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DESIGN OF AN INDUSTRIAL ELECTROCHEMICAL PLANT FOR WASTEWATER TREATMENT

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ABSTRACT

Pollution load of water from urban networks is very notable, especially in large towns and industrial sites. In this context, the search for good solutions for this problem is highly important in current studies. Many chemical, physical and biological treatment methods have been developed. However, each of these methods has specific advantages. Therefore, the search for other solutions remains an exploited area. Among these new techniques to be developed are electrochemical methods. They present significant techniques in the elimination of organic and minerals pollutants; as well as, biological contaminants. In the present work, we have contributed to the design of an industrial electrochemical plant for the treatment of different pollutants based in previous studies. Firstly: we carried out an exhaustive study on the electrochemical processes used in the elimination of different pollutants. We approved a simulation to design a simple form of an electrochemical reactor. Furthermore, we regrouped the necessary information on electrode materials and experimentally confirmed the use of a general economic electrode. This investigation allowed constructing an efficient database to design an industrial scale electrochemical processing reactor, from which we used economic and efficient materials.

Keywords: Electrode materials, electrochemical plant, wastewater treatment, C/TiO_2.

1. INTRODUCTION

Pollutants from industry sources can cause permanent contamination. Some of these pollutants are mutagenic and carcinogenic, such as antibiotics, organometallic compounds, phenolic products, cosmetics and hormones. These compounds are responsible for disorders in the aquatic environment and constitute a risk for human health. Most of them can escape from conventional wastewater treatment. Their presence in the environment is a subject of concern for most environmental agencies in industrialized countries [1,2]. To limit their existence in the environment, effective treatment strategies have been developed. Among these strategies; the application of oxidation processes, can be grouped as follows: (a) homogeneous phase chemical oxidation processes (H_2O_2/O_3 and H_2O_2/Fe^{2+}); (b) homogeneous and/or heterogeneous phase photocatalytic processes (O_3/UV, H_2O_2/UV, Fe^{2+}/H_2O_2/UV and TiO_2/UV); (c) sonochemical oxidation processes, and (d) electrochemical oxidation processes. These technologies are based on the production of reactive radical entities, particularly, hydroxyl radicals (OH*) which are the most powerful oxidizing species that can be used in the fields of water treatment and industrial effluents. Hydroxyl radicals can oxidize many organic compounds with high oxidation yields [3,4]. These oxidation processes have been applied in several sectors; for several purposes [5]. Furthermore, the electrochemical processes can produce final oxidation products (H_2O, CO_2 and small degradable molecules). Most of these technologies have only been developed and applied at the laboratory scale, however; others, such as the combination of O_3/H_2O_2, have been applied at the industrial scale. In our investigation, we carry out a review and approach of different works to simulate and propose an electrochemical industrial and economic reactor.

2. ELECTROCHEMICAL OXIDATION PROCESSES

Electrochemistry is based mainly on the transfer of electrons, which makes it particularly interesting from an environmental point of view and can be applied to a large number of pollutants. Electrochemistry has proven its efficiency for the treatment of toxic effluents containing cyanides or for the recovery of heavy metals by cathodic reduction (electrowinning). In addition, it can also treat aqueous solutions loaded with organic matter, either as a pretreatment to transform refractory compounds into biodegradable products, or as a final treatment to completely mineralize organic compounds. Electrochemical oxidation has the advantage of being able to react with pollutants in two different ways, directly or indirectly.

2.1 Direct anodic oxidation

Industrial effluents and municipal wastewater can be treated by the direct action of electric current during electrolysis. There is a direct effect on a species in solution when electron exchange takes place exclusively at the electrode. Insoluble or metallic electrodes (Ti/SnO_2, Ti/IrO_2, Pt, Ti/RuO_2, PbO_2, etc.) are usually used. When applied at the anode, these electrodes allow the complete oxidation of many molecules. The products of anodic oxidation are carbonic acid, ammonia and other products. In many cases, the anodic oxidation of organic or inorganic pollutants leads to the formation of simpler, nontoxic or less toxic and biodegradable compounds. For example, at the anode, phenols are oxidized to acids, cyanides to cyanates, sulfides to sulfates, etc. Importantly, these electrodes make it possible to oxidize compounds that are not chemically oxidized or that are difficult to oxidize [6]. Molecules known to be nonoxidized or difficult to oxidize chemically, such as ethylenediaminetetraacetic acid (EDTA) or chromium (III), can be oxidized in direct electrochemical ways. Several authors [7] have studied the mechanisms of the electrochemical oxidation of organic molecules.

In addition, direct electrolysis can also effect on bacteria. A study carried out by the authors indicated that electrolysis of concentrated suspensions of *Escherichia coli*, on porous activated carbon electrodes, led to deactivation of bacteria. The electric field can also have a bactericidal action. When a contaminated solution is subjected to an electric field greater than the field existing at the level of the bacterial membrane, a bactericidal effect is observed.

2.2 Indirect anodic oxidation

In some cases, electrolysis can be inefficient or slow. It is then necessary to electrochemically generate an oxidant that will subsequently reacts in solution with pollutants. The indirect action can be obtained by the oxidation of halides (Cl^-, Br^- or I^-) or by the reduction of dissolved oxygen, the reactions of which make it possible to generate bactericidal oxidants such as HClO, HBrO, IOH, I_3^- or H_2O_2 [8].

The oxidation of halides is often used in water disinfection. The bactericidal efficiency of these oxidants depends on their ability to cross the bacterial membrane. Bacterial membranes are said to be more permeable to molecular forms than to ionic forms [9]. Therefore, the HClO and HBrO molecules are more bactericidal than the ionic compounds ClO^- and BrO^-. Studies on the combined effect of electric current and chemical oxidants show that bacteria initially subjected to an electric current, become more sensitive to chemical oxidants and are eliminated more quickly. These bactericidal oxidants (in particular HClO); also make it possible to oxidize many organic molecules (phenol, humic acids, etc.) and inorganic ones (H_2S, CN^-, Cr(VI), etc.) contained in wastewater.

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3. ELECTROCHEMICAL OXIDATION SYSTEMS IN WATER TREATMENT PLANTS

Compared with conventional process (chemical or biologcal), the nonpolluting aspect of electricity, the ease of automation and the reduction in the volume of equipment used in wastewater treatment processes are all parameters that contribute to the industrial development of electrolytic techniques. Several electrolytic systems have been developed on an industrial scale for the treatment of effluents polluted by various organic (humic acids, phenols, fulvic acids, etc.) and inorganic (phosphates, heavy metals, sulphides, fluorides, etc.) pollutants. In contrast, very few electrochemical processes have been developed on an industrial scale for the treatment of municipal or industrial wastewater. The author of [*10*] studied the possibility of integrating electrolytic systems in wastewater treatment plants. They described an electrolytic system installed at a municipal sewage treatment plant near a coastal area. This system made it possible to disinfect and deodorize wastewater before it was discharged into the receiving environment (at sea). The beaches were thus decontaminated. The processing line consisted of an electrolytic cell (anode: Ti/Pt; cathode: Fe) in which seawater was electrolyzed to produce sodium hypochlorite (NaClO). Doses varying from 1 to 2% NaClO (relative to the flow rate of the water to be treated) were injected into the raw water pipe, which had previously undergone a disintegration treatment aimed at reducing the diameter below 3 mm of total solids. The mixture of wastewater and NaClO was subsequently passed through two reactors of stirred tank type each with a retention time of 30 min. Once treated, the effluent was discharged into the sea with a residual concentration of NaClO of 2 $mg.L^{-1}$. This residual concentration ensured a stable effect. The total energy consumption of the system (including the energy consumption of the pumps, the disintegration system and the electrolytic cell) was estimated to be 0.33 $kWh.m^{-3}$. This energy consumption is largely attributed to the operation of the electrolytic cell.

A similar study [*10*] was carried out in a municipal wastewater treatment plant, where the objective was not only to reduce the volume of installation of wastewater treatment plant equipment, but also to minimize the costs of operation by integrating an electrolytic system in the treatment chain. The water was initially subjected to screening and then grit before being subsequently sent to a tank where it was mixed with sea water (or saltwater) in adequate proportions (sea water/wastewater ratio: 1/3; 1/4). The mixture is then sent into an electrolytic cell (anode: graphite; cathode: iron) with a retention time of a few seconds (5-10 s). Once electrolyzed, the effluent was sent to a flocculation/clarification basin, inside which disinfection and sedimentation (of suspended particles) took place simultaneously.

The retention time of the clarification/flocculation basin was between 60 and 110 min. The supernatant was discharged into the sea, while the solid fraction (sludge) was dried (drying beds) before any final disposal because of its high chlorine content. Importantly, the entire process was carried out for a period of time less than half the time required for conventional physical-chemical treatment of wastewater. As a result, the processing units could be smaller and less bulky. These results were obtained from two series of tests carried out via six electrolytic cells connected in parallel, which were electrically connected in series and operated with respective retention times of 5 and 10 s, the energy consumption was halved, and the BOD_5 and turbidity were reduced by 3% and 21% respectively, when the weather retention imposed was 5 s compared with 10 s. Concentrations of 2.56 and 7.36 $mg.L^{-1}$ of residual chlorine was recorded at the outlet of the clarification/flocculation tank for retention times of 5 and 10 s. However, after 24 h (after treatment), the residual chlorine concentration in the effluent was zero when a retention time of 5 s was imposed in the electrolytic cell, with a residual concentration of 5.2 $mg.L^{-1}$ was recorded for a retention time of 10 s.

4. RESULTS AND DISCUSSION

4.1 Electrodes used in wastewater treatment

Table 1 presents a summary of the important parameters of previous works on the treatment of different water pollutants, namely, the materials of the used electrodes, the type of pollutants treated, the contact time and current density used. We can observe that (a) the common general electrode materials are graphite, Ti, TiO_2 and metallic oxides; (b) the interval of medium current density used is [5-50 $mA.cm^{-2}$]; (c) the contact time is 60 min (1 hour); and (d) the pollutants are mostly organic compounds and inorganic materials.

Table 1. Presentation of the main parameters of previous works on water treatment:

Ref.	Electrode materials	Pollutant Type	Contact time / Current density ($mA.cm^{-2}$)
[11]	Graphite composite-epoxy	hair dye (org)	30 sec / -
[12]	Ti/SnO_2-Sb/SnO_2-Sb-CeO_2	methylene blue (org)	3 $L.min^{-1}$ / -
[13]	Anion exchanger	Urban water (org-inorg)	6-7 min / -
[14]	Ti/PbO_2 doped by Ce	Esters phthalic (org)	18-24 min / 25
[15]	Al	Tannery (org-inorg)	3 $L.min^{-1}$ / 6-9
[16]	Load on *biochar*	4-chlorophénols (org)	60 min / 1000
[17]	Kaolin/steel slag	Norfloxacine (org)	30 min / -
[18]	Ti/Sb-SnO_2-Eu/Graphene	Clothianidine (org)	120 min / 5-25
[19]	Ni-Ce/activated carbon	Organic mixture (org)	- / 0.047
[20]	Fe(III)/TiO_2/graphite	Used water (org/inorg)	150 min / 100
[21]	Polyaniline	Cd (inorg)	60 min / -
[22]	Fe-chitosane-carbon	As(III) (inorg)	60 min / 0.08
[23]	TiO_2/ activated carbon	Methyl orange (org)	35-60 min / 300
[24]	Boron-doped diamond	Phenols (org)	60 min / 100
[25]	Chitosane/N/C	Used water (org/inorg)	10 min / 1000
[26]	Ti-Sn-Ce/biochar	Kitchen water (org/inorg)	150 min / 0.003
[27]	Biofilm	Nitrogen (inorg)	800 hr / 0.17-0.35
[28]	Ti/Cu_xCoMnO_4	Organic mixture	120 min / 20
[29]	Composite FeS_2/N/S/C	Cu (inorg)	- / 1000-20000
[30]	Polymer/C	NO_3^- (inorg)	2-6 min / -
[31]	SiO_2	Phosphate (inorg)	120-180 min / -
[32]	boron-doped diamond	Pharmaceutics (org)	30-240 min / 50
[33]	Al	Used water (org/inorg)	10-60 min / 2.5-10
[34]	MnO_2/graphite	Phenols (org)	60-360 min / 2-8
[35]	Graphite	Textile (org/inorg)	30-60 min / 10
[36]	Al	Escherichia Coli (bacteria)	5-30 min / 2.5
[37]	Fe_3O_4/graphene	Textile (org/inorg)	180 min / 35
[38]	Ti/RuO_2-IrO_2, Ti/RuO_2-TiO_2	Organic (org)	20-180 / 5-50
[39]	Biofilm	Used water (org/inorg)	10 min / -
[40]	TiO_2/PbO_2	dye (org)	15 min / 15
[41]	TiO_2	dye (org)	10-60 min / -
[42]	Ti/SnO_2-Sb, Ti/PbO_2, Fe/PbO_2	Methylene orange (org)	30-90 min / 30
[43]	Ti/SnO_2–Sb_2O_4	Phenols (org)	60-120 min / 20
[44]	Ti-PbO_2	dye (org)	120 min / 500
[45]	Cu/graphite	Catechol (org)	in / 100

4.2 Reactors used in water treatment

In this section we present famous reactor types used in wastewater treatment. There are several types of reactors, where the necessary characteristics taken into account are: (a) the material and form of the electrodes, which is suited to the type of pollutant, (b) the general shape of the cell: water inlet and outlet, location of electrodes, water flow, circulation path, (c) contact time, and (d) the current density.

The following works present well-known examples of electrochemical flow reactors used at the laboratory scale, which could inspire the design of an industrial-scale reactor. The authors of [12] used a fabricated titanium membrane/SnO_2-Sb/SbO_2-Sb-CeO_2 anode in a continuous tubular membrane reactor to treat methylene blue wastewater under a cell voltage of 3.0-4.5 V and a medium flow rate of 3 $L.min^{-1}$ (180 $L.h^{-1}$). In this study, titanium has good efficiency in the treatment of methylene blue (organic pollutant), which is in good agreement with results of Table 1. The used cell has a medium volume of 3 L. For an inspiring industrial scale reactor of 1 m^3, the flow rate will be V = 1*180/3 = 6 $m^3.h^{-1}$. This important quantity of water can be treated in reactor of 1 m^3, and the flow rate can be optimized on the basis of need.

A previous study [46] reported a medium flow rate of 0.35 $L.min^{-1}$ (21 $L.h^{-1}$) in 1 L cell, where the anode was a tubular porous Ti/RuO_2 (Figure 1). Therefore; titanium/metal oxides were used in the anode materials, and the current density was 5 $mA.cm^{-2}$, which is in good agreement with results of Table 1. In addition, the electrode surface area was 0.00942 m^2. With these models; we can calculate the parameters of an industrial scale reactor of size 1 m^3. The medium flow rate will reach V=1*21=21 $m^3.h^{-1}$. The electrode surface was approximately 1*1000*0.00942=9.42 m^2 for the same current density of 5 $mA.cm^{-2}$. If we propose a square electrode with a general surface area approximately 0.5 m^2 (S=0.5m*0.5m*2), we need 19 electrodes in the cell which will slow down and block the flow of water and damage the electrodes very easily. However, if we take a current density of 20 $mA.cm^{-2}$, we will need approximately 6 electrodes in a reactor of 1 m^3, where in a cubic form; the distance between them will be about d=0.17 m (17 cm), and the electrode series can be placed onto the reactor without slowing down the flux of water and assuring a high contact efficiency.

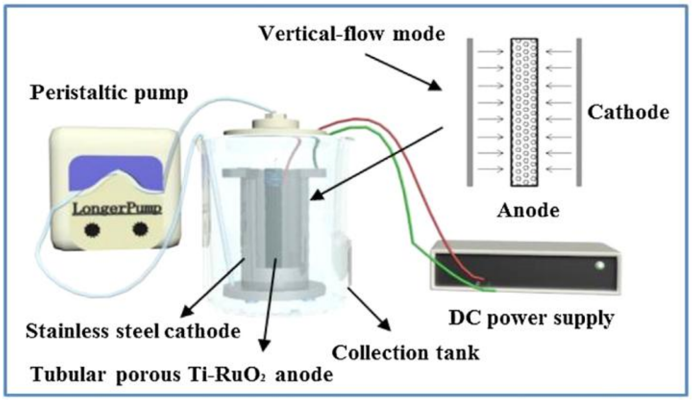

Figure 1. Reactor with a Ti/RuO_2 porous tubular electrodes [46].

The authors of [47] used a stainless steel/carbon anode of 36.4 cm^2, which is a famous and economic material. The operational cell volume was 1.6 L. The authors used this cell to remove 2.58 mg of nitrates from real wastewater at a current density of 0.035 $mA.cm^{-2}$. For an industrial plant of 1 m^3 derived from this investigation, we can treat 1.61 g of nitrate on Fe/C electrode of surface equal 2.27 m^2. The surface can be divided into 6 electrodes of a general surface of approximately 0.4 m^2 (~0.5 m^2) with the same distances between electrodes of 17 cm similar to the former example, using the same current density of 0.35 $mA.cm^{-2}$.

The authors of [48] used anode materials of Ti/IrO_2 and applied a current density of 5 $A.cm^{-2}$ for 50 h to treat antibiotics; these compounds are high refractory. These kinds of electrodes can be used as selective materials for one of difficulty degrading pollutants.

Concerning the works [49-52], electrodes used are based on graphite, titanium and metal oxides. The system of flow water between many electrodes placed in the cells as a parallel plate series (Figure 2) [50]. Therefore, the important parameters of these investigations can be summarized as follows: (a) the common general core is a cell of flow with water inlet and outlet system; (b) the electrode material is based on graphite, Titanium and metallic oxides; (c) the general electrode surface is [2.27 - 9.42 m^2]; (d) the interval of the used medium current density is [0.35 - 5 $mA.cm^{-2}$]; (e) the water flow is [6 - 21 $m^3.h^{-1}$], (f) the pollutants are organic and inorganic; (g) the single electrode surface is [0.4 - 0.5 m^2], (h) number of single electrodes in the cell is optimized to 6, which is equal to the general surface and presents a parallel electrode series with high contact efficiency.

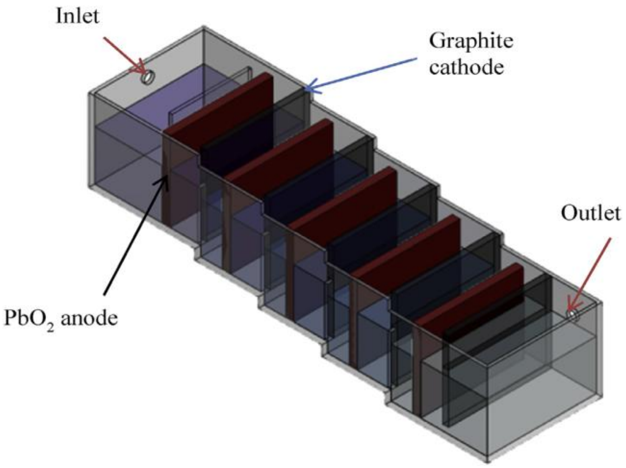

Figure 2. Parallel plate reactor with continuous serpentine flow [50].

4.3 Experimental investigation

We adopted the preparation by thermal deposition technique, as a new method to prepare electrode surfaces, where we used a simple gas torch to add a quantity of titanium oxide TiO_2 homogeneously over the entire surface of the graphite, silver and copper electrodes. This choice of C/TiO_2 was based on previous investigations as a common general electrode material; to treat organic and inorganic pollutants, the choice of silver and copper was also based on their low price as inert electrolysis electrodes compared with platinum and gold. The surface was heated to a high temperature (of approximately 700 °C) until the reddening stage for about 15 minutes, after which the TiO_2 powder was sprayed onto the heated surface and heating was continued for about 15 minutes. After cooling in air for more than 24 hours, the electrodes were washed with distilled water and acidified water (pH=1 N). Figure 3 presents the different steps of the preparation method.

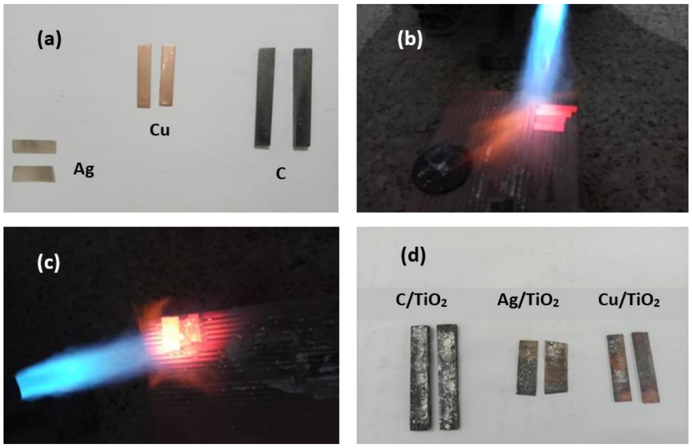

Figure 3. Thermal deposition technique of TiO_2 onto C, Ag and Cu electrodes: (a) cleaning electrode surfaces; (b) heated surfaces; (c) spraying of TiO_2 and heating; (d) raw prepared electrodes.

4.4 Validation of prepared electrodes

All C/TiO_2, Ag/TiO_2 and Cu/TiO_2 electrodes were subjected to treatment with simulated pollutants. The first simulated and examined organic pollutant was the dye tannery red (3-Amino-7-dimethylamino-2-methylphenazine hydrochloride, which is widely used in industry, with a general structure of $C_{15}H_{17}ClN_4$, pKa=6.4-7.5 and a molecular mass of 288.77 g/mol). On the other hand, the second simulated inorganic pollutant was a solution of Ni^{++}, Cu^{++} and Fe^{++} ions.

We compared the prepared electrodes with their basic precursors, namely, C, Ag and Cu, to verify their reliability and efficiency in the treatment of different pollutants.

However, we performed a quantitative kinetic study of the degradation of the simulated pollutants by decreasing their concentrations; nevertheless, because of the very fast degradation speed of approximately 10 minutes, the study was very difficult; therefore, the qualitative reliability check was performed, from which all the electrodes showed great discoloration and degradation of the tested organic solutions of tannery red. In addition, the inorganic products were rapidly reduced and precipitated. This means decomposition and reduction of materials on the surfaces of the prepared electrodes as shown in Figure 4.

The prepared electrodes were used as both as cathodes and anodes in the treatment processes, to ensure the production of all possible electrochemical treatment reactions whether, cathodic or anodic.

We also mention that the prepared electrodes are resistant to oxidation after many work runs, especially C/TiO_2 which becomes very hard and doesn't decompose compared with graphite, which oxidizes easily after several runs. With respect to Ag and Cu, we observed that TiO_2 doped layer increased the resistance of the surfaces to clogging phenomenon and extend their usage period.

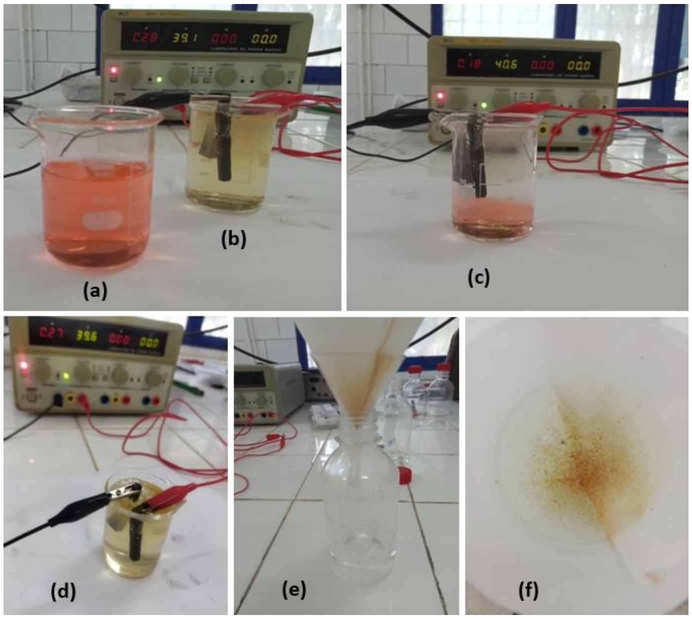

Figure 4. Different stages of depollution using C/TiO_2 general electrodes: (a) initial solution of organic pollutant (tannery red); (b) process of electro-oxidation in progress; (c) oxidation and discoloration of tannery red solution; (d) electroreduction and precipitation of iron compounds; (e) filtration of iron compounds; (f) products of electroreduction.

4.5 Simulation and design

Many recent works interested in the use of electrochemical techniques at the industrial scale using economic and long life electrodes have been published, such as the investigations of authors [53-56], who used flow reactor systems with highly resistant electrodes based on graphite/titanium/metals/metallic oxides as promising future electrodes. On the basis of collected and simulated data, we have gathered the following optimized facts:

1. The adequate industrial design is: a continuous and controlled flow plant. Here, we focus on the treatment of a large amount of water per day. Therefore, an adequate model is a series of adjacent cells (basins) with interesting volumes. The cubic form or parallelepiped can be adapted to facilitate the replacement and shape of the electrodes. The basins will be very tight in reinforced concrete such as aeration basins, connected by inert plastic pipes, ensuring the flow of water, where this flow occurs only by gravity; avoiding the use of pumps and the consumption of additional energy. The water enters through small canals to ensure low flow to the basins to prevent damage to and movement of the electrodes. Then, the water is filled and circulated from one basin to another. The passage between the basins takes an interesting time. The retention time was optimized to the range of 60 minutes. The retention time presents a sufficient period of contact between the solution and electrodes surfaces; to provide maximum time for the treatment reactions. The basins are equipped with drain valves at their bottoms; to empty the precipitated effluents after treatment.

2. The electrodes used are square shaped at approximately 0.5 m^2 for a plant of size 1 m^3 and a set of 6 square-shaped 0.5 m^2 (two-sided area of 0.25 m^2) electrodes is used, in the same basin; these large surfaces allow adequate contact with treated water. To prevent them from breaking due to the flow of interesting amounts of water; they must be supported by rigid plastic (inert) or metallic grids made of titanium oxide TiO_2 or other metallic materials depending on the need for selectivity (conductor and catalyst). The surface of these materials is very significant; because it provides a large interface for water contact. Their materials can be adapted to the types of pollutants according to the above data. Graphite with TiO_2 can be used as a general electrode for all types of pollutants. This type of electrode has been verified recently by many authors [57-61].

3. For the treatment of mineral ions, selective materials can be used to treat nonmetallic ions or recover metallic ions.

4. Diverse types of electrodes can be used in each basin to assume the selectivity of different pollutants existing in treated water, which has been verified in special industries (automotive, pharmaceutical, chemicals, etc.)

5. A single current generator or several generators can be used separately for each basin.

6. Toxic materials such as heavy metals (Pb, Cd, etc.) cannot be used as electrodes, to avoid contaminants.

7. To minimize the cost of electricity used in the process, the cells can be powered by free energy (solar or wind sources).

8. When the process of depollution on the electrodes occurs, electrolysis at a faradaic yield of approximately 40% of water and the production of hydrogen and oxygen result in secondary reactions, which has been verified by recent investigation [62].

CONCLUSION

On the basis of collected and simulated data, we propose a design example of an economic electrochemical plant for the continuous treatment of different types of pollutants, as presented in Figure 5. This model is a series of cells (basins) of optimized volume of approximately 1 m^3 that can be multiplied for industrial needs. This plant allows a circulation time of 60 min of water in each basin between the immersed series of electrodes, using a minimal current density of 0.35 to 5 $mA.cm^{-2}$. A general electrode of graphite/titanium oxide (C/TiO_2) can be used for the treatment of all types of pollutants. Additionally, it is possible to use selective electrodes for special species such as SiO_2 electrodes for recovering phosphates and composite for recovering cupper and other metals. This plant can be fed by free electricity from solar and wind sources. It can be reorganized to produce hydrogen and oxygen because of its high faradaic yield with the process of treatment.

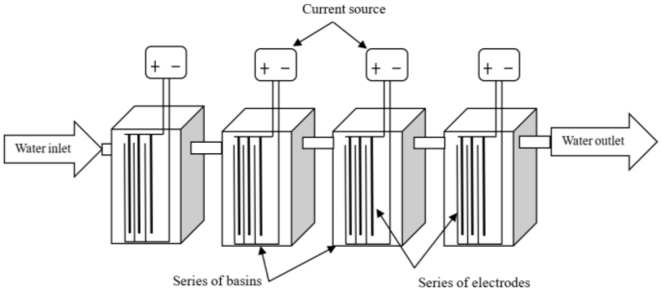

Figure 5. An industrial economical electrochemical plant with continuous flow, optimized basins and elecrode series, with general electrodes of C/TiO_2 and selective electrodes for special industries.

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