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An Analysis on Various Methods in the Treatment of Wastewater Using Electrochemical Oxidation Techniques

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Abstract – This paper reviews the development, design and applications of electrochemical technologies in water and wastewater treatment. Particular focus was given to electro oxidation. Electro oxidation is finding its application in waste water treatment in combination with other technologies. It is effective in degrading the refractory pollutants on the surface of a few electrodes. Titanium-based boron-doped diamond film electrodes (Ti/BDD) show high activity and give reasonable stability. Its industrial application calls for the production of Ti/BDD anode in large size at reasonable cost and durability.

Electrochemical oxidation (EO) as electrochemical method is unique by three aspects. The first is that is the most versatility process in water treatment area and covers: various industrial effluent treatment including, amongst others, distillery, agrochemical, pulp and paper, textile dyes, oilfield and metalplating wastes; hazardous effluent treatment including hospital wastes; removal of pathogens and persistent, pharmaceutical residues and biological from municipal wastewater treatment plant; removal of organic micro-pollutants such as pesticides and heavy metals such as arsenic and chromium from water. Another aspect is that EO is complementary with most other methods: chemical or electrochemical, and is often combined with one or more of them. And finally, this procedure is the most interdisciplinary of all. It includes: material science, (micro)biology, (electro)chemistry, environmental protection, water supply systems, etc.

Many human activities result in the production of wastewater. Usually, physical, chemical and biological processes are successfully combined for the treatment of municipal wastewater, attaining good removal efficiencies. However, some industrial processes introduce anthropogenic recalcitrant pollutants in wastewater that are quite difficult to remove or degrade using conventional means and that should be removed due to their hazardousness. In such cases, the application of an Advanced Oxidation Processes (AOP) uses to be a good and/or promising alternative to attain an appropriate effluent.

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INTRODUCATION

Using electricity to treat water was first proposed in UK in 1889. The application of electrolysis in mineral beneficiation was patented by Elmore in 1904. Electrocoagulation (EC) with aluminum and iron electrodes was patented in the US in 1909. The electrocoagulation of drinking water was first applied on a large scale in the US in 1946. Because of the relatively large capital investment and the expensive electricity supply, electrochemical water or wastewater technologies did not find wide application worldwide then. Extensive research, however, in the US and the former USSR during the following half century has accumulated abundant amount of knowledge. With the ever increasing standard of drinking water supply and the stringent environmental regulations regarding the wastewater discharge, electrochemical technologies have regained their importance worldwide during the past two decades. There are companies supplying facilities for metal recoveries, for treating drinking water or process water, treating various wastewaters resulting from tannery, electroplating, diary, textile processing, oil and oil-in-water emulsion, etc. Nowadays, electrochemical technologies have reached such a state that they are not only comparable with other technologies in terms of cost but also are more efficient and more compact. For some situations, electrochemical technologies may be the indispensable step in treating wastewaters containing refractory pollutants. In this paper, I shall examine the established technologies such as electrochemical reactors for metal recovery, electrocoagulation, electro flotation and electro oxidation. The emerging technologies such as electro photo oxidation, electro disinfection will not be

discussed. In addition, I shall focus more on the technologies rather than analyzing the sciences or mechanisms behind them. For books dealing with environmentally related electrochemistry, the readers are referred to other publications.

Nowadays, various electrochemical treatments are methods used for removal of organic and inorganic impurities from fresh, drinking and waste waters. The most usual methods are: electrocoagulation, electro floatation, electrochemical oxidation, electrochemical reduction and electrodeposition. The method consists of carrying out the oxidation reaction at the anode where pollutants are transferred into non-toxic substances, by decomposition into simpler compounds or transferring into oxidation form. It is used mostly for organic substances. Wastewater treatment by this procedure can be: direct or indirect electrochemical oxidation, as showed on Fig. 1.

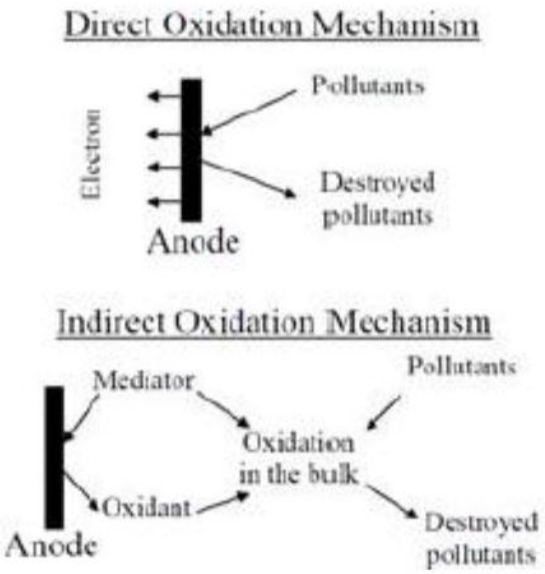

Figure 1. Pollutant removal pathway in electrochemical oxidation process.

Researches in this area are focused on increasing the process efficiency which depends from: electro catalytic activity, electrochemical stability of the electrode materials and kinetics of degradation of pollutants. The first application of the electro oxidation (EO) was cyanide destruction. The EO is mainly used for degradation: aromatic compounds, pesticides, paints, industrial pollutants, pharmaceuticals waste and other organic. The advantage of this method is that finally products are mainly CO_2 and H_2O. This is the case for some organic as aliphatic, depending on stoichiometry. Even chlorine is not the problem since it is converted in the form of chloride ion.

Indirect electrochemical oxidation

Strong oxidants are produced on anode and then chemical oxidation take place. The most effective type of the indirect electro oxidation is creation of hydroxyl radicals via Fenton reagent. In Fenton reaction, highly reactive hydroxyl radicals (·OH) are generated:

$$H_2O \rightarrow OH_{ads} + H^+ + e^-$$

$$Fe^{2+} + H_2O_2 \rightarrow Fe^{3+} + OH + OH^-$$

To avoid the disadvantages of traditional Fenton oxidation such as potential risk in transportation of H_2O_2, a loss of reactive activity and sludge production, a modified process called Electro-Fenton method (EF) has been developed. The EF method has the advantage of allowing a better control of hydroxyl radical production; in the EF method, soluble Fe^{3+} can be catholically reduced to Fe^{2+} which is known as electrochemical catalysis with E^0 = 0.77 V/SHE.

The efficiency of this process can be increased if the anodic oxidation with BDD (boron deposited diamond) is paired with a classic electro-Fenton process with carbon fiber or oxygen diffusion cathode. Reaction rate of oxidation of organics with hydroxyl radical is extremely high, for example, the reaction rate constant of second order can be 10^9–10^{10} $mol^{-1}\cdot s^{-1}$.

The indirect electro-oxidation rate is dependent on the diffusion rate of strong oxidants electroformed into solution which are able to completely convert all organic into water and carbon dioxide.

The electrochemical method of depollution presents many important advantages because it does not need auxiliary chemicals; it is applicable on a large range of pollutants and does not need high pressures and temperatures.

Further improvement could be achieved by using of UV+ radiation. UV lights (λ=365 nm) was successfully used to accelerate the mineralization process . This is the photoelectro-fenton process.

Direct electrochemical oxidation

The Direct electro oxidation takes place directly at the anode through the generate of physically adsorbed "active oxygen" (adsorbed hydroxyl radical, •OH). Direct EO process is also called electrochemical mineralization (EM) of organic contaminants. It is a relatively new technology for wastewater treatment with moderate concentrations of organic pollutants, the chemical oxygen demand (COD) up to 5 g/l.

The main advantage of technology is that it does not consume chemicals, but just electrical energy. EO does not produce secondary pollution and requiring relatively little additional equipment and maintenance. These advantages make it attractive compared to most other waste water processes. A key element of this technology is anode material. Investigated the anode: glassy carbon, Ti/RuO_2, Ti / Pt-Ir, fiber carbon, MnO_2, Pt-black carbon, porous carbon felt and stainless steel. But none of the above has sufficient activity and stability.

Therefore, Pt, PbO_2, IrO_2 and SnO were extensively studied anodes . They were the most used ones before new development of the conductive diamond films. Currently an ideal material for the anode is boron doped diamond (BDD). Its characteristics are: high reactivity for organic oxidation, high stability and efficient use of electrical energy. Specific energy consumption for this treatment with 100% current efficiency is approximately 22kWh/kgHPK.

Example of complete (electro) mineralization is a direct electrochemical oxidation of the herbicide 2,4,5Trichlorophenoxyacetic acid, also known as 2,4,5-T and Silvex (trade name) . The whole process can be represented by the following reaction:

$$C_8H_5Cl_3O_3 + 13H_2O \rightarrow 8CO_2 + 31H^+ + 3Cl^- + 28e^-$$

or by COD during electrochemical decomposition, the theoretical reaction of mineralization 2,4,5-T can be written as:

$$C_8H_5Cl_3O_3 + 7O_2 \rightarrow 8CO_2 + 3HCl + H_2O$$

Oxidation potential of the anode is directly dependent on the over potential for oxygen separation and adsorption enthalpy of hydroxyl radicals on the anode surface. It follows that for an anode material: the higher the overpotential for oxygen, the greater the oxidizing power of the anode material.

Generally, wastewater treatment is carried out using pri mary, secondary or tertiary methods, depending on the nature of the pollutants. As far as organic pollutants in wastewaters are concerned, biological abatement may sometimes be impos sible, due to the bio-refractory character of the substrates. For this reason, physical-chemical methods are preferably applied, but an oxidation with ozone or chlorine dioxide is not always effective and also transportation and storage of reactants may be a significant inconvenience for safe processing.

An alternative can be the application of electrochemical technologies for wastewater treatment, benefiting from advantages such as versatility, environmental compatibility and potential cost effectiveness among others described below.

Both direct and mediated electrochemical oxidations can be considered, and have proved to be interesting subjects for different research groups and industries seeking new technologies for wastewater treatment.

THEORIES OF ELECTROCHEMICAL TREATMENT

A typical electrochemical treatment process consists of electrolytic cell, which uses electrical energy to affect a chemical change. In simplest forms, we can say that, an electrolytic cell consists of two electrodes, anode and cathode, immersed in an electrical conducting solution (the electrolyte), and are connected together, external to the solution, via an electrical circuit which includes a current source and control device. The chemical processes occurring in such cells are oxidation and reduction, taking place at the electrode/electrolyte interface. The electrode at which reduction occurs is referred to as the cathode and conversely, the anode is the electrode at which oxidation processes occur.

The current flow in an electrochemical cell is maintained by the flow of electrons resulting from the driving force of the electrical source. In order to allow the current to flow, there must be an electrolyte, which facilitate the flow of current by the motion of its ionic charged species. Type of electrolyte has significant effect on the process in the formation of oxidizing species during the process.

Generally, oxidation of organic matter by electrochemical treatment can be classified as direct oxidation at surface of anode and indirect oxidation distant from the anode surf ace; processes are influenced significantly by the anode material. The energy supplied to an electrochemical reactor plays an important role in any electrochemical process. The energy supplied to an electrode undergoes the following steps during the process.

1. The electro active particle is transferred to the electrode surface from the bulk solution.

2. The electro active particle is adsorbed on to the surface of the electrode.

3. Electron transfer occurs between the bulk and the electrode.

4. The reacted particle is either transported to the bulk solution (desorption) or deposited at the electrode surface.

WASTEWATER TREATMENT

The effective treatment of effluents represents a serious problem, especially for the chemical industry. Over the last twenty-five years, huge efforts have beenmade to limit at the source this type of pollution, by improving processes, recycling products and controlling the treatment of wastes at the production stage.

However, considering the large amounts of industrial effluents to be treated, for example to retrieve certain solvents, there are inevitably residues requiring a final transformation, which is often delicate. Traditional destruction methods, for their part, pose problems of corrosion and, more seriously, of emissions, if the treatment conditions are not perfectly controlled.

From the industry point of view, this problem must be examined as a whole since there are no universal or simple methods in this area. The wide variety of industrial discharges means that a diversification of techniques must be sought, adapting the treatment to each situation, as much as possible.

In spite of the efforts made to develop clean processes, the increasingly severe environmental laws should encourage the research for better-performing treatments, making it possible to obtain environmentally compatible effluents. Actually, the processes for the treatment of wastewater may be divided into three main categories: primary, secondary and tertiary. Tertiary treatment, also known as advanced wastewater treatment, includes acid/base neutralization, precipitation, reduction and oxidation processes.

TECHNOLOGICAL PARAMETERS OF ELECTROCHEMICAL OXIDATION

The two most important parameters are current density and current efficiency. The process may have good efficacy removing contaminants above 80% and up to 95%, but at the same current efficiency below 50% down to 15% depending on the reaction and anode material.

The literature data given in table 1 shows the efficiency of the different indirect electrooxidation processes for treatment. The degradability of 2,4dichlorophenoxyacetic acid (2,4-D) was performed for initial concentration of 2,4-D of 230ppm in 0.05M Na_2SO_4 with H_2SO_4 at pH=3.0 and temperature of 25°4C. Pollutant (2,4-D) is common systemic pesticide/herbicide used in the control of broadleaf weeds and is chemically similar to 2,4,5-T mentioned earlier.

Table 1. Percentage of TOC removal and apparent current efficiency (ACE) obtained for the mineralization of 100ml of 230ppm 2,4-D solution

Method	Applied current, mA	% of TOC removal	ACE, %
Anodic oxidation in the presence of electrogeneration of H_2O_2	100	11	4.7
	300	23	3.1
	450	26	2.5
Electro-Fenton process	100	52	7.4
	300	63	9.4
	450	73	7.7
Photoelectro-Fenton process	100	83	37.3
	300	88	12.8
	450	82	8.9

It is obvious that photoelectro-Phenton process is the most effective one. It should be noted that even that process has not great current efficiency even for the optimal conditions. Nevertheless, removal efficiency is the more important parameter, especially for the organic with less than 500ppm concentration which is mainly the case for electro oxidation. Nevertheless, removal efficiency is the more important parameter, especially for the organic with less than 500ppmconcentration which is mainly the case for electro oxidation.

For the direct EO the best technological parametersare obtained using a borated diamond electrode(anode), BDD. This material features high hardness,strength, stability and resistance to thermal shocksand IR radiation with high thermal conductivity, highelectric field degradation and outstanding chemicalstability. Laboratory studies on the Si/BDD electrodegive very good results and are shown in Table 2 .However, in industrial conditions is not recommended for use BDD on Si substrates due to poor electricaland thermal conductivity of Si and brittleness of theelectrodes. Top features of the electrode with the filmhave BDD on Ti substrates, (Diachem® as famouscommercial and is trademark of Condias GmbH), but their prices are very high.

Table 2. Oxidation of organic pollutants on Si/BDD electrode

Pollutant	Oxidation conditions	Current efficiency, %
Phenol	Initial concentration 0.002mol/dm^3, current density 300A/dm^2, pH=2, charge 4,5Ah/dm^3, final conc. of phenol <3mg/dm^3	33.4
Cyanide, CN^-	Initial concentration 1mol/dm^3, current density 360A/dm^2, elimination more then 95% CN^- ion, 1mol/dm^3 KOH	41
Isopropanol, IPA	Initial concentration 0.17mol/dm^3, current density 300A/dm^2, efficiency about 90%.	>95
Acetic Acid	Initial concentration 0.17mol/dm^3, current density 300A/dm^2, efficiency about 90%, 1mol/dm^3 H_2SO_4	85

The current efficiency (CE) obtained on Ti/BDD was 46.9-78.5% in oxidizing acetic acid, maleic acid, phenol, and dyes, which is 1.6-4.3 fold higher than that obtained on the typical Si/BDD electrode.

ELECTROCHEMICAL PROCESSES INWASTEWATER TREATMENT

Over the last two decades, various applications of electrochemical technology have arisen in the field of environmental remediation. Treatment of liquid wastes coming from different types of industries (by electrocoagulation, electrolysis or electro dialysis) and electro kinetic soil remediation processes become the most significant.

From the scientific point of view, electrolysis and electrocoagulation have been the two most exciting research topics in this period with hundreds of very significant references.

Electrocoagulation has demonstrated to be a very interesting technology for removal of turbidity, decolonization of dyes and breakup of wastes consisting of emulsions. However, it is not a final treatment but a pretreatment and hence, it is particularly interesting for the coarse removal of pollution.

Opposite to electrocoagulation, electrolysis should not be used as a treatment for the coarse removal of pollution in industrial waste because the amount of energy required for the abatement depends directly on the concentration of pollution to be removed. Likewise, it cannot be proposed as an alternative to biological oxidation processes, because these latter processes are much cheaper (typically by one magnitude order)

and just in case biological oxidation could not be applied electrolysis can take a chance in the treatment of industrial waste. Hence, its target is the treatment of effluents polluted with anthropogenic organic species, either toxic or refractory to biological treatments, and within a concentration range which should be inside 1,000-20,000 mg Chemical Oxygen Demand (COD) dm^{-3} for direct anodic oxidations and which can be enlarge to smaller values if mediated oxidation processes are promoted (and hence mass transfer limitations are overcome).

Use of diamond electrodes has become an outreaching fact in this technology. This type of electrodes favors the production of hydroxyl radicals and their use in the production of other strong oxidants or their use in the harsh oxidation of organic species.

This explains the great efficiency obtained and the great relevance of the research results of electrolysis during these two decades. Electrolysis with diamond anodes (often called as conductive-diamond electrochemical oxidation, CDEO) exhibits three outstanding properties as compared with other advanced oxidation technologies and with electrolysis with other anodes:

- robustness, because results found in this latter years demonstrate that it can attain the complete mineralization of almost any type of organic without producing refractory final products
- efficiency, because when it is operated under the no diffusion control, current efficiencies are close to 100%
- integration capability, because it can be easily coupled with other treatment technologies and it can be fed with green energy sources such as wind mills and photovoltaics panels.

However, in spite of the large number of studies carried out, there is still a lot of work to do before its commercial application and many aspects must be enhanced. Two are of particular interest:

- The specificity of diamond electrodes and the elucidation of the influence of the diamond layer characteristics on the efficiency of the oxidation
- The potential improvements that could be obtained by the combination of CDEO with other technologies such as the irradiation of ultrasound or UV-light. This is because in the later years light irradiation and/or ultrasound irradiation have shown to significantly improve the results of many AOPs, in particular avoiding the production of refractory organics during the oxidation of complex pollutants. This is not a limitation of the robust CDEO technology, but just a clarification of how sono- and photo- CDEO can improve results of conventional CDEO is worth of investigation.

ADVANCED OXIDATION PROCESSES

Different advanced oxidation processes have been developed and investigated by several research groups for the elimination of organic pollutants from wastewater, such as Fenton processes, photoassisted Fenton processes, UV/Fe^{3+}-oxalate/H_2O_2, photo catalysis, ozone water system, Mn/oxalicacid/ozone, H_2O_2 photolysis, O_3/UV and others. These technologies consist mainly of conventional phase separation techniques (adsorption processes, stripping techniques) and methods, which destroy the contaminants by chemical oxidation and/or reduction. Chemical oxidation aims at the mineralization of the contaminants to carbon dioxide, water and inorganics or, at least, their transformation into harmless products. Obviously, the methods based on chemical destruction, when properly developed, offer a complete solution to the problem of pollutant abatement, different from those in which only a phase separation is realized with the consequent problem of the final disposal. It has been frequently observed that pollutants not amenable to biological treatments may also be characterized by high chemical stability and/or by a great reluctance to go to complete mineralization.

Also, the adoption of these oxidation treatments requires that specific conditions must be considered during the process: the influence of pH, inhibition due to scavenger presence, light wasting, mass transfer limitations, direct ozone attack and appropriate equipment. In these cases, it is necessary to adopt much more effective reactive systems than those adopted in conventional purification processes.

EXPERIMENTAL METHODS

Electrolytic system -

Electrochemical measurements were performed using a computer controlled by Potentiostat/Galvanostat model PGZ 100 associated to "Volta-Master 4" software. A conventional three electrodes cell (100 cm^3) thermo regulated glass cell was used (Tacussel Standard CEC/TH). The anode was a square plate of BDD electrode with effective surface area of 1 cm2, whereas the cathode was a platinum electrode, and the gap between electrodes was 1.5 cm. A saturated calomel electrode (SCE) was used as a reference. Galvanostatic electrolysis was carried out with a volume of 75 cm3 aqueous solution of initial COD (2498 mg/L). The range of applied current density was 10 to 70 mA/cm2 and samples were taken, at predetermined intervals during the experiment, and submitted for analysis. All tests have been performed at different temperature in magnetically stirred and aerated solutions. In all

cases sodium chloride was added to the electrolytic cell, at different concentrations. The chemical oxygen demand (COD) is measured according to the standard methods for examination of wastewater. The Chemical Oxygen Demand (COD) values were determined by open reflux, a dichromate titration method. All chemicals used in the experiments were of analytical pure grade and used without further purification. The sodium chloride used was of analytical-reagent grade and was obtained from Aldrich (Spain).

Waste water effluent -

A local wastewater cartons CMCP (from a rug industry of cartons in Agadir (Morocco)) provided the cartons effluent, which was sampled at the entrance reservoir of the industry's biological treatment station. This effluent originates from the different waste water units, thus being a complex mixture of and additives whose exact composition is unknown and dependent on the production schedule. Nevertheless, which was characterized by an intense dark yellow color and the presence of a high amount of fibers. Thus, before the electrochemical degradation experiments, the effluent was filtered two times with conventional filtering paper. Furthermore, due to the low conductivity of the effluent, NaClwas added until a 0–3g. L^{-1} concentration was reached.

RESULTS

The investigation of the mediator concentration effect has been performed in the range 0- 3g/L for NaCl. The presence of chloride ions in surface and groundwater is the principal motivation for investigating the effect of chloride on electrochemical oxidation of wastewater. During electrolysis, chloride ions are converted into chlorine and subsequently to hypochlorite ions in neutral or slightly alkaline pH. Figure 2 shows the variation of pH for wastewater cartons of electrolysis at 60 mA aunder different concentration of NaCl. Finally the pH in all cases became strong acidic (pH = 4, 2) to basic (pH = 8,9).

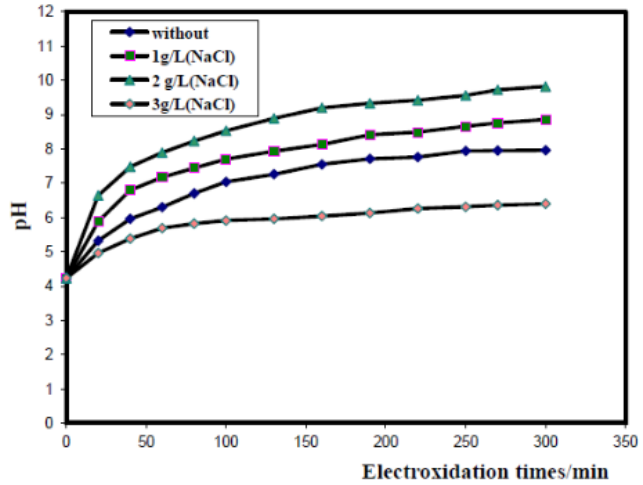

Figure 2. Variation of pH for wastewater cartons of electrolysis at 60 mA and 25° C, under different concentration NaCl.

The performance of the BDD anode in the waste effluent degradation was analyzed through the extent of decolorization and COD removal of this waste effluent. In order to compare these performances, the same total value of electric charge per unit volume of the electrolyzed effluent (Qap) was applied in all the experiments (5 $kAh.m^{-3}$). Absorbance (A) were done on 2.5 mL samples of electrolyzed effluent, collected after each 20 min of electro-oxidation times. The waste effluent absorbance was determined from 190 nm to 820 nm in a UV–vis spectrophotometer (UV1700 Pharmaspec, Shimadzou). After the absorbance determination, the COD of each sample was determined.

The Fig. 3 shown effect of NaCl concentration on the electrooxidation of waste water, carried out at $60mA/cm^2$. We observed that the application of electrolysis in this compound have the ability to reduce considerably the COD. For example, 2g/L mass NaCl and 1g/L% NaCl the achieved reduction was 86% and 47% respectively, while for without NaCl was 74%. The presence of a weak concentration of chloride ions allows to inhibit the water discharge into oxygen, and to favorise hydroxyl or chloride and oxycloride radicals, which are very powerful oxidants. It can be explain why until 2 g/L of NaCl concentration the COD removal increases with NaCl concentration.

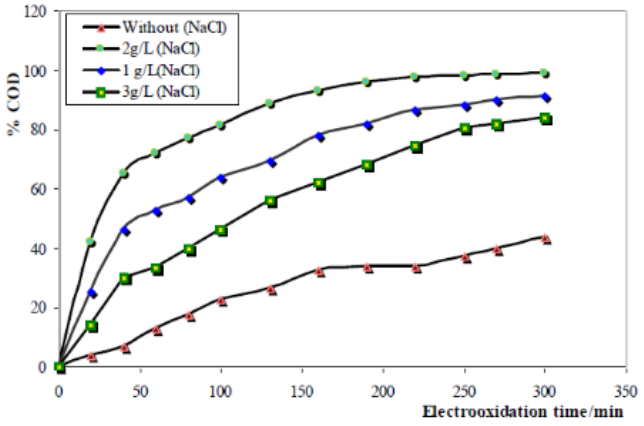

Figure 3. Influence of the concentration NaCl on the trends of %COD electrolysis of waste water cartons (COD0 = 2498 mgL^{-1}) using a $1cm^2$ BDD anode at T=25◦C.

Increasing the chloride concentration more than 3 g/L cause a "potentiostatic buffering" by the chlorine red/ox system and consequently a decrease of the anode potential. Another possibility is the presence of competitive reactions, in particular oxygen and chloride evolution due to recombination of radicals that becomes bigger with the increasing NaCl concentration.

CONCLUSION

Electrochemical technologies have been investigated as the effluent treatment processes for over a century. Fundamental as well as engineering researches have

established the electrochemical deposition technology in metal recovery or heavy metal-effluent treatment.

The electro oxidation process forms an attractive alternative and is successfully applied for industrial wastewater treatment. It does not produce any undesired reaction co-product nor use toxic or hazardous materials . It, therefore, eliminates the use of oxidation reagents to carry out the process which is very useful and suitable for on-site treatment, especially in small-scale facilities. Electro oxidation processes could be a pre-treatment step to biodegradation process. It enhances the biodegrability of wastewater containing inhibitory compounds by their decomposition resulting easily degradation of the rest in subsequent biological treatment. It could be an ideal finishing step in various combinations, virtually with all existing technologies and wastewater treatment systems.

The results of an investigation of the electrochemical degradation of a real cartons effluent using an BDD anode have been presented, in the absence and presence of chloride ions, for the system's optimized hydrodynamic conditions. The best conditions were attained applying a current density of 60 mA cm−2, at 25 ◦C, with (2 g/L) NaCl addition, when the electrochemical process is under charge transfer control and the BDD anode is stable. The rate of decolorization of the real cartons effluent was increased in the presence of chloride ions (2 g L−1NaCl). Thus, using the BDD anode the relative absorbance of the effluent could be reduced to less than 70% using an applied electric charge per unit volume of the electrolyzed effluent of only about 5 kAh m−3. This higher decolorization rate has been accounted for by the greater ability of active chlorine to degrade the organic compound of the cartons present in the effluent.

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