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Treatment of door-manufacturing factories wastewaters using CDEO and other AOPs A comparison

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ABSTRACT

In this work, three advanced oxidation technologies have been studied to improve the quality of the effluents of a physicochemical process and of a combined physicochemical–biological process during the treatment of actual industrial wastes of wooden door-manufacturing factories. From the treatment point of view (neglecting costs), advanced oxidation processes can be successfully used to treat both, coagulated and biologically treated wastes. Conductive-diamond electrochemical oxidation (CDEO) was found to be the more effective technology because it can reduce completely the chemical oxygen demand (COD) (no production of refractory compounds) with a very high current efficiency. However, from the economic viewpoint, the direct treatment of the coagulated wastes can not be recommended because it is very expensive. Only Fenton oxidation or conductive-diamond electrochemical oxidation can be cost-efficiently used to refine the quality of the effluent of the biological process.

1. Introduction

A small town in the centre of Spain concentrates an important number of door-manufacturing factories. Contrary to what it can be expected, the production of wastes in this type of industrial activities is not limited to solids particles coming from the wood machining, but it also includes small flowrates of a very-highly polluted liquid waste coming from the cleaning of the machinery used in the gluing process, whose purpose is to join different wooden pieces with adhesive substances. The main pollutants of these aqueous effluents are formaldehyde, urea, resorcinol, vinyl polyacetate and other additives used in the formulation of glues, and the organic load is usually over 10,000 mg dm^{-3} of COD.

Due to the small flowrate produce in every factory (typically 150–200 L/day/gluing machine), this type of effluent is usually merged with the domestic wastewaters produced in the factories, and after that, the resulting wastewater is directly discharge into the municipal sewers, where it is expected to merge with other industrial effluents and especially with the domestic wastewaters of the town, producing the raw wastewater that influents in the municipal wastewater treatment plant. In Spain, the typical limit for the discharge into municipal sewers is fixed by the municipal authority, which normally takes into account the capacity of the municipal wastewater treatment plant. A typical chemical oxygen demand (COD) restriction is 1500 mg dm^{-3}, value that can be easily accomplished following the described procedure.

However, the direct discharge of these effluents during long periods into the municipal sewer system have produced serious problems, because of the blocking of the sewer pipes and also because of the bad operation of the biological process of the municipal wastewater treatment plant (WWTP). It is important to take in mind that polymerization reactions take place during the transport of the effluent along the sewer, forming compact solids that in some points reduce importantly the free cross-sections of the pipes. In addition, the complex nature of the pollutants can affect importantly to the operation of the biological process of the municipal WWTP, especially during winter due to the low temperatures of this zone and their influence on the activated sludge performance.

Hence, it is necessary to integrate in the factories processes for the pretreatment of these wastes before its discharge to the municipal sewer system. Several papers have been published in the literature during the recent years about the treatment of the wastes produced in a wooden-door manufacturing factory [1–5]. Initially, the use of a coagulation-flocculation technology followed by a biological oxidation stage appears as a promising alternative from the economy viewpoint, but due to the presence of toxic and/or biorefractory species, biological techniques are not initially considered to be efficient enough for the treatment of these effluents [6–8],

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and although the combination of a coagulation-flocculation process with a sedimentation or a flotation process is able to reduce the colloidal adhesives contents [9,10], this treatment does not act against soluble pollutants, and consequently it leads to a low COD removal. So, it can only be used as a complementary treatment and not as a final one. Other kinds of technologies that can be applied to the treatment of these effluents are chemical oxidation [11] and advanced oxidation processes [12], although they are more expensive. Both techniques are based on the addition of oxidants to the industrial effluent in order to oxidize the organic matter to carbon dioxide. The main different between both techniques is the generation (or not) of hydroxyl radicals (advanced oxidation process). In this context, the use of these techniques in combination with biological or chemical processes can be interesting [13,14] as they can obtain the complete removal of the pollutants reducing the cost of the process. Two commonly used AOP are Fenton oxidation and ozonation and very recently, an electrochemical technology has joined this group: the conductive-diamond electrochemical oxidation (CDEO). Conductive-diamond is an emergent material with very good properties for the electrochemical treatment of wastewaters polluted with organic compounds, including great chemical and electrochemical stability [15] and generation of large amounts of hydroxyl radicals [16]. Recently, this electrolytic technology has been widely studied with both actual [15,17,18] and synthetic industrial wastes [19–27]. The main results are that this technology allows the almost complete mineralization of the organics contained in the wastes with very high current efficiencies.

With this background, the aim of this work has been to study the improvement in an conventional treatment (consisting of coagulation-floculation and biological oxidation) of an actual industrial waste produced in the gluing process of the wooden-door manufacturing factories with three AOPs: conductive-diamond electrochemical oxidation (CDEO), ozonation and Fenton process. The efficiency in COD removal has been monitored in order to compare their technical and economic feasibility.

2. Experimental section

2.1. Analytical procedure

The COD was used to monitor the organic load of the wastes. It was determined using a HACH DR200 analyzer. Measurements of pH and conductivity were carried out with an InoLab WTW pH-meter and a GLP 31 Crison conductimeter, respectively.

2.2. Determination of the oxygen-equivalent chemical-oxidation capacity (OCC)

To compare the performance of different AOPs it is desirable one parameter which quantifies in arbitrary units the oxidants added to the waste. In the literature [17] it is proposed to use the oxygen-equivalent chemical-oxidation capacity (OCC). This parameter is related to the different oxidants used in the three advanced oxidation processes studied in this work according to Eqs. (1)–(3).

$$\mathrm{OCC}(\mathrm{kgO_2\,m^{-3}}) = 0.298Q \tag{1}$$

$$\mathrm{OCC}(\mathrm{kgO_2\,m^{-3}}) = 1.000[\mathrm{O_3}] \tag{2}$$

$$\mathrm{OCC}(\mathrm{kgO_2\,m^{-3}}) = 0.471[\mathrm{H_2O_2}] \tag{3}$$

where Q is the specific electrical charge passed (kA h m^{-3}), $[O_3]$ and $[H_2O_2]$ are the concentrations of O_3 and H_2O_2 (kg m^{-3}) respectively, and the numerical values are dimensional factor for unit consistence (0.298 kg O_2/kA h; 1 kg O_2/kg O_3; 0.471 kg O_2/kg H_2O_2). These equations are obtained from stoichiometrical calculations, taking into account the number of electrons exchanged in the reduction of the different oxidants (for the case of ozone and hydrogen peroxide) and also the faraday number in the case of CDEO.

2.3. Conductive diamond electrochemical oxidation

CDEO assays were carried out in a single-compartment electrochemical flow-cell working under a batch operation mode [28]. Diamond-based material (p-Si-boron doped diamond) was used as anode and stainless steel (AISI 304) as cathode. Both electrodes were circular (100 mm diameter) with a geometric area of 78 cm^2 and an electrode gap of 9 mm. The wastewater was stored in a glass tank (0.6 dm^3) and circulated through the electrolytic cell by means of a centrifugal pump (flowrate 2.5 dm^3 min^{-1}). A heat exchanger coupled with a controlled thermostatic bath (Digiterm 100, JP Selecta, Barcelona, Spain) was used to maintain the temperature at the desired set point. The experimental setup also contained a cyclone for gas–liquid separation, and a gas absorber to collect the carbon dioxide contained in the gases evolved from the reactor into sodium hydroxide. Boron-doped diamond films were provided by Adamant Technologies (Switzerland) and synthesized by the hot filament chemical vapour deposition technique (HF CVD) on single-crystal p-type Si (1 0 0) wafers (0.1 Ω cm, Siltronix). Electrolyses were carried out in galvanostatic mode. Prior to use in galvanostatic electrolysis assays, the electrode was polarized during 30 min in a 1 M Na_2SO_4 solution at 50 mA cm^{-2} to remove any kind of impurity from its surface. During the electrolyses no control of pH was carried out.

The COD method was used for the determination of the average current efficiency (ACE), which was calculated using Eq. (4), where COD_0, COD_t and $COD_{t+\Delta t}$ are the COD (in g O_2 dm^{-3}) at times 0, t and $t+\Delta t$ (in seconds), respectively, I is the current intensity (A), F is the Faraday constant (96487 C mol^{-1}), V is the volume of the electrolyte (dm^3) and 8 is a dimensional factor for unit consistence (32 g O_2 mol^{-1} O_2/4 mol e^{-1} mol^{-1} O_2).

$$\mathrm{ACE} = \frac{[\mathrm{COD}_0 - \mathrm{COD}_t]FV}{8It} \tag{4}$$

2.4. Ozonation

Ozonation experiments were carried out by continuously feeding an ozone–oxygen gas stream in a mixed semi-batch bubble reactor (continuous for gas and batch for liquid). The experimental set-up consists of an ozone generator (Ambizon, Model GMF-10, Sistemas y Equipos de Ozonización S.L., Madrid, Spain) and a jacketed cylindrical reactor (2.5 dm^3) equipped with a porous gas distribution plate and baffles to increase the capacity of absorption of ozone. The ozone-oxygen mixture gas stream was sparged with a constant flow rate of 0.5 dm^3 min^{-1} (flow controller Cole Parmer, model #: 32907-39) and the average production of ozone was around 1 g h^{-1}. The volume of wastewater treated in each assay was 2 dm^3. The pH was maintained in a set point close to 12 ± 0.1. A jacketed reactor coupled with a controlled thermostatic bath (Tectron, model 3473200 Selecta, Madrid, Spain) was used to maintain the temperature at the desired set point (25 °C).

2.5. Fenton process

Fenton oxidation assays were carried out in lab-scale thermostated mixed batch reactors. The experimental set-up consists of a multistirrer device (Ikamag RO 5 power, IKA-WERKE GmbH & Co. KG, Staufen, Germany) with fifteen mixing sites coupled with a controlled thermostatic bath (Digiterm 100, JP Selecta, Barcelona, Spain). Pyrex flasks (250 cm^3) hermetically sealed and equipped with magnetic stirrers were used as reactors. In every assay, the reactors were filled with 100 cm^3 of wastewater. Then, the iron

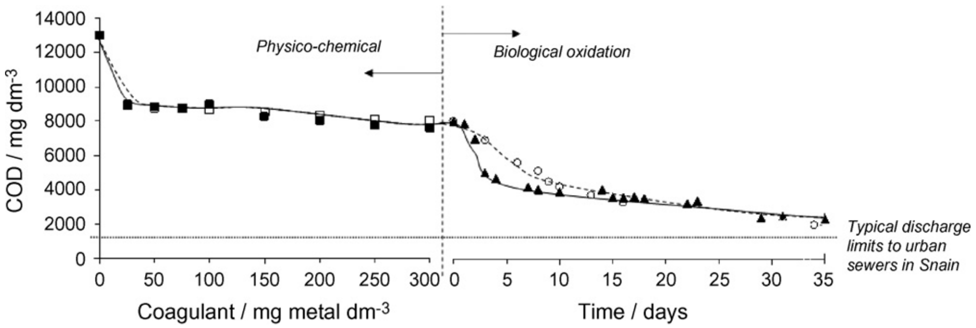

Fig. 1. Treatment of a raw effluent of a wooden-door manufacturing plant by a combined coagulation floculation ((■) aluminium, (□) iron) and biological oxidation process ((▲) non-acclimatated biological culture, (○) acclimatated biological culture).

dose was added (as $FeSO_4{\cdot}7H_2O$) and the pH was adjusted to 3 ± 0.1 with sodium hydroxide or sulphuric acid. Hydrogen peroxide was measured by colorimetric determination [29].

2.6. Biological oxidation

Discontinuous biological aerobic oxidation experiments were carried out in tanks of 5 L capacity provided with several air diffusers that produce fine bubbles. Prior to the experiments, phosphorous analyses were carried out to the effluents coming from the coagulation/flocculation process, and its value was corrected, as this nutrient is essential for the bacterial growth. The experiments started with the inoculation of the bacteria (extracted from the sludge of the secondary treatment of the municipal waste water treatment plant (WWTP)) to the waste. Then the air was continuously supplied to the tank, and samples were extracted periodically.

2.7. Conventional coagulation

The coagulation experiments were carried in discontinuous operation mode. These experiments were carried out in a standard jar test experimental set-up. In these experiments, different amounts of coagulant (0–300 ppm of $FeCl_3$ or $Al_2(SO_4)_3$) were added to the wastewater, and the pH was adjusted to 7 ± 0.1 with sodium hydroxide or sulphuric acid. Initially, solutions were vigorously stirred during 5 min and after that left 30 min to allow the sedimentation of solid phase. COD was measured to the final aqueous phase.

2.8. Wastewater characterization

The wastewater used in this work is the effluent of an actual door-manufacturing factory. It is mainly composed of urea and formaldehyde with different additives. This effluent contains a high concentration of COD (13,068 $mg\,dm^{-3}$), total nitrogen (347 $mg\,N\,dm^{-3}$) and suspended solids (13,574 $mg\,dm^{-3}$). Its conductivity is 7.49 $mS\,cm^{-1}$ and the pH is around 7.

3. Results and discussion

Fig. 1 shows the changes in the COD during a conventional treatment consisting of a sequence of coagulation-flocculation and biological oxidation. As it can be observed, both treatments are very efficient, and they can reduce the pollutant concentration close to the limits typically regulated in Spain for the discharge into municipal sewers (they depend on the municipality, and 1500 mg COD dm^{-3} is usually a very representative value). However, it is clear that a complementary treatment should be used to refine the quality of the effluent because the concentration of COD after the treatment is still over the limit and, in addition, the retention time of the biological process is too huge to be used practically.

Results of the physico–chemical treatment do not depend on the coagulant reagent (iron and aluminum curves are overlapped) and a dose of 25 $mg\,dm^{-3}$ seems to be enough to assure a significant removal of COD (greater than 30%). Higher doses do not improve the results and hence they only lead to an increase in the operation cost without further benefits.

The biological oxidation is a more complex process. In the figure, they are compared the results obtained in the batch biological oxidation using directly activated sludge from a municipal wastewater treatment plant and using previously-acclimatized activated sludge. As it can be observed, results obtained are very similar and they only differs during the three first days: the raw activated sludge does not oxidize COD during this stage (acclimatation of the biological culture to the industrial waste composition) while the acclimatized one starts the biological oxidation almost immediately. After this initial difference, in both cases there are two clear stages during the treatment, a first stage in which the COD disappears rapidly from 8000 ppm down to around 4000 (0.6 kg COD $m^{-3}\,d^{-1}$), and a second stage in which the oxidation became sluggish and the oxidation rate is smaller (0.05 kg COD $m^{-3}\,d^{-1}$). Obviously, this second stage increases very significantly the sizing of the biological operation and the use of other treatment technology can be advice. This recommendation can be encouraged if you take into account that the results of a biological process can be influenced by many parameters (pH, presence of a toxic compound, etc.) which even can inhibit the oxidation capability of the biological culture, leading to a discharge that can exceed significantly the regulated limits.

Hence, the physicochemical process seems to be mandatory in the treatment of door-manufacturing process (DMP) waste (great removal and low cost) and the biological one can be of a good help if it is used conveniently, but it should not be used alone because it can not warrant a complete treatment. In this point, it is clear that AOP can be used to treat directly the effluent of the physico–chemical treatment or to treat the effluent of the biological process. Figs. 2 and 3 compare the results obtained by CDEO, ozonation and Fenton in both cases.

As it can be observed, only CDEO is able to reduce completely the COD of the physico–chemically-treated waste (Fig. 2). Ozonation and Fenton oxidation lead to a significant removal but they are not able to diminish the COD under the discharge limits fixed typically by the municipal authority. This observation is very important but not new. It has been done in the treatment of other kind of wastes [15,17,18], and it indicates that CDEO combines (in addition to the hydroxyl radicals mediated oxidation) some powerful oxidation mechanisms that give this technology a clear advantage as compared with Fenton or ozonation. This is also supported by Fig. 4 in which it can be seen that the refractory compounds formed in

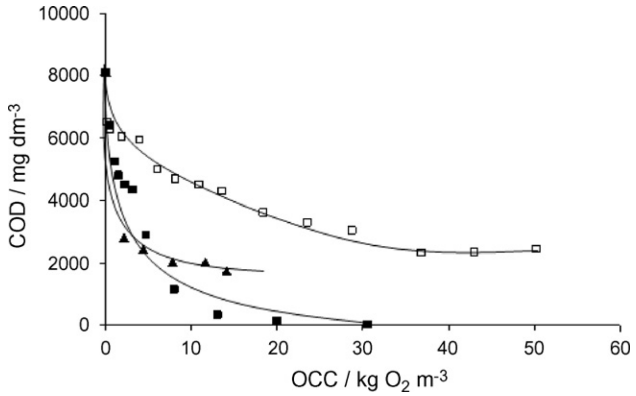

Fig. 2. Treatment of the effluent of the coagulation process of a wooden-door manufacturing wastewater by (■) CDEO (□) ozonation and (▲) Fenton oxidation.

these later technologies are easily oxidized by CDEO. One important observation in the comparison of the advanced oxidation processes is that during the first stages the three technologies have the same efficiency (the three curves shown in Fig. 2 are overlapped) but after this initial stage, even for low oxidant-doses the ozonation becomes in the less efficient process. Conversely, Fenton efficiency is similar to that of CDEO up to the oxidant-dose in which all COD of the Fenton process is transformed into refractory COD.

With regard to the biologically-treated waste (Fig. 3), it can be observed a similar behaviour of the three AOPs: similar efficiencies of Fenton oxidation and CDEO for low oxidant-doses, lower efficiencies of the ozonation process in the whole range of OCC, and formation of refractory COD in Fenton and ozonation processes. In this case, the presence of refractory COD is not as important as in the case shown in Fig. 2 because all techniques can obtain concentrations below the typical discharge limits. It is important to note in this point that the concentration of COD used in these essays is lower than the obtained by the biological treatment. This is due to the dilution used to compare the three technologies in the same conditions (in the Fenton process it is necessary to add liquid reagents that dilute the waste).

One important point concerning the use of AOP is the combination of AOP with biological oxidation. In many works [12,18,30,31], it has been stated that AOP can be used to increase the biodegradability of biorefractory industrial wastes, and later these wastes can be treated by the cheaper biological process. To check this possibility in the treatment of the secondarily-treated waste, the ratio BOD_5/COD was monitored during the treatment of the biologically-treated waste with the three AOP. Results are shown in Fig. 5. As it can be observed the biodegradability of the effluent of the three AOPs does not increase significantly but it remains in values lower than 2% (detail on the onset) during the treatments. This means that a combination of any of the three technologies with the biological oxidation is useless in this circumstance, because there will not be an improvement in the biological process efficiency (BOD_5 indicates the amount of organic matter that can be oxidized biologically within a period of 5 days).

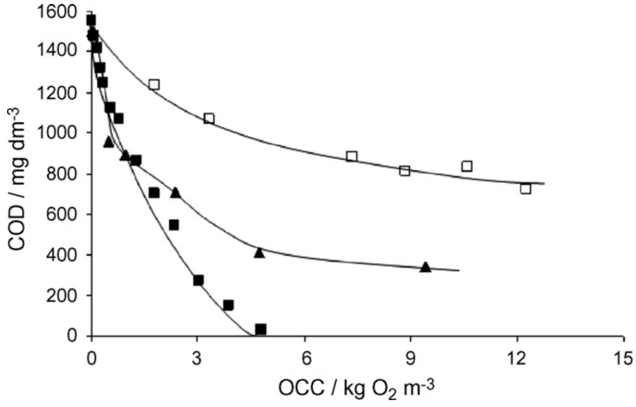

Fig. 3. Treatment of the effluent of the biological oxidation process of a door-manufacturing process wastewater by (■) CDEO (□) ozonation and (▲) Fenton oxidation.

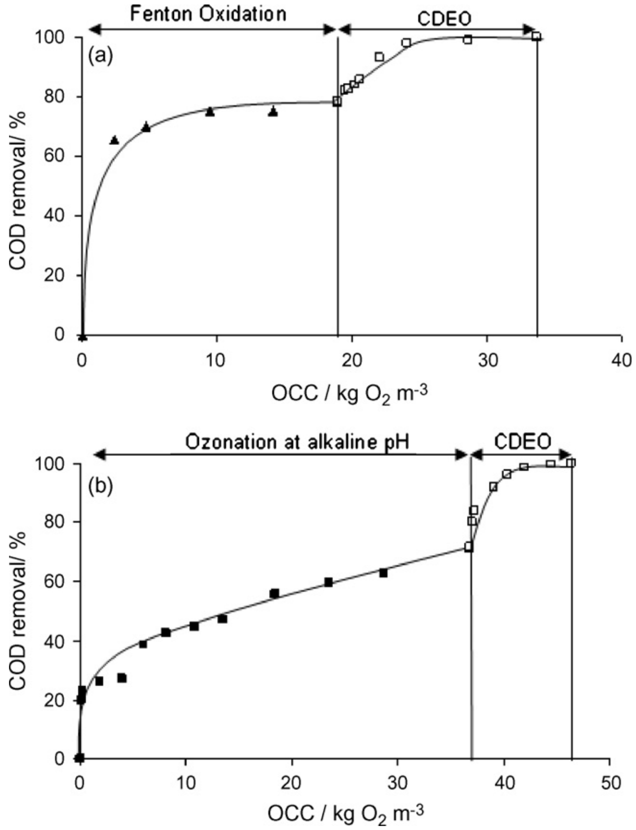

Fig. 4. Improvements in the quality of the effluent of ozonation (■) and Fenton (▲) processes by a refining treatment with CDEO (□).

Fig. 6 compares the average efficiency of the advanced treatment technologies with the progress in the removal of pollutant. As expected, the treatment of a highly-polluted influent is more effi-

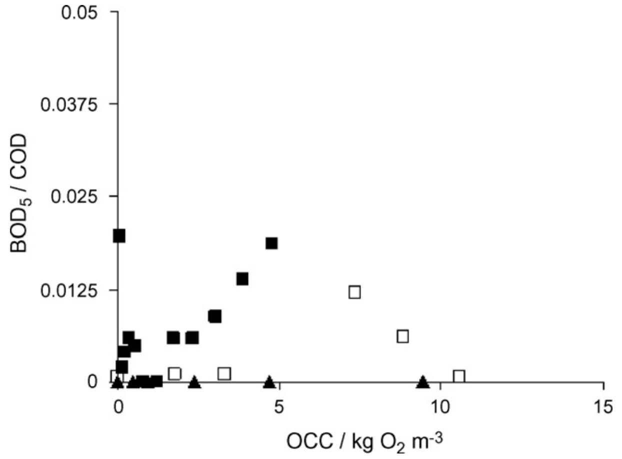

Fig. 5. Changes in the biodegradability of the wastes during the treatment with (■) CDEO (□) ozonation and (▲) Fenton oxidation of the effluent of a biological oxidation process.

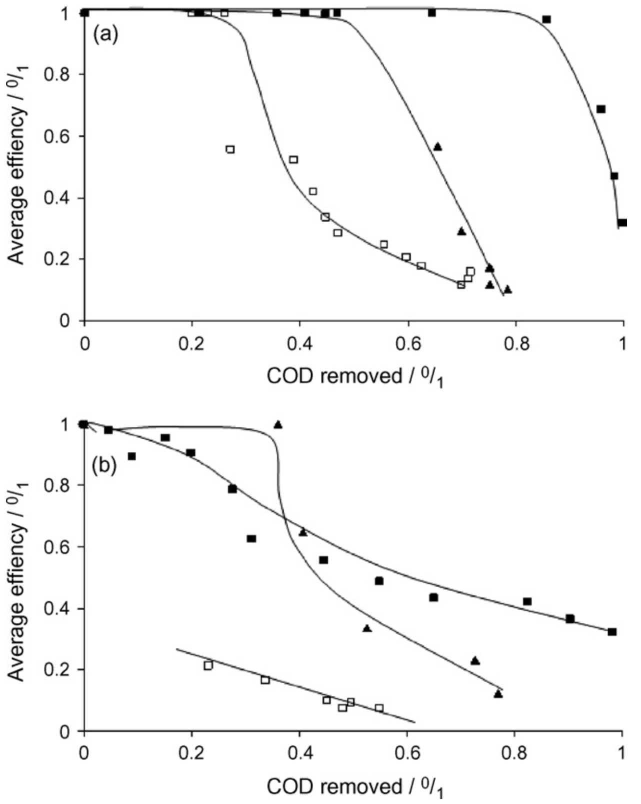

Fig. 6. Changes in the average efficiency during the removal of COD with (■) CDEO (□) ozonation and (▲) Fenton oxidation ((a) treatment of a coagulated door-manufacturing process waste; (b) treatment of a biologically-treated waste).

cient. Thus, it can be removed up to 90% of the pollutants contained in the coagulated wastewater with the maximum global efficiency using CDEO. The percentage of COD removal which allows maintaining the maximum efficiency decreases for ozonation and Fenton and it is still smaller for the treatment of a biologically-treated waste due to the smaller organic charge of this later waste.

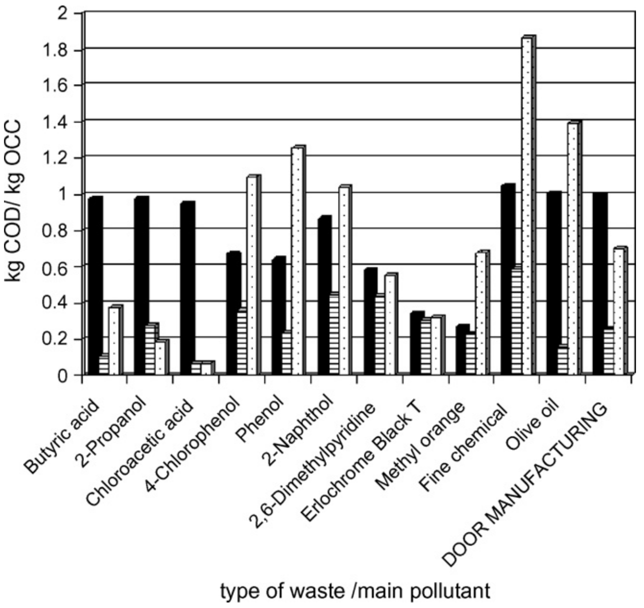

Fig. 7. Effectiveness of the oxidant dose in the removal of the door-manufacturing wastes by the three oxidation technologies. A comparison with other wastes and pollutants tested (solid: CDEO; dashed: ozonation; pointed: Fenton oxidation).

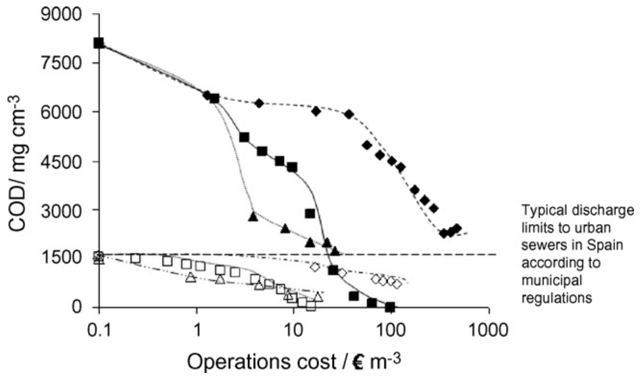

Fig. 8. Estimation of operation cost as a function of the treatment results for the technologies studied in this work. (■, □) CDEO (♦, ◇) ozonation and (▲, △) Fenton oxidation (full symbols: treatment of the coagulated waste; empty symbols: treatment of the biologically-treated waste).

Results obtained in this work are compared to those obtained in other works [15,17,18,24,28,32] in Fig. 7. This figure shows the effectiveness of the oxidant dose in the removal of COD by three oxidation technologies during the treatment of different types of actual and synthetic wastes in conditions in which mass transfer does not limit the rate of the process. Results obtained for the door-manufacturing wastewater are among the obtained for the other types of wastes. In general, it can be stated that the oxidants are less efficiently used in ozonation and more efficiently used in CDEO and Fenton oxidation. In the case of complex molecules, Fenton oxidation seems to be the most efficient process. It is important to note that the maximum value of this ratio should be 1. The higher values obtained in some cases (for Fenton oxidation essays) can be explained in terms of non-oxidative side reactions like the coagulation processes caused by the action of iron ions added to the solution and/or sedimentation processes. On the other hand, in the case of aliphatic pollutants the values obtained are completely different and these results confirm the lower efficiency of the oxidants generated by Fenton oxidation in the treatment of wastes polluted with carboxylic acids. Therefore, this oxidation technology should not be used as a final treatment but is better suited as a pre-treatment process.

From this, it seems clear that the three studied AOP allows obtaining good results, especially CDEO. However, to compare the results obtained it is important to quantify operation cost, and to do this it is important to have a good value of the unit cost of the oxidant dose. This study has been carried out by our group and recently it has been published [33].

Taking into account these data, Fig. 8 shows an estimation of the operation cost as a function of the treatment results. From these results it seems clear that Fenton and CDEO can be efficiently used to refine the quality of the biologically-treated waste with operation costs under 1 € m^{-3}. On the contrary, the direct treatment of the effluent of the coagulation process leads to a non cost-worthy process with operation costs over 25 € m^{-3}.

4. Conclusions

From this work, the following conclusions can be drawn:

- Coagulation–flocculation is a very effective treatment for the wastes produced in the gluing process of door-manufacturing factories. Biological oxidation can also reduce significantly the COD but it is a very time-consuming process and very long retention times are necessary to achieve a good removal.
- From the treatment point of view (neglecting costs), advanced oxidation processes can be successfully used to treat both,

coagulated and biologically treated wastes. Conductive-diamond electrochemical oxidation is the more effective technology because it can reduce completely the COD (no production of refractory compounds) with a high current efficiency.

- From the economic viewpoint, the direct treatment of the coagulated wastes should not be recommended because it leads to cost higher than 20 € m^{-3}. Only Fenton oxidation or conductive-diamond electrochemical oxidation can be cost-efficiently used to refine the quality of the effluent of the biological process.

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