Research Article

Treatment of a Textile Effluent by Electrochemical Oxidation and Coupled System Electrooxidation–*Salix babylonica*

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The removal of pollutants from textile wastewater via electrochemical oxidation and a coupled system electrooxidation–*Salix babylonica*, using boron-doped diamond electrodes was evaluated. Under optimal conditions of pH 5.23 and $3.5 \text{ mA}\cdot\text{cm}^{-2}$ of current density, the electrochemical method yields an effective reduction of chemical oxygen demand by 41.95%, biochemical oxygen demand by 83.33%, color by 60.83%, and turbidity by 26.53% at 300 minutes of treatment. The raw and treated wastewater was characterized by infrared spectroscopy to confirm the degradation of pollutants. The wastewater was oxidized at 15-minute intervals for one hour and was placed in contact with willow plants for 15 days. The coupled system yielded a reduction of the chemical oxygen demand by 14%, color by 85%, and turbidity by 93%. The best efficiency for the coupled system was achieved at 60 minutes, at which time the plants achieved more biomass and photosynthetic pigments.

1. Introduction

The textile industry is one of the greatest generators of liquid effluent pollutants due to the high quantities of water used in the dyeing processes. The chemical composition involves a wide range of pollutants: inorganic compounds, polymers, and organic products [1–3]. Treatment of textile dye effluent is difficult and ineffective with conventional processes because many synthetic dyes are very stable in light and high temperature, and they are also nonbiodegradable. Moreover, partial oxidation or reduction can generate very toxic by-products [4–6].

Advanced oxidation processes (AOPs) have emerged as potentially powerful methods that can transform recalcitrant pollutants into harmless substances. AOPs rely on the

generation of very reactive free radicals and very powerful oxidants, such as the hydroxyl radical, $\text{HO}\cdot$ (redox potential $E^\circ = 2.8 \text{ V}$) [7, 8]. These radicals react rapidly with most organic compounds, either by addition to a double bond or by the abstraction of a hydrogen atom from organic molecules [9, 10].

The resulting organic radicals, then, react with oxygen to initiate a series of degradative oxidation reactions that lead to products, such as CO_2 and H_2O [1, 11]. Electrochemical oxidation is carried out by indirect and/or direct anodic reactions in which oxygen is transferred from the solvent (water) to the product to be oxidized [12]. The main characteristic of this treatment is that it uses electrical energy as a vector for environmental decontamination [13]. During direct anodic oxidation, pollutants are initially adsorbed on the

TABLE 1: Percentage of removal performance of color, turbidity, nitrogen, and COD by electrochemical or electrochemical with other electrochemical or biological procedures.

Treatment	Sample	Optimal operating conditions	Removal performance	References
Vertical-flow constructed wetlands with planted <i>Phragmites australis</i>	Acid Blue 113 (AB113), Basic Red 46 (BR46)	7 mg·L ⁻¹ (AB113); 208 mg·L ⁻¹ (BR46) 48 h (AB113); 96 h (BR46) contact times	Nitrate nitrogen (NO ₃ -N) 85–100%	Hussein and Scholz (2018)
Electrochemical oxidation	Malachite green oxalate (MG)	pH = 3; stainless steel cathode Boron-doped diamond (BDD) anode 32 mA·cm ⁻² current density Na ₂ SO ₄ supporting electrolyte	COD 98% (60 min) COD 91% (180 min)	Guenfoud et al. [1]
Electrocoagulation-phytoremediation (<i>Myriophyllum aquaticum</i>)	Industrial wastewater	pH = 8; Fe electrode; 45.45 A·m ⁻² current density	COD 94%, color 97% Turbidity 98%	Cano-Rodriguez et al. [35]
Electrochemical Fenton (EF) Chemical Fenton (CF)	Textile wastewater	H ₂ O ₂ 1978 mg·L ⁻¹ 350 mA electrical current	COD 70.6% (EF) COD 72.9% (CF) (60 min)	Eslami et al. [36]
Electrochemical oxidation	Textile dyehouse	pH = 1; 8 mA·cm ⁻² current density Boron-doped diamond (BDD) anode 0.25 M HClO ₄ supporting electrolyte	Color 100% Mineralization 85% (180 min)	Tsantaki et al. [37]
Electrochemical	Dyestuff effluent	Boron-doped diamond (BDD) anode; pH = 10; 60°C, 40 mA·cm ⁻² current density	COD 100%, color 100% (15 h)	Martínez-Huitile et al. [15]
Combined electrochemical, microbial, and photocatalytic	Procion blue dye	RuO _x -TiO _x catalytic anode 2 A and 30 V power source	COD 80% to 95%	Basha et al. [16]
Electrochemical	Novacron Deep Red C-D (NDRCD) Novacron Orange C-RN (NOCRN)	pH = 3; 170 A·m ⁻² current density Graphite carbon as anode and cathode Potential: +1.0 to -0.4 V (NDRCD) and +0.5 to -0.2 V (NOCRN); 300.15 K NaCl (7 g·L ⁻¹) supporting electrolyte	Color 99% (NDRCD); 97% (NOCRN) COD 88% (NDRCD); 82% (NOCRN)	Kariyajiannavar et al. [6]

FIGURE 1: A schematic diagram of the electrochemical reactor.

surface of the anode, where the anodic electron transfer reaction degrades them [6]. In indirect anodic oxidation, strong oxidants, such as hypochlorite, chlorine, ozone, or hydrogen peroxide, are electrochemically generated.

The pollutants are degraded via the oxidation reactions with these strong oxidants [11]. Boron-doped diamond (BDD) thin films are electrode materials that possess several technologically important characteristics, including an inert surface with low adsorption properties, an acceptable conductivity, and remarkable corrosion stability even in strongly acidic media and extremely high O_2 evolution overvoltage [14, 15].

On the other hand, biotechnology continues to be used to solve environmental problems [16–18]. Phytoremediation (PR) is a green technology that uses plant systems for the remediation and restoration of contaminated sites [19]. PR's advantages are solar energy dependence and an esthetically pleasant method of treatment [20]. Plants have inbuilt enzymatic characteristics that are capable of degrading complex structures, and they can be used for cleaning contaminated sites [17].

Plants, however, remove pollutants predominantly via adsorption, accumulation, and subsequent enzyme-mediated degradation [20]. Therefore, plants are considered organisms with complex metabolic activity when referring to the assimilation of toxic substances. Plant species that have different growth forms have been proposed for the treatment of textile effluents, for instance, *Glandularia pulchella*, *Phragmites australis*, *Tagetes patula*, *Alternanthera philoxeroides*, *Eichhornia crassipes*, *Nasturtium officinale*,

TABLE 2: Experimental design of the electrooxidation process.

Experiment	pH	Current density
1	5.23	3.5
2	5.23	7
3	5.23	10
4	7	3.5
5	7	7
6	7	10
7	10	3.5
8	10	7
9	10	10

Hydrocotyle vulgaris, *Petunia grandiflora*, and *Gaillardia grandiflora* [17, 18, 21–24].

Another alternative is to use species of fast-growing woody plants with high biomass production and high genetic variability [25–27]. Trees from the Salicaceae family with the genera *Salix* and *Populus* are suitable candidates for this purpose [28–30]. Willows (*Salix* spp.) have several characteristics that make them ideal plant species for PR application, including easy propagation and cultivation, a large amount of biomass, a deep root system, a high transpiration rate, tolerance to hypoxic conditions, and high metal accumulation capability [30, 31].

Salix babylonica has been used to solve the problems associated with aquifers contaminated with ethanol-blended gasoline [32] and studies of the biotransformation and

metabolic response of cyanide and dieldrin [33, 34]. In recent years, several authors have described dye removal by electrochemical (EC) or coupled electrochemical with other chemical, electrochemical, or biological procedures (Table 1). However, no studies have been conducted on the implementation of coupled electrochemical oxidation-phytoremediation with weeping willow in the remediation of textile effluents, and because it is an introduced species, is noninvasive, and is widely distributed in Mexico, the aim of this study was to evaluate the removal of the pollutants of a textile effluent using an electrochemical oxidation process and to compare their performance with *Salix babylonica*.

2. Materials and Methods

2.1. Wastewater Sampling. A textile wastewater sample was collected from a textile industry whose business is the dyeing and washing of denim garments in Almoloya del Río, State of Mexico, Mexico. The wastewater that this industry discharges does not receive any treatment and is discharged into the sewage system, so it is necessary to give it some kind of treatment to improve its quality. The textile wastewater sample was placed in plastic containers and transported to the laboratory, where it was refrigerated at 4°C for analysis and for conducting the electrochemical oxidation and coupled system electrooxidation-*Salix babylonica*.

2.2. Electrochemical Reactor. In this study, a batch electrochemical reactor was used. The reactor contained five vertical parallel electrodes of BDD (titanium/BDD) that CONDIAS DIACHEM manufactured, two as cathodes and three as anodes. Each electrode was 20.5 cm long and 2.5 cm wide, resulting in an area of 102.5 cm² for each electrode and a total anodic area of 307.5 cm². A schematic diagram of the electrochemical reactor is shown in Figure 1. The tests were carried out in a 1 L cylindrical reactor. The reactor was operated at different pH values (5.23, 7, and 10). A current density power supply provided 1, 2, and 3 A and 5–6.75 V, corresponding to a current density of 3.5, 7, and 10 mA·cm⁻².

The experiment design used included the two factors of pH and current density. The levels of each of the factors are listed in Table 2. Different aliquots were taken, and the chemical oxygen demand (COD), biochemical oxygen demand (BOD₅), color, turbidity, and conductivity were analyzed. The boron-doped diamond electrodes (BDD) were cleaned for one hour in Na₂SO₄ (0.03 M) after each experiment to remove adsorbed molecules at the electrode surface, and then they were rinsed with distilled water.

2.3. Coupled System with *Salix babylonica* Treatment. For *Salix babylonica* treatment, secondary branches of weeping willows located in five regions near the discharge site were collected based on some defined phenotypic characteristics: intense green color, wide coverage, height greater than 8 meters, absence of pests, and straight shaft. Branch cuttings of 20 cm in length were placed in hydroponics [28] in 1 L containers with 300 mL of distilled water. They

TABLE 3: Physicochemical characterization of textile wastewater.

Parameter	Raw wastewater
pH	5.23
Acidity (mg/L CaCO ₃)	962.8
Alkalinity (mg/L CaCO ₃)	1000
BOD (mg/L)	1400
BOD/COD	0.7
COD (mg/L)	2022
Color (Pt-Co U)	3000
Chlorides (mg/L Cl ⁻)	843.71
EC (mS/cm)	2.811
Hardness (mg/L CaCO ₃)	546.2
N-NO ₂ (mg/L)	0.848
N-NO ₃ (mg/L)	17.28
N-NH ₃ (mg/L)	4.72
Phosphorus (mg/L P)	715.1
Sulfates (mg/L SO ₄ ²⁻)	429.5
Turbidity (NTU)	735
TOC (mg/L)	1396.6
TDS (mg/L)	1367
Ca ²⁺ (mg/L)	36.537
K ⁺ (mg/L)	64.43
Mg ²⁺ (mg/L)	17.346
Na ⁺ (mg/L)	392.79

were kept at room temperature (19–22°C) for a normal photoperiod (12 h light, 12 h dark). Ten willows per region were placed in jars of 1 L and were then placed in 500 mL of textile wastewater. They remained in contact with wastewater for 15 days, and water aliquots were taken at baseline and at intervals of eight days. Likewise, the development of plants during those time periods was assessed.

3. Methods of Analysis

3.1. Physicochemical Characterization. The characterization of textile wastewater was performed. During both electrochemical and phytoremediation treatment, COD, BOD₅, color, turbidity, pH, and electrolytic conductivity analyses were performed as indicated in the standard methods procedures by the American Public Health Association [38]. In addition, infrared spectroscopy of the raw and treated water was performed.

3.2. Biological Parameters. Once roots and leaves were developed in hydroponics, they were weighed on an analytical balance (BEL Engineering), and the lengths of the plants and roots were measured using a vernier. The numbers of roots and leaves were counted, and the leaf areas and photosynthetic pigments were measured by using the method that Val et al. [39] and Moisés et al. [40] established. These measurements were performed at the beginning of biological treatment and every eight days.

FIGURE 2: Behavior of (a) BOD₅, (b) color, (c) turbidity, and (d) COD. Applying three current densities: 3.5 mA·cm⁻² (●), 7 mA·cm⁻² (▲), and 10 mA·cm⁻² (X), at pH 5.23.

4. Results and Discussion

4.1. Wastewater Characterization. Physicochemical characterization of textile wastewater is shown in Table 3. The organic parameters indicate for the BOD₅ a value of 1400 mg/L. According to Mexican regulation, the allowed limit for discharging wastewater into rivers is 150 mg/L. The COD was 2022 mg/L; in this situation, the BOD/COD ratio (0.7) indicates good biodegradability [9]. The TOC was 1396.6 mg/L, and the color was 3000 Pt-Co U; this high level of color stemmed from the indigo blue dye in the textile effluent. Regarding inorganic matter, different ions contribute to high conductivity (2.811 mS/cm). This parameter could be beneficial to the electrooxidation process because it was not necessary to add any support electrolyte. However, the presence of ions as nitrates, phosphates, and alkalinity could reduce the oxidation speed of organic compounds; on the other hand, chlorides (843.71 mg/L) could improve the indirect organic oxidation.

4.2. Electrooxidation Treatment

4.2.1. Current Density Effect. An important operating variable of the electrochemical process is the current density, which is the input current divided by the surface area of

TABLE 4: Removal efficiencies of different parameters in the electrooxidation process.

Experiment	pH	Current densities	BOD (%)	Color (%)	Turbidity (%)	COD (%)
1	5.23	3.5	83.33	60.83	26.53	41.95
2	5.23	7	69.95	98.88	98.95	62.01
3	5.23	10	91.81	99.81	99.91	82.39
4	7	3.5	58.34	96.95	98.77	47.73
5	7	7	91.15	99.66	99.77	63.14
6	7	10	91.58	99.82	99.90	94.66
7	10	3.5	58.56	92.45	98.09	82.10
8	10	7	94.73	99.01	99.04	98.11
9	10	10	94.38	97.04	93.87	95.03

the electrode [11]. From other variables effective in the electrochemical process is current density as the rate of electrochemical reactions is controlled by this parameter. Further, the performance of electrodes is highly dependent on this parameter. Three different current densities were applied (3.5, 7, and 10 mA·cm⁻²) to investigate the effect in the oxidation process. All experiments were carried out at pH 5.23 (sample pH), and in all cases, a direct effect of the current densities was observed: If the current increases,

FIGURE 3: Instantaneous current efficiency (ICE) for the anodic oxidation process: 3.5 mA/cm² (●), 7.0 mA/cm² (▲), and 10 mA/cm² (X).

the removal efficiency increases. This could be due to the increased rate of the generation of oxidants, such as hydroxyl radicals and chlorine/hypochlorite at higher current densities [6]. The results at different densities are shown in Figure 2. The best removal efficiency was when 10 mA·cm⁻² was applied. BOD₅ was reduced considerably from 1400 mg/L to 114 mg/L with 92% of efficiency; COD was 2022 mg/L and was reduced to 356.05 mg/L with 82% of removal efficiency; color was reduced from 3000 Pt-Co U to 5.5 Pt-Co U (99.8% removal efficiency); and initial turbidity was 735 NTU and at the end of the process was 0.65 NTU, achieving 99.99% of removal efficiency. Efficiency was measured during 300 min of treatment time (Table 4).

The instantaneous current efficiency (ICE) for the anodic oxidation was calculated from the values of COD using

$$ICE = FV \frac{COD_i - COD_t}{8I\Delta t}, \quad (1)$$

where F is the Faraday constant (96487 C/mol), V is the volume (L), COD_i and COD_t are the chemical oxygen demand (g/L) at initial time and time t , I is the applied current (A), Δt is the treatment time (s), and 8 is the equivalent mass of oxygen (g·eq⁻¹). The instantaneous current efficiency (ICE) decreased during the electrolysis as wastewater was oxidized. This behavior is shown in Figure 3.

The best ICE percentage was when the lowest current was applied 1 A (3.5 mA·cm⁻²) in the middle stage of electrooxidation (15–45 min). This may be attributed to the presence of a higher concentration of organics near the electrodes. This indicates that the electrooxidation was under the current control regime at least in the middle stage of electrooxidation. The ICE decreased after 60 min of the electrooxidation process. This may be due to the depletion of the concentration of organics on the electrode surface.

The energy consumption per volume of treated effluent was estimated and expressed in kWh·m⁻³. The average cell voltage during the electrolysis (cell voltage is reasonably

constant with just some minor oscillations, and for this reason, the average cell voltage was calculated) was measured to calculate the energy consumption by using [15]

$$\text{Energy consumption} = \frac{\Delta E_c \times I \times t}{1000 \times V}, \quad (2)$$

where t is the time of electrolysis (h); ΔE_c (V) and I (A) are the average cell voltage and the electrolysis current, respectively; and V is the sample volume (m³). According to the results, 5.87 kWh·m⁻³ is required to oxidize the pollutants in the textile wastewater. In another study, a real textile effluent was treated using a BDD anode, applying a current density of 20 mA·cm⁻². The energy consumption was 20 kWh·m⁻³ [15].

The specific energy consumption (E_c) in kWh·(kg COD)⁻¹ removed was determined according to [37]

$$E_c = \frac{UIt/60}{(COD_0 - COD)V}, \quad (3)$$

where U is the mean applied voltage (V), I is the current (A), t is the treatment time (min), V is the liquid volume (L), and COD_0 and COD are the COD values (g O₂ L⁻¹) at times 0 and t . The results showed that 21.87 kWh·(kg COD)⁻¹ was required in the electrooxidation process. In a previous work, 95 kWh·(kg COD)⁻¹ was applied for the same COD removal [37].

4.2.2. pH Effect. The studies were performed at three different initial pH values (5.23, 7, and 10) to investigate their effects as depicted in Figure 4. The current density applied in these experiments was 3.5 mA·cm⁻². At alkaline pH (10), the best efficiencies were achieved: COD (85.9%), color (99.6%), BOD₅ (70.3%), and turbidity (99.7%). However, an addition of NaOH was required to adjust the pH, and this could be a disadvantage in the oxidation process.

The pH solution was an important factor for wastewater treatment. In anodic oxidation, many reports exist on the influence of pH solution, but the results are diverse and even contradictory due to different organic structures and electrode materials [1]. In an acidic solution, the degradation process of azo dyes is higher than in a basic solution, as in acidic solutions, chlorides are reduced to free chlorine, which is a dominant oxidizing agent [6]. During all experiments, the initial pH decreased during the treatment time (2.3–2.75). This could be attributed to the fragmentation of organic matter into carboxylic acids, carbonic acid, and ions as by-products of mineralization. Figure 5 shows the behavior of the conductivity during the treatment time; it increased at the end of the process probably as a result of the mineralization in the electrochemical oxidation process.

4.2.3. Degradation Mechanism. Previous research studies [41, 42] indicated that the oxidation of organics with concomitant oxygen evolution assumes that both organic oxidation and oxygen evolution take place on a BDD anode surface via the intermediation of hydroxyl radicals generated from the reaction with water shown in

FIGURE 4: Behavior of (a) BOD₅, (b) color, (c) turbidity, and (d) COD. Applying 3.5 mA·cm⁻², at pH 5.23 (■), 7 (Δ), and 10 (●).

FIGURE 5: Conductivity behavior during the treatment time at three different pH values.

Reaction (4) is in competition with the side reaction of hydroxyl radical conversion to O₂ without any participation of the anode surface as indicated in

Textile wastewater was analyzed via infrared spectroscopy before and after the electrochemical oxidation process, and the spectra are shown in Figure 6. The principal functional groups found in the aqueous solution of dye were -NH- (3305 cm⁻¹), the C-H aromatic bond (2910 and 2845 cm⁻¹), -NH₃⁺ (2340 cm⁻¹), aromatic -C=C- (1614 cm⁻¹), sulfoxides (1101 and 1022 cm⁻¹), and C-CO-C in ketones (611 cm⁻¹). The spectra of oxidized water showed that the intensity of corresponding bands to sulfoxides and secondary amines diminished after treatment, whereas the bands of R-COOH and O-C=O increased. In accordance with the above, the proposed dye degradation mechanism is shown in Figure 6.

4.3. Phytoremediation with *Salix babylonica*

4.3.1. Textile Wastewater. After oxidation treatment, the oxidized water was placed in contact with plants for 8 and 15 days, as shown in Figure 7. Parameters of the COD, color, and turbidity were minimally reduced. However, at 15 days of contact time, a visible reduction in color and turbidity was noted. According to the results, the plants assimilated better with the pollutants in the raw water than in the oxidized water due to the structural changes that the compounds suffered with the electrooxidation treatment. The coupled system (electrooxidation + phytoremediation)

FIGURE 6: Diagram of the degradation mechanism proposed. Within the figure are shown the infrared spectra of raw water and oxidized water.

yielded a reduction of the COD by 14%, color by 85%, and turbidity by 93%.

4.3.2. *Salix babylonica* Biomass. The willow biomass tolerance was analyzed by using Minitab 15.1.20 statistic program

analysis of variance (ANOVA) to find significant differences between treatments. As shown in Figure 8(a), significant differences were found in the leaf numbers, leaf areas, and root numbers among the plants that were in contact with oxidized water for different amounts of time ($P < 0.05$; $F = 8.20$).

FIGURE 7: Behavior of (a) COD, (b) color, (c) turbidity, and (d) conductivity, with *Salix babylonica* contact at initial time (▲), 8 days (×), and 15 days (●).

FIGURE 8: Willow tolerance (a) biomass parameters: weight (g), root number, root length (cm), leaf number, and leaf area (cm²); (b) pigment concentration (mg·L⁻¹) at different oxidized water contact times.

Willow plants in contact with oxidized water for 60 minutes reached a biomass close to that of the control plants. The same behavior was observed in the pigment concentration as shown in Figure 8(b). Willow plants tend to lose leaves in a stressful environment, but the root system and photosynthetic metabolism remain.

With respect to the contact time, willow plants reduced their photosynthetic metabolism and lost leaves at eight days

of contact time, but after this time, such plants recovered their photosynthetic metabolism to some extent as shown in Figure 9. This could be because willow plants became adapted to the new environmental conditions. The mechanism by which *Salix babylonica* decreases color and pollutant concentration is unknown, but an increase in the concentration of chlorophylls indicates that the plant is photosynthesizing and thus absorbing nutrients from wastewater.

FIGURE 9: Willow tolerance (a) biomass parameters: weight (g), root number, root length (cm), leaf number, and leaf area (cm²); (b) pigment concentration (mg·L⁻¹) at different contact times.

FIGURE 10: Willow tolerance (a) biomass parameters: weight (g), root number, root length (cm), leaf number, and leaf area (cm²); (b) pigment concentration (mg·L⁻¹) from different regions: north (N), south (S), east (E), west (W), and center (C).

Furthermore, the adsorption of contaminants in plant roots has been documented [20]. The results may indicate that the willow phenotype of the western region is characterized by very dense foliage and root system, by genetics, or by environmental influence, but in terms of photosynthetic metabolism, they are the same as the willows of the other regions (Figure 10).

5. Conclusions

Textile wastewater composition was favorable for carrying out electrochemical oxidation due to the high salt content. All experiments were carried out at the original pH (5.23), and it was determined that if the current density was increased, the removal efficiency increased. However, the current efficiency decreased during this process. For this reason, the lower current density was chosen (3.5 mA/cm²) as optimal. The infrared spectroscopy of the wastewater before and after electrooxidation showed a degradation of dye. The proposed degradation mechanism showed carboxylic acids and sulfates as degradation products. In the coupled system, a reduction of the COD was decreased by 14%, color by 85%, and turbidity by 93%. The biomass and pigment of

willow *Salix babylonica* demonstrated that this species has the ability to adapt to adverse conditions very quickly.

Conflicts of Interest

The authors declare that they have no conflicts of interest.

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