mW/cm²                 | LED World         |
| Digital multiparameter (Sension + MM150) | Hach              |
| heating and stirring plate (PC-420D)  | Corning           |
| Potentiostat/Galvanostat (series G 750) | Gamry             |
| Radiometer (HD 2102.2)                | Delta OHM         |
| Probe, 315 nm—400 nm, (LP 471 UVA)   | Delta OHM         |
| UV–Vis Spectrophotometer UV1800       | Shimadzu          |
Table 7

Operation levels of the experimental design.

| Initial pH | $C_0$ (mg/L) | $\phi_a$ (V) |
|------------|--------------|--------------|
| Low level (-1) | 5  | 9  | 0.80  |
| High level (+1) | 10 | 25 | 1.60  |

$$\text{EEO} = \frac{1000 \times P \times t}{V \times \log \left( C_i / C_f \right)}$$  \hspace{1cm} (3)

Where $P$ is the power source that is supplied to the system (kWh), $t$ is the total time of treatment (h), $V$ is the total volume of reaction (L) and $C_0$ and $C$ are the initial and final concentrations of the contaminant respectively (M) [3].

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Transparency document

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.dib.2019.103949.

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