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# Sustainable recovery of wastewater to be reused in cooling towers: Towards circular economy approach

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## ABSTRACT

Circular economy approach is needed in order to move towards sustainable development. In this frame, the current work is aimed at improving the sustainability of a food industrial plant through circular economy approach, using a waste stream, coming from the washing and disinfection of bottles before product packaging, as source of water for the cooling process. The implementation of this approach is much more relevant in sectors that are water-intensive, such as food and beverages industry. The wastewater produced as results of the washing process has quite high quality, therefore, there reuse in the cooling process is justified. However, it is needed to previously remove the hydrogen peroxide present in that wastewater stream to protect refrigeration circuits from oxidation. Hydrogen peroxide is produced as a result of the decomposition of peracetic acid, which is used as disinfection agent in the washing process, being acetic acid the other byproduct. In order to remove the hydrogen peroxide, catalytic decomposition of hydrogen peroxide with activated carbon was performed, studying both required contact time for different activated carbon/hydrogen peroxide ratios and kinetic models of hydrogen peroxide decomposition at lab scale. Subsequently, the proposed solution was applied at industrial scale, achieving 100 % water replacement in cooling towers. The solution performed in this case can be easily replicated in other plants using peracetic acid as disinfectant.

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## 1. Introduction

Circular economy model is based on increasing resource efficiency, minimizing waste production, in order to decoupling economic growth from resource use. Implementation of circular economy model would contribute to many of the UN's Sustainable Development Goals [1]. In this context, water has been identified as critical input resource, whose circular use should be promoted, being essential to the circular economy [2]. Water is one of the main resources used by industry, particularly food industry is considered within the four more water intensive sectors along with chemical, paper and metal industries. Wastewater is produced in different processes at the food industry, such as washing (food and equipment), refrigeration, rejected streams from osmosis plants, heating circuits, flushing installations and filtration circuit washing. The water consumption of the juice sector must be considered on the basis of the demand for juice in its different packaged variants. In fact, 40 % of the citrus fruits harvested are destined for processing and subsequent packaging [3,4]. In the case of Spain, between 2008 and 2018 there was

a significant growth (approximately 10 % of all varieties) in the daily intake of most processed and packaged citrus products [5]. As a consequence, the more packaging containers were needed, the greater consumption of water in its cleaning and disinfection steps were used [6,7].

It is important to consider that the possibility of water recovery and reuse depends mainly on the particular sector and on the wastewater characteristics, always following appropriate technical criteria [8]. Best Available Techniques document for the particular case of nectar/juice recommends water recycling or reusing of water streams preceded or not by water treatments to reduce water consumption in the process [9]. Furthermore, sustainable use of water resources is key in water scarcity areas, where restrictions due to freshwater limitations can take place affecting the competitiveness of the industries. All these reasons have led food industry to implement water recovery, looking for an integral sustainable water use. Water reuse has achieved reduction of water consumption and improvement of competitiveness within the sector [10]. However, many projects are not viable because of the difficulty of quantifying the real economic balance between investment costs and

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economic and environmental benefits [11].

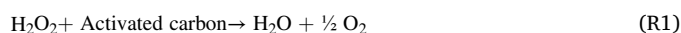
Although wastewater from the juice industry is characterized by high organic loads (5,500 mg/L of COD and 2,500 mg/L of BOD) [12], the amount and type of pollutants in the wastewater varies according to the different processes in the mill. Less polluted wastewater, as the one coming from rinsing bottles, are usually mixed with more concentrated streams to be treated [13]. An efficient management of the water would imply the reuse/recycling of segregated streams within the process [9], according to the principle of water-fit-for-use. Potential streams for reuse or recycle, based on both its high demand during use and its high quality after it, are those used for the product, or for cleaning the containers in the washing/rinsing step [14]. Table 1 shows technologies implemented to reuse water in the food industry. Studies about presence of biocides in the washing machine disinfection process were not found and all of them are focused on reusing the water in the washing machine itself. However, due to the high quality of these effluents, they can be regenerated to be used in cooling towers. Regenerated water used in cooling towers reduces water consumption, achieving a suitable performance of the refrigeration process [12], although the prevention of *Legionella* must be carefully considered [14]. Cooling towers is one of the largest consumers of water in the food industry, followed by the production process itself [11]. The adequate performance of the cooling tower depends on suitable feed water according to its operating regime, the geographical location and the chemical properties of the feed water used [3].

Wastewater generated in rinsing bottles has very low amount of pollutants and sometimes has been directly reused [15–18]. However, the presence of residual biocides and oxidant chemicals can damage the installations in the long term [19]. Therefore, this stream should be treated to avoid the damage. One of the most common biocides in food industry is peracetic acid [20], whose decomposition results in hydrogen peroxide and acetic acid (the three species are in chemical equilibrium). The biocide must comply with current legislation, must be adequate for the installation and the bottles' material (for example, metal containers are more susceptible to corrosion phenomena and migration of metal to the final foodstuff). For this reason, peracetic acid is a common biocide in the washing and disinfection of polymeric containers, such as PET.

Therefore, rinsing wastewater, containing hydrogen peroxide, should be treated to remove this oxidant in order to avoid corrosion of installation as a consequence of the Fenton reaction between hydrogen peroxide and iron at the acidic pH of the wastewater [19]. This reaction would subsequently lead to the generation of hydroxyl radicals, which are highly oxidizing and potentially corrosive for the cooling tower installation [21]. In order to remove hydrogen peroxide, membrane technologies can be considered; however, besides the drawback of the high investment and maintenance costs, its application to remove hydrogen peroxide is limited since the polymeric materials are very susceptible to oxidation in short intervals of time [22]. Transition metals can be used to decompose hydrogen peroxide [6], as well as enzymes, such as catalase [23], and microorganisms, such as *Saccharomyces cerevisiae* [24]. However, none of them seems to be adequate for the treatment of high flows of water, which can suffer concentration peaks of hydrogen peroxide due to the dosing of biocide used. Besides, the use of enzymes or microorganisms requires an exhaustive control and monitoring of the operating conditions because the microbiota can

easily be altered by any change in the reaction conditions.

Looking for a simpler and cost effective treatment for the hydrogen peroxide present in the wastewater from the washing and disinfection of bottles, a different approach was considered in this study. Hydrogen peroxide, as an oxidizing agent, is used to modify properties of the carbons in the activation processes [24,25], and to analyze catalytic properties of the carbons [26], such as acid and active sites. Based on that, the removal of hydrogen peroxide from a wastewater is proposed by its interaction with activated carbon to produce oxygenated water (reaction R1) [27].



Hydrogen peroxide has been used for the obtaining of activated carbon, i.e.: hydrogen peroxide for the activation of carbon with nitric acid mixtures [28] or hydrogen peroxide for final acid washings to increase mesoporous properties of the activated carbon [29,30]. Paternina et al. [27] used an activated carbon for the catalytic decomposition of hydrogen peroxide, using acid washed activated carbon with a low methylene blue value [28,31]. Furthermore, it is expected that chemical simplicity of a wastewater, in terms of chemical species content, will minimize the activated carbon saturation by adsorption and will favor the decomposition process [32]. Referring to the previous reaction 1, hydrogen peroxide in the presence of activated carbon undergoes catalytic decomposition to form radicals, which are reaction intermediates with a short half-life [27]. Once the radicals are gone, it is safe to use the water in the cooling towers.

Some of the advantages of the catalytic decomposition of hydrogen peroxide in heterogeneous phase include that there is no immediate generation of wastes and that the decomposition products do not have toxicity [33]. It is important to consider that activated carbon is not considered toxic according to its quality certificates, although it may have health effects, especially if inhaled. Furthermore, the waste generated in the long term, mainly deactivated carbon is easily managed and classified according to current European legislation [34]. It is a process that can operate in stationary continuous regime facilitating the internal factory processes.

However, there are not studies about the catalytic decomposition of hydrogen peroxide in presence of peracetic acid with real process samples, evaluating the influence of its physical-chemical composition. Furthermore, this potential solution has not been implemented up-scale yet. Besides, there is a need of studying the performance of commercially available activated carbons for this purpose, such as those selected in this study. Therefore, based on the state of the art, it is needed to generate the fundamental knowledge to validate and to scale-up this solution in the food sector to reuse the bottle washing water in the cooling towers.

As the water quality for the rinsing of clean containers process is very high, osmotic water with low chemical load (low electrical conductivity, low organic load and absence of metals and pesticides), the main residual compound present in the wastewater after the process is hydrogen peroxide. The rinsing process leads to low sustainability, which could be increased by regenerating and reusing the wastewater for other needs. Therefore, the objective of this study is to regenerate a wastewater coming from the rinsing of clean containers to be reused as water for the

**Table 1**  
Technologies implemented to reuse water in industrial washing processes of the food industry.

	Beverages / Food Industry	Consider disinfection besides washing	Main treatment technologies	Economic assessment	Reference
General industrial washing processes	Food	No	Sand filtration, chlorination and membrane technologies	No	[11]
	Food	No	Membrane technology, distillation and ion exchange	Qualitative	[21]
	Food	No	Chlorination, ozone and membrane technology	Qualitative	[45]
Specific to the bottle washing process	Beverages	No	Filtration + adsorption + reverse osmosis and filtration + adsorption + ion exchange	Quantitative	[57]
	Beverages	No	Reverse osmosis, microfiltration and ion exchange	Yes	[58]

cooling towers. In order to accomplish this goal, activated carbon has been used to catalytically degrade hydrogen peroxide. Optimization of the process and its kinetics has been studied. The current research proposes a sustainable process at industrial scale that does not generate hazardous wastes, with a solution that promotes the circular economy by the recovery of a large flow (5 m<sup>3</sup>/h), with excellent physical-chemical properties to be used in cooling towers. Besides, it is simple to install and maintain by plant maintenance personnel.

## 2. Materials and methods

### 2.1. Material and analytical methods

Wastewater was sampled after the rinsing and disinfection process of the bottles in a juice industry in Spain. As the bottles are going to be in contact with the juice, this process requires high quality water, which is obtained in a reverse osmosis process (recovery rate of 60 %) from the municipal water supply network. The permeate, with average conductivity of 120 µS/cm, is the influent water for the rinsing and disinfection process. Currently, the average water consumption for this step is 0.2 L per bottle, regardless the volume of the bottle (information taken from the washing machine manufacturer's technical data sheet). If an average of 25,000 bottles/h are washed, the flow that can be recovered is 5,000 L/h. During the washing process, peracetic acid is added in order to keep around 196 mg/L of hydrogen peroxide in the wastewater after the process (the final owner does not authorise for industrial confidentiality to specify the characteristics of commercial peracetic acid, considering under these conditions that it operates with a hydrogen peroxide residual of 196 mg/L). Factory staff usually modifies the dosing pumps as a function of the contamination degree of the bottles, determined by the qualitative criteria of the different maintenance managers who regulate the dosage. Therefore, peaks in the concentration of residual hydrogen peroxide can occur.

Commercial activated carbons were used in this study, and they were selected considering both their availability in significant nominal quantities (tonnes) from regular suppliers, and their cost. Additionally, they are commonly used for the adsorption treatment of industrial wastewater. This study may broaden the applications of these activated carbons in water treatment, with the possibility of being used for catalytic processes. Two activated carbons were selected, which mainly differ in the pretreatment applied during their preparation. Activated carbon 1 underwent acid washing pretreatment, whereas activated carbon 2 did not. Table 2 shows a comparison of their properties. The physical and chemical properties have been extracted from the Technical Data Sheet of each of the commercial products:

The acid-washed activated carbon was additionally characterized by FTIR analysis (Fig. 1), using the Nicolet Magna 750 FT-IR spectrometer, with 0.5 cm<sup>-1</sup> resolution and equipment (detectors and beam splitters) to work in near, medium and far infrared (between 15000 and 50 cm<sup>-1</sup>). The maximum signal was observed at 3450 cm<sup>-1</sup>, which corresponds to

**Table 2**  
Physical-chemical properties of the activated carbons used in the study and provided by the suppliers.

	Acid washed activated carbon (1)	No acid washed activated carbon (2)	Units
Iodine number	1,055	1,060	mg/g
Granulometry	12*40	12*45	US std. Sieve
Humidity when bagging	5	6	%
pH	5.0	8.1–10.2	pH units
Apparent density	440	450	kg/m <sup>3</sup>
BET surface	1100	1200	m <sup>2</sup> /g
Methylene blue	230	340	g/Kg
Mean particle diameter	0.95	0.98	mm

hydroxyl groups [35], from phenolic and/or carboxylic groups, and N–H groups (pyroles) [35]. Another characteristic peak was observed at approximately 1130 cm<sup>-1</sup>, indicative of a characteristic point in carbonaceous materials activated with phosphoric acid [36,32]. The absorbance values in the spectrum for peak intensity are low due to the existence of covalent links whose total dipole moment is zero, which makes difficult to represent the results.

PVC was the selected material for the industrial scale process and its technical specifications are shown in Table 3. The data were provided by the manufacturer (Baeza, Spain) in their quality certificates.

Acetic acid was supplied by Sigma Aldrich (United States) with more than 99 % of purity, sodium hydroxide solution was supplied by Sigma Aldrich, (United States) at 70 %v and more than 99.5 % of purity, potassium acid phthalate was supplied by Merck (Germany) with more than 99.5 % of purity and nitric acid was supplied by Merck (Germany) at 70 %v with more than 99.5 % of purity. Aluminum sulfate, iron (III) chloride, both at 40 %v, polyaluminum chloride hydroxide sulfate at 35 %v and polyacrylamide at 40 %v were supplied by Fupinax (Spain).

pH, electrical conductivity and temperature were measured with HANNA HI98130 (Spain), turbidity with HANNA HI93703 C (Spain), following ISO 7027, and hydrogen peroxide with HANNA HI3844 (Spain) through a titration method. Total organic carbon (TOC) was measured with a TOC analyzer QbD1200 from HACH (Spain). Firstly, TIC was removed from the water sample in presence of H<sub>3</sub>PO<sub>4</sub>. Complete oxidation of organic carbon into CO<sub>2</sub> gas was carried out in presence of UV light and powerful oxidizer, (NH<sub>4</sub>)<sub>2</sub>S<sub>2</sub>O<sub>8</sub>, and then measured in NDIR detector and quantified by integrating the area under the curve. Chemical oxygen demand (COD) was measured with the photometer HI83399-02 from HANNA (Spain), using the dichromate method (Standard Method 5520 C). Acetic acid was measured by titration with 1.235 M sodium hydroxide solution, previously standardized with potassium acid phthalate, using phenolphthalein indicator [37].

Methylene waste index was measured in the activated carbon at the industrial phase because it provides information about the catalytic and surface properties of the activated carbons [38,39], following the method describe by Rojas et al. [40]. Although this measurement is related to the adsorption capacity of the activated carbon, it will allow to determine the effectiveness of the catalyst along their industrial use, being a simple and low cost method.

Oxidation induction times were determined in the PVC pipes to assess their corrosion times before their use, as a control sample, after contact with the wastewater and after contact with the treated water. It was determined the interaction of the material on an industrial scale according to Standard UNE-EN ISO 11357-6:2013 [41], using differential scanning calorimeter DSC PT 1000 from Linseis (Germany).

### 2.2. Experimental procedure

A conceptual map with the sequence of the experimental procedure is shown in Fig. 2. All tests have been repeated three times.

The possibility of applying a pretreatment to the wastewater was assessed to improve the subsequent catalytic decomposition of hydrogen peroxide with the activated carbon. Therefore, coagulation/flocculation pretreatment was applied to test their effect on the wastewater quality, particularly on the turbidity. Initially, pH was adjusted to 7.5 by adding the theoretical stoichiometric amount of sodium hydroxide, which is the pH recommended by the coagulant and flocculant supplier. Two different coagulants, aluminum sulfate and iron (III) chloride, which were added at the dose of 500 mg/L, and two different flocculants, polyaluminum chloride hydroxide sulfate and polyacrylamide were added at different doses between 30 and 60 mg/L. The process was kept under magnetic agitation in a beaker at 300 rpm during 15 min. After the coagulation/flocculation process, the treated sample was left to stand for 5 min. to allow the separation of the solid and clarified phases. The clarified water was then analyzed.

Hydrogen peroxide decomposition process was performed putting in

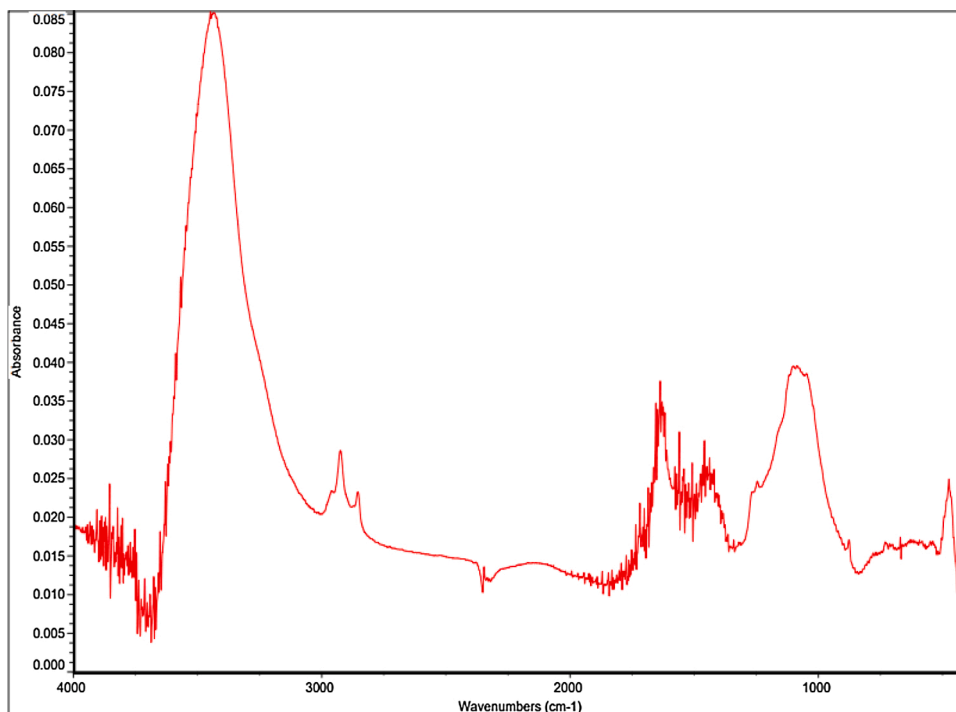


Fig. 1. Fourier Transformed IR spectrum of acid washed activated carbon.

Table 3

Technical specifications of PVC provided by the supplier.

Property	Value
Nominal diameter (cm)	15
External diameter (cm)	16
Nominal pressure (kg/cm <sup>2</sup> )	4
Thickness (mm)	3.2
Color	Grey RAL701
Specific weight (g/cm <sup>3</sup> )	1.40

contact 11, 22 and 33 g of activated carbon with 100 mL of the sampled industrial wastewater, containing around 196 mg/L of hydrogen peroxide. The treatment was performed in Erlenmeyer flasks under slow

magnetic agitation (120 rpm) without alter the natural pH of the wastewater (3.8 when no pretreatment was applied and 7.5 when the pretreatment was applied). Aliquot samples were withdrawn every minute to measure the concentration of hydrogen peroxide along the experiments. Simultaneous measurements of pH, COD and TOC have been carried out every 5 min. Different ratios of activated carbon (adsorbent)/hydrogen peroxide (adsorbate) were evaluated to determine the optimum decomposition conditions and the specific kinetic models for each condition.

The influence of pH on the catalytic decomposition of peroxide was studied by acidifying and neutralizing the pH in the interval 3.20–8.12 for an adsorbent/adsorbate ratio of 0.75 in the time interval of 15 min. pH adjustments have been carried out through the use of diluted nitric acid at 3% and diluted sodium hydroxide at 5%.

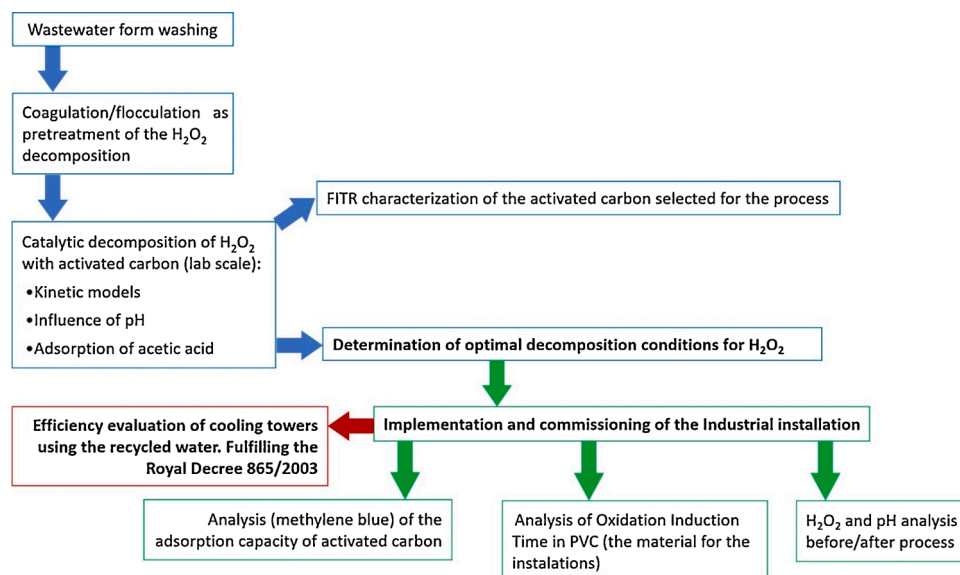


Fig. 2. Conceptual map of the experimental processes performed in this study.



Additionally, the adsorption of acetic acid on the activated carbon was evaluated to determine its influence on the efficiency of the process. Adsorption tests were carried out with an initial concentration of acetic acid between 0.0057 and 0.013 M and at different temperatures, in an interval like the one of the industrial process, between 15 and 35 °C.

The industrial process was run along almost one year to monitor the functionality of the process. pH and hydrogen peroxide concentration were analyzed daily along 48 weeks, before and after the wastewater treatment. Microbiological and physical-chemical parameters of the treated water used in the cooling tower were measured monthly in the mill, according to the current legislation [42–44]. Methylene blue was measured weekly in the mill to assess the performance of the activated carbon.

### 3. Results and discussion

#### 3.1. Industrial wastewater characterization

The parameters that should be analyzed depend on both the characteristics of the effluent and the intended end-use of the regenerated water [21,45]. There are 4 washing machines in the plant that converge to a common mixing tank where the wastewater is collected. It is in this tank that the physical-chemical characterization has been carried out (Table 4). Microbiological analyses were not performed because the use of peracetic acid, which is a powerful biocide, in an osmotized water, used for the washing and disinfection process, allow assuming pathogens and fecal indicators are absent and heterotrophic bacteria concentration is low with respect to regulations or guidelines.

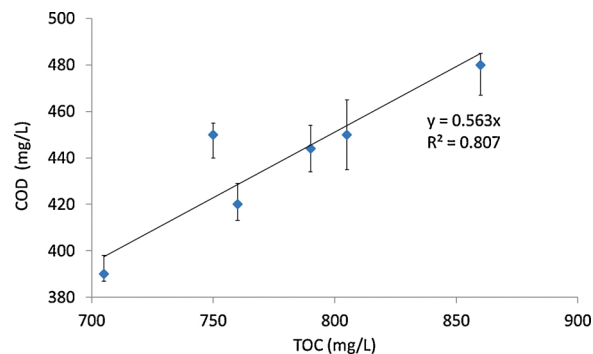
Considering the high quality of this water, it is surprising the high values of COD and TOC. The main source of organic matter is the biocide, peracetic acid, added during washing. Additional COD and TOC measurements were performed in random samples, taken from the “mixing tank” during a period of 6 weeks, obtaining almost linear correlation between COD and TOC (Fig. 3). TOC concentration in the wastewater varies depending on the amount of peracetic acid added to the osmotized water, which is the main source of organic carbon in the wastewater. Peracetic acid decomposes into hydrogen peroxide and acetic acid, thus the TOC in the wastewater is the result of both the remaining concentration of peracetic acid and the concentration of the produced acetic acid. On the other hand, COD concentration in the wastewater varies according to the remaining concentration of peracetic acid and the concentration of both produced hydrogen peroxide and produced acetic acid. The linear correlation between TOC and COD confirms that the COD concentration is the result of the chemical balance between peracetic acid, acetic acid and hydrogen peroxide (Fig. 3).

However, TOC values obtained in all the random samples are quite higher than the ones of COD. Calculated theoretical values of TOC and COD are 32 mg/L and 63 mg/L, respectively (for each 100 mg/L of peracetic acid). The degradation of peracetic acid into acetic acid and hydrogen peroxide would increase the value of COD, as hydrogen peroxide contributes to the COD value, but not to the TOC, and 100 mg/L of acetic acid would contribute with 40 mg/L to the TOC and with 107 mg/L to the COD. Therefore, there has to be another source of carbon in the wastewater that would contain organic compounds that are partially or not oxidized during the COD measurement, which can include

**Table 4**

Physical-chemical characterization of the industrial wastewater after the washing and disinfection process.

Parameter	Mixing tank
pH	3.8
Electrical conductivity (µS/cm)	108
Turbidity (NTU)	40
COD (mg/L)	444
Hydrogen peroxide (mg/L)	196
TOC (mg/L)	790



**Fig. 3.** Linear correlation between COD and TOC for the analyzed random samples of wastewater.

aromatic and aliphatic organic compounds [46]. There are three possible sources of organic carbon: remains of machinery lubricant, i.e. blower, timing belts, bearings or shut-off valves; remains of polymers or raw materials from the production process, such as PET (the bottles are manufactured in the same mill by a blowing process), ethylene glycol and/or terephthalic acid. Rojas et al. [40] analyzed the existence of traces of PET in drinking water contained in PET containers. Therefore, it would be possible to find PET also in the studied wastewater. The third possible source are remains of other products used in the preventive/corrective maintenance of the washing machine, highlighting among them detergents and cleaners for CIP, industrial sanitizers, high foam detergents and additives for washing containers. It has not been possible to determine the exact cause of this deviation in the COD-TOC ratio due to the unknown details of the process that belongs to the know-how of the company.

#### 3.2. Assessment of the effect of coagulation/flocculation pretreatment

The motivation for selecting coagulation/flocculation treatment was to keep the sustainable approach of the project, using already existing materials, chemicals and installations in the industrial site for carrying the treatment. Furthermore, this technology is well known, easy to control and to install. Results of the coagulation/flocculation pretreatment are summarized in Table 5. The pretreatment was carried out at pH of 7.5, according to the manufacturer indications. The performance of the process was determined by measuring turbidity of the clarified water.

Polyacrylamides used in combination with iron (III) chloride did not promote any formation of flocs, as a result there was no reduction of turbidity. However, the use of iron (III) chloride as a coagulant with polyaluminum chloride hydroxide sulfates as a flocculant resulted in 50 % reduction of turbidity. Therefore, the use of polyacrylamides was ruled out. The combination of aluminum sulfate as coagulant and polyaluminum chloride hydroxide sulfate as flocculant resulted in reductions of turbidity between 50 and 70 %, depending on the dose of flocculant. The optimum dose of flocculant was found at 40 mg/L, which achieved the lower value of turbidity. Besides, the sedimentation processes adding aluminum sulfate were quicker than the one with iron (III) chloride.

The best performance of aluminum sulfate may be related to the lower solubility of most of the sulfates salts that can be formed and sediment during the process in comparison with the salts formed with chloride. However, Cogollo et al. [47] obtained the opposite results, studying chloride and sulfate-based coagulants, showing that the first one provides better results. On the other hand, Barlares [49] carried out a comparative study between these both coagulants in water for human consumption, observing better results for aluminum sulfate than for iron chloride, achieving 100 % reduction of turbidity. Results previously published in the literature are indicative that the best performance of

**Table 5**

Comparison of the efficiency of coagulation/flocculation chemicals.

Coagulant	Coagulant dose (mg/L)	Flocculant	Flocculant dose (mg/L)	Turbidity (NTU)	Turbidity reduction (%)
Iron (III) chloride	500	Polyacrylamide	30	40	0
Iron (III) chloride	500	Polyacrylamide	40	40	0
Iron (III) chloride	500	Polyaluminum chloride hydroxide sulfate	30	20	50
Aluminum sulfate	500	Polyaluminum chloride hydroxide sulfate	40	12	70
Aluminum sulfate	500	Polyaluminum chloride hydroxide sulfate	50	14	65
Aluminum sulfate	500	Polyaluminum chloride hydroxide sulfate	60	20	50

one coagulant or the other would depend on a variety of factors, such as pH or selected flocculant. In this case, for the studied options, the best coagulation/flocculation conditions treatment would be the combination of 500 mg/L of aluminum sulfate followed by 40 mg/L of polyaluminum chloride hydroxide sulfate, at a pH of 7.5.

During the sedimentation process at the optimum conditions, it can be seen, after some time, that the flocs tend to float. Therefore, a flotation treatment would be recommended for the separation process after the coagulation/flocculation treatment.

### 3.3. Catalytic decomposition of $H_2O_2$ with activated carbon

The catalytic decomposition of  $H_2O_2$  with activated carbon was initially performed with the wastewater without coagulation/flocculation pretreatment, as it was the preferred solution at industrial scale. Complete removal of  $H_2O_2$  was achieved after 15 min of contact time, regardless the adsorbent/adsorbate ratio applied with the acid washed activated carbon (Fig. 4). However, at shorter times, the removal of  $H_2O_2$  was higher when the adsorbent/adsorbate ratio was higher. For the activated carbon not washed with acid, complete decomposition of hydrogen peroxide was not achieved after 15 min of contact time (Fig. 4). That could be related to the acidity of the effluent to be treated (3.8, Table 4) as a result of the residual acetic acid in the wastewater. Acid wastewater is neutralized by the basicity of the surface of the non-acid washed activated carbon (8.1–10.2, Table 2). Consequently, in the active sites of this kind of activated carbon the fastest stage would be the neutralization, limiting the decomposition of the hydrogen peroxide.

For the determination of the kinetic models, the residual hydrogen peroxide for the highest adsorbent/adsorbate ratios (0.50 and 0.75) was measured at one-minute time intervals using the acid washed activated carbon (Fig. 5), which was the one that obtained the best results in the preliminary experiments. The non-acid washed activated carbon has not provided a complete decomposition of the hydrogen peroxide; therefore, it was discarded from the kinetic study. The selection of adsorbent/adsorbate ratios was conditioned by operational advantages on an

industrial scale (less fluidization of the activated carbon, higher content in active sites and longer operating life, which will avoid later stoppages due to maintenance of the deactivated bed). Additionally, the comparison with the pretreated wastewater has been performed to establish if the pretreatment was able to improve the results.

Removal of hydrogen peroxide from the pretreated wastewater was much lower than in the case of the no pretreated wastewater (Fig. 5). As it can be observed, there are not meaningful differences between 0.50 and 0.75 adsorbent/adsorbate ratios for the no pretreated wastewater, being the tendencies for both ratios the same, with the curve acquiring an almost infinite slope in the first minute of reaction time. That is due to the high degradation rate of hydrogen peroxide due to its high reactivity (the activated carbon acts as a catalyst promoting the decomposition of the hydrogen peroxide), considering also the sensitivity of the hydrogen peroxide analysis method. These patterns have also been observed by other authors [27]. The worsen behavior of the pretreated wastewater could be related to the initial pH of the wastewater. Whereas the original wastewater had a pH of 3.8 due to the presence of peracetic acid, hydrogen peroxide and acetic acid, the pretreated wastewater was conditioned to pH 7.5, which was the adequate for the coagulation/flocculation process. As the activated carbon used was the acid-washed one, the acid pH of the wastewater is recommended. The addition of sodium hydroxide during the pretreatment of the wastewater affects the performance of the acid-washed activated carbon, due to the neutralization of the acid sites, as previously found by Cortés [48], which implies that the kinetics of the neutralization is quick enough to affect the kinetics of the hydrogen peroxide decomposition, resulting in a less effective decomposition process, approaching the behavior with that of the not acid washed activated carbon. As a conclusion, there was not improvement in the catalytic treatment when this pretreatment was applied, in fact, worse results were obtained. Therefore, this pretreatment process can be ruled out for industrial implementation.

The kinetic models have been obtained using the integral method. The correlation coefficients show the reliability of the fit, similar to those previously obtained by Paternina et al. [27]. From the slopes of

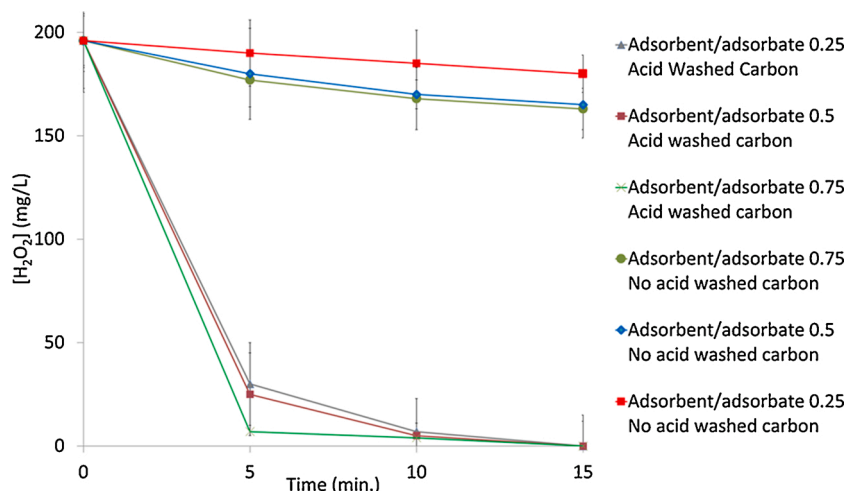


Fig. 4. Catalytic decomposition of hydrogen peroxide with acid washed activated carbon and not acid washed activated carbon, without wastewater pretreatment.

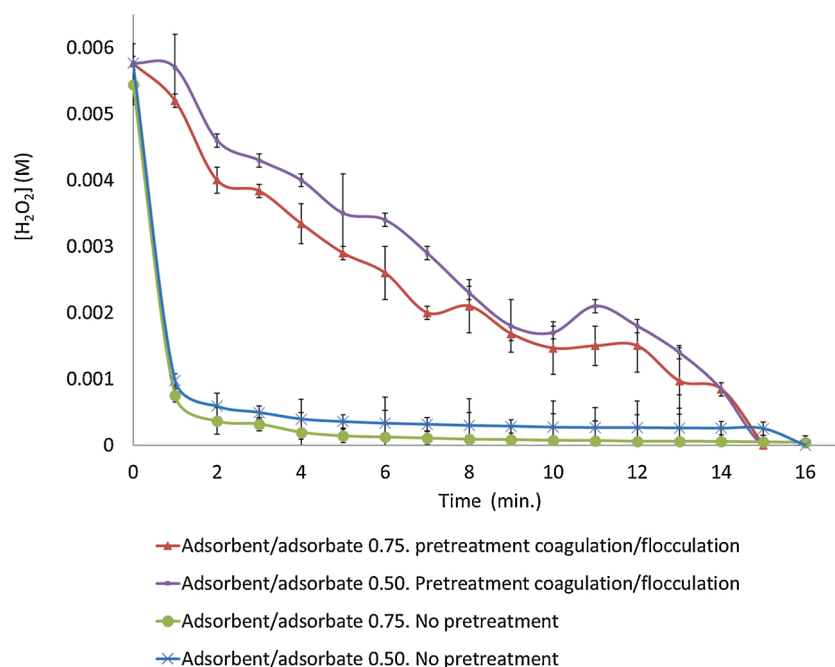


Fig. 5. Hydrogen peroxide concentration profiles along the experiments at different conditions (Acid washed activated carbon).

each linear adjustment, the kinetic constant was obtained, which provides an idea of how fast the hydrogen peroxide is decomposed. In this case, first order reaction was considered according to Eq. (1) and the integrated Eq. (2), whose graphical representation is shown in Fig. 6.

$$\frac{-dC_A}{dt} = kt \quad (1)$$

$$\ln\left(\frac{C_{A0}}{C_A}\right) = kt \quad (2)$$

Table 6 shows the kinetic constants for first order reaction. According to Fig. 5, higher values of kinetic constant were obtained for the untreated water compared to the values of the kinetic constant of the pretreated water. Other kinetic models were tried, but the best fit was obtained for order 1 using integral method, although when the amount of adsorbent increased, the adjustment at the beginning of the reaction to order 1 was worse. Looking at Fig. 5, it can be seen a deep decrease of hydrogen peroxide during the first minute of reaction, which is more pronounced when the amount of adsorbent is higher. That first step could be considered as zero order kinetics. The pattern drastically changed thereafter, showing the proposed first order kinetics clearly.

Considering that the neutralization of the acidic active sites because of the pH of the medium competes with the decomposition of hydrogen

Table 6  
Kinetic constants and correlation coefficients.

Decomposition conditions		k (min <sup>-1</sup> )	R <sup>2</sup>
Adsorbent/adsorbate ratio	Pretreatment		
0.75	Yes	7.716	0.915
0.75	No	8.754	0.985
0.50	Yes	7.674	0.971
0.50	No	7.830	0.976

peroxide, effect of wastewater pH on the process efficiency was studied. The influence of pH was analyzed between 3.20 and 8.12 along 15 min of treatment time (Fig. 7). This range has been selected to avoid corrosion and/or calcification phenomena in the final recovery process. Several authors have studied the influence of the pH in adsorption processes [50–52], focusing on the thermodynamics of adsorption, not evaluating cases of competition between chemical reactions as it is the present case.

At the beginning of the experiments (time = 0) there was differences

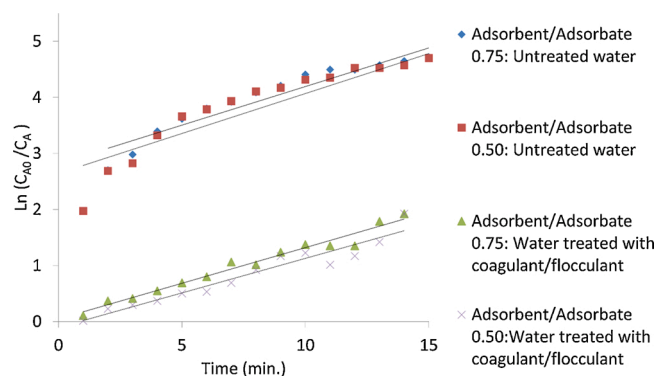


Fig. 6. Adjustment of data to first order kinetics.

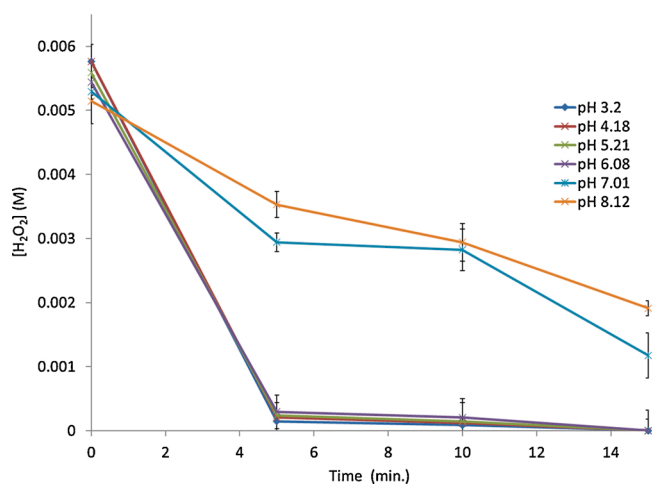


Fig. 7. Influence of pH on the catalytic decomposition of hydrogen peroxide.

in the initial hydrogen peroxide concentration, which are consequence of the influence of pH on the chemical equilibrium of peracetic acid, moving towards the formation of hydrogen peroxide as pH decreases. Results show that at higher pH values, the decomposition of hydrogen peroxide was reduced. However, it is not necessary to work at extremely low values of pH because at pH 6 or below complete degradation of hydrogen peroxide was achieved in 15 min, obtaining the same patters between 6.08 and 3.20 (Fig. 7).

In addition to the measurement of hydrogen peroxide, pH, COD and TOC were analyzed every 5 min along the experiment with acid washed activated carbon at adsorbent/adsorbate ratio of 0.75 without any pretreatment to assess their evolution (Fig. 8). pH decreased along the reaction due to the acetic acid generated from the decomposition of peracetic acid. In the case of COD and TOC, no significant differences can be address based on the results obtained. Therefore, no significant adsorption of organic compounds took place on activated carbon surface, being the catalytic decomposition of hydrogen peroxide the main process.

### 3.4. Adsorption of acetic acid on the activated carbon

Although previous results indicated that there is no adsorption of organic compounds, according to the literature, acetic acid adsorption is common in water treatment processes using activated carbons [53,54]. Therefore, it is necessary to determine the degree of adsorption of this molecule with the selected activated carbon because that can determine the saturation of the activated carbon pores, which would lead to a reduced lifespan of the activated carbon. This degree of adsorption will depend on the type of activated carbon used, and mainly, on the type of activation.

Adsorption tests were carried out with an initial concentration of acetic acid between 0.0057 and 0.013 M and at different temperatures, in an interval like that of the industrial process, between 15 and 35 °C.

The Freundlich isothermal model that follows Eq. (3) has been used to adjust the adsorption models based on the results obtained from previous studies [52]. Other models such as Langmuir's model were analysed, however, the best fit was Freundlich's model:

$$\frac{X}{m} = K \cdot C^{1/n} \quad (3)$$

Where:

- X: Adsorbate mass (g).
- m: Adsorbent mass (g).
- C: Equilibrium concentration of adsorbate in solution (mg/L).
- K and 1/n: Constants for adsorbent and adsorbate, dependent on the temperature.

Fig. 9 shows the Freundlich isotherms at three temperatures for different adsorbate/adsorbent ratios. The acetic acid analysis was performed after 10 min of contact time to maintain a value slightly below

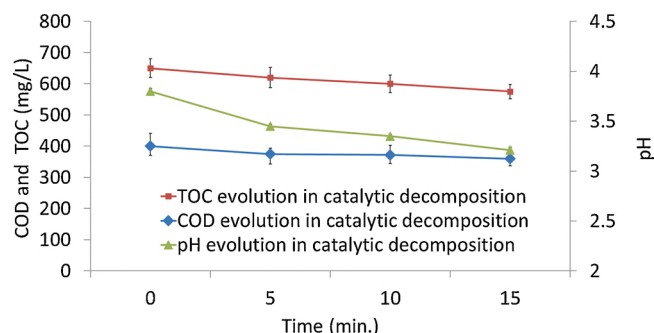


Fig. 8. Evolution of pH, COD and TOC in catalytic decomposition.

the optimal for the effluent. According to the Freundlich model, the parameters of the adjustment and their corresponding correlation coefficients were obtained (Table 7). It can be observed that the pattern of the isotherms is similar for 15 and 25 °C, however for 35 °C is different. In general, as the temperature decreased, the concentration of acetic acid at the equilibrium is lower; therefore, the adsorption is more effective at lower temperature, which agrees with the exothermic behavior of the acetic acid adsorption process previously reported by Freitas et al. [54].

At each temperature, the removal of acetic acid during the adsorption was measured. At adsorbate/adsorbent ratio of 0.968, which is the closest ratio to the adsorbent/adsorbate ratio of 0.75 (selected for the industrial process) removal of acetic acid only achieved 9.17 % at 15 °C, which is negligible compared to the catalytic decomposition of hydrogen peroxide that is the controlling stage of the process.

As it was previously mention, the type of activation of the carbon affects the adsorption of acetic acid, which was clearly reflect in this study. Whereas the acid sites in the activated carbon favored the decomposition of hydrogen peroxide, the adsorption of acetic acid was not favored. That agrees with the generally hydrophobic character of activated carbons with little affinity for polar substances [55], such as the acetic acid. However, activated carbon with zinc chloride leads to high adsorption of acetic acid and excellent results in the adjustment to adsorption models [52].

### 3.5. Industrial application of the catalytic degradation with activated carbon

Initially, the annual consumption in the cooling towers over several years was studied, observing that 100 % recycled water can be used from the wastewater for washing and disinfection process, establishing in 5 m<sup>3</sup>/h the needs for the cooling towers. If that flow is not enough due to higher demand in the towers, a mixing plan with drinking water would be used. Even in this case, economic savings and adequate functional performance in the towers would be achieved. Any surplus of wastewater from bottle washing would be sent to the wastewater treatment plant, although other possible reuse should be assessed in compliance with current legislation.

Compatibility of the effluent with PVC was assessed due to its utmost importance for the economics of the industrial project. PVC is commonly used in different applications, including sanitary water, greywater, reverse osmosis machines or other water treatment processes. One of the physical parameters that allow to characterize the chemical resistance of polymeric materials is the oxidation induction time (OIT) [41]. This test establishes the time needed for a material to be oxidized under oxygen containing environment, at a specific temperature, and in contact with different additives, including antioxidants and stabilizers among others.

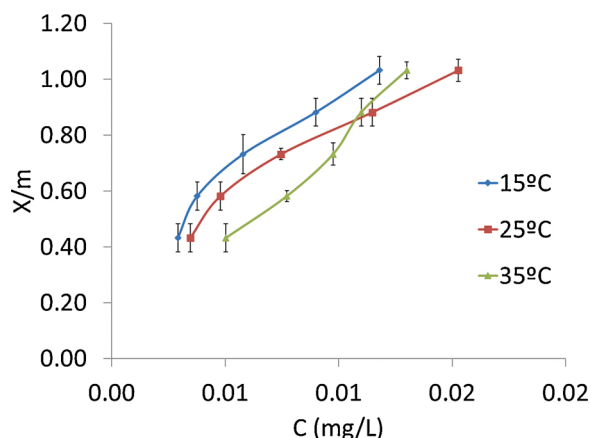


Fig. 9. Freundlich isotherms obtained at different temperatures.



**Table 7**  
Freundlich isotherms coefficients.

T° (°C)	K	n	Correlation coefficient
15	0.0106	0.6013	0.9693
25	0.0138	0.5956	0.9800
35	0.0129	0.9425	0.9809

Therefore, OIT was measured for the PVC material selected for the industrial process. The analysis of the material prior to be used was carried out as a control to make a comparison (Fig. 10). Additional measurements were performed for the PVC material in contact with the wastewater before and after the treatment with the activated carbon, after an operation regime of one year, working 8 h per day (Table 8).

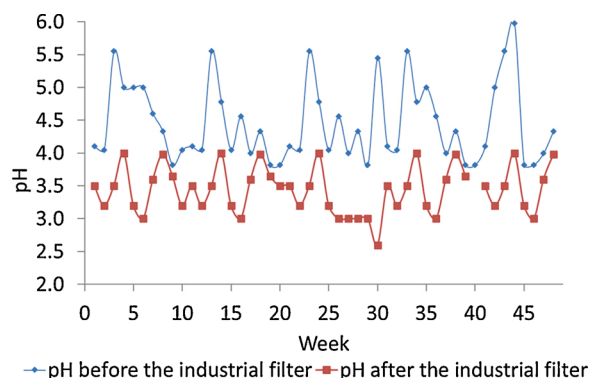
The results show minimal variation in OIT times, noting that there is not oxidation of the treated effluent. On the other hand, there is a slight effect of the wastewater containing hydrogen peroxide, more accentuated in the inner radius, with a percentage of reduction of 4.88 %. Expert associations, such as ASETUB [56], recommends a maximum reduction of the OIT of 40 %. Thus, the minimal oxidation of the material indicates the full functionality and compatibility of PVC in the industrial process. Even though more expensive materials could be selected, OIT results are satisfactory, being safe to select PVC, one of the most common polymers, in the design of the water reuse process. This value is relevant because it depends on factors such as the temperature, the type of fluid circulating, the incidence of radiation, etc.

The industrial operation along one year proved the effectiveness of the catalytic decomposition of hydrogen peroxide using commercial acid washed activated carbon, as well as the suitability of the regenerated water as source for the cooling towers. Fig. 11 shows the variation of the pH before and after the treatment, observing the expected acidification as a consequence of the acetic acid generated. Fig. 12 shows the variation of hydrogen peroxide before and after the treatment, observing the almost constant concentration of hydrogen peroxide in the influent to be treated, and the total absence of hydrogen peroxide in the regenerated water. The results shown in Figs. 11 and 12 corroborate the analysis and orders of magnitude of the samples studied at the laboratory scale.

After one year using the recovered effluent, monthly results show values within the permitted ones. The average values over a year with monthly measurements are shown in the Table 9.

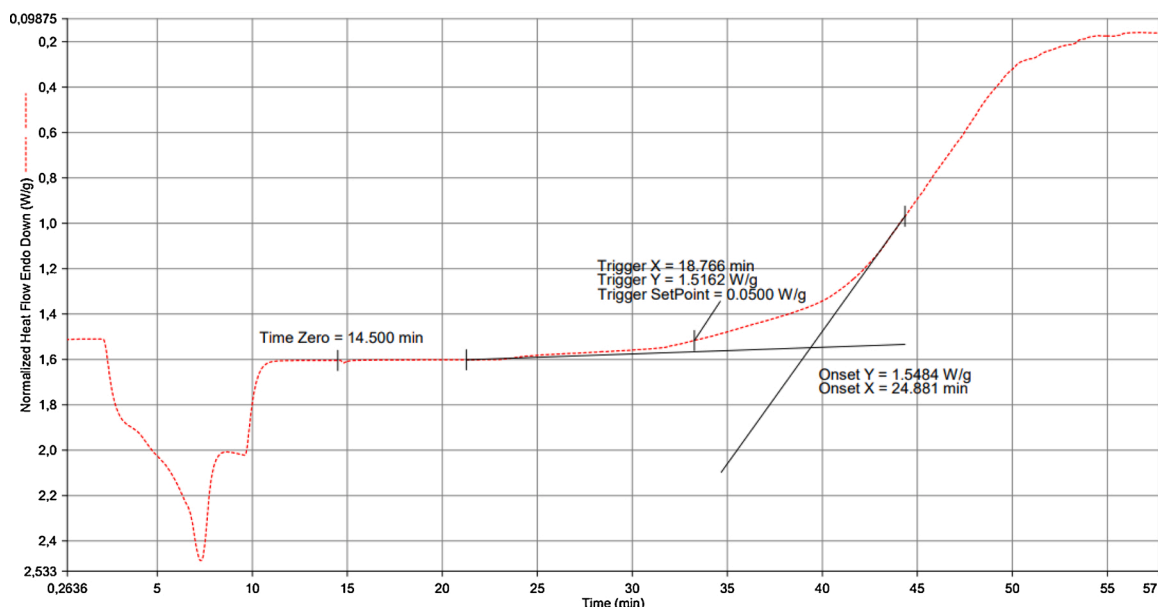
**Table 8**  
OIT results in PVC with untreated and treated effluent.

	PVC	PVC installed for one year	
		OIT (min/13 g) Untreated effluent	OIT (min/13 g) Treated effluent
External radius		24.880	24.880
Average radius	24.881 (for all)	23.709	24.800
Internal radius		23.667	24.876



**Fig. 11.** Variation of the pH per week (each weekly average includes 5 working days).

In order to verify the functionality and capacity of the activated carbon in the industrial treatment, methylene blue was analyzed. This property does not provide a detail of the catalytic capacity of the activated carbon, but rather its adsorption capacity, an aspect related to its specific surface. This technique has been selected due to the low cost and simplicity of the measurement, considering that weekly replications must be made in the factory where it is installed. Fig. 13 shows a slight reduction after one year that did not affect the catalytic capacity of the process, according to the complete removal of hydrogen peroxide.



**Fig. 10.** Thermogram of the PVC used before contact with the effluents.

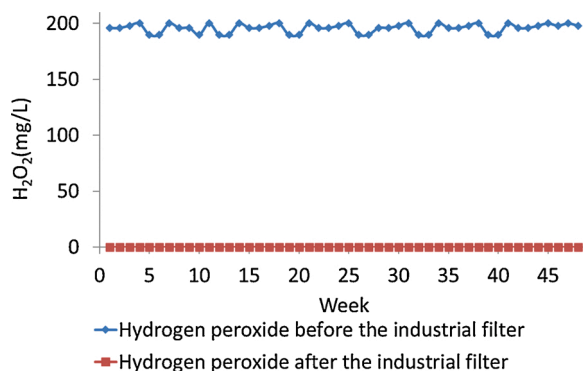


Fig. 12. Variation of hydrogen peroxide weekly (each weekly average includes 5 working days).

Table 9

Monthly measurements (12 months) in cooling towers.

PHYSICAL-CHEMICAL PARAMETERS (Monthly)	
Temperature (°C)	20.5 ± 3.7
pH (6.5–9.0)	7.4 ± 0.3
Conductivity (µS/cm) (Setpoint 3,500 µS/cm)	1,700 ± 850
Iron (mg/L) (<2 mg/L)	0.70 ± 0.15
Isothiazolone biocide (minimum 50 mg/L)	226.67 ± 10.43
Turbidity (NTU) (<15 NTU)	1.4 ± 0.3
AEROBIC MICROORGANISMS (ISO 6222)(Monthly)	
Aerobic microorganisms at 22 °C (<10,000 CFU/mL)	1,769 ± 340
Aerobic microorganisms at 36 °C (<10,000 CFU/mL)	1,775 ± 365
<i>Legionella</i> (ISO 11731) (Quarterly)	
<i>Legionella spp</i> (CFU/L)	Not detected

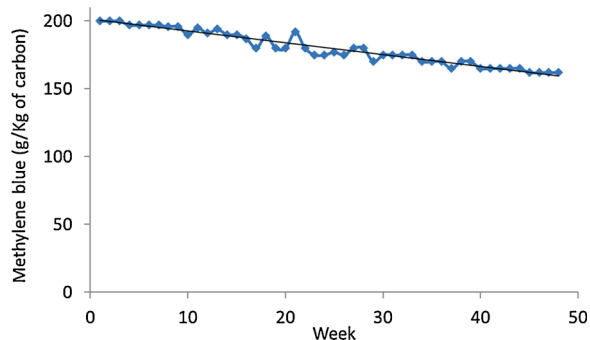


Fig. 13. Weekly variation of methylene blue index in industrial filter.

### 3.6. Economic analysis

To determine the economic viability, a quantitative analysis of the investment cost was carried out (Fig. 14), considering the water savings achieved with this solution. For this purpose, a project investment of 21,663 € was considered for the installation and start-up of the treatment. The recovered flow is 5 m<sup>3</sup>/h, obtained during 253 working days with 8 h of operation per day, which are the operating conditions of the washing process. Considering that, the catalytic treatment with activated carbon is able to substitute all the fresh water, whose cost summed up 2.64 €/m<sup>3</sup>, resulting in annual saving of 26,716 €. The performance of the activated carbon treatment after one year of operation indicates that it still has catalytic activity. It is assumed that by the 30th month the catalytic bed (acid washed activated carbon and support) will have to be changed, mainly due to the loss of granulometric and textural properties, which could lead to a reduction in particle size and its loss with the treated effluent. Therefore, the cost of catalytic bed replacement has

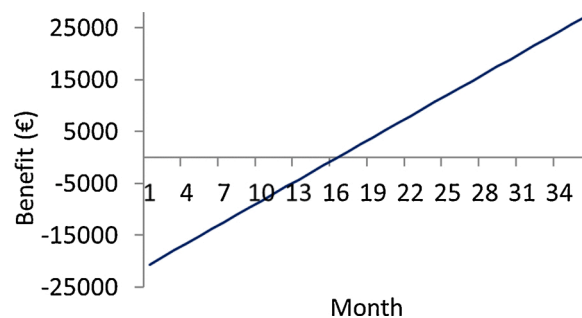


Fig. 14. Benefit vs. time of the industrial installation considering the investment, monthly preventive maintenance and catalytic activated carbon change.

been considered as depreciation. Furthermore, preventive maintenance was considered on a monthly basis for an amount of 865 €/month for the verification of the most significant variables, the sampling of activated carbon and the analysis of deviations by two qualified technicians. Under these economic conditions, it can be concluded that the profit acquires positive values approximately in the 16th month since its installation and start-up.

## 4. Conclusions

Catalytic decomposition of hydrogen peroxide, coming from the chemical equilibrium with peracetic acid and acetic acid, was successfully achieved by using commercially available activated carbon. The acid washed activated carbon achieved the complete decomposition of hydrogen peroxide in 15 min, regardless of the adsorbate/adsorbent ratio. However, unwashed activated carbon was not as efficient for this acid wastewater. First order kinetics was obtained for the hydrogen peroxide decomposition, being the decomposition rate faster when the adsorbate/adsorbent ratio was higher. Physical-chemical coagulation/flocculation pretreatment, which improved water quality, do not result in better performance of the catalytic decomposition, mainly due to the high pH of the pretreated water. It is recommended to work below pH 6 since above this value there is a neutralization of acidic active sites of activated carbon that competes with the catalytic decomposition of hydrogen peroxide.

No interference of acetic acid due to adsorption on the activated carbon has been observed. The characteristics of the treated water reached the quality to be used in the cooling towers. The selected material for the installation was PVC, which presents a total compatibility with the effluent and it is an economical and resistant material. The activated carbon does not lose its catalytic properties at least in the period of one year, so the project is economically viable.

The proposal was successfully implemented at industrial scale, with 5 m<sup>3</sup>/h of water savings, 100 % of the water needs in the cooling towers. The plant can be amortized in 16 months from its installation and start-up, taking into account the monthly scheduled maintenance, and considering the change of the activated carbon bed in the third year.

The circular economy approach for using regenerated water can be easily replicated in other plants in the food industry that use peracetic acid as disinfection agent in some of their processes.

## Declaration of Competing Interest

The authors report no declarations of interest.

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