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Color Removal of Textile Wastewater Using Indirect Electrochemical Oxidation with Multi Carbon Electrodes

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Abstract

The color removal of textile wastewater was studied using the indirect electrochemical oxidation technique with multi carbon electrodes in a parallel construction. The main objective of the study was to investigate the effects of the various operating parameters, such as initial pH, the amount and distance between electrodes, table salt concentration and cell voltage on color removal of artificial textile wastewater. The experimental result showed that 96.05% of color removal was observed for artificial textile wastewater of 150 mg/L in electrochemical cell containing 8 bars of carbon electrode with the inter-electrode distance of 10 cm, 3.5 g/L table salt, initial pH 6 and cell voltage of 12 V within 60 min of operation. The result showed the use of multi electrode in the indirect electrochemical cell is an effective solution to increase the performance of electro-degradation process. This study proves that this method is a promising technology for remediation of organic pollutant from industrial wastewater.

Keywords: Indirect electrochemical oxidation; Multi carbon electrodes; Color removal efficiency; Artificial textile wastewater

1. Introduction

Textile wastewater contains high organic and inorganic contaminants with extreme color intensity resulting from the use a large amount of chemicals including dyes in the textile dyeing process. It was predicted that more than 10^4 ton per year of the synthetic dyes were consumed for textile industry (Tan *et al.,* 2010) and it was estimated about 10-50% lost to environment as wastewater (Ben Mansour *et al.,* 2012). Discharge of textile wastewater into the environment without treatment reduces the environmental aesthetics and triggers the disruption of human health. The release of colored effluents into the surface water bodies could directly effect on aquatic ecological system due to dissolved oxygen depletion in the water resulting in an increased activity of anaerobic microorganisms that produce odorous compounds.

A number of methods have been developed to remove contaminants from textile wastewater such as chemical method by using active chlorine (Massoudinejad *et al.,* 2015), coagulation (Sabur *et al.,* 2012; Solanki *et al.,* 2013), electrochemical oxidation (Kavitha, 2012; Najafpoor *et al.,* 2017), by biological means using bacteria consortium attached to volcanic stone (Sastrawidana *et al.,* 2008), marine *Cyanobacteria lyngbya* sp. (Bella and Malliga, 2015), and fungal isolate includes *Aspergillus terreus* (Neeratanaphan *et al.,* 2015), *Aspergillus ninger*, *Trichoderma virens, Talaromyces stipitatus, and Phlebiopsis raveneli,* (Sweety *et al.,* 2017), Most of the available methods are not fully applicable to industry, especially in small scale industries because they require high operational costs and need a qualified staff for their operations.

In recent years, the electrochemical oxidation technology has got a great attention due to the fact that the process is quite simple, low cost but resulting in high degradation efficiency. The electrochemical oxidation process of pollutants can be performed through two different mechanisms: (1) direct electro-oxidation, in which the pollutants are oxidized on the anode surface without adding other substances in bulk solution, and (2) indirect electro-oxidation, where mediators such as NaCl, H_2SO_4, and $H_2S_2O_8$ are used electro chemically generating oxidants at the anode surface and subsequently the oxidants convert organic or inorganic pollutants in the bulk solution into less harmful products.

Electrochemical oxidation at laboratory scale commonly uses a small reactor with two electrodes in which one electrode functioning as cathode and the other as anode. The laboratory wastewater degradation study result showed that this method proved to be effective for degradation of textile wastewater, cyanide and ammonia (Akarsu *et al.,* 2017 ; Bhatnagar *et al.,* 2014; Felix-Navarro *et al.,* 2011; Hu *et al.,* 2009). Electrochemical cell reactors with two electrodes are predicted to be ineffective when applied in industrial scale with relatively large sized wastewater treatment reactors due to the decrease in electro-catalytic activity to generate the oxidants. This is caused by the minimum area contact from electrolyte to the electrodes in bulk solution.

Therefore, in this research, the indirect electrochemical cell reactor was modified by using multi carbon electrodes and then it was investigated in terms its ability to decolorize artificial textile wastewater. The performance of electrochemical cell reactor was evaluated

based on several parameters, i.e., the amount and inter-electrode distance, initial pH, electrolyte concentration, and cell voltage. From the optimum conditions obtained from the removal of artificial textile wastewater was then used to degrade the real textile wastewater.

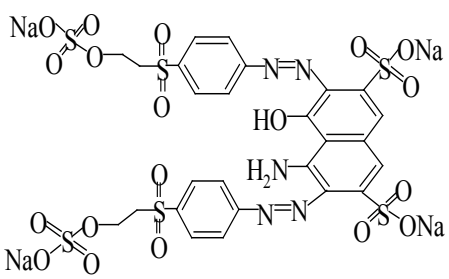
2. Materials and Methods

2.1 Dyes

The dyes which were used to make the artificial textile wastewater were azo dyes reactive group comprising remazol black B, remazol red RB, remazol yellow G and remazol blue. The IUPAC name and chemical structure of these dyes are illustrated as follows;

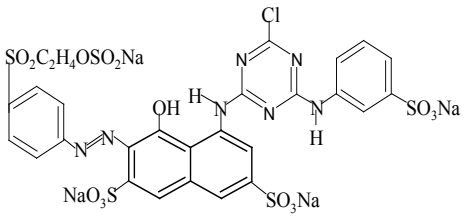
Remazol black B/Reactive black 5

IUPAC name: tetrasodium 4-amino-5-hydroxy-3,6-bis[[4-[[2-(sulphonatooxy)ethyl] sulphonyl]phenyl]azo]naphthalene-2,7-disulphonate. Molecular formula $C_{26}H_{21}N_5Na_4O_{19}S_6$.



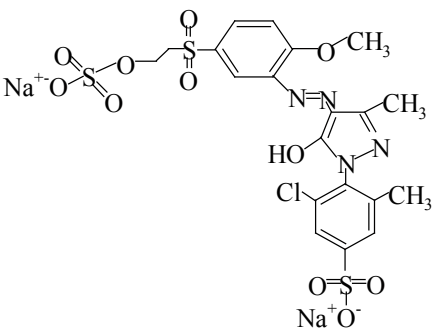
Remazol red RB/Reactive red 198

IUPAC name: tetrasodium;(3Z)-5-[[4-chloro-6-(3-sulfonatoanilino)-1,3,5-triazin-2-yl]amino]-4-oxo-3-[[4-(2-sulfonatooxyethyl-sulfonyl)phenyl]hydrazinylidene]naphthalene-2,7-disulfonate. Molecular formula: $C_{27}H_{18}ClN_7Na_4O_{16}S_5$.



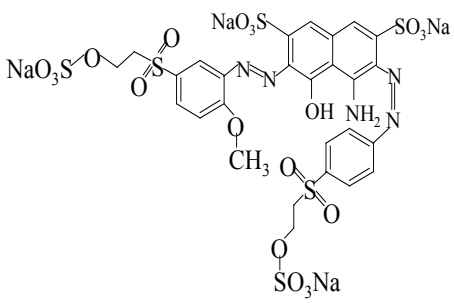
Remazol yellow G /Reactive yellow 14

IUPAC name: disodium;3-chloro-4-[4-[[2-methoxy-5-(2-sulfonatooxyethylsulfonyl) phenyl]diazenyl]-3-methyl-5-oxo-4H-pyrazol-1-yl]-5-methylbenzenesulfonate. Molecular formula: $C_{20}H_{19}ClN_4Na_2O_{11}S_3$



Remazol blue/Reactive blue 250

IUPAC name: tetrasodium;5-amino-3-[[2-methoxy-5-(2-sulfooxyethylsulfonyl)phenyl] diazenyl]-4-oxido-6-[[4-(2-sulfonatooxyethyl -sulfonyl)phenyl]diazenyl]naphthalene-2,7-disulfonate. Molecular formula: $C_{27}H_{23}N_5Na_4O_{20}S_6$



2.2 Artificial textile wastewater preparation

Artificial textile wastewater with concentration of 150 mg/L was prepared by weighing 37.5 g of each dye and dissolving it in distilled water and stirring it for 10 min at room temperature. The dyes solution was put into the volumetric flask of 1000 mL and diluted to 1000 mL with distilled water.

2.3 Real textile wastewater collection

The real textile wastewater sample was collected from a textile factory in Denpasar, Bali.

2.4 Experimental setup

Batch experiments were carried out at room temperature (28 ± 2°C) using electrochemical cell which is made from acrylic container with internal dimensions of length x width x height (50 x 35 x 25 cm). Carbon from battery with diameters of 1 cm and length of 10 cm were used as an electrode and a DC power supply was used as an electric source. The experimental setup is shown in Figure 1.

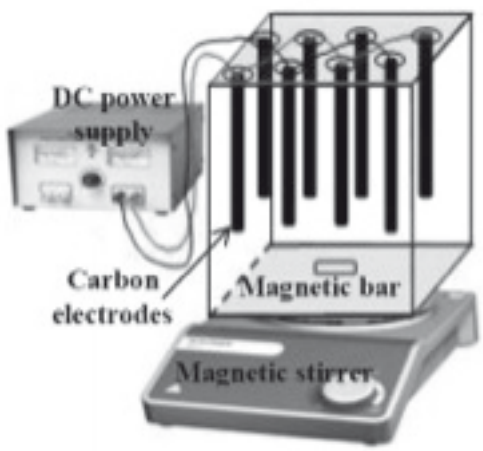


Figure 1. Schema of electrochemical oxidation for artificial textile wastewater.

2.5 *Artificial textile wastewater treatment on indirect electrochemical oxidation reactor*

A volume of 2000 mL of artificial textile wastewater with concentration of 150 mg/L was placed in electrochemical cell and was stirred using automatic magnetic stirrer constantly at a 150 rpm to keep the homogeneity of electrolyte distribution. The solution of NaOH or HCl was used to adjust the pH of wastewater. The influences of the operating treatment parameters include the effect of the inter-electrode distances; initial pH, table salt concentration, and cell voltage on color removal were investigated. All treatments were replicated three times

2.5 *Determination of color removal*

After treatment, all samples were filtered through Whatman No. 1 paper filter. The color removal of artificial textile wastewater was analyzed by measuring their absorbance at before and after treatment using a double beam Shimadzu-1700 UV-Visible spectrophotometer at 480 nm. The percentage of color removal was calculated by following formula:

$$\text{Color removal}(\%) = \frac{Ao_o - A_1 1}{Ao_o} \times 100\%$$

Where Ao and A1 are the absorbance values of artificial textile wastewater at before and after treatment, respectively.

2.6 *Real textile wastewater treatment*

The degradation of the real textile wastewater was carried out in electrochemical oxidation reactor for 60 min at the optimum performance conditions. The parameters degradation of textile wastewater which was analyzed included COD, BOD_5, phenol and ammonia. All parameters were analyzed using Indonesian National Standard for water and wastewater analysis. The analysis result is shown in Table 1.

2.7 *Statistical Analysis*

The experimental data were analyzed using IBM SPSS statistics version 20. One-Way ANOVA was used to analyze the effect of treatment. The difference was considered significant if $p < 0.05$.

Table 1. Chemical characteristics of real textile wastewater.

Parameters	Value	Analysis method
BOD_5 (mg/L)	38.45 ± 3.6	Iodometric titration
COD (mg/L)	65.25 ± 5.4	Close reflux titrimetric
Phenol (mg/L)	nd	Amino antipyrine
Ammonia (NH_3-N) mg/L)	0.45 ± 0.09	Phenate method

3. Result and Discussion

3.1 Effect of initial pH

To observe the effect of initial pH on color removal efficiency, the experiments were adjusted at different pH from 3 to 10 in the indirect electrochemical cell for 60 min with two carbon electrodes by inter-electrode distance of 5 cm, 4 g/L of table salt and cell voltage of 12 volt. The extents of color removal at different initial pH are illustrated in Figure 2.

The results showed that the effect the initial pH on the color removal efficiency of artificial textile wastewater increased from 3 to 6 and then slightly decreased from 6 to 10. Statistical analysis with One-way ANOVA at confidence level of 95% indicated that color removal efficiency has been significantly influenced by pH treatment. The color removal was a significant increase at pH treatment from 3 to 6 while it was not significant from pH 6 to 9. Figure. 2 showed that the best pH for artificial textile wastewater treatment was pH 6 with color removal efficiency of 83.31%. The reason may be due to presence of active chlorine species as oxidants which were pH dependent. In indirect electrochemical oxidation, sodium chloride from table salt was oxidized on anode surface to generate active chlorines by the following reactions.

$$2\ Cl^- \longrightarrow Cl_2\ (aq) + 2\ e^-$$

The chlorine gas dissolves in water which subsequently undergoes hydrolysis resulting in hypochlorous acid. Hypochlorous acid is a weak acid, it tend to undergo partial dissociation to form hypochlorite ion.

$$Cl_2 + H_2O \longrightarrow HClO + Cl^- + H^+$$

$$HClO \longrightarrow OCl^- + H^+$$

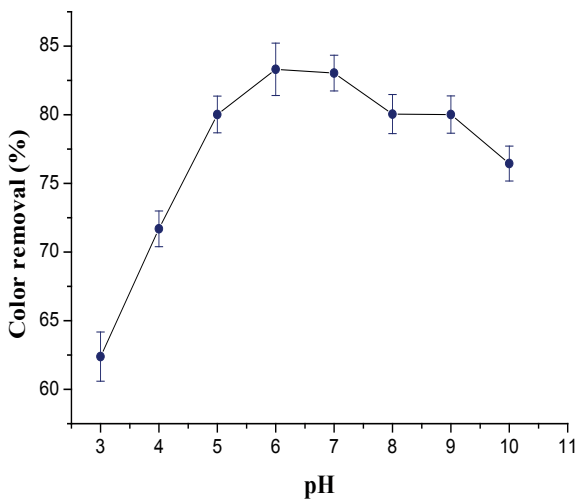


Figure 2. Percentage color removal of artificial textile wastewater at different pH.

Based on the above reaction, the relative amount of each active chlorine forms was dependent on the pH. In general, at room temperature, Cl_2 was dominant species when the electrolysis carried out under very acidic conditions, whereas the HClO species predominates at 3.3 < pH<7.5, while OCl^- was present at pH>7.5 (Azeroual et al., 2017). Chlorine, hypochlorous acid and hypochlorite ion were strong oxidants whose reduction potential (E^o_{red}) value was 1.36 V; 1.49 V and 0.89 V vs. SHE, respectively. This means, oxidation of organic pollutants by indirect electrochemical method using sodium chloride as a supporting electrolyte was effective at slightly acidic to neutral conditions. However, in our study result, that color removal efficiency of artificial textile wastewater at pH treatments from 5 to 9 are not significant different. Perhaps during the degradation process at the base pH a decrease occurs which is caused by the formation of Hydrogen ion. This is proven by the fact that at the end of the process we observed a decrease pH. The treatment at initial pH of 3, 4, 5, 6, 7, 8, 9 and 10, became 2.90; 3.95; 5.25; 5.28; 6.15; 6.25 and 7.15, respectively. This finding is consistent with Murthy *et al.* (2011), who reported that the pH of textile wastewater decreased from 9.4 to 8.564. The effect of initial pH on color and COD removal of textile effluent also was examined by Rathinakumaran and Meyyappan, (2015) who reported that the best pH conditions for the maximum COD and color removal was initial pH 6.25 with addition of 4 g/L NaCl.

3.2 *Effect of amount and inter-electrodes distance*

Indirect electrochemical oxidation process was carried out by varying amount of carbon electrode from 2 to 8 with inter-electrode distance differences of 5, 10 and 15 cm. The other parameter conditions were set at pH 6, table salt of 4 g/L and cell voltage of 12 volt. The experiment result at different amounts and inter-electrode distances are shown in Figure 3.

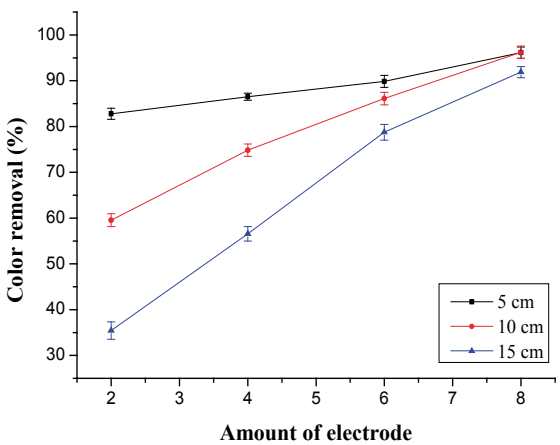


Figure 3. Percentage color removal of artificial textile wastewater at different amount and inter-electrode distance.

The number of electrodes and inter-electrode distances showed a significant influence on dye removal by indirect electrochemical cell. At a fixed size of indirect electrochemical reactor and a constant inter-electrode distance, an increased number of electrodes produces a higher color removal efficiency because the electrolysis process of chloride ion on anode surface more effective to generate the active chlorines, mainly hypochlorous acid and hypochlorite ion which act to oxidize the dyes. Color removal efficiency of artificial textile wastewater by indirect electrochemical oxidation for 60 min using 2; 4; 6 and 8 electrodes were 35.71-83.32%; 56.48-87.47%; 78.25-90.25% and 92.51-96.12%, respectively. In this study, it was also observed that the decrease in color removal was in line with increase the inter-electrode distance. The reason of this observation may be caused by the decreasing current density when inter-electrode distance increases. Upon decreasing current density, the amounts of active chlorines for oxidation of organic pollutants also decreased. This finding agrees with Ghalwa et al., 2016, who reported that the removal efficiency of dyes and COD increased with the increasing the current densities in electro-degradation process. Sastrawidana et al., (2018) in a previous study reported that the degradation of organic pollutants from restaurant wastewater using indirect electrochemical oxidation with batch technique was more effective by using four pairs of carbon electrodes compared to two pairs.

3.3 Effect of table salt concentration

The effect of table salt concentration on color removal using indirect electrochemical oxidation process was investigated over range concentration up to 5 g/L wastewater. Batch electro-degradation experiment was conducted for 60 min using 8 bars of carbon electrodes (4 anodes and 4 cathodes) by inter-electrode distance of 10 cm, initial pH 6 and applied voltage of 12 V. The chloride ion content on table salt was obtained 50.63 % (w/w) through Morh method of Argentometric titration. The effect of table salt concentration on color removal of artificial textile wastewater is shown in Figure 4.

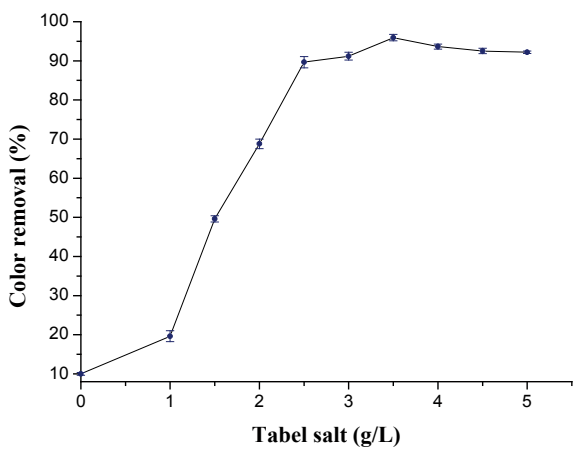


Figure 4. Percentage color removal of artificial textile wastewater at difference of table salt concentration.

The result showed that the increase in table salt dosage (from 1 to 3.5 g/L) leads to corresponding increase in the dye degradation rate. It can be seen, that electro-degradation of dye in the absence of table salt (chloride ion) was very slow. When chloride ion was present in wastewater, electro-degradation process improved significantly and the color removal efficiency increased with the increasing of table salt concentration. Basically, the important roles of chloride ions in the electro-oxidation process of contaminants are seen in two aspects; to increase the oxidant concentration (hypochlorous acid and hypochlorite ions) and to increase cell conductivity (Maljaei et al., 2009). The hypochlorous acid and hypochloriteion formed during electrolysis are strong oxidants and responsible to dye degradation. The degradation of dye effluent via active chlorine such as hypochlorite ion generated from electrolysis of chloride ion through the following reaction (Mohan et al., 2007)

At anode:

$2\ Cl^- \longrightarrow Cl_2 + 2e^-$

At cathode:

$2\ H_2O + 2\ e^- \longrightarrow H_2 + 2OH^-$

Net reaction in bulk solution:

$Dye + OCl^- \longrightarrow CO_2 + H2O + Cl^- + P$

Since dye molecules are electrochemically inactive, the reaction occurring at the anode is the oxidation of chloride ions with the liberation of Cl_2, which is a strong oxidizing agent. Hydrolysis of chlorine gas forming the hypochlorous acid, then it tends to dissociate partially to form hypochlorite ion. The slightly alkaline or neutral pH conditions are more favorable for reactions involving Cl_2 to form HOCl and OCl^-. Aromatic compounds including dyes are broken down electrochemically into intermediate products for further processing. In electrochemical oxidation, these organic compounds are completely oxidized to CO_2 and H_2O. Besides, the presence of table salt in wastewater causes a rise of current density at the same voltage which provides more chance for the production of active chlorine (Jara et al., 2007). The maximum color removal was achieved on table salt concentration of 3.5 g/L. The addition of table salt higher than 3.5 g/L does not have a significant effect on color removal increases.

3.4 *Effect of cell voltage*

The experiments were carried out at six different of cell voltages keeping the other parameters constant. The effects of different cell voltage on color removal efficiency are illustrated in Figure 5.

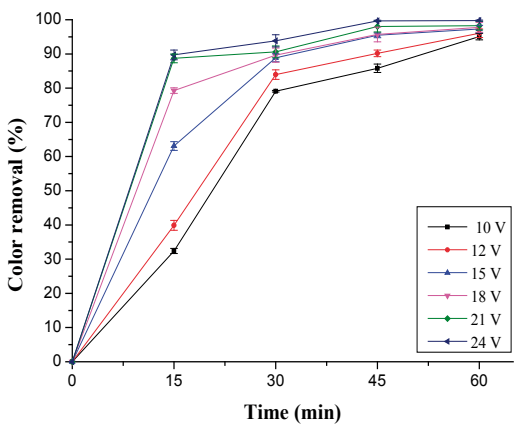


Figure 5. Percentage color removal as function of electrolysis time at difference cell voltage.

The color removal efficiency of artificial textile wastewater at varying cell voltage of 10-24 within 60 min of electro-degradation time was slightly different. Within 15 min of electrolysis time, degradation degree of dye for cell voltage of 10, 12, 15, 18, 21 and 24 volt were 32.39; 39.88; 63.09; 79.29; 88.72 and 89.72%, while after 60 min, color removal efficiency of 95.06-99.75% was reached in all varying cell voltage. The electrolysis of electrolyte on anode surface was effectively conducted at a high cell voltage because of the increased rate of generation of oxidants. However, the electro-oxidation process at cell voltage higher than 12 V with inter-electrode distance of 10 cm causes the abrasion of the anode part of carbon electrodes. Therefore, the optimum of cell voltage selected for color removal by electrochemical oxidation was 12 Volt.

3.5 Treatment result of real textile wastewater

Decolorization of textile wastewater was performed under optimum condition in terms of amount carbon electrode (8 bars), the distance between electrodes (10 cm), initial pH (6) and chloride ion from table salt (3.5 g/L). The characteristics of real textile wastewater after treatment are listed in Table 2.

The analysis result of the real textile wastewater before treatment is listed in Table 1. It can be noticed, that the high content values of BOD5 and COD in real textile wastewater textile are related to a large quantity of chemicals used in dyeing process. The pollutant content of textile wastewater can vary, but the main characteristics are high organic content, presence of dyes, and other inorganic compounds like sodium hydroxide, detergents, sodium hypochlorite and solvents. Major contaminants load from textile industries come from the steps processing operation like scouring, bleaching, mercerizing and dyeing (Radha *et al.,* 2009) As a result various processes, considerable amount of pollutant are released into environment.

It can be seen from Table 2 that the quality of textile wastewater after treatment in indirect electrochemical cell with multi carbon electrodes is acceptable category according to Indonesian wastewater quality standard for textile wastewater. Therefore, this technology has proven effective and efficient to reduce BOD_5, COD, phenol and ammonia contained in the real textile wastewater. Removal efficiency of BOD_5, COD, and ammonia within 60 min electrolysis time in this study was achieved up to 92.07, 94.88 and 82.00%, respectively. The use 3.5 g/L of table salt equal to 0.1 mol/L of chloride ion was selected for wastewater treatment using electrochemical oxidation method. Excessive use of salt will also have a negative impact on the environment.

Table 2. Chemical characteristic of real textile wastewater after treatment

Parameters	Value	Standar value
BOD5 (mg/L)	38.45 ± 3.6	≤ 60
COD (mg/L)	65.25 ± 5.4	≤ 150
Phenol (mg/L)	Nd	≤ 0.5
Ammonia (NH_3-N) mg/L)	0.45 ± 0.09	≤ 8.0

Remark: Indonesian wastewater quality standard, nd: not detected.

4. Conclusion

The electrochemical oxidation method has a great potential to be developed as an alternative technology for textile wastewater treatment. Color removal efficiency is dependent on the amount and inter-electrode distance, supporting electrolyte concentration, initial pH and cell voltage. The experiments were carried out in a batch electrochemical oxidation reactor within 60 min electrolysis time. The result revealed that the color removal of artificial textile wastewater was obtained 96.05% at optimum parameters condition of 8 rods carbon electrodes with inter-electrode distance of 10 cm, initial pH 6.0, 3.5 g/L table salt, and cell voltage of 12 Volt.

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