

# Optimization on the Hydrolysis Process of Cellulose from Corn Husk to Glucose with Activated Carbon Catalyst Sulfonated

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## ABSTRACT

The design of this research consisted of four stages, that is manufacture of catalysts, cellulose hydrolysis process, glucose yield test and optimization process. The research data is plotted in a mathematical model that is optimized using software of Statistica 10 with *Response Surface Methodology* (RSM) and ANOVA methods. From the RSM method was obtained mathematical equation model for the relationship of the combination of temperature, time and amount of catalyst to glucose levels, that is:  $Y = -20,0457 + 5,341x_1 - 3,245x_1^2 + 6,471x_2 - 2,798x_2^2 + 4,697x_3 - 2,965x_3^2 + 1,241x_1x_2 - 0,996x_1x_3 + 0,675x_2x_3$ . ANOVA method produces a value of determination coefficient ( $R^2$ ) as big 0.91545. In this research, the optimum temperature is at 70°C, the optimum time is at 2 hours, and the optimum amount of catalyst is at 11 grams. Results of Glucose yield obtained from the optimal operating conditions is 31%.

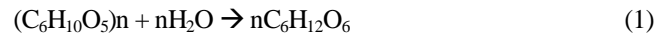
**Key words:** RSM, ANOVA, Cellulose, Activated Carbon Catalyst, Glucose

## 1. INTRODUCTION

During this time corn husk waste is not utilized maximally, so that it disturbs the surrounding environment. Usually unused corn husk waste is immediately burned. The consequence is air pollution everywhere which can interfere with breathing. So the corn waste, especially the husk should be used to reduce environmental pollution. Based on BPS data for 1993 - 2018, the average corn production in Indonesia reaches 15 million tons per year. On 1 kg of corn, contained corn husk waste as much as 200 grams of corn husk [1].

In this research, corn husk will be hydrolyzed cellulose to get glucose. Cellulose is an organic compound which is a straight chain polysaccharide of 1,4 $\beta$ -glycosidic which binds in a D-glucose unit [2]. This cellulose can replace fossil sources used as fuel, because cellulose is a renewable source of biomass [3].

The Generally, reaction of cellulose hydrolysis can be expressed as follows:



The degree of cellulose polymerization is indicated by the length of the polymer chain, which is n. Decreasing 1 mole of cellulose will produce n moles of glucose. Based on this reaction equation, cellulose hydrolysis can actually be done using only water, but hydrolysis like this requires a very long time. To speed up the reaction, a catalyst needs to be added, which can be done using a catalyst of sulfonated activated carbon [4].

The technology used in hydrolyzing cellulose is acid hydrolysis and enzyme hydrolysis. Both have drawbacks in the process of hydrolysis which the drawbacks that generate waste which is very dangerous and also make use of acid and enzyme costs are very expensive. Hydrolysis technology using sulfonated activated carbon catalyst is the right solution, because it does not cause hazardous waste and in terms of cost is also very relatively cheap [5].

Based on research of rispiandi (2015) [6], Explain that the results of the sulfonated activated carbon catalyst performance test from a coconut shell have a very significant effect on the reaction of cellulose into glucose. this is due to the more catalysts, the more protons that play a role in chemical reactions. In accordance with Xiang et al (2005) [7] who concluded that the higher the concentration of acid, the faster the reaction time because more and more available H + groups. Thus, so many catalysts are used in this variable, resulting in% yield and cellulose conversion to glucose also increases.

Based on the research of Ashadi (2013) [8], glucose levels produced from the hydrolysis process are influenced by the hydrolysis temperature, hydrolysis time and the addition of the amount of catalyst. Increasing the reaction temperature in the hydrolysis process would lower glucose levels resulting from glucose that is formed will be degraded further [9]. Therefore an optimization is needed to determine the optimum conditions of hydrolysis which includes hydrolysis temperature, hydrolysis time and the addition of the amount of catalyst [10]. Determination of optimization in the process of cellulose hydrolysis from corn husk using the Response Surface Methodology (RSM) method with the help of software Statistica 10.

## 2. RESEARCH METHODOLOGY

### 2.1 Materials

The materials used in this study are as follows: solution of technical H<sub>2</sub>SO<sub>4</sub>96%, dried corn husk, aquades, benedict solution, activated carbon from coconut shell.

### 2.2 Experimental Procedure

In this research, three variables are used to be tested. These variables are the hydrolysis temperature, hydrolysis time, and the addition of the amount of catalyst. After carrying out the hydrolysis process, glucose results from the hydrolysis filtrate were tested qualitatively using benedict solution and quantitatively using a spectrophotometer. Glucose test results were optimized by the Response Surface Methodology (RSM) method with the help software of Statistica 10 so that the optimum conditions of cellulose hydrolysis from corn husk obtained in the form of temperature, time and the addition of catalysts.

## 3. RESULTS AND DISCUSSION

### 3.1 Experimental Results

The results of this research are listed in table 1 which shows that the highest glucose yield were obtained 31%.The relationships between the three independent variabels (temperature of hydrolysis, time of hydrolysis and additional amount of catalyst) and glucose yield were research [11].

### 3.2 Characteristics of sulfonated activated carbon

Test of SEM (*Scanning Electron Microscope*)

The surface of activated carbon can be seen using a Scanning Electron Microscope (SEM) to determine the presence of large pores on the surface of activated carbon. Test results of SEM that appear and shape of the catalyst surface morphology are amorphous so that the chance for a reaction is even greater. The shape of the catalyst surface influences the interaction of the reaction process [12]. From the SEM test results for 3000x magnification obtained the following results.

The SEM test results show that the surface morphology of the catalyst is amorphous (arranged irregularly) so that the chance of a reaction is even greater (Figure 1). The shape of the catalyst surface influences the interaction of the reaction process. For SEM sulfonated activated carbon the surface structure appears more open compared to activated carbon before disulfonation [6].

With the same magnification of 3.000x it is seen that the morphological structure of the activated sulfonated carbon is more open, so that reactants (cellulose) more easily enter the surface of the catalyst so that it is possible to interact more easily with H + groups that are bound to the surface and form glucose [6].

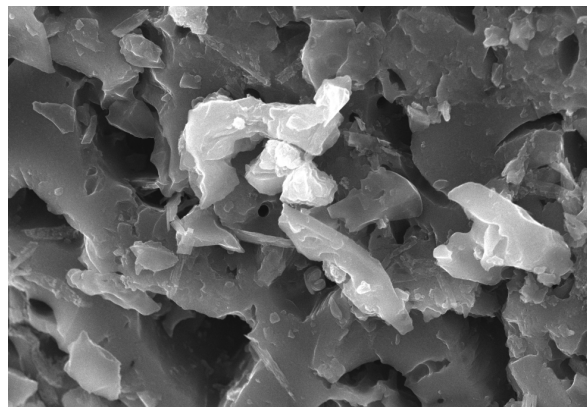


Figure 1: Result Test of Active Carbon With SEM

### 3.3 Test of BET (Brunaur Emmet Teller) Surface Area

Identification of surface area of sulfonated activated carbon was carried out by a BET (Brunaur Emmet Teller) test. Based on the analysis of these test results it is known that activated carbon from coconut shell has a surface area of 51.372 m<sup>2</sup>/g.

### 3.4 Effect of temperature, time and amount of catalyst on glucose yield

From this table the results of the glucose yield test using this spectrophotometer can be seen that the highest glucose yield values were obtained with variables with operating conditions at a temperature of 70<sup>0</sup>C, 2 hours' time and the amount of catalyst 11 grams. This is due to the increase in reaction temperature, the length of reaction time and the addition of excess catalysts which can accelerate the hydrolysis process which results in breaking the lignin and cellulose bonds (Fan et al, 2014).Other than that, In addition, increasing the temperature, time and amount of catalyst can increase the rate of hydrolysis reaction. An increase in the rate of this reaction can affect the operation of the hydrolysis process. If the operating conditions are made in excess, then the glucose yield will be degraded, thereby causing glucose yield can be decreased [13].Therefore, look for the value of the most optimum conditions, so that the glucose yield can be obtained results the most. Research data shows that the most glucose yield is produced under optimal conditions is not excessive (Number of Experimental Run 13) [20-21].

### 3.5 Optimization Using the RSM Method

The results of the research were analyzed by the RSM method with the help software of statistical 10 to find out the most optimal conditions. The results of the optimization process obtained matamatis equation 1 is a model that shows the relationship between the hydrolysis temperature, hydrolysis time and the weight of activated carbon catalyst of the glucose content is expressed as the following equation:

$$Y = -20,0457 + 5,341x_1 - 3,245x_1^2 + 6,471x_2 - 2,798x_2^2 + 4,697x_3 - 2,965x_3^2 + 1,241x_1x_2 - 0,996x_1x_3 + 0,675x_2x_3(1)$$

Description: Y = Yield of glucose (%)

x<sub>1</sub> = Temperature of hydrolysis (°C)

x<sub>2</sub> = Time of hydrolysis (hours)

x<sub>3</sub> = amount of catalyst (grams)

**Table 1:** Test results for glucose yield

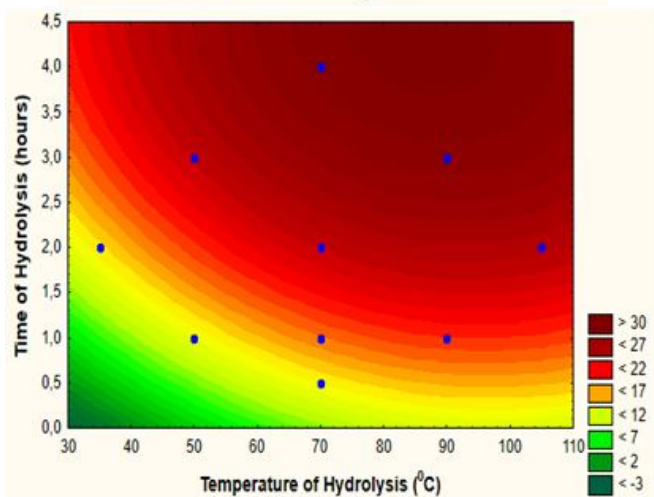
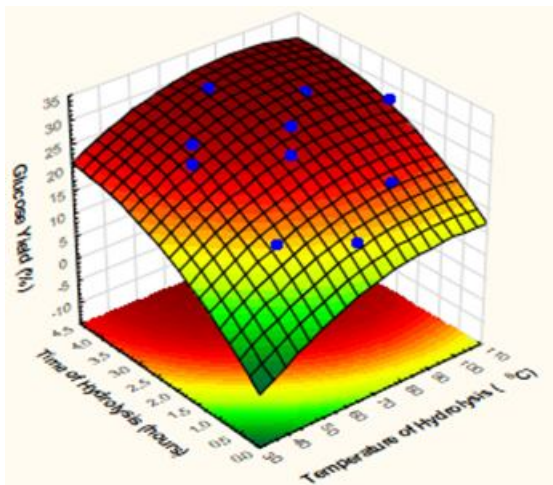
Experimental Run	Temperature of hydrolysis(°C)	Additional amount of catalyst (grams)	Time of hydrolysis (hours)	Glucose yield (%)
1	50	3	1	11
2	90	3	1	14
3	50	9	1	17
4	90	9	1	20
5	50	3	3	23
6	90	3	3	25
7	50	9	3	27
8	90	9	3	28
9	70	6	1	16
10	70	6	0,5	15
11	70	6	4	28
12	70	1	2	15
<b>13</b>	<b>70</b>	<b>11</b>	<b>2</b>	<b>31</b>
14	35	6	2	9
15	105	6	2	29
16	70	6	2	25

**Table 2:** Variant Analysis with ANOVA Method

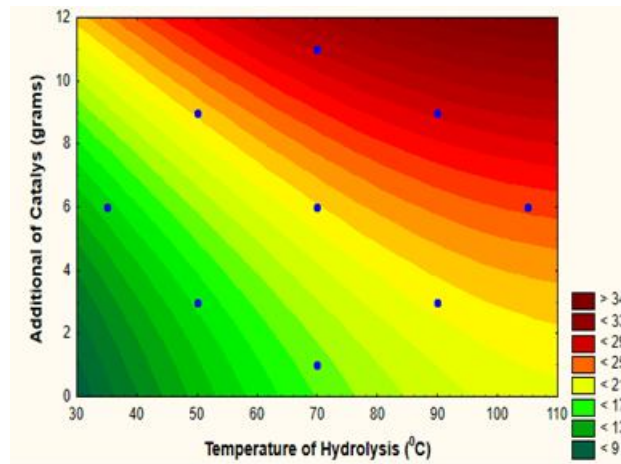
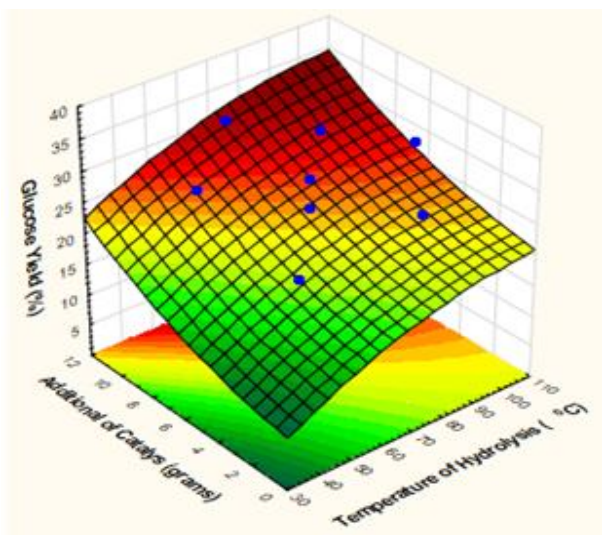
Effect	SS	Df	MS	F	P	R <sup>2</sup>
$x_1$	15,9730	2	15,9730	1,7588	0,4330	0,91545
$x_1^2$	50,1514	2	50,1514	7,3481	0,0551	
$x_2$	55,8993	2	55,8993	9,0455	0,0112	
$x_2^2$	53,1018	2	53,1018	7,2393	0,0560	
$x_3$	0,5534	2	0,5534	0,0621	0,9836	
$x_3^2$	57,8076	2	57,8076	8,2020	0,0865	
$x_1 \cdot x_2$	5,0957	2	5,0957	0,5923	0,5945	
$x_1 \cdot x_3$	20,1526	2	20,1526	3,8995	0,3911	
$x_2 \cdot x_3$	18,7354	2	18,7354	5,1217	0,2554	
Error	42,5262	6	6,887			
Total SS	303,3013	24				

The accuracy of the mathematical model can be analyzed with ANOVA which is shown in table 2. The accuracy of this method can be seen from the coefficient of determination (R<sup>2</sup>), which reached 0.91545. Value of R<sup>2</sup> the closer it is to number 1, the better the ANOVA analysis results related to the results of research conducted (Borglum et al, 2105). This indicates that 91.545 % of the total variation in the results obtained is represented in the model. The accuracy of this model can also be seen from the results of the calculation of the F (*ratio of mean square*) value is greater than the value of P (*probability*) [14]. The values of F (*ratio of mean square*) showed statistically significant regression results at the level of 5%. For a value of P (*probability*) less than 0.05, then the variable is very influential in getting the yield [15]. Analysis of variants obtained from software of Statistica 10 can be seen in Table 2.

Analysis of the optimum operating conditions can use that response surface analysis using charts and graphs of 3-dimensional optimization surface contours. Graph 3 dimensional optimization consists of two independent variables and one dependent variable, so that one other variable is a constant number [16][18]. The axis of x and y are the independent variable and the dependent variable z axis shows. In the contour graph of surface areas of color, so that it can be seen from this graph the point - the point of interaction of two variables results in a clear, whereby the most optimal interaction is located in the oldest red area [17-20]. The graph can be seen in the image below:



**Figure 2:** Graph contour of hydrolysis temperature vs time hydrolysis reaction



**Figure 3:** Graph contour of temperature hydrolysis reaction vs addition amount of catalyst

Figure 2 shows that the most optimum glucose level is in the temperature range of 70<sup>0</sup>C to 90<sup>0</sup>C and the optimum time on the range of 2 to 4 hours. Whereas in Figure 3 shows that the addition of amount the most optimum on catalyst is in the range of 10 grams - 12 grams.

#### 4. CONCLUSION

The process of cellulose hydrolysis reaction from corn husk using sulfonated activated carbon catalyst resulted in optimum operating conditions at 70<sup>0</sup>C, 2 hours and 11 grams of catalyst. In that optimum conditions, glucose levels reached 31% with mathematical equations  $Y = -20,0457 + 5,341x_1 - 3,245x_1^2 + 6,471x_2 - 2,798x_2^2 + 4,697x_3 - 2,965x_3^2 + 1,241x_1x_2 - 0,996x_1x_3 + 0,675x_2x_3$ . Values of R<sup>2</sup> predicted with the model can approach the values obtained from the results of experimental which is 0,91545.

#### ACKNOWLEDGEMENTS

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