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Modeling of Photocatalytic Activity of ZnO/AC by Using Linear Probability Model, Logit and Complementary Log Transformation

Jaka Nugraha\textsuperscript{a,}\textsuperscript{*}, Is Fatimah\textsuperscript{b}

\textsuperscript{a}Statistics Department, Islamic University of Indonesia, Campus Terpadu UII, JL Kaliurang Km 14, Yogyakarta, Indonesia
\textsuperscript{b}Chemistry Department, Islamic University of Indonesia, Campus Terpadu UII, JL Kaliurang Km 14, Yogyakarta, Indonesia

Abstract

This current research is aimed to study the modeling of photocatalytic activity of ZnO immobilized activated carbon (ZnO/AC) by using statistical model: linear, exponential, logistic transformation and complementary log model. ZnO/AC was prepared with varied Zn content which is affect to its physicochemical properties and the materials were tested in phenol photooxidation. Quantitative relationship of physicochemical character and varied photocatalytic conditions to the photocatalytic activity of materials is simulated. From the simulation it is found that exponential model is the fittest model and representing the effect of time of treatment, Zn content in catalyst and catalyst dosage significantly contribute to the linear model.

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Keywords: Linear model; Photocatalyst; Exponential model; Complementary log model; Logistic transformation.

Nomenclature

<table>
<thead>
<tr>
<th>Symbol</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>Act</td>
<td>Photocatalytic activity</td>
</tr>
<tr>
<td>t</td>
<td>time</td>
</tr>
<tr>
<td>Zn</td>
<td>Zn content in catalyst</td>
</tr>
<tr>
<td>R²</td>
<td>coefficient of determination</td>
</tr>
<tr>
<td>PRESS</td>
<td>prediction sum of squares</td>
</tr>
<tr>
<td>m</td>
<td>dosage of catalyst</td>
</tr>
<tr>
<td>F</td>
<td>statistic F value</td>
</tr>
<tr>
<td>SS</td>
<td>sum of square</td>
</tr>
<tr>
<td>d</td>
<td>Durbin–Watson statistic</td>
</tr>
<tr>
<td>MS</td>
<td>mean of square</td>
</tr>
<tr>
<td>β</td>
<td>coefficient of regression</td>
</tr>
<tr>
<td>π</td>
<td>probability function</td>
</tr>
<tr>
<td>ρ</td>
<td>correlation</td>
</tr>
<tr>
<td>df</td>
<td>degree of freedom</td>
</tr>
</tbody>
</table>

\* Corresponding author.
E-mail address: jnugraha@uui.ac.id

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1. Introduction

Advance oxidation process (AOPs) becomes important process in wastewater treatment technology. A combination between photocatalytic material, oxidant and light source called as photooxidation treatment is one important technique in this scheme. Reusability and low cost investment of the method are the factors, and in other side those are depend on the photocatalytic activity of catalyst(photocatalyst) materials. Some researches and investigations are efforted to look for some important aspects within the technology and one of these is the photocatalyst. The activity of photocatalyst is mainly related to the ability to catch light, to adsorb the pollutant in the treated solution and destroy the pollutant over releasing radicals and active oxidizing agent due to photocatalysis mechanism[1][2]. By these steps, some researches combined the utilization of adsorbent and photocatalytic semiconductor metal oxide such as TiO_2 and ZnO. Refer to some feasibilities of activated carbon(AC) as adsorbent and ZnO as photocatalyst, this research aimed to evaluate the utilization of zinc oxide immobilized onto activated carbon (ZnO/AC) composite as photocatalyst[3-6]. Photocatalytic activity of the materials is studied over phenol photooxidation reaction. In order to develop, improve and optimize processes in order to fixing the activity in larger scale, this research focuses on evaluation of the photocatalytic activity by using statistical modelling. As reported by some works on photocatalyst preparation and activity testing, many parameters affect to the activity.

Usually response surface methodology (RSM) was reported for the simulation on catalytic data. However actually other modeling methods can be adopted, studied and compared in order to determining the optimum model in applications. However the use of RSM and other methods usually are not available to interpret some relationship between parameters within the model. As well as in other models, quantitative variables on are employed for analyzing the fitness of photocatalytic activity[7][1][3-6]. The objective of this study was to develop a statistical model that relates the photocatalytic activity of ZnO/AC in phenol oxidation and subsequently, to assess the effects of the different factors on the degradation rate constant. Another objective was to determine the optimum modeling condition for ZnO/AC utilization with respect to the degradation rate.

Modeling will be used to express the relationship between a set of independent variables and a dependent variable. For example, we may want to compute the relationship between the dose of a drug and its effectiveness, the relationship between training and subsequent performance on a task, the relationship between the price of a house and the time it takes to sell it, etc. We may recognize research issues in these examples that are commonly addressed by such techniques as multiple regression or analysis of variance. Multiple regression and analysis of variance assume that the relationship between the independent variable(s) and the dependent variable is linear in nature. Nonlinear Estimation leaves it up to you to specify the nature of the relationship; for example, we may specify the dependent variable to be a logarithmic function of the independent variable(s), an exponential function, a function of some complex ratio of independent measures, etc[9]. Some of non-linear modeling and statistical approach for catalyst preparation and application is also reported from previous research[9-11]. Included within this type of modeling is multiple regression analysis.

The regression model for independent variables \(x_1, ..., x_p\) and a dependent variable \(y\) can be expressed

\[
y = \beta_0 + \beta_1 x_1 + ... + \beta_p x_p = \mathbf{\beta} \mathbf{x} \tag{1}
\]

We assume the dependent variable to be a linear function of the independent variables, this is called a linear model. let \(x=(x_1, ..., x_p)^T\) and \(\beta=(\beta_0, \beta_1, ..., \beta_p)^T\). Multiple Regression does not “know” that the response variable is binary in nature or percentage (0%-100%). Therefore, it will inevitably fit a model that leads to predict values greater than 1 or less than 0. However, predicted values that are greater than 1 or less than 0 are not valid; thus, the restriction in the range of the binary variable (e.g., between 0 and 1) is ignored as the standard multiple regression procedure used. Nonlinear Estimation allows to specify essentially any type of continuous or discontinuous regression model. Some of the most common nonlinear models are probit, logit, exponential growth, and breakpoint regression.

In the logit regression model, the predicted values for the dependent variable will never be less than (or equal to) 0, or greater than (or equal to) 1, regardless of the values of the independent variables. The model formula is

\[
\pi(x) = \frac{\exp(\beta_0 + \beta_1 x_1)}{1 + \exp(\beta_0 + \beta_1 x_1)} \tag{2}
\]
As \( x \rightarrow x_0, \pi(x) \rightarrow 0 \) when \( \beta_1 < 0 \) and \( \pi(x) \rightarrow 1 \) when \( \beta_1 > 0 \). The S-shaped curves in Fig. 1. are typical.

![Logistic regression Function](image)

Fig. 1. Logistic regression Function

The model for multiple variables is as follow:

\[
\pi(x) = \frac{\exp(\beta x)}{1+\exp(\beta x)}, \quad \text{and} \quad \ln\left(\frac{\pi(x)}{1-\pi(x)}\right) = \beta X
\]  

there is no simple way to express the effect on the probability of increasing a predictor by one unit while holding the other variables constant. The effect of the \( j \)-th predictor on the probability \( \pi \) depends on the coefficient \( \beta_j \) and the value of the probability (Agresti, 2005). The response curve for \( \pi(X) \) are a symmetric appearance about the point where \( \pi(X) = 0.5 \), so \( \pi(X) \) approaches 0 at the same rate it approaches 1.

Logit models are inappropriate when this is badly violated. The other model is called the complementary log-log. The model formula is

\[
\pi(x) = 1 - \exp[-\exp(\beta_0 + \beta_1 x)]
\]  

The response curve has the shape shown in Fig. 2. \( \pi(X) \) approaching 0 fairly slowly but approaching 1 quite sharply. Linear transformations for this model is

\[
\ln[-\ln(1-\pi(x))] = \beta_0 + \beta_1 x
\]  

![Complementary log-log regression Function](image)

Fig. 2. Complementary log-log regression Function

2. Evaluating Model

There are several sizes to determine the model fit to the data. In general, a good model is the difference between predictive and observations are very small. Usable size value devian and coefficient of determination. To get a good model, some of the things that must be considered are multicolliner and autocorelasi. Multicolliner can be measured
by statistics and prediction sum of squares (PRESS). Auto correlation can be measured using Durbin-Watson Test.

2.1. Coefficient of determination

Tests thus far have shown if a linear relationship exists; it is also useful to measure the strength of the relationship. This is done by calculating the coefficient of determination – $R^2$. One criterion that is commonly used to illustrate the adequacy of a fitted regression model is the coefficient of determination, or $R^2$\([12]\).

\[
R^2 = \frac{\sum_{i=1}^{n} (\hat{y}_i - \bar{y})^2}{\sum_{i=1}^{n} (y_i - \bar{y})^2}
\]  

(6)

The quantity $R^2$ merely indicates what proportion of the total variation in the response $Y$ is explained by the fitted model. Often an experimenter will report $R^2 \times 100\%$ and interpret the result as percent variation explained by the postulated model. The square root of $R^2$ is called the multiple correlation coefficient between $Y$ and the set $x_1, x_2, \ldots, x_p$. Adjusted $R^2$ is a variation on $R^2$ that provides an adjustment for degrees of freedom. The coefficient of determination cannot decrease as terms are added to the model.

2.2. Prediction sum of squares

Another approach is to measure the discrepancy between each observation and its prediction but where that observation was not used in the development of the prediction equation. The sum of squares of these discrepancies is the PRESS statistic given by Allen (1971b). The term PRESS is an acronym for prediction sum of squares. The PRESS statistic is

\[
PRESS = \sum_{i=1}^{n} (y_i - \hat{y}_{\text{pred}(i)})^2
\]  

(7)

Let $\hat{y}_{\text{pred}(i)}$ be the prediction of observation $i$, where the $(i)$ indicates that the $i$th observation was not used in the development of the regression equation\([13]\). In addition to the PRESS statistic itself, the analyst can simply compute an $R^2$ like statistic reflecting prediction performance. The statistic is often called $R^2$ of prediction (\(R^2\text{-pred}\)) and is given as follows:

\[
R^2_{\text{pred}} = 1 - \frac{PRESS}{\sum_{i=1}^{n} (y_i - \bar{y})^2}
\]  

(8)

2.3. Durbin-Watson test

The presence of a serial correlation in the residuals is also detected by the Durbin-Watson test for independence (Durbin and Watson, 1951). The Durbin-Watson test statistic is

\[
d = \frac{\sum_{i=2}^{n} (e_i - e_{i-1})^2}{\sum_{i=1}^{n} e_i^2} \approx 2(1 - \rho)
\]  

(9)

where $\rho$ is the sample correlation between $e_i$ and $e_{i-1}$. The Durbin-Watson statistic $d$ gets smaller as the serial correlation increases. The one-tailed Durbin-Watson test of the null hypothesis of independence $H_0: \rho = 0$, against the alternative hypothesis $H_A: \rho < 0$, uses two critical values $d_U$ and $d_L$ which depend on $n$, $p$, and the choice of $a$. The test procedure rejects the null hypothesis if $d < d_L$, does not reject the null hypothesis if $d > d_U$, and is inconclusive if $d_L < d < d_U$. Tests of significance for the alternative hypothesis $H_A: \rho > 0$ use the same critical values.


$dU$ and $dl$, but the test statistic is first subtracted from 4.

3. Experimental

3.1. Materials and Method:

Materials used for preparation of ZnO/AC consist of zinc acetate dehydrate, isopropanol, activated carbon and also phenol were supplied from Merck-Millipore.

3.2. Instrumentation:

Gas Sorption analyzer of NOVA 1200e and EDX JEOL were used for surface profile and elemental analysis. Gas chromatography Buck Scientific was used to determine phenol concentration in initial and treated solution.

ZnO/AC was prepared by impregnation method. Zinc precursor solution obtained by diluting zinc acetate in isopropanol:water(1:1) was mixed with activated carbon powder followed by stirring for a night. The Zn content in the ZnO/AC was varied by Zn percentage of 2.5, 5.0, 7.5 ad 10%wt. The solvent was then evaporated before the dry solid were calcined at 450°C for 4 h. From varied Zn content the materials were designated as ZnO/AC-2.5 to ZnO/AC-10 respectively. The physicochemical character of the materials was performed by gas sorption analysis to get specific surface area(SSA), pore volume(PV) and pore radius(PR) data, while the Zn content was identified by energy dispersive x-ray fluorescence(EDX). Photocatalytic testing of the materials was performed in a batch photoreactor complimented with UV Lamp Philips 40W. The UV lamp is immersed in the tested solution. For each solution photocatalyst powder at varied weight was evaluated and at certain time of sampling (0-120 mins) phenol concentration was sequentially analyzed by using gas chromatography.

Photocatalytic activity(Act) of materials was calculated by using following equation (10):

$$
Activity(Act) = \frac{[\text{phenol}]_0 - [\text{phenol}]_t}{t}
$$

(10)

With $[\text{phenol}]_0$ and $[\text{phenol}]_t$, are phenol concentration at initial and time sampling $t$.

4. Result and Discussion.

The character of prepared ZnO/ACs is listed in Table 1.

<table>
<thead>
<tr>
<th>Parameter Code</th>
<th>Parameter</th>
<th>AC</th>
<th>ZnO/AC-2.5</th>
<th>ZnO/AC-5.0</th>
<th>ZnO/AC-7.5</th>
<th>ZnO/AC-10.0</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zn content (%wt)</td>
<td>Zn</td>
<td>0</td>
<td>2.4</td>
<td>5.2</td>
<td>7.6</td>
<td>9.8</td>
</tr>
<tr>
<td>Pore radius (Å)</td>
<td>PR</td>
<td>16.79</td>
<td>17.48</td>
<td>20.71</td>
<td>24.24</td>
<td>22.24</td>
</tr>
<tr>
<td>Specific surface area (m²/g)</td>
<td>SSA</td>
<td>618</td>
<td>557</td>
<td>323</td>
<td>177</td>
<td>206</td>
</tr>
<tr>
<td>Pore Volume (cc/g)</td>
<td>PV</td>
<td>0.5817</td>
<td>0.4865</td>
<td>0.3341</td>
<td>0.2141</td>
<td>0.2288</td>
</tr>
</tbody>
</table>

As can be seen from Table 1 Zn content in the precursor solution increased, the measured Zn content is also increased while for SSA and PV parameters the higher Zn content reflects the lower values. This phenomena is related to the impregnation mechanism used in the preparation. The dispersed Zn is only affected by the concentration of inserted molecule as there is no washing during the preparation. The inserted ZnO seems to be
aggregated on the AC surface and affecting porous distribution. As consequent to the amount of ZnO the lower surface parameters is found. Those parameters are theoretically and reported to be related with the photoactivity. The higher ZnO as photoactive agent presented in the system it will affect to catch more photon to convert and produce radicals, similarly the higher SSA and PV parameters will contribute to increase adsorptivity of target molecules[14][15]. The variables of Zn, SSA, PPA, PV and PR have strong correlation and significance as exhibited by the data in Table 2.

<table>
<thead>
<tr>
<th>Variable</th>
<th>Correlation</th>
<th>P-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zn</td>
<td>PR</td>
<td>0.900</td>
</tr>
<tr>
<td>Zn</td>
<td>SSA</td>
<td>-0.956</td>
</tr>
<tr>
<td>Zn</td>
<td>VP</td>
<td>-0.969</td>
</tr>
<tr>
<td>PR</td>
<td>SSA</td>
<td>-0.962</td>
</tr>
<tr>
<td>PR</td>
<td>VP</td>
<td>-0.972</td>
</tr>
<tr>
<td>SSA</td>
<td>VP</td>
<td>0.994</td>
</tr>
</tbody>
</table>

By these considerations, modeling on photocatalytic activity is performed based on related parameters. Photocatalytic activity (Act) engaged under varied time of treatment(t), catalyst dosage (weight of catalyst per L solution) and also considering Zn content in each photocatalyst (Zn) is listed in Table 3.

<table>
<thead>
<tr>
<th>Varied Condition</th>
<th>Variable Code</th>
<th>Values</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zn content in photocatalyst (% wt.)</td>
<td>Zn</td>
<td>0.8; 2.4; 5.2; 7.6; 9.8</td>
</tr>
<tr>
<td>Catalyst dosage (g/L)</td>
<td>m</td>
<td>0.1; 0.2; 0.3</td>
</tr>
<tr>
<td>Time of treatment(mins)</td>
<td>t</td>
<td>0; 15; 30; 45; 60; 12</td>
</tr>
</tbody>
</table>

Effect of variable can be seen by graph in Fig. 3.

![Graph showing photocatalytic activity vs time of treatment](image1)

![Graph showing photocatalytic activity vs Zn content](image2)

![Graph showing photocatalytic activity vs catalyst dosage](image3)

Fig.3. Effect of Zn, time and catalyst dosage

Due to F values in Table 4 it can be concluded that significant difference in photocatalytic activity among Zn, time
and catalyst dosage. This means that the activity of photooxidation (Act) is affected by those variables.

<table>
<thead>
<tr>
<th>Source</th>
<th>df</th>
<th>SS</th>
<th>MS</th>
<th>F</th>
<th>P</th>
<th>P-value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zn</td>
<td>4</td>
<td>5.26951</td>
<td>1.31718</td>
<td>58.32</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td>t</td>
<td>5</td>
<td>5.43228</td>
<td>0.6646</td>
<td>35.60</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td>m</td>
<td>2</td>
<td>0.56744</td>
<td>0.28372</td>
<td>14.71</td>
<td>0.000</td>
<td></td>
</tr>
<tr>
<td>Error</td>
<td>78</td>
<td>1.59396</td>
<td>0.01928</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Total</td>
<td>85</td>
<td>10.77319</td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

From the figure 3 it is found that Zn and m variables affect to the photooxidation activity in negative quadratic (polynomial 2nd order) while t variable affect exponentially. Furthermore this significant effect of the variables can be modeled by several model (a) linear model (b) exponential transformation (c) logistic transformation (d) complementary log transformation.

4.1. Linear Model

Simple regression model of each variable is listed in Table 5.

<table>
<thead>
<tr>
<th>No</th>
<th>Model</th>
<th>P(α=0.05)</th>
<th>P-value</th>
<th>R²</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>Act = 4.96 m - 11.89 m² (7.90) (−4.45)</td>
<td>74.93</td>
<td>9.979E−10</td>
<td>63.00%</td>
</tr>
<tr>
<td>2</td>
<td>Act = 0.281 Zn - 0.0266 Zn² (2.88) (−11.68)</td>
<td>165.97</td>
<td>1.376E−10</td>
<td>79.04%</td>
</tr>
<tr>
<td>3</td>
<td>Act = 0.409 + 0.0297 ln t (13.32) (96.41)</td>
<td>41.05</td>
<td>7.10E−10</td>
<td>31.81%</td>
</tr>
</tbody>
</table>

The values in parenthesis reflect the t value of each regression coefficient. By the values it is concluded that found that m and Zn formed in polynomial 2nd and t variable affect in exponential model. The equation of regression model on photoactivity (Act) is as follow:

\[
\text{Act} = 0.232 \text{Zn} + 1.31 \text{m} - 0.023 \text{Zn}^2 - 3.20 \text{m}^2 + 0.0295 \ln t \\
(14.17) \quad (2.89) \quad (-14.10) \quad (-2.24) \quad (11.97)
\]

4.2. Exponential transformation model

Exponential transformation is calculated by following equation:

\[y = \ln (b_0 + b_1x_1 + \ldots + b_px_p) \text{ atau } \exp(y) = b_0 + b_1x_1 + \ldots + b_px_p\]

Regression equation is as follow:

\[
\exp(\text{Act}) = 0.00450 \text{ t} + 0.403 \text{ Zn} + 10.4 \text{ m} - 0.0398 \text{ Zn}^2 - 25.1 \text{ m}^2 \\
(4.99) \quad (11.28) \quad (9.71) \quad (-11.23) \quad (-7.69)
\]

4.3. Logistic transformation model

The equation used for logistic transformation is as follow:

\[
\ln[y/(1-y)] = b_0 + b_1x_1 + \ldots + b_px_p
\]

and the regression equation obtained is:

\[
\ln[\text{Act} / (1 - \text{Act})] = -0.00571 \text{ t} - 6.50 \text{ m} + 15.2 \text{ m}^2
\]
4.4. Complementary Log transformation model

Complementary log transformation was conducted by using the equation: \( \ln[-\ln(1 - y)] = \beta_0 + \beta_1 x \) and the regression equation obtained is:
\[
\ln[-\ln(1 - Acf)] = 0.139 t - 131 m + 321 m^2
\]
\[
(5.42) \quad (-5.61) \quad (3.99)
\]

The comparison of four models based on \( R^2 \), PRESS, Durbin watson values is listed in Table 6.

<table>
<thead>
<tr>
<th>Model</th>
<th>( R^2 )</th>
<th>PRESS</th>
<th>( R^2_{pred} )</th>
<th>F</th>
<th>P-Value</th>
<th>Durbin-Watson</th>
</tr>
</thead>
<tbody>
<tr>
<td>Linear</td>
<td>92.74%</td>
<td>2.33081</td>
<td>91.58%</td>
<td>217.11</td>
<td>7.6490E-47</td>
<td>1.34214</td>
</tr>
<tr>
<td>Exponential</td>
<td>96.47%</td>
<td>10.6278</td>
<td>96.60%</td>
<td>464.92</td>
<td>3.8248E-60</td>
<td>1.66552</td>
</tr>
<tr>
<td>Logistic</td>
<td>86.40%</td>
<td>11.7139</td>
<td>85.42%</td>
<td>184.28</td>
<td>1.4106E-37</td>
<td>1.65852</td>
</tr>
<tr>
<td>Complementary log</td>
<td>39.52%</td>
<td>8448.19</td>
<td>35.12%</td>
<td>18.95</td>
<td>1.5174E-09</td>
<td>1.66382</td>
</tr>
</tbody>
</table>

From Table 6, it can be concluded that exponential transformation model is the best model among others. Linear model has several limitations, one of these is the predicted value that possible lay at out of the interval of 0<Ac<1 while logistic transformation and complementary log models have \( R^2 \) and \( R^2_{pred} \) less than 90%. Durbin-Watson statistic on the linear model is on \( d=1.34214 \) and less than \( dL=1.41 \) (\( \alpha=5\% \)) means that fail to reject \( H_0 \) and indicating that there is a serial correlation. Meanwhile by exponential transformation model the value of \( d =1.66552 > dU=1.64 \) indicating that there is no serial correlation as well as in logistic transformation and complementary transformation models.

The best way to predict the Acf in photooxidation process is stated as following equation:
\[
\exp(\text{Acf}) = 0.00450 t + 0.403 \text{Zn} + 10.4 m - 0.0398 \text{Zn}^2 - 25.1 m^2
\]
\[
(4.99) \quad (11.28) \quad (9.71) \quad (-11.23) \quad (-7.69)
\]

The model express the effect of time, Zn content and catalyst dosage within the examined range. Due to chemical kinetic perspective the degradation of treated target molecule is exponential function as time variable. By this model, the contribution of Zn content as photoactive agent and also catalyst dosage are also figured out.

5. Conclusions

Modeling on photocatalytic activity of ZnO/AC in this research give theoretical approach on the relationship between physicochemical character of the material, time and catalyst dosage toward photoactivity in photooxidation. Zn content in catalyst, catalyst dosage, and time of the treatment are significant variables influencing the photooxidation activity while the surface parameters of materials are strongly related with Zn content. By using four models: linear, exponential, logistic transformation and complementary log model it is found that the exponential model is the best model for predicting photoactivity of ZnO/AC in phenol photooxidation.

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