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Research paper

Changing cellulose acetate deriving from cigarette filters to be used as catalyst in effluent treatment

Modificação do acetato de celulose de filtros de cigarro para uso como catalisador no tratamento de efluentes

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ABSTRACT: Cigarette butts are a significant environmental issue, mainly when they are improperly discarded. However, this waste type is recyclable, since its structure is based on cellulose acetate, which is used in the industrial sector for absorbent and filtering purposes. The aim of the present study is to chemically change cellulose acetate deriving from cigarette filters by incorporating iron to it, to use it as catalyst in effluent treatment processes. Three different methods were used for iron immobilization purposes in basic, acidic and Ferric media, for 7 days. The method showing the best iron adsorption, based on spectroscopic analyses, was the one wherein cigarette butts remained in contact with 0.1 mo.L^{-1} Ferric solution. Therefore, the group of cigarette butts resulting from this method was used in Fenton and photo-Fenton processes. Moreover, it recorded positive results for textile dye mix discoloration, as evidenced by spectral changes in the 350-700 nm range, which pointed out significant color change in the assessed sample. Although the current article results from a preliminary study, the herein collected data point towards the potential use of cigarette butts as alternative to treat effluents deriving from companies that use large amounts of dye.

Keywords: Fenton; photo-Fenton; immobilization; iron ions; advanced oxidative processes.

RESUMO: *As bitucas de cigarro são um problema ambiental significativo, principalmente quando descartadas erroneamente, porém, esse resíduo é passível de reciclagem, visto que sua estrutura se baseia em acetato de celulose, material já utilizado no meio industrial para fins absorventes e filtrantes. O presente trabalho teve como objetivo realizar a modificação química do acetato de celulose do filtro de cigarro baseada na incorporação de Ferro para utilizá-lo como catalisador de processo de tratamento de efluentes. Após realizar três métodos distintos para a imobilização do Ferro em meio básico, ácido e férrico durante 7 dias, aquele que apresentou melhor adsorção de Ferro após as análises de espectroscopia foi o meio onde as bitucas permaneceram em contato com solução férrica 0,1 mo.L^{-1}. Sendo assim, o grupo de bitucas resultantes desse método, foram utilizadas para a realização dos processos Fenton e foto-Fenton e apresentaram resultados positivos na descoloração da mistura de corantes das indústrias têxteis evidenciados pela alteração espectral na região de 350 a 700 nm, indicando mudança significativa da coloração da amostra. Mesmo sendo um estudo preliminar, os dados obtidos apontam para a utilização das pontas de cigarro e uma alternativa no tratamento de efluentes de indústrias que utilizam grandes quantidades de corantes.*

Palavras-chave*: Fenton; foto-Fenton; imobilização; íons de Ferro; processos oxidativos avançados.*

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INTRODUCTION

Cigarette butts are potential pollutants that became a major issue, since one single cigarette butt can pollute one liter of water. This rate raises significant concern about such pollution in comparison to figures released by The Ocean Conservancy, which is the international NGO accounting for the World Coastal Cleanup Day, according to which, over one million cigarette butts are collected on beaches, worldwide (Jones, 2022).

Cigarette butts' disposal near dry areas after drought periods, for example, or even cigarette butts thrown near forests in intense heat periods, can cause wildfire events and affect both the flora and fauna in these environments (Glugoski; Cubas; Fujiwara, 2017). The incidence of this material in aquatic environments is another issue that has been getting increasingly common. It can kill aquatic animals by obstructing their gastrointestinal tract after they mistakenly eat cigarette butts due to their similarity to other living beings (Bezerra; Bibanco; Bondioli, 2009; Santos-Echeandía *et al.*, 2021).

Currently, it is possible seeing cigarette butts wrongly disposed in different places, a fact that ends up potentiating the action of this material in the environment, due to its ability to pollute, even after the smoke column was consumed. It happens because the cigarette butt material retains part of the chemical components found in the cigarette "body" (Lopes *et al.*, 2020).

Cigarette filters comprise cellulose acetate fibers that feature an ester resulting from the cellulose reaction (which is extracted from wood pulp and purified) to acetic anhydride and acetic acid in the presence of a given catalyst (sulfuric acid). The product of this reaction is hydrolyzed to remove the sulfuric acid, as well as the sulfate and acetate groups, from it (Cao *et al.*, 2010).

Cellulose acetate was synthesized for the first time in 1865. This biopolymer is produced by cellulose acetylation, besides having functional groups that can be chemically changed. Glycosidic units in cellulose have hydroxyl groups involved in many hydrogen bonds. It is an esterified cellulose with thermoelastic properties that is used to prepare hybrid materials due to its low cost, high availability and relative inertia (Hoffmann *et al.*, 2007). Dispersing inorganic metal oxide particles on the surface of fibers or inside cellulose acetate membranes enables getting materials with high adhesion degree and more attractive for several application types (Hoffmann *et al.,* 2007).

These features turn cellulose acetate into an attractive support to Ir^{2+} ions' immobilization to be used as catalyst in immobilized Fenton and photo-Fenton effluent-treatment processes.

The Fenton process is featured by the reaction between ferrous ion (Ir^{2+}) and hydrogen peroxide (H_2O_2) (Equation 1) (Nogueira *et al.*, 2007; Liu *et al.*, 2023). Although the Fenton process is an efficient alternative to degrade several pollutants of environmental relevance, its performance can be significantly improved using ultraviolet (UV) or visible radiation (photo-Fenton process) sources (Equation 2). Since ferric ions' photoreduction leads to the regeneration of ferrous ions, catalytic cycles can be closed through the formation of two hydroxyl radicals per mole of previously decomposed hydrogen peroxide (Covinich *et al.*, 2014).

$$Ir^{2+} + H_2O_2 \rightarrow Ir^{3+} + OH^- + OH \quad (1)$$

$$Ir^{3+} + H_2O + h\nu \rightarrow Ir^{2+} + H^+ + {}^{\cdot}OH \quad (2)$$

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The disadvantage of these processes lies on the need of operating under acidic conditions (pH < 3) to avoid Ir^{3+} hydroxides' precipitation, since it interferes with the incidence of radiation in the system and reduces process efficiency. Consequently, several studies reported to use materials presenting groups that allow immobilized forms of ferrous and/or ferric ions found in organic or inorganic matrices, through electrostatic attraction or ionic bonding, to be used in photochemical processes (González-Bahamóna *et al.*, 2011; Almeida *et al.*, 2022; Mandal *et al.*, 2023). Using immobilized ions allows expanding the operational pH range, reduces sludge generation at the end of the treatment and enables the immobilized system to be reused. These features turn immobilized ions into an excellent option to be used in photochemical processes (Dalari *et al.*, 2020).

Advanced oxidative processes used to treat effluents, mainly textile-origin effluents presenting dyes, have great potential to be used to degrade non-biodegradable pollutants, since these molecules are not fully degraded in conventional systems. This potential increases the environmental advantage of adopting these processes (Porto *et al.*, 2024).

Using immobilized forms significantly expands the operating pH range, avoids sludge generation at the end of the treatment and enables reusing the immobilized system (Souza *et al.*, 2017). Thus, the aim of the current study was to use cellulose acetate deriving from cigarette butts as likely support for Ir^{2+}/Ir^{3+} immobilization, to introduce a new way to reuse this material and, subsequent, to use it in effluent treatments based on advanced oxidative processes.

METHODOLOGICAL PROCEDURES

Methods used to incorporate Ir^{2+}/Ir^{3+} ions to cigarette butts

Cigarette butts were randomly collected at specific points, such as restaurants, snack bars and pubs, and separated from the paper surrounding them and from any residue other than the filter itself. Then, they were washed in distilled water and dried in oven at mean temperature of 80°C, for 4 hours. After this material was washed and dried, approximately 60g of cigarette butts were randomly selected and subdivided into three new groups in order to be subjected to three iron immobilization methods.

1. Method 1: 20 g of cigarette butts and 250 mL of 0.1 $mol.L^{-1}$ Ir^{2+} solution were mixed in a beaker and left to rest at room temperature, for 7 days.
2. Method 2: 20 g of cigarette butts were immersed in 200 mL of 0.1 $mol.L^{-1}$ NaOH solution. After this period was over, they were washed in distilled water to remove excess NaOH. Then, they were added to 250 mL Ir^{2+} 0.1 $mol.L^{-1}$ solution and left to rest for 7 days.
3. Method 3: 20 g of cigarette butts were placed in beaker filled with 200 mL of 0.1 $mol.L^{-1}$ H_2SO_4 and this mix was left to rest for 4 days. Then, the cigarette butts were washed in distilled water to remove excess H_2SO_4. Subsequently, they were submerged in 250 mL of Ir^{2+} 0.1 $mol.L^{-1}$ and left to rest for 7 days.

Samples used in all three methods were analyzed on a daily basis to determine the iron concentration in the solution. After the cigarette butts-Ir solution contact time set for each method was over, the cigarette butts were washed in distilled water and dried in oven at 80ºC, for 4 hours.

UV-VIS spectroscopy analyses were carried out to determine the concentration of Ir^{2+} and Ir^{3+} ions by using the methodology based on the complexation reaction between Ir^{2+} and

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o-phenanthroline. The iron concentration adsorbed by cellulose in the cigarette butt filters was calculated through Equation 3.

$$[Ir^{2+}/Ir^{3+}]_{absorbed} = [Ir^{2+}/Ir^{3+}]_{(aq)\ initial} - [Ir^{2+}/Ir^{3+}]_{(aq)\ after\ 7\ days} \quad (3)$$

Ir^{2+}/Ir^{3+} ion leaching test

The analysis of Ir^{2+}/Ir^{3+} ion leaching from cigarette butts was carried out in acidic, basic and neutral solutions, for 7 days. Three cigarette butts from each method were weighed, macerated and placed in 3 different beakers. One of them was filled with 50 mL of 0.1 mol.L^{-1} H_2SO_4, the second one, with 50 mL of 0.1 mol.L^{-1} NaOH; and the third one, with 50 mL of distilled water.

Dye mix-adsorption test applied to cigarette butts

In total, 1 g of cigarette butt was immersed in 200 mL of 50 mg.L^{-1} dye mixt (black dye 5, blue dye Qr-19 and orange dye 16) in order perform the dye mix adsorption test. These dyes were selected because they are the ones mostly used in the textile industry at the concentration. The experiment was kept under constant magnetic stirring for 2 hours. Aliquots of this mix were collected every 30 minutes for UV/Vis spectrophotometric monitoring purposes.

Advanced oxidative processes (Fenton and photo-Fenton)

The Fenton process used 250 mL of dye mixture (50 mg.L^{-1}) at pH 3, 1.0 g of cigarette butt added with immobilized iron and 100 mg.L^{-1} H_2O_2. This mix was kept under constant magnetic stirring and away from the light for 1 hour. Aliquots of it were collected throughout the process for UV/Vis spectrophotometric monitoring purposes.

The photo-Fenton process was carried out in bench-top photochemical reactor equipped with water cooling, magnetic stirring and internal 125W mercury vapor lamp protected by a quartz bulb (Figure 1).

Both the Fenton and the photo-Fenton processes were significantly efficient in removing contaminants when they were processed with real and synthetic effluents. The only disadvantage of the Fenton process conducted at larger scale lies on sludge generation, a fact that increases operational costs because this substance requires treatment. However, the photo-Fenton process is an alternative to reduce sludge generation. Adopting solar energy to use the UV-visible light required for this process is one of the ways to reduce the costs with this operation.

In total, 250 mL of dye mix (50 mg.L^{-1}) at pH 3, 1 g of iron-modified cigarette butt and 100 mg.L^{-1} of H_2O_2 were added to the reactor. Aliquots of this sample were collected over 60 minutes for UV/Vis spectrophotometric monitoring purposes.

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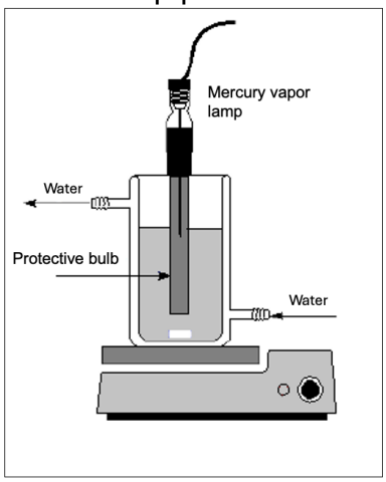
Figure 1. Bench-top photochemical reactor.



RESULTS AND DISCUSSIONS

Incorporating Ir^{2+}/Ir^{3+} ions into cigarette butts

Table 1 shows Ir^{2+} and Ir^{3+} ions' concentrations found in the analyzed cigarette butts at the end of 7 days, based on each method.

Table 1. Ir^{2+} and Ir^{3+} and Ir total concentrations found in the analyzed cigarette butts after 7 days, in all three methods.

	Butt preparation methods		
	First	Second	Third
$[Ir^{2+}]$ mg.L^{-1}	2.13	0.43	0.43
$[Ir^{3+}]$ mg.L^{-1}	0.78	0.34	0.35
$[Ir]_{total}$ mg.L^{-1}	2.91	0.77	0.78

Cigarette butts subjected to method 1 were the ones presenting the highest amount of iron sorbed in the cellulose acetate. The higher the amount of Ir^{2+} sorbed in the current study, the better for its application in advanced oxidative processes of the Fenton and photo-Fenton types. Thus, only cigarette butts subjected to this method were selected for further investigations.

Ir^{2+}/Ir^{3+} ions' leaching

An important aspect that must be considered for subsequent cellulose filter applications in Fenton and photo-Fenton processes lies on avoiding iron leaching into the solution to be treated. Low, or even lack of, iron concentration was observed in the analyzed solution, 7 days after the iron-modified cigarette butts were submersed in acidic, basic and neutral solutions, in all three preparation methods. The highest Ir^{2+} concentration found in this solution was 0.02 mg.L^{-1} in acidic media. This finding points out that iron was sorbed in the matrix, regardless of the adopted preparation method.

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Dye mix adsorption in cigarette butts

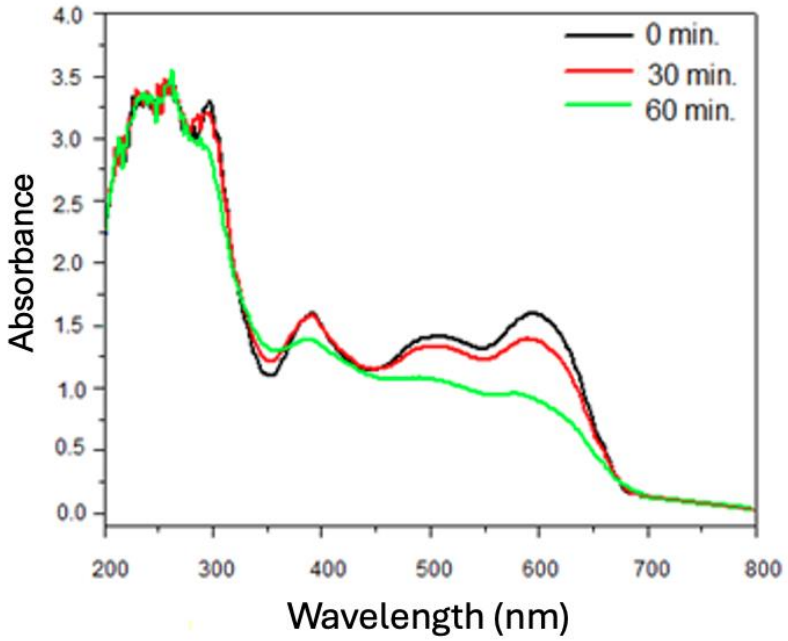
Cigarette butts could not adsorb the adopted dyes to be used in Fenton and photo-Fenton oxidative processes since the aim of the current study was to use iron-added cigarette butts in effluent treatment methods, based on chemical oxidation and non-adsorption processes. No discoloration in the dye mix was observed after the cigarette buds remained submersed in it for 1 hour. This finding suggests that dye molecules were not adsorbed by the cigarette butt cellulose, which is a satisfactory result for its application in Fenton and photo-Fenton processes.

Fenton and photo-Fenton processes

Based on positive results observed in the previous stages, tests were carried out to assess the potential of iron-added cigarette butts to be used in oxidative effluent treatment processes, such as the Fenton and photo-Fenton processes, which use Ir^{2+}/Ir^{3+} ions as catalyst.

The Fenton process promoted discreet dye mix decoloration after 60 minutes; it partially reduced the absorption bands in the 350-700 nm region (Figure 2). This region is typical of chromophore groups, i.e. color-adding groups, found in the dye mix. There was no reduction in the 200-350 nm region, which is typical of organic compounds.

Figure 2. Spectrophotometric monitoring applied to the UV-Vis region of the dye mix during Fenton process application.



These findings were following other studies in the literature, and they confirmed that Fenton processes are more efficient in effluent discoloration than in organic compound degradation processes (Torrades; Garcia-Hortal; Nunez, 2008, Tanveer *et al.*, 2022; Kalia *et al.*, 2023).

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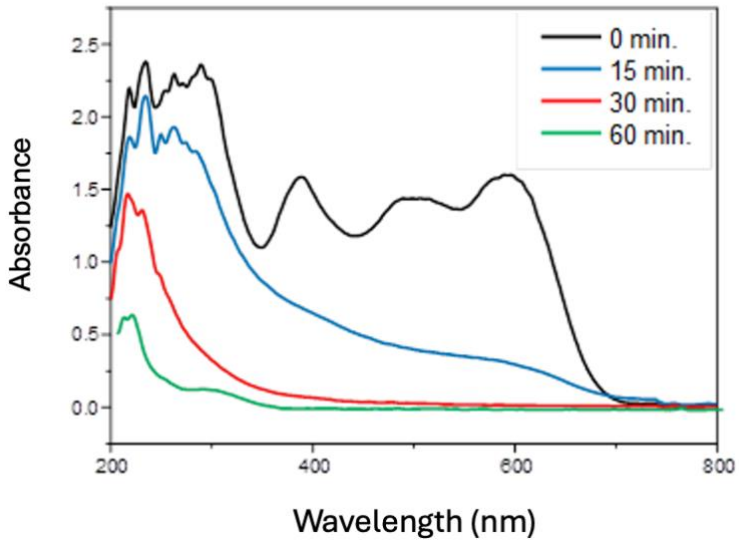
Significant spectral change in the 350-700 nm region (Figure 3), which corresponds to the visible region, could be seen in the first 15 minutes of the photo-Fenton process and it evidenced significant dye mix discoloration. Moreover, there was total signal reduction in this region after 30 minutes of this process, i.e., it took 30 minutes for the photo-Fenton process to fully decolorize the dye mix. In addition, the solution did not show yellowish color after 30 minutes, and it evidenced lack of iron leaching from the cigarette butt cellulose into the solution.

Figure 3. Spectrophotometric monitoring applied to the UV-Vis region of the dye mix during photo-Fenton process application.



Unlike what happened in the Fenton process, there was significant reduction in absorbing bands in the 200-350 nm region, and it indicated that, in addition to decoloring, there was also significant degradation, i.e., the breaking of other bonds in the organic compounds, as well as the breaking of the chromophore group in the dye mix.

The chemical structure of the dyes forming the analyzed sample is quite complex, since they comprise several organic groups, such as aromatic amines and sulfonate groups, among others, which absorb in the 200-350 nm region. The reduced absorption signal in this region points out that these groups were broken down during the photo-Fenton process. The signal remaining in this region after 60 minutes is typical of aldehydes and carboxylic acids, among other low-molecular-weight species (Pavia *et al.*, 2013).

It is worth emphasizing that the cigarette butts did not show physical changes after the advanced oxidative processes were carried out. This finding suggests that they could be reused, and this is one of the advantages of working with immobilized Ir^{2+} and Ir^{3+} ions.

The analyzed results have evidenced the efficiency of the herein performed processes, even at bench scale. The current results were like those reported in other studies in the literature, such as the one conducted by Lan *et al.* (2015), who focused on investigating the reuse of carbon fibers in dye degradation processes. The authors observed that the activity of the investigated material remained unchanged until its fourth use, as well as recorded 100% acid red B dye and 43% total organic carbon removal rates.

Martins (2022) also pointed out cellulose acetate's efficiency in removing dyes from

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industrial effluents by adding it to dye solutions under magnetic stirring from 0 to 10 hours. UV/VIS reading enabled finding dye removal rate higher than 90%.

Huang *et al.* (2022) tested carbonized cigarette filters' efficiency in degrading from 95% to 100% dye Rhodamine B, after 20 minutes in batch reactor system, to show that cigarette butts can be used in all their forms.

Soon and Hameed (2013) immobilized iron in silica to be used in photo-Fenton process to degrade dyes. They recorded degradation efficiency equal to 88% within 30 minutes and observed that silica could be reused in 4 cycles.

Galvão *et al.* (2023) investigated the use of cigarette filters to produce membranes capable of immobilizing iron ions to be applied in advanced oxidative processes.

Based on the existing literature and on the herein observed results, it is possible making significant progress in the advanced oxidative process field based on using cigarette butts as iron ions' immobilizers. However, it is important focusing on issues, such as alternation in cigarette butt samples, among other parameters like temperature.

CONCLUSIONS

Based on the herein reported results, it is possible impregnating Ir^{2+} and Ir^{3+} ions in cellulose acetate of cigarette filters. In addition, iron ions did not leach from cellulose in neutral or basic media. This leaching was only observed in acidic media to a minimal degree, and there was no dye mix adsorption on the cigarette filter. These important factors allow these filters to be used as catalyst in oxidative effluent treatment processes.

The application of Fenton and photo-Fenton processes presented promising results. The Fenton process promoted discreet dye mix decoloring within 60 minutes, without reducing the characteristic band of aromatic compounds (UV).

On the other hand, the mix of dyes was fully decolorized after 30 minutes into the photo-Fenton process. There was significant spectral reduction in the UV region after 60 minutes into it. This finding suggests organic compounds' degradation.

In addition to the herein observed positive effluent treatment results, using cigarette butts in this environment can help preventing the environmental pollution caused by the incorrect disposal of this material.

The herein conducted investigation of advanced oxidative processes was just a preliminary study. Thus, further studies should be performed to check the efficiency of the system focused on using immobilized iron for the large-scale application of cigarette butts to treat industrial effluents, as well as to reuse this material in the aforementioned system, since the current study was conducted at bench scale. In addition to investigating such efficiency at other scales, it is necessary assessing the feasibility of getting cigarette butts enough to carry out these processes, as well as checking their operability from the economic perspective.

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