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Submission date: 30-Jul-2021 09:43AM (UTC+0700)
Submission ID: 1625649924
File name: Paper_IEEE_Ni_Ketut_Sari_FIK_2021_Inggris.edited.docx (256.82K)
Word count: 4171
Character count: 22523

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Abstract*—The starch content contained in tapioca flour liquid waste can be processed into glucose by a hydrolysis process, which was previously carried out by a chemical pretreatment process. The purpose of this research is to find the optimization of pH in the pretreatment process with Liquid Waste of Tapioca Flour (LW-TF) and Hydrochloric acid (H-Cl) volume, using Minitab software with Response Surface Method (RSM). Before the hydrolysis of tapioca starch liquid waste, pretreatment and filtration processes were carried out to remove impurities, the chemical pretreatment process used LW-TF with a variation of 200 to 1000 ml and a hydrolysis temperature of ± 40 ^{0}C, H-Cl variable 5 to 25 ml, and a stirring time of 30 minutes using MS-20D type digital magnetic stirrer. In the fermentation process the addition of Amylase enzyme as much as 11 %w/v and Maltose enzyme as much as 9%w/v to glucose contains 24.9 %v/v. Optimization of pH using Response Method Surface showing similarities to Regression Equation, obtained optimum pH of 8.85, Liquid Waste of Tapioca Flour volume of 1165.69 ml, and volume of H-Cl 0.859 ml, used as a reference for the next fermentation process to obtain optimum glucose levels.

***Keywords*—glucose, hydrolysis, response surface method, liquid waste of tapioca flour**

I. INTRODUCTION

The first technology bioethanol feedstocks which include corn and sugarcane can't meet the worldwide call for ethanol manufacturing, some other opportunity to agricultural waste, however, has demanding situations and obstacles withinside the biomass process, and the glucose and xylose production tactics require new fermentation technologies [1]. Waste from the manufacture of sago for bioethanol manufacturing acquired glucose containing 0.47 (g ethanol in step with g glucose), and 15.6 ethanol in step with a hundred grams of dry sago [2]. The starch issue may be very complicated withinside the shape of disaccharides, earlier than being fermented, the starch issue withinside the shape of maltase is hydrolyzed with enzymes to achieve glucose [3]. Rice flour liquid waste as uncooked cloth for bioethanol, the hydrolysis system produces 5-10% glucose with bacilli, and the fermentation system produces 11-16% bioethanol the usage of Saccharomyces Cerevisiae [4], the fermentation system makes use of Saccharomyces Cerevisiae and Zymomonas Mobilis produces bioethanol 10-15 % [5], to gain bioethanol content material of 36% v/v through batch distillation system [6].

Ethanol with purple sage as raw material derived from cellulose carried out a pretreatment process with acid (HNO3), and a delignification process to remove lignin content in the raw material, before the fermentation process, reducing the lignocellulose fraction to obtain good cellulose biomass [7]. Ethanol with sago pulp as raw material uses a microwave hydrothermal hydrolysis process to remove carbon dioxide [8]. Higher energy savings compared to the previous strategy, without the use of enzymes in the fermentation process, without acid or catalysts in the hydrolysis process, a simple process including the pretreatment process and the distillation process, obtained 15.6% ethanol content [9]. Optimization of the hydrolysis process utilizing enzymes, optimizing the parameters of Ammonia Fiber Explosion to obtain higher ethanol levels [10]. Bioethanol with cellulose as raw material in the ethanol purification technique, using batch and flash distillation techniques, the results received showed almost the same bioethanol yield of about 95-96% v/v [11].

The study was conducted to evaluate the acid pretreatment of waste raw materials for bioethanol production, optimization of sulfuric acid hydrolysis [12]. Pretreatment of biomass containing Lignocellulose with sulfuric acid (H2SO4) and phosphorus (H3PO4) is widely used because it is relatively inexpensive and efficient in hydrolyzing lignocellulose, although the beautiful effect is lighter and more environmentally friendly. Hydrochloric acid (H-Cl) is more volatile and more easily recovered and attacks biomass better than H2SO4 [13]. Similarly, nitric acid (HNO3) has a good conversion rate of cellulose to sugar [14]. However, both acids are expensive compared to sulfuric acid. Lignocellulose pretreatment has received considerable research globally due to its impact on the technical, economic, and environmental sustainability of cellulosic ethanol production, reviewing the emerging knowledge and methods of chemical pretreatment, combining chemical pretreatment with other methods to improve carbohydrate preservation, reducing formation to degradation products. , achieves high sugar yields under mild reaction conditions, reduces solvent load and enzyme requirements, and reduces waste build-up [15].

The application of the basic type of fuzzy control with a closed-loop system formula is built and hidden in a discrete form, taking into account the input variables and output variables, sending set-point errors and performance indexes, respectively. The quadratic optimization problem

formulation is adopted to obtain a feedback controller output, which can bring about a slight change in the index number dynamically compensated by the set-point controller, which can be changed according to the changing conditions of the bottom layer [16]. Theoretically the reservoir tank controller, computer simulation were carried out with the reservoir tank controller, the numerical results obtained were used to validate the performance of the reservoir tank [17]. The concentration of ethanol that needs to be considered in deionized water and white wine in the fermentation process, by using an increase in the limit of detection, the results obtained show the limit of detection is 140 times better than the PCA method which is widely used in biochemical sensing systems [18]. Clostridia enzymes are used for the purification of bio-acetone and bio-butanol, impurity molecules in the form of bio-molecules, providing additional and sustainable benefits by applying Clostridia fermentation and increasing the yield of the next generation [19].

Starch is a more complex component than disaccharides, before the fermentation process, starch must be broken down using the amylase enzyme (which is abundant in germinated wheat) into disaccharide components, namely maltose. By using the enzyme maltase, maltose will be hydrolyzed into glucose.

$$(C_6H_{10}O_5)_n + 1/2n \xrightarrow{\text{E. Amylase}} 1/2n\ C_{12}H_{22}O_{11}$$

$$1/2n\ C_{12}H_{22}O_{11} + 1/2n\ H_2O \xrightarrow{\text{E. Maltase}} 1/2n\ C_6H_{12}O_6$$

The hydrolysis process is influenced by several factors, including the amount of starch content in the raw material, the degree of acidity (pH), the concentration of acid used, the hydrolysis time, the hydrolysis temperature, and the catalyst [20].

In the formation of bioethanol through the fermentation process, the role of microbiology is very large, and usually, the type of microbiology used greatly affects the fermentation process. Alcoholic beverages resulting from the fermentation process that is produced without distillation, usually have an alcohol content between 3–18%, to increase the alcohol content in the product, often the result of the fermentation process is carried out by a distillation process, and the resulting alcohol content is between 29–50% [21]. The reaction of the ethanol formation process with the fermentation process:

$$C_6H_{12}O_6 \xrightarrow{\text{Yeast}} 2\,C_2H_5OH + 2CO_2$$

The result of the fermentation process is usually a dilute alcohol solution because the yeast cells will die when the ethanol content exceeds 12-15%. The ideal result of the fermentation process is 51.1% ethanol and 48.9% carbon dioxide. The results of the optimum alcoholic fermentation process include Ethyl alcohol 48.8%, Carbon dioxide 46.6%, Glycerol 3.3%, Succinic acid 0.6%, Cellulose and so on 1.2% [22]. Other factors that influence the fermentation process include pH, a good pH for the fermentation process between pH 4-5 because good lactic acid is a good acid for yeast growth, but the bad thing is that butyric acid bacteria can grow, which can be detrimental to yeast growth [23]. The time required for the fermentation process depends on the temperature and concentration of sugar, in general, the time required is between 36-50 hours, generally, a good temperature for the fermentation process is between 25-30 ^{0}C, the lower the fermentation temperature the higher the alcohol content produced, at low temperatures will lose alcohol because it is carried away by carbon dioxide gas [24].

The results of the technical glucose and bioethanol levels experiment were sought optimal results using the Response Surface Methodology (RSM) method. RSM is a combined method of mathematical and statistical engineering, used to model and analyze a Y response influenced by several X-free variables to optimize the response.
The relationship between the Y response and the X-free variable is:

$$Y = f(X_1, X_2, X_k) + \varepsilon \quad (1)$$

Description:
Y is a response variable, f (X_1, X_2, X_k) are free variables/factors, and ε is an error.

The first step of RSM is to find a relationship between the response and the x-factor through the first-order polynomial equation and used linear regression models, otherwise known as first-order models (first-order models) are:

$$Y = \beta_o + \sum_{i=1}^{k} \beta_o X_i \quad (2)$$

The design of the first-order experiment suitable for the factor filter stage is the 2k factorial design (Two-Level Factorial Design). Second-order model, there is usually curvature and used polynomial models of the second-order whose squared functions are:

$$Y = \beta_o + \sum_{i=1}^{k} \beta_o X_i + \sum_{i=1}^{k} \beta_u X_i^{\,2} + \sum_{i=1,j=2}^{k-1,k} \beta_{i,j} X_i X_j + \quad (3)$$

Equations and optimization results are obtained using Minitab software. In this software, the optimal results will be shown using graphs as well as numbers of calculation results.

The application of the basic model on fuzzy controls with closed-loop system formulas is built and hidden in discrete form, taking into account input variables and output variables, each sent by set-point and performance index errors. Formulation of quadratic optimization issues was adopted to obtain a feedback controller output that could bring about slight changes in economic index numbers as well as the dynamic realization of set-point compensation by the controller, replaced according to the change in lower layer conditions [25]. The agitator tank controller theoretically, as a computer simulation is performed with the agitator tank controller, numerical results to validate the performance of the agitator tank, obtained superior results [26].

The concentration of ethanol is noteworthy in deionized water and white wine in its fermentation process, using the WSRM method focused on increasing the limit of detection, the results obtained show 140 times better the limit of detection than the PCA method, which is widely used in biochemical sensing systems [27]. Clostridia solvents are used for bio-acetone and bio-butanol purification, bio-based

impurity molecules such as chemical petro, providing additional benefits and ongoing benefits, by applying clostridia fermentation will increase yields on the production of the next generation of bio-molecules [28]. They produce maximum technical glucose (18 %v/v) with polynomial equations of the order of two, $y = -0.0028x^2 + 0.2226x + 13.2$ with proximity to the result $R^2 = 0.9611$, and bioethanol with optimization in the process of hydrolysis and fermentation using Minitab software with Surface Response. Chemical hydrolysis process using solid Na-OH with MS-20D digital magnetic stirring, fermentation process using turbo yeast [29].

From previous studies, to produce optimum glucose levels and pH by optimizing pH in the pretreatment process using Minitab software with Surface Response. In the chemical pretreatment process using tapioca flour liquid waste with variable (200-1000) ml and variable HCL (5-25) ml, and stirring time of 30 minutes using a digital magnetic stirrer MS-20D type. The next process is a filtration process to separate the filtrate and sediment. The filtrate obtained was then subjected to a hydrolysis process at a hydrolysis temperature of ± 40 ^{0}C, the addition of the Amylase enzyme as much as 12% w/v, and the Maltose enzyme as much as 9 % w/v to produce glucose levels between 20-25 %v/v. Optimization of pH using Response Method Surface showing similarities to Regression Equation, to produce an optimum pH of 7 to 8.

II. METHODOLOGY

The stages of making glucose from LW-TF with a variable (200–1000) ml were carried out with a pretreatment process with a variable H-CL (5–25) ml and a stirring time of 30 minutes using a digital magnetic stirrer MS-20D type, obtained a pH between 4.5-8,5. The next process is a filtration process to separate the filtrate and sediment. The obtained filtrate was then hydrolyzed at a hydrolysis temperature of ± 40 0C, the addition of Amylase enzyme as much as 11 %w/v, and Maltose enzyme as much as 9%w/v to remove glucose levels between 20-25 %v/v.

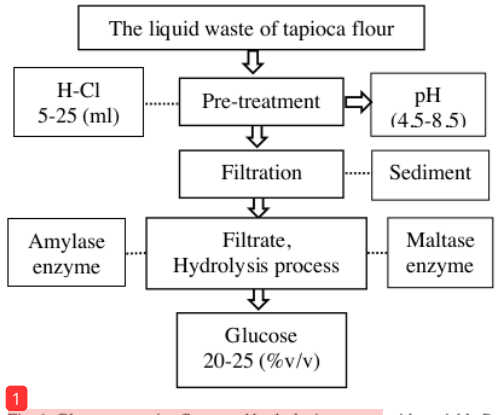

Fig. 1. Glucose contains flow used hydrolysis process with variable Pre-treatment

III. RESULTS AND DISCUSSION

In the hydrolysis process with a hydrolysis temperature of ± 40 ^{0}C, the stirring time is 30 minutes using a digital magnetic stirrer MS-20D type. In Table 1, the pretreatment process and hydrolysis process are carried out, where the pretreatment process with LW-TF is between 200-1000 (ml), H-Cl volume is 5-25 (ml), and pH is between 4.5-8.5. The optimum glucose levels were 24.9 %v/v, Amylase Enzyme 12 %w/v, Maltase Enzyme 9%w/v, with a volume of 600 ml LW-TF, 600 ml H-Cl addition, and a pH of 7.1. When compared with previous studies on the biological hydrolysis process using Bacillus, glucose levels were obtained from 3-9 (%v/v) [6].

TABLE 1. THE EFFECT OF LW-TF AND HCL VOLUME ON pH IN PRETREATMENT PROCESS

The volume of liquid waste of tapioca flour	**Volume H-Cl**	**pH**
(ml)	(ml)	
200	5	7.6
	10	7.4
	15	6.8
	20	5.4
	25	4.5
400	5	7.4
	10	7.1
	15	6.9
	20	5.6
	25	5.2
600	5	7.3
	10	7.2
	15	7.1
	20	5.8
	25	4.9
800	5	7.8
	10	7.7
	15	7.6
	20	6.1
	25	5.2
1000	5	8.5
	10	8.3
	15	8.1
	20	7.2
	25	6.3

TABLE 1 showed the change in substrate pH in each process. LW-TF dissolved in distilled water (substrate) has an initial pH of about 10, therefore variations of H-Cl are added to achieve a pH variation of 4.5 to 8.5. In the pretreatment process, the larger the volume of LW-TF, the smaller the addition pH. In volume variations of 200 - 1000 ml, with the addition of 5 ml of H-Cl volume, a pH increase in the range of 7.5 to 8.5 was obtained, and the addition of 25 ml H-Cl volume obtained an addition of pH in the range of 4.5 to 6.5. The greater the addition of H-Cl volume the more significant the decrease in pH. The final pH of the process has decreased due to the bleaching process with the addition of a little powdered activated charcoal and also due to the

filtration process which allows contamination from the environment.

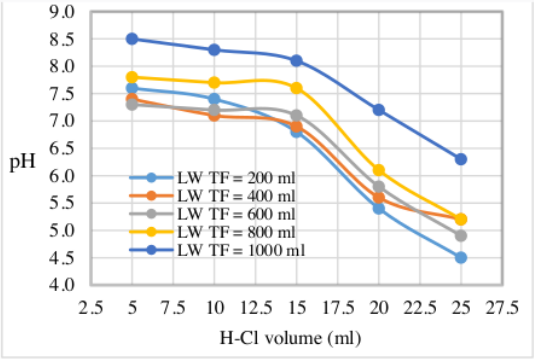

Fig. 2. Effect liquid waste of tapioca flour volume and HCL volume on pH

In Fig. 2, the effect of pH in the pretreatment process greatly determines the results of glucose levels in the hydrolysis process. For the LW-TF volume between 200-600 ml, the pH ranges from 4.5 to 7.5, with the optimum being at pH 7.1. Relatively significant in the linear and quadratic coefficients of the interactive effect coefficient. The real response data plotted against the predicted response is presented in Fig. 2. The predicted response was the optimum pH 7.1, the LW-TF volume was 600 ml, and the H-Cl volume was 15 ml. From the results of the pretreatment process, starch hydrolysis was used, the process of breaking down starch into simpler glucose structures, which was carried out enzymatically. In the starch hydrolysis process, there are several stages, namely gelatinization, liquefaction, and saccharification. At all stages using heating with a water bath so that the hydrolysis process takes place evenly. Enzymatic hydrolysis of starch (starch) aims to break down starch into simpler constituent parts such as dextrin, isomaltose, and glucose using enzymes. The starch used comes from tapioca flour solid waste which has a starch content of 76.055% consisting of 15.8429% amylose and 60.2121% amylopectin.

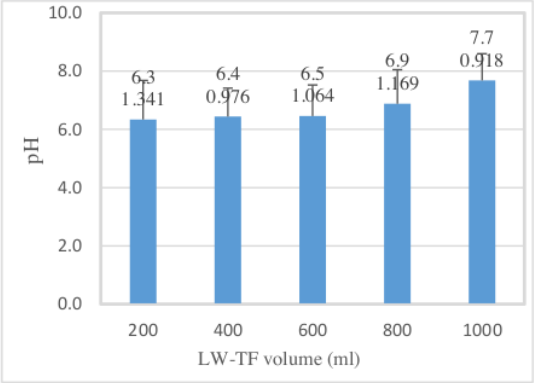

Fig. 3. The error bar on a liquid waste of tapioca flour volume variable to pH

In Fig. 3, it is shown that the smallest pH error bar of 0.918 is in the LW-TF volume of 1000 ml, and the largest pH error bar of 1.341 is in the LW-TF volume of 200 ml. The difference in pH between the average pH and the error bar from a volume variation of 200-1000 (ml) indicates a pH of about 5 to 6.5. The highest glucose level was found at pH 7.1 at 24.9 %v/v, the best pH for the pretreatment process was at pH 5.5. The optimal pH for the glucoamylase enzyme is 4-5 [20], while the optimum pH for glucoamylase enzyme activity is 4.5 [12].

Before entering the starch hydrolysis stage using enzymes, the cassava is gelatinized first. The gelatinase process for tapioca flour is 52-64 ^{0}C, so the gelatinization process on solid waste tapioca flour continues until the temperature of the substrate solution reaches about 50 ^{0}C, or when the substrate begins to thicken. The temperature of the substrate solution should not be too high, because if the temperature is too high then the substrate solution will become a very thick gel (like glue). If the substrate solution is very thick, it will complicate the liquefaction process, the added enzymes will not work optimally because there is a change in the structure of the starch which causes the substrate solution to become very thick. On the other hand, if the temperature at the time of gelatinization is too low, the starch swelling process will be limited because the amount of water absorbed is limited, the water absorbed is only 30% [29].

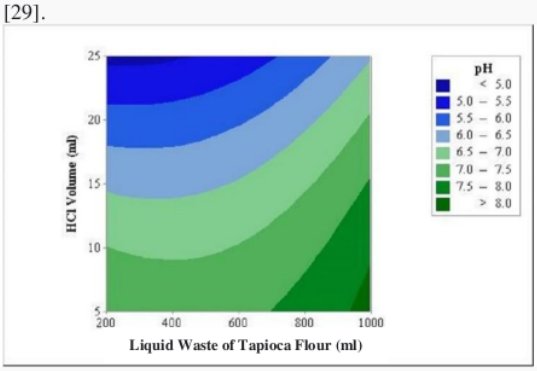

Fig. 4. Effect of volume of liquid waste of tapioca flour and volume of H-CL on pH to two-dimensional

Regression Equation in Uncoded Units:

pH = 8.30 - 0.00274 Liquid Waste of Tapioca Flour - 0.073 HCl Volume -0.000003 Liquid Waste of Tapioca Flour * Liquid Waste of Tapioca Flour - 0.00233 H-Cl Volume*H-Cl Volume + 0.000056 Liquid Waste of Tapioca Flour *H-Cl Volume

$pH = 8.30 - 0.00274\ X_1 - 0.073\ X_2 - 0.000003\ X_1^2 - 0.00233\ X_2^2 + 0.000056\ X_1X_2$

When:

X_1 = Wheat Flour Liquid Waste

X_2 = H-Cl Volume

The complex effect of the process variables ie, LW-TF volume (X_1), H-CL volume (X_2) on pH was investigated using a pretreatment process in a stirred tank to visualize the effect of independent factors on responses. RMS was used to determine the significance of the parameter regression coefficients. The coefficient with the larger the value of the LW-TF volume and the smaller the volume of H-Cl, the more significant it is for all variables of an interaction.

In Fig. 4 for two dimensions and Fig. 5 for three dimensions list the regression coefficients, values of X_1 and X_2 for all linear, quadratic, and interaction effects of the parameters. The coefficient of the linear and quadratic effect of the reaction time of LW-TF volume (X_1), H-CL volume (X_2) on pH (Composite Desirability = 1) is very significant as shown in Fig. 5 with the prediction of an optimum pH value of 8.84921, LW-TF volume of 1165.69 ml, and H-Cl volume of 0.857864 ml.

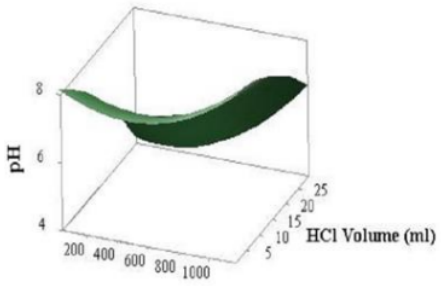

Fig. 5. Surface characteristics of contour plot response between the volume of liquid waste of tapioca flour and HCL volume on pH

Response Optimization: pH

Parameters

Response	Goal	Lower	Target	Upper	Weight	Importance
pH	Maximum	4.5	8.5		1	1

Solution

Solution	Wheat Flour Liquid Waste (ml)	HCl Volume (ml)	pH Fit	Composite Desirability
1	1165.69	0.857864	8.84921	1

Multiple Response Prediction

Variable	Setting
Wheat Flour Liquid Waste (ml)	1165.69
HCl Volume (ml)	0.857864

Response	Fit	SE Fit	95% CI	95% PI
pH	8.85	1.17	(6.07, 11.62)	(5.50, 12.20)

CONCLUSION

Optimum pH in the pretreatment process of pH 7.1, the LW-TF volume was 600 ml, and the H-Cl volume was 15 ml, and the stirring time of 30 minutes using a digital magnetic stirrer MS-20D type. Prediction using Response Method Surface an optimum pH value of 8.84921, LW-TF volume of 1165.69 ml, and H-Cl volume of 0.857864 ml. and showing similarities to Regression Equation: $pH = 8.30 - 0.00274\ X_1 - 0.073\ X_2 - 0.000003\ X_1^2 - 0.00233\ X_2^2 + 0.000056\ X_1X_2$ when X_1 = liquid waste of tapioca flour, X_2 = H-Cl volume.

ACKNOWLEDGMENT

The author would like to thank the financial support from the Ministry of Education, Culture, Research and Technology, the Republic of Indonesia with Applied Grants, Contract Number: 293/E4.1/AK.04.PT/2021.

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