Optimization of the Fenton treatment of 1,4-dioxane and on-line FTIR monitoring of the reaction

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**HIGHLIGHTS**

- 1,4-Dioxane can be totally removed applying an optimized Fenton process.
- FTIR-based monitoring of compounds presence along treatment was accomplished.
- A degradation route is proposed for the Fenton treatment of 1,4-dioxane.

**ABSTRACT**

1,4-Dioxane is a non-biodegradable, toxic, hazardous, and priority pollutant widely used in the chemical industry as a solvent; as well as it is a resulting by-product of many industrial processes. The optimization of the Fenton treatment of 1,4-dioxane, and the on-line FTIR monitoring of its degradation route, including the assessment of the enhancement of the biodegradability of the solution along treatment are herein addressed. Besides the full removal of 1,4-dioxane, an 80% reduction of the chemical oxygen demand (COD) was achieved at the best tested treatment conditions. Whether the used concentration of H₂O₂ was expectedly addressed as the reaction factor most influencing the achieved COD removal at the end of the process; the performance of the treatment under acid pH conditions showed to have just a slight influence, thus supporting this process may suitably be performed at neutral pH value. On-line FTIR monitoring of the process novelty provided the degradation route of 1,4-dioxane along its oxidation treatment, as well as a comprehensive optimization of the Fenton process based on the increase of the biodegradability of the solution and the reduction of the consumption of reagents.

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1. Introduction

1,4-Dioxane is an organic compound used as solvent in many industrial processes, as well as it is generated as a by-product in several industrial chemical reactions [1]. The main environmental concern regarding the potential release of this compound to the environment has recently increased due to its major negative effects on health. Correspondingly, 1,4-dioxane is classified as a priority pollutant by the US EPA [2]. Furthermore, it is a bio-recalcitrant and persistent organic molecule that cannot be treated by conventional bio-treatment technologies [1,3]. In fact, physical treatments, such as adsorption on activated carbon or air stripping, have not been successful removing dioxane from water because of this inherent high aqueous solubility and its low vapor pressure [3,4]. Moreover, it has been addressed that the chemical oxidation of 1,4-dioxane by chlorine could lead to the formation of even more toxic compounds [1]. In conclusion, traditional treatments have not been shown as effective degrading this substance. Only distillation technology has efficiently been proved to separate 1,4-dioxane from the solution due to its high volatility: but
its use has a very high associated cost, particularly at very low concentration levels [1,3,4].

Advanced oxidation processes (AOPs) may represent a feasible treatment alternative for 1,4-dioxane [4] because they can be applied at a lower economical cost just transforming this persistent compound into simpler more biodegradable compounds that might be further degraded by traditional biological treatment [5]. Particularly, Fenton method is one of the most commonly used AOPs. It is based on the electron transfer between hydrogen peroxide and ferrous ion, which acts as a homogenous catalyst, yielding hydroxyl radicals (OH•) that are able to degrade a wide range of organic compounds [6]. In general, the Fenton process is usually very efficient, and it even implies a lower economical cost than the application of others AOPs [7]. Regardless a high chemical oxygen demand (COD) removal efficiency could be achieved and a relative overall environmental friendliness, a Fenton treatment of wastewater is limited by the final production of iron sludge, which requires ultimate disposal [8], and the optimal pH range of application, which is usually set at acid values (pH=3) [9,10].

There are very few limited studies considering the treatment of 1,4-dioxane by Fenton’s reagent [1,3,11]. Although all these essays have assessed the improved elimination of 1,4-dioxane and COD from wastewater, an increased biodegradability of the final solution, and the reduced consumption of reagents; a comprehensive process optimization has not been addressed yet. In fact, the identification of which by-products are formed along the degradation of 1,4-dioxane by Fenton has not been addressed to date. Some authors have previously reported the removal of 1,4-dioxane and other by-products that are formed along its treatment, but the production of other chemical species along the reaction has not really been assessed in full yet [1,11,12]. Only Stefan and Bolton [13] have previously proposed a degradation mechanism for 1,4-dioxane treating this chemical by UV/H2O2 and taking periodical samples that were analyzed by gas chromatography (GC), solid phase microextraction (SPME), gas chromatography/mass spectrometry (GC/MS), ion chromatography (IC), and high-performance liquid chromatography (HPLC), in order to identify intermediate chemical species and by-products. The advantage of monitoring similar processes using Fourier transform infrared spectroscopy (FTIR) has previously been addressed identifying the compounds that are appearing and disappearing in the solution along the reaction sampling aliquots at preselected time intervals [14,15].

Therefore, the main objectives of this research are: (1) optimizing conventional Fenton method as a promising alternative for the treatment of 1,4-dioxane; and (2) developing an FTIR-based methodology enabling the on-line control of the use of reagents, and the production of intermediates and by-product along the degradation of 1,4-dioxane.

2. Material and methods

2.1. Materials and analytical methods

All used chemicals were of analytical grade and supplied by PAN-REAC S.A. (Barcelona, Spain) or Sigma-Aldrich (Highland, USA). The solutions were prepared in deionized water and kept in the dark until use. All the analyses were made according to the standard methods for the examination of water and wastewaters [16]. COD was measured by the colorimetric method at 600 nm using an Aquamate-spectrophotometer (Thermo Scientific AQA 091801, Waltham, USA). Hydrogen peroxide concentration was analyzed by the titanium sulphate spectrophotometric method [17].

In order to confirm FTIR results, 1,4-dioxane and ethylene glycol were identified and quantified along the reaction by gas-liquid chromatography (GLC) using a 7980A instrument (Agilent Technologies Inc., Palo Alto, CA) equipped with a flame ionization detector. Injector and detector were respectively set up at 310 and 280–300 °C. Samples (2 μL) were injected using the pulsed-split mode (split ratio 5:1) and analyzed in a TRB-FFAP (Teknokroma, Sant Cugat del Vallès, Spain) fused silica column (30 m × 0.25 mm internal diameter × 0.25 μm film thickness), with He (43 psi) as carrier gas, and the following temperature program: 60 °C to 240 °C at a 15 °C min⁻¹ ramp rate after a 9 min initial hold. Peaks were identified on the basis of sample coincidence to relative retention times of commercial standards. Quantification was performed according to peak areas that were corrected by response factors that were calculated for each compound using 1-butanol (250 mg L⁻¹) as internal standard, and GC-ChemStation Rev.B.04.02 (96) software from Agilent.

Formic, oxalic, acetic, glycolic, and methoxyacetic acids were also complementarily identified and quantified by ion chromatography (IC) using a Dionex DX-500 device (Thermo Scientific, Sunnyvale, CA) equipped with a conductivity detector. A 40 to 60 mM NaOH gradient was used as the eluent for measurement keeping the flow at 1.5 L min⁻¹. The injection loop was 75 μL. An AS11HC Ion Pac ionic resin column was used aided with a previous Anion Trap Column (ATC3) and an AG11-HC guard column. Peaks were identified and quantified on the basis of sample coincidence to relative retention times and standard concentrations of commercial standards.

2.2. FTIR analytical equipment

Mettler-Toledo ReactIR iC10 (Columbia, USA) is a Fourier transform infrared (FTIR) spectrometer that measures chemical species as they react over a period of time. It uses a Mercury Cadmium Telluride (MCT) detector that is cooled by liquid nitrogen. Measurements are optically taken using a diamond tipped probe with a 1 mm fiber optic conduit. The system should be purged (using instrument grade air, nitrogen or other suitable inert gas) in order to prevent water vapour from collecting inside the optics, which might otherwise obscure spectral data.

Data acquisition in the absorbance scale was taken from 2000 to 750 cm⁻¹ with an 8 cm⁻¹ nominal resolution; and 256 scans were co-added for each spectrum. A background on pure water was carried out just before performing each spectral record under the same resolution and scanning conditions that were used for the trials. Real-time component analyses were run using ConcIR software (Mettler-Toledo, Columbia, USA), which calculates associated component spectra, and relative concentration profiles.

2.3. Experimental procedure

Experiments were performed in a 500 mL glass reactor placed on a magnetic mixing device. Temperature was adjusted to the desired value (25 °C) using a water heater and circulator. pH was monitored along the process, and adjusted to the desired value (±0.1) using 1 mol L⁻¹ sodium hydroxide, or 1 mol L⁻¹ sulphuric acid, as required. Considering 1,4-dioxane is degraded to ethylene glycol at a very slow rate at acid pH values, whereas it is ionized and keeps a stable structure under basic ones, pH adjustment was carried out after adding dioxane (247.8 mg L⁻¹; initial COD = 450 mgO₂ L⁻¹) when performing trials under acid conditions, and before its incorporation to the solution otherwise. After temperature and initial pH adjustment, ferrous sulphate was added to the solution at the ferrous ion concentration specified in the experimental design described next to optimize treatment results. Hydrogen peroxide (30% w/v) was then added in batch mode until the designed concentration was also reached. Treatments were run until all the added H₂O₂ was totally consumed, so maximum COD removal was achieved for a set of reaction variables values.
Fig. 1. Reference spectra for the main chemicals that are expected to be found along the Fenton oxidation treatment of 1,4-dioxane.

Aliquots of the treated solution were periodically sampled with a syringe along the trials. These samples were adjusted to pH=9.0 adding 40% sodium hydroxide (NaOH), and then centrifuged during 15 min at 2000 rpm. COD and H2O2 concentrations were measured in the collected supernatant. H2O2 concentration values were used to correct COD values according to Hermosilla et al. [18].

Initial reaction conditions and concentrations of reagents chosen to perform on-line FTIR control experiments were determined after optimizing the Fenton treatment of 1,4-dioxane. In general, the experimental procedure used for FTIR trials was the same, despite the concentration of 1,4-dioxane was 0.07 mol L⁻¹, and the addition of hydrogen peroxide was carried out in continuous mode (8.8 mmol H2O2 min⁻¹ to a total 100 mL of reaction solution) because it allows to understand better the reaction process; besides it has previously shown better results than batch mode in this particular process [18,19].

The main compounds that actual scientific literature proposes to be considered in the degradation route of 1,4-dioxane by oxidation are: ethylene glycol diformate, glycolic acid, glyoxylic acid, methoxiacetic acid, formic acid, acetic acid and oxalic acid [1,3,11,13,20]. Their reference spectra were first experimentally collected by FTIR (Fig. 1). Despite the likeness of these compounds, the proper interpretation of mixture spectra (recorded during Fenton processes) have been successfully carried out with the help of the mathematical algorithms integrated in ConClRT software, and comparing spectra of pure solutions with those obtained along the process. Water spectra were subtracted from the obtained on-line spectra in all experimental runs.

2.4. Experimental design

Response surface methodology (RSM) was used to optimize the Fenton treatment of 1,4-dioxane statistically assessing the significance of the effects of the considered individual factors, and the interactions between them, that influence the process [21,22]. In addition, Pareto charting was used to highlight the most important factors that influenced the treatment efficiency. In short, this methodology allows determining optimum operation conditions for the system, as well as the region in which the operating specifications are satisfied [22]. Experimental design, regressions, response surfaces, and Pareto charts, were run by Systat 13 software (Systat Software Inc., Chicago, USA). pH, temperature, the concentrations of reagents, and reaction time, are the main factors that may influence the Fenton treatment of an organic compound. Among them, temperature has previously been reported not to affect much between 25 to 45 °C, and reaction time is really dependent on the concentrations of reagents [18]. Therefore, experiments were designed to optimize pH, hydrogen peroxide and ferrous ion concentrations. Based upon previously reported results for optimizing Fenton treatment of organics [23], RSM was actually performed considering pH values of 2.8, 5.0 and 7.2; and H2O2 concentration dependent ratios of H2O2/COD = 1.063, 2.125 and 4.250; and [H2O2]/[Fe²⁺], molar ratios of 1.0, 5.0 and 10.0.

In short, RSM regression analysis was performed according to a full factorial design (N=27) using the following quadratic model:

\[ Y = b_0 + \sum_{i=1}^{k} b_i X_i + \sum_{i=1}^{k} b_{ii} X_i^2 + \sum_{i=1}^{k} \sum_{j=1}^{k} b_{ij} X_i X_j \]

where \( Y \) (COD removal) is the process response dependent variable; \( X_i \) are the considered independent variables \( X_1 = [\text{H}_2\text{O}_2]/\text{COD} \); \( X_2 = [\text{H}_2\text{O}_2]/[\text{Fe}^{2+}] \); and \( b_i, b_{ii} \) and \( b_{ij} \) are the corresponding regression coefficients.

3. Results and discussion

3.1. Optimization of the conventional Fenton treatment of 1,4-dioxane

pH is one of the most important factors influencing the final result of a Fenton process because it mainly controls the speciation of iron and the stability of hydrogen peroxide [24,25].

In short, iron precipitation as Fe(OH)₃ is avoided under acid conditions because the solubility of iron increases [26] and, as a consequence, the efficiency of the process also increases. In addition, hydrogen peroxide decomposition is also impeded under acid environments, favoring its stabilization [27]; as well the scavenger function of inorganic carbon is also prevented because it should mainly have already been removed. Nonetheless, further significance may be found in the case of the Fenton treatment of 1,4-dioxane because of the structural changes this compound shows in relation to the pH value of the solution.

The resulting estimations of the regression coefficients of the quadratic model used to optimize the Fenton treatment of 1,4-dioxane by RSM (Fig. 2) are shown in Table 1. Whether a positive value of one of these coefficients indicates a positive relation of the associated factor to COD reduction; negative coefficients mean a lower COD removal would be achieved at a higher value the related factor. In particular, the negative coefficient related to the pH factor means that higher COD reductions were achieved working at lower pH values (Fig. 2); as well as higher doses of H2O2 (a higher
the optimum amount of H₂O₂ is added to the solution (Fig. 2). In addition, the molar rate between reagents ([H₂O₂]₀/[Fe²⁺]₀) resulted the second most important parameter influencing process efficiency. Its associated negative coefficient in the resulting RSM model (Table 1) implies that COD removal was enhanced when this ratio between reagents was lower. Therefore, considering certain amount of H₂O₂ is added to perform treatment, the higher tested related supply of Fe²⁺ yielded the higher removal of COD.

In summary, the best tested reaction conditions achieved a 65% COD removal (pH = 2.8, [H₂O₂]₀/COD₀ = 4.250 and [H₂O₂]₀/[Fe²⁺]₀ = 1). The COD remaining in the solution after this optimum treatment was characterized by the high content of volatile fatty acids (72.5 mg L⁻¹), which represents a high increase of biodegradability in the final solution. Therefore, the oxidative degradation of 1,4-dioxane at least resulted in the final persistence of more biodegradable organic acids, which is in agreement with previous results reporting an almost total destruction of 1,4-dioxane and a progressive decrease of the pH of the solution due to the formation of organic acids [1]. In addition, a poor elimination of the DOC further supported the degradation of 1,4-dioxane into more biodegradable products rather than to CO₂, which is also in agreement with previous related results [11].

In addition, more feasible industrial treatment conditions were chosen to perform further trials exploring the potential biodegradability enhancement of wastewater containing dioxane, namely: pH = 2.8, [H₂O₂]₀/COD₀ = 2.125 and [H₂O₂]₀/[Fe²⁺]₀ = 5. Higher doses of the reagents involve a proportional increase in the costs of chemicals and disposing iron sludge. Therefore, optimum reaction values resulting from RSM modeling are not really justified for real applications. As a result, a 43% COD removal was achieved performing the Fenton treatment of 1,4-dioxane under these reaction conditions; as well as the final presence of volatile fatty acids reached 58 mg L⁻¹, which also means a significant increase of the biodegradability of the final solution.

### 3.2. On-line FTIR monitoring of the degradation of 1,4-dioxane by Fenton oxidation

Concentration profiles of the main compounds that were generated in the solution along the Fenton oxidation of 1,4-dioxane (Fig. 4) were obtained monitoring the process with an on-line FTIR probe. The ConcIR software identified the compounds that were being on-line detected by FTIR comparing them to the previously obtained reference FTIR spectra of these compounds (Fig. 1). Initially, FTIR spectra did not show any change after the addition of a low concentration of H₂SO₄ at the studied wavenumber spectral region; as well as it was shown that the degradation of 1,4-dioxane into ethylene glycol under acid reaction conditions resulted too slow to be detected at the beginning of the reaction (Fig. 5). In fact,

![Fig. 2. Response surface and contour plot for the reduction of the COD resulting in the Fenton treatment of 1,4-dioxane (247.8 mg L⁻¹) being performed at different pH and reagents concentrations values (T=25 °C).](image1)

![Fig. 3. Pareto chart including the degree of influence of each considered factor in the Fenton treatment of 1,4-dioxane.](image2)

| Table 1 Estimated regression coefficients and standard errors for quadratic model used in RSM analysis. |
|---------------------------------------------------------|----------------|
| **Coefficient**                                          | **Standard error** |
| Constant                                                | 39.509          | 3.851          |
| pH (X₁)                                                 | −5.449          | 1.671          |
| [H₂O₂]₀/[COD₀] (X₂)                                     | 11.503          | 1.656          |
| [H₂O₂]₀/[Fe²⁺]₀ (X₃)                                    | −6.046          | 1.669          |
| X₁²                                                    | 4.000           | 2.866          |
| X₂²                                                    | −2.625          | 3.283          |
| X₃²                                                    | −4.550          | 2.908          |
| X₁ X₂                                                   | −2.679          | 1.990          |
| X₁ X₃                                                   | 0.083           | 1.986          |
| X₂ X₃                                                   | 3.836           | 2.022          |
the absence of ethylene glycol at this point was also checked by chromatographic analysis before the addition of Fenton’s reagent, after which certain very early changes can be noticed in the spectra. The dissolution of ferric sulphate heptahydrate reported a peak at about 1100 cm$^{-1}$, which surely represents sulphate ion [29]. The evolution of iron content cannot be followed separately.

1,4-Dioxane and FeSO$_4$·7H$_2$O were already present in the solution before any H$_2$O$_2$ was added. A red-brown color appeared just after hydrogen peroxide was incorporated, denoting iron oxidation from pale-green ferric ion to red-orange ferric one. A final intense orange color remained in the solution during almost all the process. When 8.8 mmol of H$_2$O$_2$ were already added to the reaction medium, ethylene glycol began to increase its presence, as well as glycolic acid and oxalate anion; whereas 1,4-dioxane began to decrease (Fig. 5). After 35 mmol of H$_2$O$_2$ were fully added, glycolic acid and oxalate anion reached a maximum of absorbance and began to decrease their presence thereafter. On the other hand, the presence of formic acid was newly detected at this point, and increased much its content until reaching a constant value at the end of the reaction. Thereafter, the reaction evolved until the complete reduction of 1,4-dioxane just when all the H$_2$O$_2$ dosage (70.5 mmol) was added. In addition, the profile of ethylene glycol showed a slightly decreasing tendency at the same time that glycolic acid and oxalate anion increased their presence at a similar linear pace, denoting certain relationship between both trends toward the end of the process.

![Fig. 4](image)

**Fig. 4.** Evolution of FTIR spectra within the 2000–750 cm$^{-1}$ region along the Fenton treatment of 1,4-dioxane. Reaction conditions: [1,4-dioxane] = 70 mmol, pH = 2.8 ± 0.2, [H$_2$O$_2$]/[Fe$^{3+}$]$_0$ = 5.

Chromatographic analyses confirmed the presence of all these compounds at the same moments of the reaction and the full removal of 1,4-dioxane at the end of the Fenton treatment. Therefore, the same process evolution shown in Fig. 5 was confirmed by the performed chromatographic determinations at singular reaction moments. Particularly, IC analyses indicated that, whether glycolic acid was produced in a small quantity, the presence of oxalate was very high. This might have been the result of a fast degradation of glycolic acid into oxalic acid, which may actually be the main responsible of the detected trend.

The pH-dependence of oxalic acid caused the appearance of the corresponding oxalate anion [30]. Some other peaks appeared along the reaction indicating the presence of other possible intermediates that the ConcIR software could not identify, such as ethylene glycol diformate and methoxyacetic acid (Fig. 6). Complementarily, the chromatographic analyses detected the production of a low concentration of methoxyacetic acid and a negligible amount of acetic acid; whereas ethylene glycol was actually found along the reaction, which pointed out that the pathway of degradation mainly progressed through the formation of ethylene glycol diformate.

As a consequence, a mechanism for the degradation pathway that was followed along the Fenton treatment of 1,4-dioxane may be proposed based on a radical reaction mechanism (Schemes 1–3).

First, hydroxyl radicals that were generated by the reaction between Fe$^{2+}$ and H$_2$O$_2$, attacked 1,4-dioxane molecules to form 1,4-dioxanyl radical. Thereafter, this reaction proceeded until 1,4-dioxan-α-oxyl radical was obtained by the reaction of 1,4-dioxanyl radical with available dissolved oxygen (Scheme 1), first resulting in the formation of the peroxy radical that next underwent termination reactions to generate the tetroxide precursor of 1,4-dioxan-α-oxyl radical, which was finally produced releasing oxygen [2,3,13]. Thereafter, 1,4-dioxan-α-oxyl radical progressed splitting the C–C bond, which was stabilized by ring opening. This linear peroxy radical evolved then reacting with oxygen to produce a linear tetroxide that may undergo two further reactions: (1) the electrocyclic process of this linear tetroxide, which finally yields ethylene glycol diformate [2,13]; or (2) the decomposition in two alkoxyl radicals and oxygen, followed by β-fragmentation and H abstraction, finally producing ethylene glycol monoformate [13] (Scheme 1).

![Fig. 5](image)

**Fig. 5.** FTIR-absorbance profiles of the main identified by-products that are produced during the Fenton oxidation of 1,4-dioxane. Reaction conditions: [1,4-dioxane] = 70 mmol, pH = 2.8 ± 0.2, [H$_2$O$_2$]/[Fe$^{3+}$]$_0$ = 5.

![Fig. 6](image)

**Fig. 6.** FTIR-absorbance profiles representing the evolution of representative peaks along the degradation of 1,4-dioxane by Fenton’s reagent within the spectral region of 2000–750 cm$^{-1}$: (989 cm$^{-1}$: ethylene glycol and methoxyacetic acid; 1631 cm$^{-1}$: glycolic acid, oxalate anion, and oxalic acid; 1581 cm$^{-1}$: oxalate anion; 1380 cm$^{-1}$: 1,4-dioxane, ethylene glycol diformate, and formic acid; 1715 cm$^{-1}$: ethylene glycol diformate, methoxyacetic acid, and formic acid). Reaction conditions: [1,4-dioxane] = 70 mmol, pH = 2.8 ± 0.2, [H$_2$O$_2$]/[Fe$^{3+}$]$_0$ = 5.
Scheme 1. Degradation mechanism generating ethylene glycol mono- and diformate from 1,4-dioxane.
Stefan and Bolton [13] also proposed an alternative degradation pathway from 1,4-dioxan-α-oxyl radical consisting on the formation of methoxyacetic acid, and finally resulting in the formation of acetic acid. Although some peaks that were recorded along the reaction could be assigned to methoxyacetic acid (Fig. 6), and chromatographic analyses showed this by-product was somewhat produced, the almost completely absence of acetic acid along the reaction actually denotes that this degradation alternative was not significantly followed in comparison to the degradation pathway through ethylene glycol. Nevertheless, Beckett and Hua [2]
reported that the degradation of methoxyacetic acid may also result in the generation of glycolic acid and formic acid.

Finally, ethylene glycol mono- and dimate may have progressed by acid hydrolysis to yield ethylene glycol and formic acid as by-products [31] (Scheme 2). Whereas ethylene glycol may be degraded to glycolic acid, and then evolve to oxalic acid by deformalization [32]; oxalic acid itself really shows up as oxalate anion under the acidic pH values predominating in the solution (Scheme 3).

In summary, during the oxidation of 1,4-dioxane, several by-products were produced as the consequence of the ring opening of 1,4-dioxane molecules, namely: ethylene glycol, glycolic acid, oxalate anion and formic acid. As a result, all 1,4-dioxane was removed, resulting in the appearance of more biodegradable by-products, which may allow a further biological treatment stage when necessary. An 80% total COD removal was finally achieved during these trials thanks to the previously identified optimum reaction conditions for this treatment and the further selection of a continuous addition mode for \( \text{H}_2\text{O}_2 \) during the process, which has previously been reported to address a similar treatment efficiency enhancement [18,19].

4. Conclusions

The treatment of 1,4 dioxane by Fenton’s reagent achieved the total removal of this compound from wastewater, as well as 80% COD reduction efficiency under the best tested treatment conditions. In addition, wastewater biodegradability may be further enhanced, which has been addressed by a significant high presence of volatile fatty acids at the end of the reaction. Therefore, although the total mineralization of 1,4-dioxane was not achieved, final persisting by-products were highly biodegradable; thus allowing further conventional treatment if final disposal requirements make it necessary.

FTIR technique allowed the identification of the main compounds involved in the degradation of 1,4-dioxane by conventional Fenton treatment, as well as the control of the behavior of the target compound, denoting its presence and evolution along the reaction. This methodology also allowed addressing a new degradation mechanism, monitoring the formation of by-products, and optimizing the process identifying the presence of biodegradable compounds that may easily be treated by conventional biological technologies, which may furthermore represent a saving in the overall treatment cost.

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