

Effects of Operational Parameters of Electrocoagulation Using Zinc Electrode on the Treatability of Textile Wastewater

Electrocoagulation of Textile Wastewater

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Abstract: *Electrocoagulation, in its optimum conditions, could effectively replace conventional treatment processes for textile wastewater. Zinc electrode has the potential to increase the treatment efficiency, but the right operating parameters should be identified. The electrocoagulation process using zinc electrodes for the treatment of synthetic textile wastewater (containing reactive blue 261 dye) and real textile wastewater was investigated. Current density (2-8 mA/cm²), initial dye concentration (20-100 mg/L) and initial pH (5-9) were the investigated variables. The best conditions were found at a current density of 4 mA/cm², an initial dye concentration of 100 mg/L, and an initial pH of 9. The highest dye removals for treating synthetic textile wastewater and real textile wastewater were 99.8% and 83.7 %, respectively, under the best-operating conditions. The electricity consumption for the treatment of synthetic wastewater and real textile wastewater was found to be 0.66 kWh/m³ and 0.69 kWh/m³, respectively.*

Keywords: Electrocoagulation; reactive blue dye; textile wastewater; zinc electrode; operating conditions; energy consumption.

1. INTRODUCTION

Textile industry is one of the industries that present significant environmental pollution problems. This industry uses chemicals and dyes in various fabrication processes (dyeing and finishing processes) where a large quantity of these dyes (up to 50 %) are lost in wastewater.^{1,2} Different types of dyes used in the textile manufacturing process are direct, vat, acidic, sulfur, metal complex, and reactive dyes.^{2,3} Reactive dyes are the most important class of dyes. They are used for dyeing cellulosic and protein fibers such as wool and silk. Wastewater containing reactive dyes is usually toxic and sometimes carcinogenic, as well as causing an aesthetic problem due to the color.⁴ Wastewater from the reactive dyeing process usually

has high alkalinity ($> 200 \text{ mg CaCO}_3/\text{L}$), inorganic (total dissolved solids (TDS) ($> 5,000 \text{ mg/L}$)), and organic matter content (COD $> 1,000 \text{ mg/L}$).⁵

Methods to treat textile waste include biological processes, chemical coagulation, activated carbon adsorption, ultrafiltration, ozonation, electrocoagulation, flotation processes, and other physical processes.⁶ Biological treatment is usually subject to low efficiency due to the toxicity characteristic of the wastewater and the need for a continuous aeration system.^{7,8} However, the low degradability of the organics in the wastewater renders limited treatability. Meanwhile, a chemical coagulation process involves the use of many chemicals, needs accurate pH control, and generally leads to secondary pollutants in the form of sludge.⁹ Activated carbon adsorbent is difficult to be regenerated and its performance is limited by adsorption equilibrium and adsorptive capacity of the adsorbent.¹⁰ In ultrafiltration and reverse osmosis processes, high operating costs and low treatment capacity are the main limitations.¹¹ Due to the drawbacks of current treatment processes, the development of new and effective technology for the treatment of textile wastewater is critically needed.

Electrocoagulation (EC) process has been demonstrated to effectively treat paper mill wastewater,¹² metal plating wastewater,¹³ battery industry wastewater,¹⁴ heavy metal-containing wastewater,¹⁵ and landfill leachate.¹⁶ It uses an electrochemical process in combination with chemical coagulation in which the applied potential releases the coagulant species into the solution by gradually dissolving the metal anodes such as aluminum or iron.¹⁷ The produced metal coagulants are active and efficient for charge neutralization of colloidal and suspended particles, leading to floc formation.¹⁸ The settling of the flocs removes significant pollutant loads from the effluent while allowing the adsorption of some soluble constituents. Compared to the conventional coagulation process, the gradual release of the metal coagulant during the electrocoagulation process allows effective coagulation while avoiding excessive metal coagulant remaining in the treated effluent. Soluble metal ions such as Al and Fe ions are produced at the anode, and they will react with the hydroxide ions that are formed at the cathode to form metal hydroxides. These insoluble metal hydroxides will react with suspended and colloidal solids in the wastewater and precipitate.^{17,19}

Several studies on electrocoagulation technology for synthetic wastewater treatment have been successfully conducted by using iron and aluminum electrodes.^{5,11,12} However, the search for a new material that could lead to high dye removal is still being undertaken while the performance in the treatment of a real textile wastewater containing other normal constituents is hardly reported in the literature. Until recently, despite being a good chemical coagulant, zinc electrode has not been used for EC treatment of textile wastewater as the sacrificial

electrode. The performance of an electrode can be influenced by many factors, such as the number of charges per ion, the solubility of their hydroxides in the water, the pH of the water, and the presence of other water constituents to interact with the metal coagulant.⁵ In this study, zinc was chosen as the electrode material on the grounds of its high ability to undergo redox reactions in water and its low cost. The location of zinc in the galvanic series theoretically allows the generation of zinc ions more readily as compared to iron and aluminum. Synthetic textile wastewater was prepared by using reactive blue 261 dye to demonstrate the effects of operational variables. The electrochemical behaviors of the system under different current densities, pH, and concentrations of the dye were elucidated. The best treatment conditions were subsequently used to treat a real textile wastewater for comparison. The practicality of the process was also evaluated based on energy consumption during the process.

2. EXPERIMENTAL DETAILS

2.1 Materials and wastewater samples

The materials and chemicals involved in this study were used without any purification. The H_2SO_4 (98.0 %) and HCl (37.5 %) were purchased from Sigma-Aldrich while NaOH (99.5 %) was sourced from Merck. The dye used in the preparation of synthetic textile wastewater was Reactive Blue 261 dye, and it was also obtained from Sigma-Aldrich. The synthetic wastewater was prepared by dissolving different concentrations of the dye (20-100 mg) into one liter of distilled water. The real textile industry wastewater was also used in this study for comparison, and it was collected from the dyeing process at Penfabric textile industry in Penang, Malaysia. The main characteristics of the real wastewater are presented in Table 1.

Table 1: Characteristics of the real textile industry's wastewater.

Basic property	Value
pH	9
Temperature (°C)	28
Electrical conductivity ($\mu\text{S}/\text{cm}$)	28,600
COD (mg/L)	1,100
Turbidity (NTU)	90
λ_{max} (nm)	527
Absorbance at max wavelength	1.49

2.2 Electrocoagulation set-up

The electrocoagulation set-up used consisted of a reactor vessel, an electrode set, a DC power supply, a stirrer, a pH meter, and an electroconductivity meter (Figure 1). The reactor was fabricated using double-walled perspex with dimensions of 100 mm x 100 mm x 110 mm and has a capacity of 1.1 liters. Ten square electrodes (i.e., five anodes and five cathodes) of the same dimensions (70 mm x 70 mm x 1 mm) were used. The submerged effective surface area for each electrode was 55 mm x 70 mm. The distance between electrodes was maintained at 5 mm while the gap between the electrodes and the bottom of the reactor was set at 30 mm. Zinc electrodes with a purity of 99.5% were used in this study. In all experiments, the anode and cathode materials were the same materials to avoid passivation of the anode surface or the formation of carbonate films on the surfaces of the cathode. The electrodes were connected to each other in a parallel monopolar mode. Two outer electrodes were connected to a DC power supply (Dazheng PS-305D; 32V, 5A) to vary the current during the experiment. A magnetic stirrer was used during the experiment, and the solution was stirred at 250 rpm to ensure homogeneity.

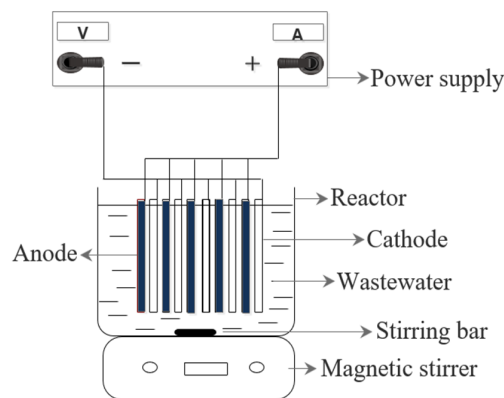


Figure 1: The experimental set-up used.

2.3 Experimental procedure

Before starting each run, the electrodes were washed with acetone to remove any contaminants such as oil, grease, scales, and dust from the surface. Then, they were dipped in a dilute HCl solution (10%) for 5 min for electrode activation and subsequently rinsed with distilled water, dried, and weighed. 1,000 mL of wastewater solution was then fed into the reactor. The electroconductivity of the solution was adjusted by adding NaCl to the solution and was measured using a conductivity meter, while the pH of the solution was adjusted using 0.1 M NaOH

or 0.1 M H₂SO₄ solution. The solution was continuously stirred at 250 rpm in all experiments, and the electrode set was placed inside the reactor at a depth of 55 mm and connected to a DC power supply. The current density was adjusted to the desired values (2-8 mA/cm²) and 60 min of operating time was set for each run.

Samples of the treated wastewater were taken using a pipette from the middle of the reactor at 5, 10, 20, 30, 40, 50, and 60 min of operating time. The collected samples were filtered using a 0.45 µm membrane filter paper for further analysis. The operating parameters investigated in this study were the current density (2, 4, 6, and 8 mA/cm²), the initial pH of the sample (5, 6, 7, 8, and 9), the dye concentrations (20, 30, 50, and 100 mg/l) and the processing time up to 60 min. The Reactive Blue 261 dye absorbance was measured using a UV-vis spectrophotometer (Agilent 2010) at its maximum wavelength of 620 nm. Meanwhile, the dye removal of the real textile wastewater was calculated based on the absorbance of the untreated and treated samples.

2.4 Electrical energy consumption

The consumption of energy during the electrocoagulation process was calculated at the best electrocoagulation conditions using Equation 1 as proposed by Pirkarami & Olya.²⁰

$$E = UIt/V \quad (1)$$

Where, E is the amount of energy consumed during the EC process (kWh/m³), U is the operating voltage (volt), I is the operating current (ampere), t is the EC process time (h) and V is the volume of wastewater (m³).

2.5 Characterization of the electrode surface

The characterization of the electrode surface enables the understanding of the effect of wastewater treatment using the EC process on the surface morphology, composition, and microstructure of the electrode. Electrode surface characterization before and after wastewater treatment was conducted using a scanning electron microscope (SEM) (Philips model XL30S).

3. RESULTS AND DISCUSSION

3.1 Effect of current density

In this study, all measurements were made in triplicate, and the data reported are the means of the respective set of measurements. It was also found that the reproducibility of the data was measured to be within $\pm 5\%$ to indicate a sufficiently good method of dye concentration measurement. However, a quite heavy overlap in the curves could be seen in certain sections especially when the dye removals were high. As such, more emphasis was given to the characterization of the overall trend shown by the curves rather than questioning the accuracy of the data points with the help of the error bar. Furthermore, the authors were of the opinion that error bars would result in quite messy data plots with heavily overlapped curves, especially at high process times when the removals were high.

Current density could determine the production rate of coagulant, bubble production, and affect the growth rate of the flocs.^{18,21} The electrical current introduced into the electrocoagulation system determines the amount of Zn^{2+} ions that can be released from the electrode. According to Faraday's law, increasing electrical current will accelerate the release of metal ions into the solution, as indicated by;

$$\Delta m = MQ / FZ \quad (2)$$

where, Δm is the mass of the substance liberated at an electrode surface (g), Q is the total electrical charge passing through the electrode, F is the Faraday's constant (96,845 C/mole), Z is the number of electrons transferred during the anodic reaction, and M is the molar mass of the electrode substance.²² In the case of zinc electrodes, 1 mole of Zn^{2+} acting as a coagulant will be produced for every 2 moles of electrons released. To demonstrate the effect of current density, four levels of current density i.e., 2, 4, 6, and 8 mA/cm² were studied at the natural pH of synthetic textile wastewater (pH of 6.8), and an initial dye concentration of 50 mg/L for up to 60 min using the zinc electrode.

The dye removal efficiency gradually increased as the current density was increased (Figure 2). Almost complete removals were achieved in just about 10 min when the current density was increased above 4 mA/cm². Even a low current density of 2 mA/cm² managed to remove almost 90 % of the dye within this time frame. These results were significantly better than some reported results for dye removals using iron,³ mild steel,¹¹ and aluminum electrodes.⁵ No significant differences were detected in the dye removals of the runs with different current densities after 20 min as almost complete removals were achieved.

The dye removal was associated with the generation of dissolved Zn^{2+} ions to neutralize charges of suspended and colloidal particles in the solution to form flocs that could readily settle. The zinc ions then formed insoluble zinc hydroxide ($Zn(OH)_2$) that could also settle together with the flocs. Additional removal of dye could be associated with the adsorption of soluble dyes in the solution onto the settling flocs.⁷ With increasing current density, the amount of generated $Zn(OH)_2$ also increased to have a better ability to remove dye molecules through flocs formation and adsorption mechanism.²³ Increasing process time beyond 10 min did not result in any noticeable changes as nearly 100 % removals were recorded. However, to avoid the excessive evolution of hydrogen and to minimize the cost of operation, the current density should be as low as possible.²⁴ Therefore, 4 mA/cm² was decided to be the lowest current density that led to reasonably high dye removal.

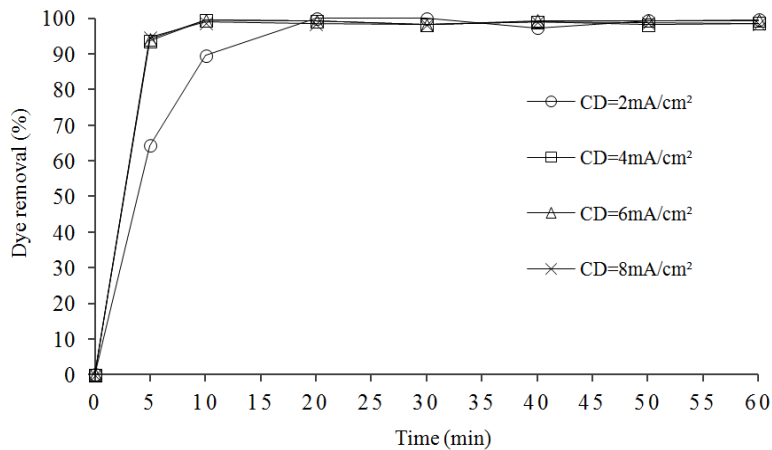
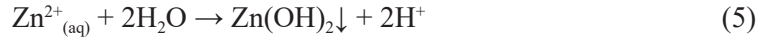


Figure 2: Dye removal of synthetic textile wastewater at various current densities (pH = 6.8 and dye concentration = 50 mg/L).

3.2 Effect of initial pH

The initial pH of the solution is another important parameter that could affect the electrocoagulation process. Different metal ions and their hydroxides have different solubility at different pH levels.⁷ A specific pollutant could achieve the highest removal at a certain optimum value of solution pH and this point should be determined experimentally. The trend of removal efficiency of pollutants is system-specific and it may decrease by either increasing or decreasing the value of solution pH.² The main reactions for the zinc electrode are;



Zinc hydroxide ($\text{Zn}(\text{OH})_2$) flocs could adsorb soluble organic particles before undergoing sedimentation if the pH is favorable.²² The increase in the interfacial pH during the electrocoagulation process could favor the formation of zinc hydroxide.²¹ According to the Zn-H₂O Pourbaix diagram and thermodynamics of zinc material, the precipitation of $\text{Zn}(\text{OH})_2$ would only be significant at pH values higher than 8.6.²³

The removal of dye was found to increase with increasing pH value and 99++ % of dye removals were achieved at initial pH between 6 and 9 (Figure 3). An acidic condition evidently resulted in relatively poor removals, as shown by the curve for pH 5 and it was associated with the adsorption of H^+ ions on the surface of the flocs to cause their restabilization. In addition, the hindered formation of $\text{Zn}(\text{OH})_2$ also led to poor adsorption of dyes in the solution. The dye removal reached its highest value in 10 min and insignificant changes were detected afterward. The Zinc Pourbaix diagram suggests that the formation of zinc hydroxide and the minimum solubility of zinc hydroxide occur in a pH range from about 7 to 9. Most of the zinc complexes for the adsorption of dye were formed within this pH range, and they were responsible for the increases in dye removal as compared to those at lower pH values.²⁰

The electrocoagulation process was observed to be virtually unaffected in the pH range of between 6 and 9. This suggested the stability of the operation, bearing in mind the difficulty of accurately maintaining the pH near neutrality. Slight differences were reported for other electrode materials within this pH range.⁵ At longer process times, no added benefit in terms of dye removal was achieved as most of the flocculated dyes were already separated from the solution in the form of settleable solids, while the adsorption of soluble dyes in the solution could have reached equilibrium. In this respect, pH 9 was therefore chosen to be used for further study.

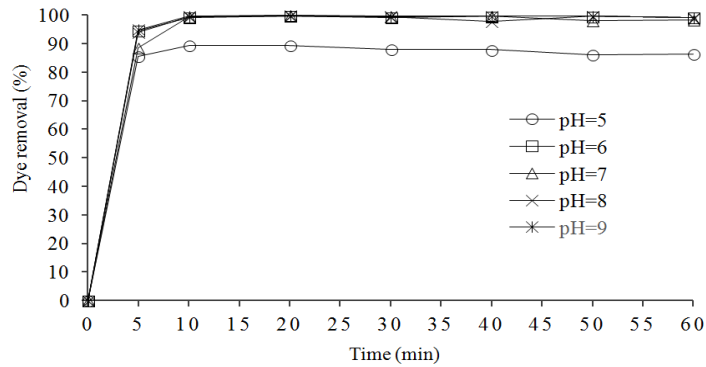


Figure 3: Dye removal of synthetic textile wastewater at different initial pH (current density = 4 mA/cm² and dye concentration = 50 mg/L).

3.3 Effect of initial dye concentration

Figure 4 illustrates the profiles of dye removal efficiency as a function of initial dye concentration. A five-time increase in the concentration from 20 mg/L to 100 mg/L seemed to result in an almost similar degree of treatment. This observation suggested that the electrocoagulation system managed to demonstrate near-complete removal of the dyes in about 10 min and initial concentrations within this range had little influence on the degree of treatment. This observation concluded the role of coagulation as the main mechanism leading to dye removal rather than adsorption of the flocs. In this study, more than 99 % of dye removal was obtained for all studied dye concentrations, indicating the potential of the zinc electrode for the electrocoagulation process.

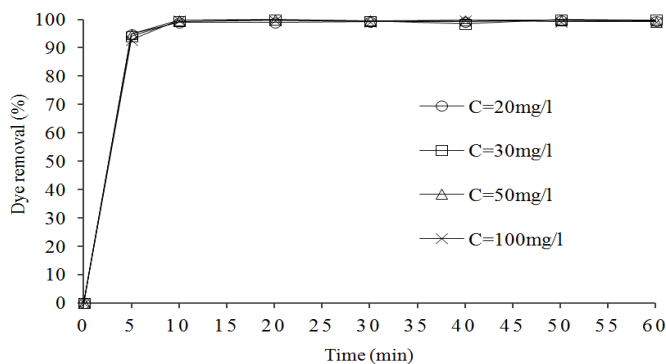


Figure 4: Dye removal of synthetic textile wastewater at various dye concentrations (current density = 4 mA/cm² and initial pH = 9).

It was noted that the dye removal efficiency did not show noticeable changes at varying dye concentrations from 20 to 100 mg/L using zinc electrodes. Thus, it could be concluded that the system was able to cause almost complete dye removal within this concentration range, so that the effect of concentration was virtually insignificant. As a result, the overall trends of the removal were rather similar. This might be due to the fact that the flocs that were produced by the zinc electrodes were able to effectively bring about charge neutralization and sweep-adsorb nearly all the dye molecules from the solution. In the meantime, the dye removal remained at the highest value during the 60 min of treatment, suggesting that flocs restabilization did not occur significantly at prolonged treatment times. It was also interesting to highlight that at an initial dye concentration of 100 mg/L, the produced flocs were sufficient to adsorb dye molecules, leading to high dye removal. However, the objective of wastewater treatment is always to treat the highest concentration of feed with the highest treatment efficiency. Therefore, a concentration of 100 mg/L was selected for further demonstration of the capability of zinc electrodes in this process.

3.4 Dye removal efficiency

The dye removal efficiency was then investigated at an initial pH of 9, a current density of 4 mA/cm², and an initial dye concentration of 100 mg/L (Figure 5). Consistent with earlier results, a high value of dye removal efficiency (99.8 %) was obtained in 10 min for the treatment of this synthetic textile wastewater. By extending the treatment time up to 60 min, almost complete decolorization of the blue solution was noted. For comparison, the electrocoagulation treatment of the real textile wastewater was also attempted, and the results are plotted in the same figure. It can be clearly seen that the removal efficiencies of the real wastewater were about 20 % lower than those of the synthetic wastewater.

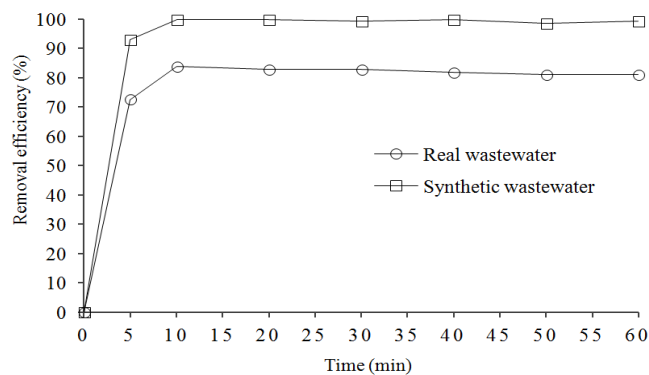


Figure 5: Dye removal for synthetic and real textile wastewater (current density = 4 mA/cm², initial pH = 9 and initial dye concentration = 100 mg/L).

Slight reductions in dye removal of real textile wastewater compared to synthetic textile wastewater were ascribed to the interactions between the components of the wastewater leading to its partial restabilization.³ This was due to the presence of other organic compounds such as sequestering, chemical, and auxiliary agents in real textile wastewater which could affect the performance of the electrocoagulation process. These chemicals could influence the charge of colloidal particles in the solution as well as possibly compete with the adsorption onto the settling flocs. Meanwhile, synthetic textile wastewater might contain only reactive dye compounds, which could be easily removed. It can be concluded that the dye removal of synthetic wastewater was higher than that of real textile wastewater. It should also be borne in mind that the real wastewater contained a COD value of 1,100 mg/L at the beginning of the process. Therefore, the marginal drop in the removal efficiency of the real textile wastewater was still deemed acceptable considering the significantly higher initial load of pollutants it contained. The degree of removal was still higher than some reported results with iron electrodes⁴ and stainless-steel electrodes.^{5,14}

3.5 Energy consumption during the treatment

Besides the costs for the electrodes, the operating cost of the electrocoagulation process is directly influenced by the cost of electrical energy consumed during the treatment process. In this study, the electrical energy required to treat the wastewater was estimated to evaluate its practicality as a means of treatment. The calculation of energy consumption for the treatment of synthetic and real textile wastewater under the best-operating conditions was determined using equation 1 as reported in section 2.4. The current and cell voltage were measured after 10 min of treatment, as concluded by the data in Figures 2-5. The electrical consumption for the treatment of real textile wastewater (0.69 kWh/m³) was found to be slightly higher than that for the treatment of synthetic textile wastewater (0.66 kWh/m³). The difference was attributed to the additional components in the real wastewater and the higher strength of the real textile wastewater. In this study, current and cell voltage used for the calculation of energy consumption were actually measured after 10 min of treatment, as concluded by the data in Figures 2-5. The result was then compared with a result reported by Pirkarami and Olya²⁰ in their study under the same conditions but using Al electrodes. Significantly lower energy consumption was demonstrated by the zinc electrodes. Thus, this type of electrode material could also offer a considerable saving in terms of electrical energy consumption during the electrocoagulation process.

3.6 Changes in the morphology of the anode

The typical SEM images of the electrode surface microstructure before and after the electrocoagulation treatment under the best conditions are shown in Figure 6. The original anode surface was found to be rather smooth and uniform. After the electrocoagulation treatment, the anode electrode showed a relatively rough surface with the appearance of micro-voids void or crevices that originated from the dissolution of zinc ions from the electrode surface. It was clear that the dissolution of the anode did not occur evenly on the surface. Instead, it was found to be more localized in the crevices and pits.

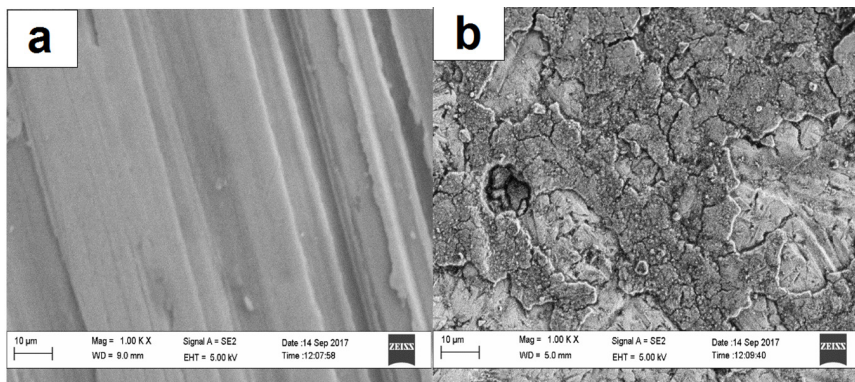


Figure 6: SEM images of zinc anode (a) before and (b) after the EC treatment.

It was theoretically anticipated that more intense dissolution occurred in those spots due to the increased surface area caused by the dissolution of earlier zinc atoms from the surface. Zinc electrodes initially were in a passivated form with the formation of an oxide layer. Initial dissolution created micro crevices that gradually enlarged and spread throughout the surface. Crevices and pits that were initially formed upon zinc dissolution would experience oxygen deficiency as compared to the bulk solution. This oxygen deficiency would limit the formation of a protective oxide layer, leading to the higher susceptibility of those spots to undergo further localized zinc dissolution.⁵ Depending on the pattern of zinc dissolution, deep pits could be created at certain spots.

4. CONCLUSION

Current density, initial pH, and, initial dye concentration were demonstrated to have specific effects on the dye removal efficiency of the synthetic textile wastewater. Those effects were successfully elucidated in this study. The treated

wastewater under the best operating conditions showed high dye removal (99.8 %) and reasonable energy consumption (0.66 kWh/m³) after just 10 min. Meanwhile, the dye removal and energy consumption were found to be at 83.7 % and 0.69 kWh/m³, respectively, for the treatment of real textile wastewater. The real textile wastewater showed a slightly lower reduction, which was attributed to its other constituents that could interfere with the dye removal process. A zinc electrode was demonstrated to be an effective electrode material for the treatment of textile wastewater.

5. ACKNOWLEDGEMENT

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