Optimization of Wood Vinegar from Pyrolysis of Jelutung Wood (Dyera lowii Hook) by Using Response Surface Methodology

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Abstract. The optimization of vinegar produced from ‘jelutung’ wood (Dyera lowii Hook) was investigated. The pyrolysis temperature and pyrolysis time are primary parameters for wood vinegar yield. Effects of pyrolysis temperature and pyrolysis time on wood vinegar yield were evaluated through experimental and statistical analysis. The objective of this study was to optimize experimental parameters to obtain the maximum wood vinegar yield from pyrolysis of jelutung wood by using response surface methodology (RSM). Important pyrolysis process parameters such as pyrolysis temperature (X1) and pyrolysis times (X2) were optimized. The regression equation obtained for the wood vinegar yield was Y = 30.24 + 2.11 X1 - 0.72 X2 - 2.10 X1² - 1.27 X2² - 0.53 X1.X2. The maximum wood vinegar yield of 30.97 % was obtained at the optimum parameters of pyrolysis temperature of 462.5 °C and pyrolysis time of 152 minutes.

1. Introduction

Pyrolysis of wood is promising way to produce wood vinegar, charcoal, tar, and other gaseous. Wood vinegar is crude condensate, highly oxygenated compound obtained from a wood carbonization process with absence of oxygen at a high temperature [1-2]. As concerns for the environment grow, botanical pesticides including wood vinegar are increasingly emphasized as alternative solution for fungal and insect attacked. The use of wood vinegar as a pesticide needs to be optimized for the production process in wood vinegar yield.

The wood vinegar yield during pyrolysis depends on different parameters such as pyrolysis temperature, pyrolysis times, and particle size of wood. To optimize the maximum wood vinegar yield from pyrolysis, the parameters influencing its production should be optimized [3]. Several studies have been described the effects of pyrolysis temperature, pyrolysis times, and particle size of wood on the wood vinegar yield [4-6] and chemical compound of wood vinegar [7-8]. The pyrolysis temperature is the primary factors for the yield of wood vinegar [4].

The variation in operational process as well as biomass type that give the maximum yield of wood vinegar. For instance, the maximum wood vinegar yield from Pinus brutia Ten was 33.25 % at 450 °C [9], meanwhile, the maximum wood vinegar yield from palm fruit shell was 37 % at 300 °C [10]. The response surface methodology (RSM) has been effectively used to maximize yields of liquid from the pyrolysis of waste mixtures [11]. A previous work reported the effect of wood particle size, temperature of pyrolysis, and time of pyrolysis on wood vinegar yield. The vinegar obtained from Shorea laevis Ridi [6].
At present, no studies on optimization of wood vinegar yield from jelutung wood (*Dyera lowii* Hook). Therefore, in this study, RSM was used to optimize pyrolysis parameters for maximizing wood vinegar yield from *D. lowii*. The temperature of pyrolysis and time of pyrolysis was selected as the optimization parameters.

### 2. Material And Method

#### 2.1. Materials

The ‘jelutung’ wood was collected from a saw mill in Pontianak, West Kalimantan. Its was converted into particle with a disk mill. The pyrolysis process of wood vinegar was determined according to literature [8,12].

#### 2.2 Experimental Design and Statistical Analysis

An RSM was used to determine the experimental design and the optimal operating process in producing wood vinegar from ‘jelutung’ wood. A three coded level, two-factors RSM design and the coded levels of 425, 450, and 475 °C temperature and 140, 160, and 180 minutes were marked with -1, 0, +1 is presented in Table 1.

| Factor                          | Symbol   | Range and levels |
|---------------------------------|----------|------------------|
| Temperature of pyrolysis (°C)   | $X_2$    | 425 450 475      |
| Times of pyrolysis (min)        | $X_3$    | 140 160 180      |

For optimum point prediction of wood vinegar yield, the second-order polynomial equation is the following:

$$Y = \beta_0 + \sum_{i=1}^{k} \beta_i x_i + \sum_{i=1}^{k} \beta_{ii} x_i^2 + \sum_{i=1}^{m} \sum_{j=1}^{m} \beta_{ij} x_i x_j + \epsilon$$  \hspace{1cm} (1)

where $\beta_0$ is the constant regression coefficients and $\beta_i, \beta_{ii} and \beta_{ij}$ are the regression coefficients for linear, quadratic and interaction effects, respectively, the term $x_i, x_j$ are the independent factors, and $\epsilon$ is the error.[13]

The STATISTICA software package (version 6.0) and SAS software (version 8.2, SAS Institute Inc., NC, USA) was used perform statistical analysis of the RSM design, as well as perform the regression analysis and analysis of variance (ANOVA). The significance of each model parameter were analyzed by determined by F-test with $p$-value less than 0.05 were considered significant with 95% confidence.

### 3. Results and Discussion

Summarized in Table 2 are the result of experimental runs based on the different pyrolysis temperature and pyrolysis time conditions generated from the RSM. To maximize the yield of vinegar obtained from ‘jelutung’ wood, the combination of the independent variable were selected. It varied from 23.89 to 32.22 % of vinegar yield obtained from ‘jelutung’ wood.

Interaction relationships of treatment pyrolysis temperature ($X_1$) and pyrolysis time ($X_2$) on wood vinegar yield are shown in Table 3. It also indicated that one independent variables contribute significantly effect on vinegar yield obtained from ‘jelutung’ wood, whereas the effect on variable $X_2$ was insignificant.

The ranges in optimal operating conditions for wood vinegar yield were obtained using Eq. (1) derived from the surface response experiments was the STATISTICA Software 6.0. The Wood vinegar yield was attended to investigated the effects of pyrolysis temperature (425, 450, and 475 °C) and times of pyrolysis (140, 160, and 180 min). Higher vinegar yield obtained from ‘jelutung’ wood can be achieved with a temperature near 475 °C at pyrolysis time of 160 minute. The prediction...
optimal conditions for operating condition were pyrolysis temperature of 462.50 °C and pyrolysis time of 152 minute. Under these conditions, the model predicts a maximum wood vinegar yield of 30.97 %.

Table 2. The RSM design in coded terms showing the levels of tested variables, the yield of vinegar from ‘jelutung’ wood in terms of experimental and predicted values

| Run | Coded variable level | Wood vinegar yield (Y, %) |
|-----|----------------------|---------------------------|
|     | X1       | X2       | Experimental | Predicted |
| 1   | -1       | -1       | 26.67        | 25.19     |
| 2   | 1        | -1       | 29.89        | 27.14     |
| 3   | -1       | 1        | 25.00        | 25.19     |
| 4   | 1        | 1        | 26.11        | 27.14     |
| 5   | -1       | 0        | 23.89        | 26.40     |
| 6   | 1        | 0        | 32.22        | 30.62     |
| 7   | 0        | -1       | 28.33        | 29.24     |
| 8   | 0        | 1        | 29.44        | 26.97     |
| 9   | 0        | 0        | 30.56        | 29.93     |
| 10  | 0        | 0        | 28.89        | 29.93     |
| 11  | 0        | 0        | 31.67        | 29.93     |
| 12  | 0        | 0        | 30.00        | 29.93     |

Table 3. Regression coefficients of the predicted second order polynomial model for the vinegar yield obtained from ‘jelutung’ wood

| Sources of variation | Coefficient of polynomial | Error | t-value | Pr>t |
|----------------------|---------------------------|-------|---------|------|
| Intercept            | 30.24                     | 0.87  | 34.89   | <0.000 |
| X1                   | 2.11                      | 0.78  | 2.72    | 0.035 |
| X2                   | -0.72                     | 0.78  | -0.93   | 0.387 |
| X1 * X1              | -2.10                     | 1.16  | -1.80   | 0.122 |
| X2 * X1              | -0.53                     | 0.95  | -0.56   | 0.599 |
| X2 * X2              | -1.27                     | 1.16  | -1.09   | 0.319 |

Coefficient of variation = 6.65 %, \( R^2 = 0.72 \)

To further analysis the effect of the two factors on wood vinegar yield, the relationship between the variables and response was illustrated in a representation of contour plots (2D) and response surface (3D) were generated model for wood vinegar yield. The independent variables used in this work were pyrolysis temperature (X1) and pyrolysis time (X2) within the range of 425-475 and 140-180 minutes, respectively, trials were arranged by RSM.

The empirical models for the wood vinegar yield wood were obtained as follows and the contour plots and three-dimensional response surface curves (3D) and is illustrated in Fig. 1. The wood vinegar yield equation comprised a second-order term, illustrating the yield of wood vinegar, is given as follows:

\[
Y = 30.24 + 2.11 X1 - 0.72 X2 - 2.10 X1^2 - 1.27 X2^2 - 0.53 X1X2
\] (2)

Where, \( Y \) is the predicted wood vinegar yield and X1 and X2 are coded values of pyrolysis temperature and pyrolysis time, respectively.

Statistic significance of the model equation, individual variable, and their interaction was examined using an ANOVA. The yields of wood vinegar was determined from the equations 2, where the wood vinegar yield is given as a function of pyrolysis temperature and pyrolysis time. Based on the \( R^2 \), the quality of the model can be judged. The \( R^2 \) for wood vinegar is 0.72 which gives that 72.00 % of the total variation in the wood vinegar yield can be attributed to the experimental variables.
observed. Some similarities were recognized between the experimental values and those predicted by the model. Similar results were observed with the other biomass raw materials [14-17].

Figure 1. The contour plots (a) and surface chart (b) to show the interactive effect pyrolysis temperature (X1, °C) and pyrolysis time (X2, minutes) on yield of vinegar obtained from 'jelutung' wood

From the regression, the linear term of the temperature pyrolysis (X1) had significant role ($p<0.05$), which advised that the pyrolysis temperature (X1) variables was the most important factor for wood vinegar yield ($p<0.05$). ANOVA was employed for the determination of significant parameters and to estimate the yield of wood vinegar. ANOVA model exhibited the linear effects of pyrolysis temperature for wood vinegar yield was significant ($p<0.05$), but the linear effects of pyrolysis time was not significant ($p<0.05$). These indicated that the term had an impact on wood vinegar yield. Wei et al. [18] stated that wood vinegar prepared from walnut tree branches at three range temperature of 90-230 °C, 230-370 °C, and 370-450 °C and found the wood vinegar yield was 11.44, 31.49, and 1.45 %, respectively.
4. Conclusions
This study has investigated the effects on wood vinegar yields of various experimental variables such as the pyrolysis temperature and pyrolysis times. The pyrolysis temperature variable was the most significant factor in wood vinegar yield. Optimization by the RSM the pyrolysis temperature condition for the optimal vinegar yield obtained from ‘jelutung’ wood (*Dyera lowii*) was pyrolysis temperature and pyrolysis time were 462.50 °C and 152 min, respectively and the optimal wood vinegar yield was 30.97 %.

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