

Degradation of Sucralose by Peroxidation Assisted with Ultraviolet Radiation and Photo-Fenton

Gabriela F. Ferreira, Milena G. Maniero, and José R. Guimarães

Abstract—Substances discharged into bodies of water must be studied due to possible risk of contamination and consequent damage to nature and human health. Sucralose is an artificial sweetener widely used to produce diet food and beverage. Most of this substance is expelled intact from the human body; therefore, its concentration in rivers and wastewaters is increasing worldwide. In this work, sucralose degradation was performed using advanced oxidation processes (AOPs): peroxidation assisted by ultraviolet radiation ($\text{H}_2\text{O}_2/\text{UV}$), Fenton's reagent ($\text{Fe(II)}/\text{H}_2\text{O}_2/\text{H}^+$), and photo-Fenton ($\text{Fe(II)}/\text{H}_2\text{O}_2/\text{H}^+/\text{UV}$). Mineralization of the sweetener was measured using total organic carbon (TOC). In addition, AOPs were applied to different water matrices, such as ultrapure, synthetic, and surface waters. Experiments were performed to evaluate the toxicity of the solution during the degradation processes.

Index Terms—AOPs, sweetener, toxicity, UV.

I. INTRODUCTION

Sucralose is a sweetener that is being used more frequently by the food industry. Sucralose was intended to be a substitute for aspartame, which may be carcinogenic. The sweetener was discovered in 1976 by researchers at Queen Elizabeth College, University of London, in a program in collaboration with UK sugar producer Tate & Lyle, PLC. It is produced by a multi-step process with sucrose where three chlorine atoms replace three hydrogen-oxygen groups. It is sold under the trade name Splenda®. Very little sucralose is metabolized by the body and it is excreted in its original form. It is considered a stable anti-carcinogenic compound at cooking temperatures. This gives it a wide range of applications [1], [2].

Although there have been various toxicity tests and studies that ensure the safety of sucralose in food and beverages [3]–[5], there have been few studies of its fate and behavior after it's been excreted by the human body. Sucralose has been detected in municipal wastewater and surface water in Europe. In the United States, its concentration has been increasing in recent years [6], [7]. Due to the presence of this substance in bodies of water, some scientists have turned their attention to its possible toxic effects on non-target species [8].

Since sucralose is found in drinking water, it is possible to suggest that in addition to the molecule itself, there may be

many others present in the environment: that is, Wastewater Treatment Plants (WWTP) are not completely efficient at removing some recalcitrant compounds. Advanced oxidation processes (AOPs) may be a good option to degrade this artificial sweetener. AOPs are based on generation of hydroxyl radicals ($\bullet\text{OH}$), which have a high reduction potential ($\sim 2.80\text{ V}$) [9], capable of causing mineralization of organic matter, i.e., turning it into carbon dioxide, water, and inorganic ions. The most common processes are peroxidation assisted by UV radiation ($\text{H}_2\text{O}_2/\text{UV}$), Fenton's reagent ($\text{Fe(II)}/\text{H}_2\text{O}_2/\text{H}^+$), and photo-Fenton ($\text{Fe(II)}/\text{H}_2\text{O}_2/\text{H}^+/\text{UV}$) [10], [11]. Peroxidation assisted by ultraviolet radiation is a simple and efficient process for producing a large number of $\bullet\text{OH}$ radicals; therefore, it is suitable for degradation of organic molecules. Photo-Fenton consists of a combination of Fe(II) and hydrogen peroxide and application of UV radiation [12].

During AOPs, a common degradation pathway is the addition of a hydroxyl group to carbons of the molecule. Analyzing the structural formula of sucralose, shown in Fig. 1, the potential reaction sites are where the three chlorine atoms are located [5].

In addition to studies confirming the safety of sucralose consumption, there have been papers that show that AOPs are viable options for sucralose degradation [13], [14]. However, intermediate products from its degradation can be toxic; so, toxicity assays can show how toxic the byproducts formed are by inhibiting the activity of a microorganism. The sucralose molecule has chlorine atoms, so its degradation could generate toxic intermediates, such as toxic chlorinated compounds. Therefore, in addition to degradation assays, bioassays could be conducted to evaluate its toxicity. Studies with aerobic and anaerobic biological reactors have shown that these processes were not able to degrade the molecule [15].

The aim of this study was to evaluate the degradation of sucralose by $\text{H}_2\text{O}_2/\text{UV}$, Fenton's reagent and photo-Fenton in ultrapure water, a synthetic water matrix, and surface water. Acute toxicity tests using the bacteria *V. fischeri* were also performed to predict possible environmental impacts.

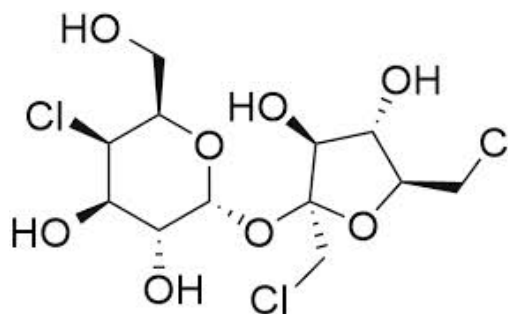


Fig. 1. The structural formula of sucralose.

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II. MATERIALS AND METHODS

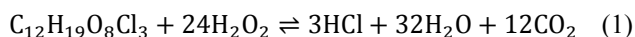
A. Reagents

Sucralose ($\geq 98\%$) was purchased from Sigma Aldrich (São Paulo, Brazil), hydrogen peroxide (30% m/m) from Synth (Diadema, Brazil), and ferrous sulphate heptahydrate from Synth (Diadema, Brazil). Ultrapure water used for the preparation of the solutions was obtained using a Milli-Q system (Millipore). The synthetic water was prepared with sodium bicarbonate from 'Cinetica Química Ltda' (São Paulo, Brazil), magnesium sulfate from Vetec (Rio de Janeiro, Brazil), and calcium sulfate and potassium chloride from Synth (Diadema, Brazil). Concentration are shown on Table I. Surface water was collected from 'Fazenda Rio das Pedras' lake near the city of Campinas in Brazil.

Hardness, alkalinity, and pH were measured for the synthetic water matrix; hardness, alkalinity, pH, conductivity, color, and turbidity were measured for surface water, as shown on Table II.

B. Experimental Conditions

The concentration of sucralose ($C_{12}H_{19}O_8Cl_3$) in aqueous solutions was approximately $55 \mu\text{g}/\text{cm}^3$, corresponding to $20 \mu\text{g}/\text{cm}^3$ of total organic carbon (TOC). In the peroxidation (H_2O_2) and H_2O_2 /UV processes, hydrogen peroxide concentrations were based on the stoichiometry presented in (1). The reagents were used at $C_{12}H_{19}O_8Cl_3:H_2O_2$ molar ratios of 1:24, or $3.3 \mu\text{mol}/\text{cm}^3 H_2O_2$; 1:48 $C_{12}H_{19}O_8Cl_3:H_2O_2$, or $6.6 \mu\text{mol}/\text{cm}^3 H_2O_2$; and 1:96 $C_{12}H_{19}O_8Cl_3:H_2O_2$, or $13.2 \mu\text{mol}/\text{cm}^3 H_2O_2$. The 1:600 $C_{12}H_{19}O_8Cl_3:H_2O_2$ molar ratio was also used in the H_2O_2 /UV process to evaluate if the excessive increase in H_2O_2 concentration significantly alters the results.



For Fenton and photo-Fenton processes, Fe(II) concentrations of 1.1, 2.2 and $4.4 \mu\text{mol}/\text{cm}^3$ were used. These Fe(II) concentrations were tested for molar ratios $C_{12}H_{19}O_8Cl_3:H_2O_2$ of 1:24 and 1:96. The Fe(II) ions were obtained from $FeSO_4 \cdot 7H_2O$. The tests proposed were all performed in duplicate and the graphs presented in Results and Discussion show the error bars calculated using the values' standard deviation.

TABLE I: SYNTHETIC WATER COMPOSITION

Substance	Concentration ($\mu\text{g}/\text{cm}^3$)
$NaHCO_3$	12.0
$CaSO_4$	6.0
$MgSO_4$	7.5
KCl	0.5

TABLE II: SYNTHETIC WATER MATRIX AND SURFACE WATER PROPERTIES

Parameter	Synthetic Water Matrix	Surface Water
Hardness, in $\mu\text{g}/\text{cm}^3 CaCO_3$	11.0	22.0
Alkalinity, in $\mu\text{g}/\text{cm}^3 CaCO_3$	11.6	38.4
pH	7.17	6.59
Conductivity, in $\mu\text{S}/\text{cm}$	-	65.4
Color, in Pt-Co	-	Real: 30 Apparent: 49
Turbidity, in TU	-	1.23

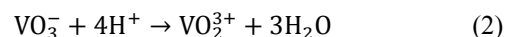
C. Experimental System

The laboratory setup consisted of a cylindrical photochemical reactor made of borosilicate glass (4 cm inner diameter and 42.5 cm length) with a germicidal lamp (2.5 cm inner diameter, 15 W, and $\lambda_{max} = 254 \text{ nm}$) inserted in the center; the lamp was in direct contact with the solution. The working volume of the reactor was about 300 cm^3 and a magnetic stirrer was used to homogenize the solutions. As shown in Fig. 2, the system was operated in batches with recirculation of the solution.

D. Analytical Methods

Sucralose degradation was evaluated by a TOC analyzer (Shimadzu TOC 5000A, Sao Paulo, Brazil), by monitoring the content of total organic carbon. The spectrum of the substance was not obtained because it does not absorb in the UV/visible range because it does not have a chromophore group [16].

The concentration of residual hydrogen peroxide was also monitored throughout the reaction time. This control is based on an reaction between the substance and a metavanadate ion (VO_3^-), which is yellow. Peroxovanadium cation (VO_2^{3+}) is formed in the presence of H_2O_2 , so the solution turns red and has a maximum absorbance at the wavelength of 450 nm. Maximum absorbance was measured by a spectrophotometer (HACH, DR4000). The oxidation-reduction reaction is shown in (2) [12], [17].



E. Toxicity Assays

The toxicity assays were carried out based on standard procedure L5.227 from CETESB [18] using a Microtox Model 500 Analyzer (Strategic Diagnostics Inc., Newark, Delaware, USA). The toxicity of the initial solution and solutions submitted to H_2O_2 /UV process (1:24 and 1:96 sucralose:hydrogen peroxide molar ratios) were evaluated, monitoring the changes between initial *V. fischeri* luminescence and its luminescence after 1800 seconds of exposure.

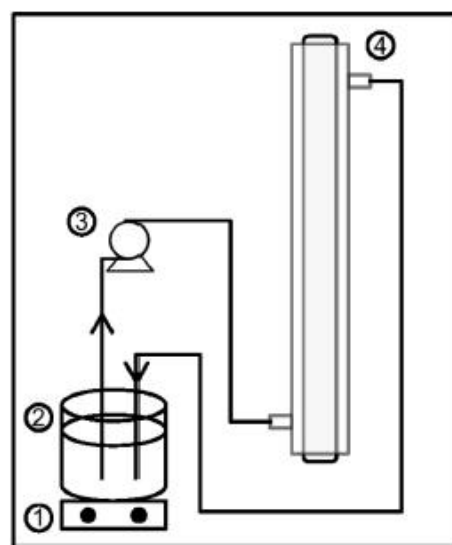


Fig. 2. Experiment setup: (1) magnetic stirrer, (2) vessel, (3) peristaltic pump, and (4) photochemical reactor.

Luminescence was measured directly from aliquots taken after 0, 900, 1800, 2700, 3600, 5400, 7200, and 9000 s of testing. Also, luminescence of the aqueous H_2O_2 solutions (concentrations of 2, 3.3, 6, 8, 10, and $13.2 \mu\text{mol}/\text{cm}^3$) was evaluated to find out if toxicity results were from sucralose degradation or the oxidant agent. The results of bacteria inhibition were calculated following the established protocol of the Microtox software (SDI Microtox Omni 4.0) (Strategic Diagnostics Inc, Newark, Delaware, USA).

III. RESULTS AND DISCUSSION

A. Degradation in Ultrapure Water

1) Experimental parameters

Temperature and pH were monitored during the experiments, although the data is not presented here. The temperature remained the same, around 26°C , in degradation using the peroxidation and Fenton processes, and increased smoothly in UV mediated processes. The pH values of the initial solution were neutral in general, around 6.8. For peroxidation and photolysis processes, pH remained nearly constant during testing; for Fenton and photo-Fenton processes, it was around 2.5; and for $\text{H}_2\text{O}_2/\text{UV}$, it was around 3.3. The recommended pH range of Fenton and photo-Fenton is 2.5-3.0, so the pH value did not need to be corrected with an acid or base solution.

The aliquots that underwent Fenton and photo-Fenton had to be centrifuged to remove the iron before the TOC analysis, because this compound could clog the equipment pathway and damage it. An Excelsa 2 Model 205 N Centrifuge from Fanem (Sao Paulo, Brazil) was used for this purpose; it was run for 300 seconds at 2000 rpm.

2) Degradation by UV, H_2O_2 , and $\text{H}_2\text{O}_2/\text{UV}$ processes

In UV and H_2O_2 processes, TOC value did not vary throughout the time of the experiment (0 to 7200 s). This shows that neither photolysis nor peroxidation processes were able to mineralize the molecule (see Fig. 3).

The mechanism of the $\text{H}_2\text{O}_2/\text{UV}$ reaction consists of photolysis of the H_2O_2 molecule into two hydroxyl radicals, as shown in (3).

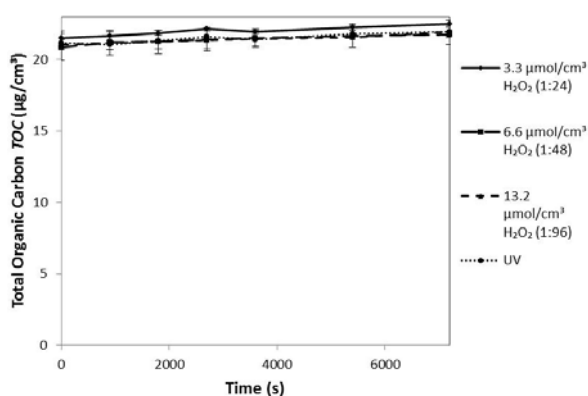
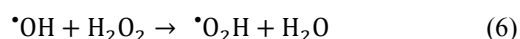
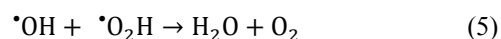
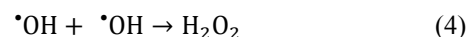


Fig. 3. Degradation of sucralose by H_2O_2 and UV processes.

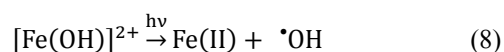
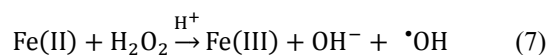
In the $\text{H}_2\text{O}_2/\text{UV}$ process, degradation tests lasted two and a half hours. As shown in Fig. 4, 88.6% mineralization of the

molecule was obtained using a sucralose:hydrogen peroxide molar ratio of 1: 96. The mineralization efficiency achieved were very close; it did not increase with a significant increase of hydrogen peroxide concentration. It is important to highlight that with excess peroxide and high concentrations of $\cdot\text{OH}$, competitive reactions occur that slow the degradation rate, as shown in (4)-(6). For this reason, optimal H_2O_2 concentrations have to be determined to avoid oxidant excess that can impair the reaction and reduce its efficiency [19], [20].



3) Degradation by fenton and photo-fenton processes

The reaction of Fenton's reagent is shown in (7). The photo-Fenton process consists of Fenton's reagent combined with ultraviolet radiation. It significantly accelerates the degradation of organic compounds due to the regeneration of Fe(II) ions shown in (8).



For Fenton's reagent, mineralization of the compound was observed in the first 1200 seconds of testing, as shown in Fig. 5. After this time, TOC concentration stabilizes, indicating that the compound did not undergo further degradation. This behavior is typical when using Fenton's reagent because the rate of the process decreases as the catalyst Fe(II) is consumed. Moreover, stable Fe(III) complexes can be formed. When the concentration of iron is too small, the degradation efficiency of sucralose is very low, even in the presence of oxidant (H_2O_2). The oxidant is unable to react with the sweetener, as previously discussed.

The results obtained using 1: 96 sucralose:hydrogen peroxide concentration had superior degradation than the 1: 24 sucralose:hydrogen peroxide molar ratio; the degradation efficiency of the 1: 96 concentration was 46.4%.

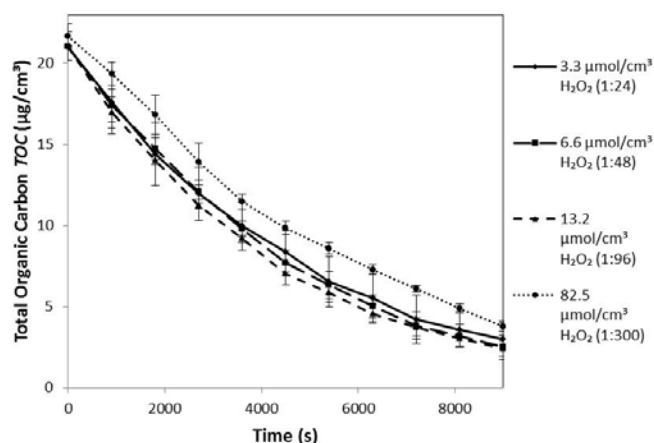
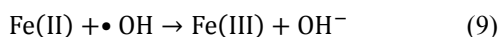


Fig. 4. Degradation of sucralose by the $\text{H}_2\text{O}_2/\text{UV}$ process.

Thus, efficiency increased as H_2O_2 concentration increased. However, when the Fe(II) concentration rose to $4.4\mu\text{mol}/\text{cm}^3$, the reaction was impaired, decreasing its efficiency. This may have been due to scavenging of hydroxyl radicals by excess Fe(II) , as shown in (9).

Finally, the results obtained with the photo-Fenton process Fig. 6 had higher degradation percentages in shorter periods of time. For two hour assays (7200 s), a maximum sweetener mineralization of 98.7% was obtained for the sucralose:hydrogen peroxide molar ratio of 1:96 and Fe(II) concentration of $2.2\mu\text{mol}/\text{cm}^3$. This means that the compound was almost completely mineralized and there were no more organic molecules present in the solution. In most cases, keeping Fe(II) concentration constant and increasing H_2O_2 concentration increased degradation. In general, keeping H_2O_2 concentration constant and increasing Fe(II) concentration decreased degradation. This is because in excess, iron ions act as hydroxyl radicals scavengers, as discussed above and shown in (9) [21].



When all of the processes studied in this work are analyzed, it can be seen that the advanced oxidation processes have the highest percentages of mineralization and are therefore best suited to the purpose. When comparing the

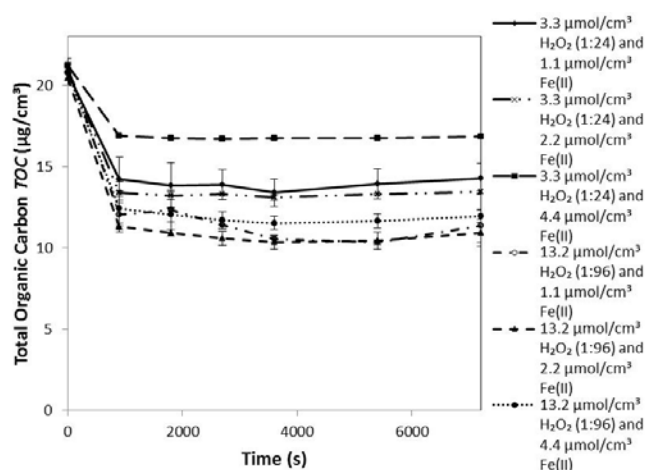


Fig. 5. Degradation of sucralose by the $\text{Fe(II)}/\text{H}_2\text{O}_2/\text{H}^+$ process.

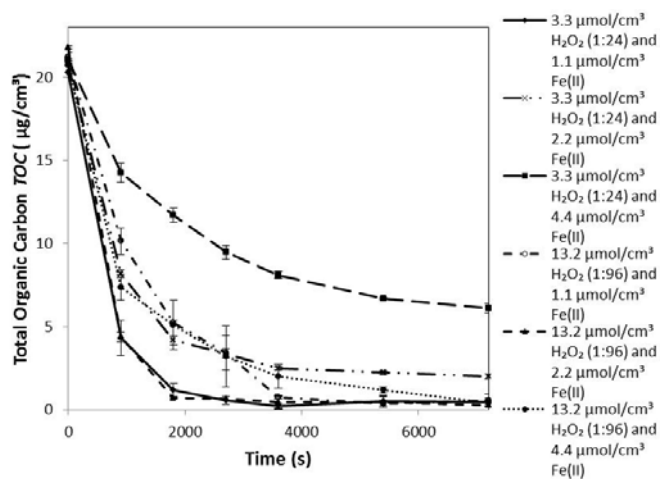


Fig. 6. Degradation of sucralose by the $\text{Fe(II)}/\text{H}_2\text{O}_2/\text{H}^+/\text{UV}$ process.

$\text{H}_2\text{O}_2/\text{UV}$ and photo-Fenton processes, the

photo-Fenton ensures higher efficiency in a shorter period of time; however, it has some disadvantages: the addition of reagents to adjust pH and the generation of sludge. The Fenton's reagent process had significant mineralization during the beginning of testing, but there efficiency leveled as the limiting reagent Fe(II) was consumed. The photo-Fenton process offers a solution to this problem, since UV radiation regenerates Fe(II) ions. It starts a cycle and makes the method more efficient. The photolytic and peroxidation processes were not able to degrade the sweetener.

4) Residual peroxide

Residual peroxide was measured for AOPs ($\text{H}_2\text{O}_2/\text{UV}$, Fenton, and photo-Fenton) and the chemical process of peroxidation. For peroxidation, H_2O_2 concentration remained nearly constant from the beginning to the end of the test; that is, it was not consumed throughout the time of the reaction, indicating that there really was no reaction between the sweetener and the oxidant. For $\text{H}_2\text{O}_2/\text{UV}$, the decrease of peroxide concentration was almost proportional with time; the minimum concentration measured in 9000 seconds was $0.14\mu\text{mol}/\text{cm}^3$. For photo-Fenton, concentration decreased exponentially, and at the end of the experiment, the residual peroxide concentration was below the detection limit ($0.0245\mu\text{mol}/\text{cm}^3$). Finally, for Fenton, the oxidant concentration decreased in inverse proportion to Fe(II) concentration; that is, the lowest concentration of Fe(II) corresponded to the highest concentration of remaining H_2O_2 because less Fe(II) reacted with the oxidant to produce hydroxyl radicals.

5) Chemical reaction kinetic

The order of the chemical reactions was evaluated to determine the kinetic parameters. For degradation using $\text{H}_2\text{O}_2/\text{UV}$, the reaction was of the first order, with a reaction rate constant of $2.223 \times 10^{-4}\text{s}^{-1}$ for a sucralose:hydrogen peroxide concentration of 1:24; the reaction rate constant was $2.454 \times 10^{-4}\text{s}^{-1}$ for a sucralose:hydrogen peroxide concentration of 1:48; the reaction rate constant was $2.399 \times 10^{-4}\text{s}^{-1}$ for a sucralose:hydrogen peroxide concentration of 1:96; and the reaction rate constant was $2.016 \times 10^{-4}\text{s}^{-1}$ for a sucralose:hydrogen peroxide concentration of 1:300. Fig. 4 shows that in general, total organic concentration does not vary significantly with hydrogen peroxide concentration. For the other processes, it was not possible to determine the reaction order because the results were not consistent; the model must follow an unconventional reaction order.

B. Ultrapure Water, Synthetic Matrix, and Surfacewater

The two most effective AOPs were used to degrade sucralose in different aqueous matrices. $\text{H}_2\text{O}_2/\text{UV}$ and photo-Fenton processes were compared using a 1:24 molar ratio sucralose:hydrogen peroxide and Fe(II) concentration of $1.1\mu\text{mol}/\text{cm}^3$.

Fig. 7 shows that the degradation behavior was almost the same for ultrapure and synthetic water. This means that salts do not interfere with the efficiency of sucralose degradation. There is a notable difference between these two matrices and surface water because it contains different dissolved organic molecules as well as other substances. Although the initial concentration of sucralose was $55\mu\text{g}/\text{cm}^3$ in all aqueous matrices, TOC concentration was higher for

surface water, as was expected. Nevertheless, at the end of the experiment mineralization of surface water was very similar to that of pure water and the synthetic matrix. This consolidates the idea that H_2O_2 /UV is a good option for degrading this sweetener and other organic compounds that are not removed by conventional wastewater treatment.

Photo-Fenton degradation with surface water had a very similar curve to ultrapure water and synthetic matrix water, as shown in Fig. 8. The outcome shows that this method is also effective in order to degrade the sweetener, even in real water sources.

Comparing both processes using surface water, it is clear that photo-Fenton is more effective, in a shorter period of time, on degrading sucralose, as well as other organic substances present in surface water. The final mineralization efficiency was 89.6% for H_2O_2 /UV in a two-and-a-half-hour reaction; final mineralization efficiency was 90.7% for photo-Fenton in a two-hour reaction. If only the first hour of the experiments is considered, the degradation efficiency was 42.1% for H_2O_2 /UV versus 90.5% for photo-Fenton. Clearly reaction time, degradation effectiveness, reagent availability, byproduct formation, and other factors have to be considered in order to select the best process.

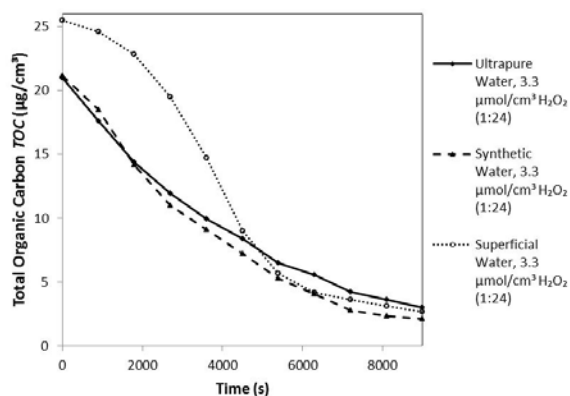


Fig. 7. Degradation of sucralose by the H_2O_2 /UV process in different aqueous matrices.

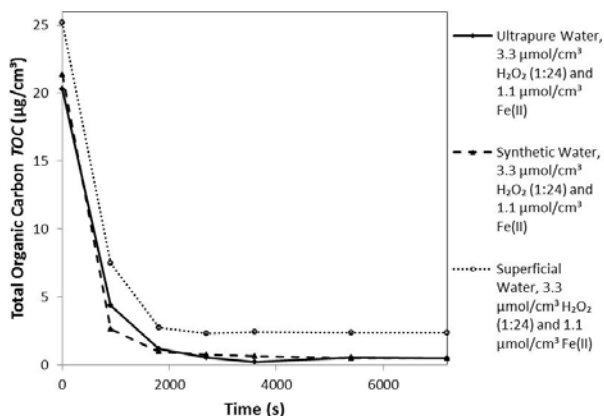


Fig. 8. Degradation of sucralose by $Fe(II)/H_2O_2/H^+/UV$ processes in different aqueous matrices.

C. Toxicity Assays

Fig. 9 shows the results of bacteria inhibitions when using a 1:24 sucralose:hydrogen peroxide molar ratio; Fig. 10 shows the results of bacteria inhibitions when using a 1:96 sucralose:hydrogen peroxide molar ratio.

Solutions of hydrogen peroxide (0 to $13.2 \mu\text{mol}/\text{cm}^3$) were also analyzed using Microtox[®]. Bacteria inhibition was directly proportional to H_2O_2 concentration (linear correlation coefficient of 0.9735). This shows that at high concentration, this reagent is toxic. Toxicity results were accurate when H_2O_2 concentration was lower. The sweetener solution ($55 \mu\text{g}/\text{cm}^3$ sucralose in ultrapure water) did not inhibit bacteria growth; that is, sucralose is not toxic to *Vibrio fischeri*. Due to the toxicity of hydrogen peroxide, the residual oxidant concentration was also plotted in Fig. 9 and Fig. 10.

Inhibition behavior was similar to the residual hydrogen peroxide curve Fig. 9. As pointed out before, toxicity (inhibition percentage) was proportional to H_2O_2 concentration; therefore, it is plausible that the toxicity was due to H_2O_2 reagent and that toxic byproducts were not formed.

For the highest H_2O_2 concentration (1:96 sucralose:hydrogen peroxide molar ratio) (Fig. 10), toxic compounds were formed during the degradation process.

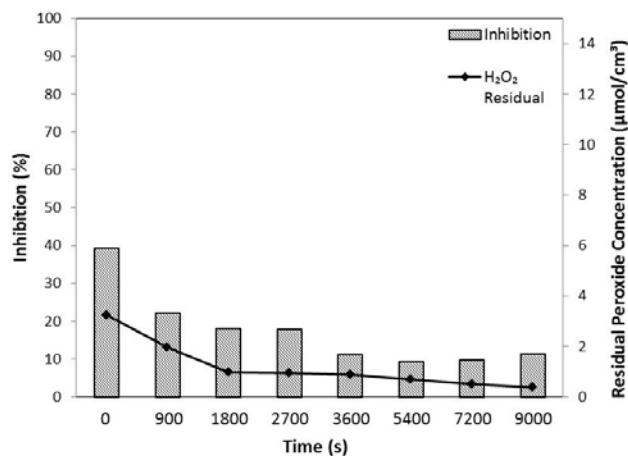


Fig. 9. Toxicity and residual hydrogen peroxide during sucralose degradation by H_2O_2 /UV (1:24 sucralose:hydrogen peroxide molar ratio).

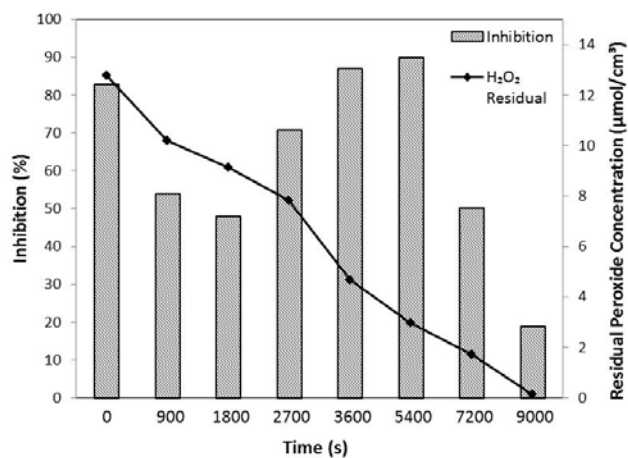


Fig. 10. Toxicity and residual hydrogen peroxide during sucralose degradation by H_2O_2 /UV (1:96 sucralose:hydrogen peroxide molar ratio).

In the first 1800 seconds of testing, *Vibrio fischeri* inhibition could be caused only by H_2O_2 , as there was a correlation between inhibition and residual oxidant concentration. However, from 2700 to 5400 seconds of

testing, toxicity increased. This means that toxic byproducts were formed, since H_2O_2 concentration continued to decrease. After two hours of testing (7200 s), toxicity begins to decrease again, with the lowest toxicity result taking place at the end of the experiment (9000 s).

Even though the TOC profile was approximately the same for the two sucralose:hydrogen peroxide molar ratios (1:24 and 1:96) shown in Fig. 4, the byproducts formed during the degradation processes were different, because for the 1:24 concentration there was no toxicity. Moreover, it is important to highlight that at the end of the experiment, the toxicity was less for the 1:24 sucralose: hydrogen peroxide molar ratio despite the residual oxidant concentration being slightly higher.

The stoichiometric concentration of sucralose to hydrogen peroxide of 1:24 was the optimum condition, since the degradation efficiency was about the same, less reagent was necessary for sweetener degradation, and the intermediate molecules formed were not highly toxic.

IV. CONCLUSIONS

Sucralose is a chemically and photolytically stable compound. It was not degraded using only UV radiation or H_2O_2 at the doses evaluated in the present work. Fenton's reagent was able to degrade the molecule for the first 1200 seconds of testing, but after this period of time, the TOC concentration reached a plateau as iron ions were consumed. H_2O_2 /UV and photo-Fenton were the best processes for sucralose degradation and mineralization, but peroxidation assisted by UV radiation had a lower mineralization efficiency and a longer reaction time.

Comparing degradation in different water matrices, it can be concluded that there were no significant difference between ultrapure water and the synthetic water matrix. Using surface water, the mineralization efficiency was slightly reduced due to the presence of organic compounds and other substances. The AOPs utilized can be used for sweetener degradation, especially those mediated by UV radiation.

Toxicity results showed that sucralose is not toxic, but intermediate compounds formed from its degradation could be. Bacteria inhibition decreased during the first 1800 seconds of testing and increased at 2700 seconds for the 1:96 sucralose:hydrogen peroxide molar ratio, because toxic byproducts were formed. For the 1:24 sucralose:hydrogen peroxide molar ratio, the byproducts formed were not toxic; so, this stoichiometric concentration is superior.

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