Plasma generation using dielectric barrier discharge reactor for phenol degradation in batik wastewater

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Abstract. Batik wastewater contains phenolic compounds. Phenolic compounds are hematotoxic, hepatotoxic, and capable of causing mutagenesis and carcinogenesis in humans and other living organisms. Therefore, phenol compounds need to be degraded. This study uses plasma electrolysis method with Dielectric Barrier Discharge (DBD) reactor to degrade phenolic compounds in Batik wastewater. The purpose of this study was to characterize the Dielectric Barrier Discharge (DBD) reactor, to determine the effect of voltage and type of catalyst on phenol concentration, and to determine the interaction between voltage and catalyst type on the response of phenol concentration through analysis of variance (ANOVA). The result obtained from the characterization of the reactor is ignition voltage at 1400 Volt. The best degradation results of phenolic compounds were obtained in the treatment of Batik wastewater with FeSO$_4$ catalyst at 2600 Volt. The phenol reduction in the best conditions reached 88.73%. Based on analysis of variance (ANOVA), voltage and quadratic catalyst variables affect the response of phenol concentrations in batik waste.

1. Introduction

Batik is a piece of cloth applied by means of a dye-resist technique using "batik-wax" as the resisting medium [1]. Generally, the process for colouring batik has 12 steps: nyungging (using a pencil to sketch and draw the pattern on a paper), njaplak (moving the drawn pattern from paper to fabric), nglowong (waxing pattern on fabric using canting filled with hot wax), ngiseni (drawing to the main pattern), nyolet (colouring some parts of the pattern using a paintbrush), mopok (covering parts that have been coloured with wax using a paintbrush), ngelir (colouring the whole fabric by putting the fabric into coloured water), nglorod (removing wax by dipping fabric into the boiling water), nglorod (removing wax by dipping fabric into boiling water) [2]. Batik colouring process consumes large amounts of discharged effluents during dyeing and finishing operations [3]. Batik is considered as one of many UNESCO’s intangible cultural heritage of Indonesia that continues to develop. Batik industry has developed for hundreds of years [4]. The cultural heritage has its own economic value dan market [5]. The export market reaches 125 million US dollars per year. Around two million people depend on batik business, starting from small to medium merchants [6]. The enthusiasm of Indonesians for batik is getting higher along with the times.

However, batik industry are not free from problems. Some problems that might occur are raw materials availability, labor skills, market competition, and wastewater treatment. 81% of batik...
industries in Indonesia do not treat their wastewater and only 19% do wastewater treatment [7]. The wastewater mainly comes from the coloring process which leads to various negative environmental problems if not being processed [8]. This occurs because the wastewater contains organic compounds, suspended solids, dyes, chromium, sulfide, ammonia, fats, oils, and phenolic compounds [9]. Synthetic dyes and phenolic compounds in batik wastewater need to be treated [10].

Phenol is a toxic compound either for plants or animals that live in water ecosystems [11]. Phenol can reduce dissolved oxygen concentration in water. Aquatic organisms are affected due to the reduction of the dissolved oxygen concentration [12]. Phenolic compounds are toxic even at low concentrations [13]. Phenolic compounds are toxic to blood cells (hematotoxic), injury or damage to the liver (hepatotoxic), and capable of causing mutagenesis and carcinogenesis in humans and other living organisms [14]. The carcinogenic property in phenolic compounds leads to cancer [15]. Therefore, phenolic compounds need to be degraded.

Several methods have been carried out as an effort to degrade phenolic compounds, such as photolysis, coagulation, and electro-fenton. The previous methods have been proven as ineffective because phenolic compounds are difficult to degrade, have a long degradation time, toxic, and produce toxic byproducts [16]. The photolysis method is limited by the clarity of the water [17]. While, electro-coagulation method increases the temperature of wastewater as a result of fluctuating voltage to current [18]. Meanwhile, the electro-fenton method is expensive because it requires large energy to produce hydrogen peroxide and to light up the ultraviolet lamp. Fenton reagent used in electro-fenton requires the handling of highly reactive materials [17]. Therefore, an effective degradation method is needed to reduce the levels of phenolic compounds in the batik industry wastewater.

An effective degradation method offered is plasma electrolysis. Plasma electrolysis can be done by using various types of reactor, such as the pulsed discharge plasma reactor, gliding arc discharge reactor, and dielectric barrier discharge (DBD). The dye degradation efficiency of the three reactors are 69.8%, 89.69%, and 90% respectively. The DBD reactor has the largest efficiency compared to the other reactors. DBD reactors have various advantages, such as stable and uniform plasma can be achieved under atmospheric pressure with wide frequency range. DBD reactors can be applied to most reaction conditions. The dielectric barrier inside the reactor can prevent the formation of spark or explosion so that the reactor is safer than other types of reactor. While the large plasma discharge area is useful for establishing full reaction contact. This characteristic is not available in other non-equilibrium plasma reactors [19]. For this reason, plasma electrolysis method using DBD reactor is used to produce the highest possible phenolic compounds reduction.

2. Research Method

2.1. Materials
Materials needed in this study are filtered batik waste derived from the Nalendra Batik in Jepara Regency, 4-Aminoantipyrine (C₁₁H₁₃N₃O), sodium hydroxide (NaOH), ammonium hydroxide (NH₄OH), buffer phosphat, potassium ferricyanide (C₆N₆FeK₃), sulfuric acid (H₂SO₄), indicator [Fe(phen)₃]²⁺, cerium(IV) sulfate (Ce(SO₄)₂), iron(II) sulfate (FeSO₄), iron(III) oxide (Fe₂O₃) and distilled water.

2.2. Instrumentation
Instrumentations needed in this study are dielectric barrier discharge (DBD) reactor, oscilloscope, HV probe, calpmeter, AC power supply, UV-Vis spectrophotometer, and a titration set.

2.2.1. Setting up the reactor. The main components of the DBD reactor are a glass tube made of pyrex with a thickness of 0.25 cm, diameter 2 cm, and height of 30 cm which acts as a barrier between two electrodes. There are two electrodes: the inner electrode which is stainless steel with 2 cm diameter and 30 cm height and also the outer electrode which is consists of 50 coils of copper electrode [20]. Reactor is connected to the AC power supply with voltage variation of 0-2.6 kV.
2.2.2. **Reactor performance test.** The performance test needs to be carried out to ensure that there are no leaks in the reactor and to observe the occurrence of plasma phenomenon.

2.2.3. **Current and voltage testing.** The effect of voltage on the current produced by using the plasma electrolysis method is needed to determine the range of plasma formation (breakdown voltage). This breakdown voltage will be used on voltage variations. In addition, the test will examine the effect of the catalyst on the formation of the gas sheath which results in the I-V graphics obtained. The voltage used is between 0-2.6 kV while the current is the parameter to be measured.

2.2.4. **Hydrogen peroxide concentration and yield measurement.** Reactive species that play a role in the phenol degradation of batik wastewater are hydroxyl (OH) radicals. Hydroxyl radical is a very reactive radical and has a very short lifetime. Hydroxyl radical is easy to recombine to form other compounds, including hydrogen peroxide (H$_2$O$_2$). The measure of H$_2$O$_2$ is based on the previous study [21], a solution that contains H$_2$O$_2$ is titrated with standard Ce(SO$_4$)$_2$ as titrant and [Fe(phen)$_3$]$^{2+}$ as the acid indicator. Reaction occurred in titration is as follows.

\[ 2\text{Ce}^{4+} + \text{H}_2\text{O}_2 = 2\text{Ce}^{3+} + \text{O}_2 \uparrow + 2\text{H}^+ \]  

(1)

2.2.5. **Phenol concentration measurement.** Measurement is done according to SNI 06-6989.21-2004 on phenol concentration test by spectrophotometry method. All phenols in water will react with 4-aminoantipyrine at pH 7.9 ± 0.1 in potassium ferric cyanide solution condition forming a brownish-red color solution. The color formed is measured for absorbance at a wavelength of 500 nm.

2.2.6. **ANOVA analysis.** This study uses the statistic method using Design Expert Version 12. The method used produces an equation model using the Analysis of Variance (ANOVA) to determine the effect of each variable and the interaction between variables. The result “p” from Analysis of Variance (ANOVA) is used to determine the significance of the variable on the response. The equation model is considered significant if the p-value is less than 0.05.

3. **Result and Discussion**

3.1. **Dielectric Barrier Discharge Reactor Characterization**

Dielectric Barrier Discharge (DBD) reactor can release plasma discharge between two electrodes separated by a dielectric barrier [22]. Plasma discharge is generated using alternating current (AC) and electrodes [23]. DBD reactor is operated at 0-2.6 kV in the atmospheric environment. DBD reactor characterization can be done by measuring the electric current, voltage, and observing the formation of plasma [24]. The relationship between electric current and voltage can be seen in Figure 1.
Figure 1. The relationship between electric voltage and current in DBD reactor

Reactor characterization using FeSO₄ catalyst, Fe₂O₃ catalyst, and without catalyst. Figure 1 shows that the three variables tested have a similar phenomenon. The current will increase as the voltage increases. It happens because the electric voltage affects the number of electron charges and the electric field generated by the DBD reactor [25]. The increased electric field can be attributed to the large number of electrons accumulating at the inner electrode. The accumulation of electrons causes electrons to have enough energy for the ionization process.

Ionization due to the accumulation of electrons causes the formation of plasma between the two electrodes [26]. The color of the plasma formed is influenced by the amount of voltage used and the amount of air being ionized [27]. Plasma electrolysis begins with an increase in the slope of the voltage-current graph [28]. As seen in Figure 1, an increase in slope occurs at 1400 Volt. This point is referred as the ignition voltage. The ignition voltage is a sign of plasma formation [29]. Figure 2 shows the plasma formed during the operation of the DBD reactor.

Figure 2. Plasma formed in DBD reactor

3.2. Hydrogen Peroxide Concentration Measurement

Figure 3 and Figure 4 show the molarity and yield of hydrogen peroxide produced in the plasma electrolysis process.
As shown in Fig. 3, the electric voltage increased with increasing of hydrogen peroxide molarity and yield that be formed. The increased voltage causes increasing electron temperature [30]. Increasing electron temperature effects on increasing of kinetic energy of ions and radical intensity of hydroxyl directly [31]. The increased ion kinetic energy causes ions could move to the anode side easily and could be excitation [32].

Hydroxyl (OH) radicals can be formed by the water molecule degradation process [33]. The water molecule was dissociation, ionization, and excitation by vibration or rotation [34]. The excitation process affected on water molecules that changed into molecules with low energy and produced radical compounds (·OH, ·H, and ·O) [35]. De-excitation of radical compounds from high energy to low energy that more stable condition causes the formation of UV radiation [36]. Hydroxyl (OH) radical is the main radical that has an important role in degradation of phenolic compounds [37]. Two inside OH radicals combined to form a hydrogen peroxide compound that more stable as seen in equation 2 [38].

\[
·\text{OH} + ·\text{OH} \rightarrow \text{H}_2\text{O}_2
\]  \hspace{1cm} (2)
Therefore, with the increasing of electric voltage, the concentration and yield of hydrogen peroxide could increase because more hydrogen peroxide could be produced and accumulated from water degradation process.

3.3. Effect of Voltage on Phenol Concentration
Figure 5 shows the effect of voltage and type of catalyst on phenol reduction. Figure 6 shows the effect of voltage on phenol concentration.

![Figure 5](image1.png)

**Figure 5.** The effect of voltage and type of catalyst on phenol reduction
Figure 6. The effect of voltage on phenol concentration.

Based on Figure 6, it showed a correlation that the higher voltage can produce the higher phenol reduction in Batik wastewater. This is due to higher voltage can produce plasma maximally. Plasma formation could increase the amount of ionized gas that causes the formation of radical compounds [33]. The decreased phenol concentration is caused by an increased amount of hydroxyl radical. This phenomenon shows that phenolic degradation was caused by the hydroxyl radical compound [39].

Hydroxyl radical has a role in the degradation of phenolic compounds in batik wastewater through some steps. Phenolic compounds were oxidized by two hydroxyl radicals became dehydroxylase ring such as pyrocatechol which then be degraded by two other hydroxyl radicals and produce benzoquinone [40]. Hydroxyl radicals have a role to open aromatic ring benzoquinone to form organic compounds such as C₆H₄O₆ or C₆H₄O₄ as seen in the following equation [41]:

\[
2 \cdot \text{OH} + \text{C}_6\text{H}_5\text{OH} \rightarrow \text{C}_6\text{H}_5(\text{OH})_2 + \text{H}_2\text{O} \\
2 \cdot \text{OH} + \text{C}_6\text{H}_4(\text{OH})_2 \rightarrow \text{C}_6\text{H}_4\text{O}_2 + 2 \text{H}_2\text{O} \\
8 \cdot \text{OH} + \text{C}_6\text{H}_4\text{O}_2 \rightarrow \text{C}_6\text{H}_4\text{O}_6 + 4 \text{H}_2\text{O}
\]

Organic compounds will undergo further oxidation to form carbon dioxide and water. It indicated by the color of the solution became clearer. Therefore, the increased voltage and hydroxyl radical compounds can produce higher phenol reduction in batik wastewater.
3.4. Effect of Catalyst Type on Phenol Concentration

The results of this study indicated that the type of catalyst affects the degradation of phenol compounds in batik wastewater. Figure 7 shows the effect of catalyst type on phenol concentration.

![Figure 7](image_url)
Based on Figure 7, it showed a correlation that the use of iron catalysts can increase the percentage reduction in phenol concentration. Batik wastewater treated with iron catalyst had a higher percentage reduction in phenol concentration than the one that was not treated with iron catalyst. Iron catalyst role to transform hydrogen peroxide compound back into hydroxyl radical (·OH and ·HO2) through fenton reaction [42,43]. Hydroxyl radicals have an important role to degrade phenol compounds [44]. The following equations show the reaction of phenol degradation by hydroxyl radicals [45]:

\[
\begin{align*}
\text{Fe}^{3+} + \text{H}_2\text{O}_2 & \rightarrow \text{Fe}^{3+} + \cdot \text{OH} + \text{OH}^- \\
\cdot \text{OH} + \text{H}_2\text{O}_2 & \rightarrow \text{HO}_2^- + \text{H}_2\text{O} \\
\text{Fe}^{2+} + \cdot \text{OH} & \rightarrow \text{Fe}^{3+} + \text{OH}^- \\
\text{Fe}^{3+} + \text{H}_2\text{O}_2 & \rightarrow \text{Fe}^{2+} + \text{HO}_2^- + \text{H}^+ \\
\cdot \text{OH} + \cdot \text{OH} & \rightarrow \text{H}_2\text{O}_2
\end{align*}
\]

Organic compound + ·OH → degraded compound

This research uses 2 types of catalysts (FeSO4 and Fe2O3). The difference between FeSO4 and Fe2O3 was found in the amount of charge of iron ions. As shown in Fig. 7, Batik wastewater treated with Fe3+ (FeSO4) catalyst had a higher percentage of decreasing of phenol concentration than Batik wastewater treated with Fe2+ (Fe2O3) due to Fe3+ catalyst has an important role to transform hydrogen peroxide to hydroxyl radical directly. Therefore, the degradation of phenolic compounds was faster [46]. Meanwhile, Fe3+ catalyst must be changed by hydrogen peroxide to Fe2+ and this reaction occurs very slow [47]. The produced Fe2+ ion would react with other hydrogen peroxides to produce a hydroxyl radical [48]. Therefore, Fe2+ produce higher hydroxyl radicals and faster than Fe3+ for the same hydrogen peroxide amount [49,50]. The best degradation results of phenolic compounds were obtained in the treatment of Batik wastewater with FeSO4 catalyst at 2600 Volt. The phenol reduction in the best conditions reached 88.73%.

3.5. Analysis of Variance (ANOVA)

Analysis of Variance (ANOVA) was carried out using Design Expert Version 12. Selection of the proper model was conducted to get ANOVA results. The model selection was carried out based on the sequential sum of the square model, the regression model suitability test using the lack of fit test, and the statistic summary model on the response of phenol concentration. In testing the sequential sum of the square model, the model will be selected if the probability value of p-value > F is less than 0.05 (p < 5%). The next evaluation process for selecting the model is based on model deviation (lack of fit test). In the lack of fit test, the model will be selected if p-value of lack of fit is more than 5% (> 0.05) which indicates that the inaccuracy of the model is not significant. The next evaluation process for selecting the model is based on statistical model testing. In this statistic model testing, there are several parameters used in choosing the proper model, including the lowest standard deviation value, the highest R-square, the highest adjusted R-square, the highest predicted R-square, and the lowest PRESS (Prediction Error Sum of Square) [51].

| Source               | Sum of Squares | df | Mean Square | F-value | p-value |
|----------------------|----------------|----|-------------|---------|---------|
| Mean vs Total        | 2956.64        | 1  | 2956.64     |         |         |
| Linear vs Mean       | 107.75         | 2  | 53.87       | 1.10    | 0.3707  |
| 2FI vs Linear        | 0.0062         | 1  | 0.0062      | 0.0001  | 0.9918  |
| Quadratic vs 2FI     | **450.02**     | 2  | **225.01**  | **38.65**| **0.0002**| Suggested|
| Cubic vs Quadratic   | 32.61          | 2  | 16.30       | 10.01   | 0.0178  | Aliased   |
| Residual             | 8.14           | 5  | 1.63        |         |         |
| Total                | 3555.17        | 13 | 273.47      |         |         |

Table 1. Sequential Model Sum of Squares
Based on the data provided in Table 1, Table 2, and Table 3, the suggested model is the quadratic model. The selected model was used for Analysis of Variance (ANOVA). ANOVA can explain the correlation between variables and responses. The results of the ANOVA for phenol concentration-response using the quadratic model can be seen in Table 4.

Table 2. Model Summary Statistics

| Source   | Std. Dev. | R²  | Adjusted R² | Predicted R² | PRESS |
|----------|-----------|-----|-------------|--------------|-------|
| Linear   | 7.01      | 0.016 | -0.2744 | 762.78 |
| 2FI      | 7.38      | -0.0933 | -0.5770 | 943.89 |
| Quadratic| 2.41 0.9319 | 0.8833 | 0.6505 | 209.18 Suggested |
| Cubic    | 1.28 0.9864 | 0.9674 | 0.7782 | 132.76 Aliased |

Table 3. Lack of Fit Tests

| Source   | Sum of Squares | df | Mean Square | F-value | p-value |
|----------|----------------|----|-------------|---------|---------|
| Linear   | 483.88         | 6  | 80.65       | 46.73   | 0.0012 |
| 2FI      | 483.87         | 5  | 96.77       | 56.07   | 0.0009 |
| Quadratic| 33.85          | 3  | 11.28       | 6.54    | 0.0507 Suggested |
| Cubic    | 1.24           | 1  | 1.24        | 0.7180  | 0.4445 Aliased |
| Pure Error| 6.90          | 4  | 1.73        |         |         |

Table 4. ANOVA for phenol concentration-response

| Source   | Sum of Squares | df | Mean Square | F-value | p-value |
|----------|----------------|----|-------------|---------|---------|
| Model    | 557.78         | 5  | 111.56      | 19.16   | 0.0006 significant |
| A-Tegangan| 54.41         | 1  | 54.41       | 9.35    | 0.0184 |
| B-Katalis| 17.69         | 1  | 17.69       | 3.04    | 0.1248 |
| AB       | 0.0062         | 1  | 0.0062      | 0.0011  | 0.9750 |
| A²       | 0.0816         | 1  | 0.0816      | 0.0140  | 0.9091 |
| B²       | 449.69         | 1  | 449.69      | 77.25 < 0.0001 |
| Residual | 40.75          | 7  | 5.82        |         |         |
| Lack of Fit | 33.85     | 3  | 11.28      | 6.54    | 0.0507 not significant |
| Pure Error| 6.90          | 4  | 1.73       |         |         |
| Cor Total| 598.53         | 12 |             |         |         |

Table 5. Fit statistics for phenol concentration-response

| Std. Dev. | 2.41 | R² | 0.9319 |
|-----------|------|----|-------|
| Mean      | 15.08 | Adjusted R² | 0.8833 |
| C.V. %    | 16.00 | Predicted R² | 0.6505 |
| Adeq Precision | 11.4529 |

The result of the ANOVA in Table 4 shows that the quadratic model is significant with F-value of 19.16. This is evidenced by the p-value which is less than 0.05. The quadratic model has a lack of fit F-value of 6.54 and p-value of 0.0507 which indicates that the inaccuracy of the model is not significant (the model is appropriate). The adjusted R² value of the quadratic model obtained is 0.8833 and it's close to R² value of 0.9319, the difference between adjusted R² and predicted R² is small, and
the Adeq precision value of 11.4529 (>4) indicates that the model can be applied in research design modeling. Based on Table 4, it can be seen that voltage and quadratic catalysts influence the response of phenol degradation in Batik wastewater. The quadratic model has a significant level of good response. Equation 12 shows the second-order polynomial equation (quadratic) from this research. This equation was obtained from data processing using software Design-Expert Version 12.

\[
\text{Phenol concentrations} = +90.29906 - (0.018113 \times \text{Voltage}) - (49.88576 \times \text{Catalyst}) - (0.000111 \times \text{Voltage} \times \text{Catalyst}) + (2.13601E-06 \times \text{Voltage}^2) + (12.14725 \times \text{Catalyst}^2)
\]  

(12)

The quadratic equation resulted from the response surface method can be used to predict the phenol concentration-response on voltage and catalyst variables.

3.6. Analysis of Surface Characteristic Response

The selected model can be displayed in surface contour graph and three-dimensional (3D) surface as can be seen in Figure 8 and Figure 9.

**Figure 8.** Surface contour graph of voltage and catalyst on phenol concentration-response

**Figure 9.** Three-dimensional (3D) surface graph of voltage and catalyst on phenol concentration-response

4. Conclusion

DBD reactor can release plasma discharge between two electrodes [22]. Plasma electrolysis process can be characterized by the presence of ignition voltage. Ignition voltage on the Dielectric Barrier
Discharge reactor used in this study occurred at 1400 Volt. Therefore, the voltage used for the degradation process of phenol compounds must be above the ignition voltage value. The higher voltage can produce a higher phenol reduction in Batik wastewater. This is due to higher voltage can produce plasma maximally. The type of catalyst can also affect the degradation process of phenol compounds. Batik wastewater treated with Fe$^{2+}$ (FeSO$_4$) catalyst has the highest percentage reduction in phenol concentration (88.73%). Fe$^{2+}$ catalyst has an important role to transform hydrogen peroxide to hydroxyl radical directly [46]. Based on analysis of variance (ANOVA), stress and quadratic catalyst variables affect the response of phenol levels in batik waste.

Acknowledgments
The authors would like to show their gratitude for Center for Plasma Research, Faculty of Science and Mathematics, Diponegoro University, Indonesia for all support and discussion throughout the research. This work was supported by research grant from the Indonesian Ministry of Research, Technology and Higher Education (Ristekdikti) under the grant number: 1949/E2/KM.05.01/2021. The authors declare no conflict of interest.

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