In situ Autohydrolysis for the Glucose Production from Sago Pith Waste with DIC Technology

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Instant controlled pressure drop (DIC) technology was utilized in the production of glucose from sago pith waste (SPW). *In situ* autohydrolysis was conducted in a DIC reactor to obtain the maximum glucose production. The influence of pressure, acid concentration, and treatment time on the glucose yield from SPW subjected to DIC-assisted *in situ* autohydrolysis was determined, and the experimental conditions were optimized using the response surface method. The results showed that the linear term of acid concentration and the quadratic terms of pressure and time had a significant effect on the glucose yield. The optimized experimental conditions for maximum glucose production (48.21%) from SPW subjected to DIC-assisted autohydrolysis were a pressure of 0.1 MPa, acid concentration of 0.1 M, and time of 4 min. The findings demonstrated that DIC technology has the potential to be utilized for the commercial production of glucose from SPW.

Keywords: Instant controlled pressure drop; DIC technology; Autohydrolysis; Sago pith waste; Glucose production; Response surface methodology

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INTRODUCTION

The sago palm of the genus *Metroxylon* and belonging to the Palmae family (*Metroxylon sago* Rottb.) grows well in the swampy, tropical rain forest of Southeast Asia between 10° north and south of the equator (Karim *et al.* 2008). The sago palm is widely utilized for starch production due to several advantages, such as being economically viable, relatively sustainable, environmentally friendly, uniquely versatile, vigorous, and promoting socially stable agro-forestry systems (Nozaki *et al.* 2004; Karim *et al.* 2008). The sago starch reserve in the pith core of the sago palm stem accumulates to its largest amount just before flowering and fruiting, which depletes these reserves (Nozaki *et al.* 2004). Large quantities of sago pith waste (SPW) are generated from sago processing plants. A small sago processing plant may produce approximately 7 tons of SPW daily. In Malaysia, 50 to 110 tons of SPW are generated daily by the starch processing factories (Karim *et al*. 2008).

SPW is a fibrous starchy lignocellulosic biomass, which is produced from the sago palm after the extraction of starch. Generally, SPW contains about 58% starch, 23% cellulose, 9% hemicellulose, and 4% lignin (Jenol *et al.* 2014). SPW is a promising starchy lignocellulosic feedstock for glucose production. Unfortunately, most SPW is either dumped into nearby rivers or deposited in factory compounds for use as boiler feed. The improper disposal of SPW into rivers causes serious water pollution that increases chemical oxygen demand, biological oxygen demand, and organic pollutants (Karim *et al.* 2008).

Glucose production from lignocellulosic biomass involves pretreatment and glucose extraction. The main concern in commercial fuel production is finding an effective method that converts starch, cellulose, and hemicellulose into fermentable sugars and is energy efficient, economically feasible, and fast (Harish *et al.* 2015). Various pretreatment methods have been utilized for glucose production from lignocellulosic biomass, including physicochemical, physical, chemical, and biological methods (Thangavelu *et al.* 2014). However, the existing methods that are utilized for glucose production have various disadvantages, including large duration of time requirements, high expense, and poor yields (Awg-Adeni *et al.* 2013). The main problem in the effective utilization of SPW is its high moisture content, which is about 400 to 500% of the solid material. The moisture content can be decreased to 200 to 300% by adding a screw press step after final washing. Moreover, the production of glucose from SPW subjected to *in situ* autohydrolysis requires depolymerization of polymers, such as polysaccharides (*e.g.*, starch, cellulose, and hemicellulose), into their monomers (Thangavelu *et al.* 2014).

A successful chemical fractionation needs to transform the structural components of biomass into valuable chemicals and products. Depolymerization can be conducted by prehydrolysis (acidic media), steam explosion, or autohydrolysis, in which water reacts with the raw material (Hendriks and Zeeman 2009). Steam explosion (SE) pretreatment process treats the biomass materials structure hydrothermally. As an example of the SE process, lignocellulosic biomass may be taken into a reactor operating at a temperature of 28 °C and under atmospheric pressure. Subsequently, the reactor is pressurized to 1 MPa to 3 MPa with an increasing temperature up to 200 °C to inject the steam into the biomass material for a given duration. Then, the reactor is depressurized to atmospheric pressure at a temperature of around 100 to 120 °C (Pan *et al*. 2004; Sarip *et al.* 2011). The significant drawback of SE pretreatment of the lignocellulosic biomass is the possibility of generating phenolic compounds and other toxic derivatives, such as furfural and 5-hydroxymethyl-2-furfural due to the high degree of severity, which can inhibit the susequent yields of enzymatic or acid hydrolysis (Mounir *et al.* 2014). Autohydrolysis treats the lignocellulosic biomass at lower temperatures than steam explosion (Kumar *et al.* 2009). It is an environmentally friendly pretreatment process for lignocellulosic biomass because it does not require additional chemicals or catalysts. The autohydrolysis pretreatment process may convert hemicellulose into high-yield soluble saccharides, while rendering cellulose into a rich, extractable solid residue (Amendola *et al.* 2012). However, the autohydrolysis of biomass may cause hemicellulose degradation due to the elevated temperatures (170 to 210 °C) (Hendriks and Zeeman 2009). Thus, it is important to define a suitable pretreatment process for lignocellulosic biomass that is both economically viable and capable of large scale operation.

The basis of the détente instantanée contrôlée (DIC) (instant pressure drop) process is based on a thermo-mechanical process that requires a material with a certain moisture level to be treated (Allaf *et al.* 1992). The process starts by subjecting process materials inside the DIC treatment chamber to a vacuum, followed by pressurizing it under steam and rapid expansion towards a vacuum (vacuum 50 to 100 mbar, with a rapid valve opening time of approximately 0.2 s). The operating temperature inside the DIC reactor is usually below 200 °C, and the treatment time during high steam pressure ranges from seconds to minutes.

The rapid pressure drop ($\Delta P / \Delta t > 2.5 \times 10^5$ Pa/s) while the material is being heated causes bursting due to evaporation inside the material. This increases the potential for blows and breaks in the cell wall cavities (Louka *et al.* 2004). The degree of structural damage inside the materials depends strongly on the nature of the material, as well as parameters of the treatment.

A successful pretreatment process for lignocellulosic biomass implies the effective separation of the components and their successive exploitation (Kumar *et al.* 2009; Amendola *et al.* 2012; Wi *et al.* 2015). The selection of a superior pretreatment process strictly depends on the downstream utilization of pretreated lignocellulosic biomass (Beukes and Pletschke 2011). To overcome the disadvantages of existing processes, studies suggest treating the lignocellulosic biomass with a combination of pretreatment processes (Akhtar *et al.* 2016; Sarip *et al.* 2016). Therefore, the present study was undertaken to produce glucose from SPW treated with autohydrolysis following DIC pretreatment. This approach is justified by both technologies having comparable characteristics in terms of pressure and temperature. In fact, autohydrolysis pretreatment with DIC technology is based on subjecting raw material to saturated steam pressure at high temperatures (up to 160 °C) for a short period of time (a few seconds to 1 min) and then dropping the pressure abruptly (Allaf *et al.* 1992). This multi-process treatment involves a heat treatment of materials inside a reactor, which is followed by abruptly releasing pressure toward a vacuum (about 5 kPa). The sudden pressure drop rapidly cools the treated material. The auto-evaporation of water during the process may protect the sugars from thermal degradation. The resulting biomass expansion and texture change helps in breaking down cell walls to make the biomass components more easily accessible.

EXPERIMENTAL

Sample Preparation

The SPW was collected at the final washing stage from a sago palm processing factory in Batu Pahat, Johor, Malaysia. The material was in the form of biomass slurry and contained water. It was dried in open air for 2 days and packed in ziplock bags for transportation. The SPW was dried further in a convection oven with a bed depth of 4 cm at 45 °C for 12 h and then stored in double ziplock plastic bags at room conditions. The dried materials were kept in double ziplock plastic bags to minimize potential moisture uptake. The dried SPW was analyzed for its moisture content, particle size distributions, starch, and cellulose content as below:

Moisture content determination was done at 105 °C for 5 min with a Sartorius M30 device in accordance with the AOAC 930.15 method. Particle size distribution were conducted by sieving using vibratory FRITSCH Sieve Shakers. Then the particle size distribution was calculated as below,

$$Particle\ size\ (\%) = \frac{m}{M} \times 100 \tag{1}$$

where *m* is weight of SPW retain on respective sieve, and *M* is the total weight of SPW. Starch and cellulose content SPW was determined using HPLC following the method shown by Sun *et al.* (1999).

Glucose Production using DIC Technology

The experimental setup of the DIC pretreatment method is presented in Fig. 1. The DIC system consisted of three main elements, which were the treatment chamber, vacuum tank, and pneumatic valve. The pneumatic valve opened the connection between the treatment chamber and vacuum tank in less than 0.2 s to ensure an instant pressure drop in the treatment chamber. The material was placed in an aluminium container in a stainless steel holder and placed in the DIC reactor for treatment. The experiments were conducted according to the central composite design and followed a random sequence to minimize the effect of variability in observed response.

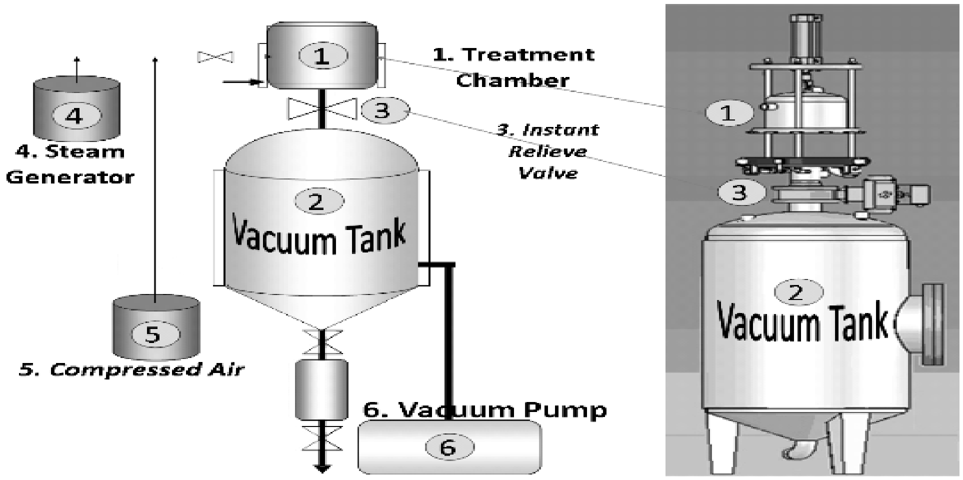


Fig. 1. Schematic diagram of the DIC system

Response surface methodology (RSM) with a central composite design was used in the investigation of the effect of DIC pretreatment on the autohydrolysis of sago pith waste (SPW) into glucose and low degree of polymerization (DP) starch polymers. The polynomial second degree quadratic equation for the RSM model was represented by the following equation,

$$Y = B_0 + \sum_{i=1}^{n} \beta_i X_i + \sum_{i=1}^{n} \beta_{ii} X_i^2 + \sum_{i \neq j}^{n} \beta_{ij} X_i X_j + \varepsilon \qquad (2)$$

where Y is the response, β_0 is a constant, β_i is the linear regression term, β_{ii} is the quadratic regression term, β_{ij} is the interaction regression term, n is the number of variables, and ε is random error. The data obtained from the experimental trials was numerically fitted into the equation using the commercial statistical software Statgraphics (Centurion XV, version 15.2.11, Warrenton, VA, USA). The response surface and contour plots for the model based on Eq. 1 were plotted as a function of any two variables at a time. The name of the variables and their level in the experiments are presented in Table 1.

Table 1. Coded and Uncoded Levels of the Independent Variables

Factor	Symbol	Level		
		Low (-1)	Intermediate (0)	High (+1)
Pressure (MPa)	X_1	0.089	0.275	0.461
Sulphuric acid (M)	X_2	0.04	0.06	0.08
Time (min)	X_3	2.08	3.25	4.42

Glucose Analyses

The glucose analyses were conducted to collect depolymerized starch and glucose after the SPW was pretreated by DIC, which used hot water at 110 °C and a solid to liquid ratio of 10:90 for 60 min. Two mL of hot water extract was placed in a centrifuge vial, closed, and centrifuged at 10,000 rpm for 10 min. One mL of a clear supernatant was carefully transferred into a glass vial for glucose content determination with a HPLC system. The Agilent 1100 HPLC system used a G1311A Quaternary Pump (Agilent Technologies, Santa Clara, CA, USA). ICE-COREGEL 87H3 (Transgenomic) column was used at 35 °C to separate glucose and certain organic acid in the solution. Wherein, 5 mM dilute sulfuric acid was used mobile phase at a flow rate of 0.5 mL/min. Finally, the extracted glucose concentrations were determined and expressed as a mg/g or percentage glucose production, as shown below,

$$Y({}^{mg}\!/_{g}) = \frac{W_{glue}}{W_{SPW}} \tag{3}$$

$$\%Y = \frac{W_{glue}}{W_{SPW}} \times 100 \tag{4}$$

where Y represents the yield of glucose separation from SPW, W_{glue} is the weight of glucose, and W_{SPW} is the weight of SPW.

Environmental Scanning Electron Microscopy Image Analyses

Environmental scanning electron microscopy (ESEM) was used to collect an electron micrograph of a sample with minimal sample preparation. This was done to increase the ability to inspect the actual elemental percentage (*i.e.*, without interference from coating element), type of material porosities, and effect of the thermal process on the biomass without any interference from conventional SEM sample preparation requirements. The ESEM detection was done at LASAT, La Rochelle, France and used a configuration under a low vacuum of 1.30 mbar with two detectors. A secondary electron detector (SED) was used for solid state or morphology detection, and a backscattered electron detector (BSED) was used to analyze the chemical/elemental composition with a maximum resolution of 2.5 nm at 20 kV. The ESEM was done with a FEI Quanta 200 FEG (Hillsboro, OR, USA) that was equipped with Oxford Inca Energy Dispersive X-ray (EDX) software (Oxford Instruments, High Wycombe, UK) for elemental analysis. In this work, the BSED micrograph was presented as it gives better contrast than the SED micrograph.

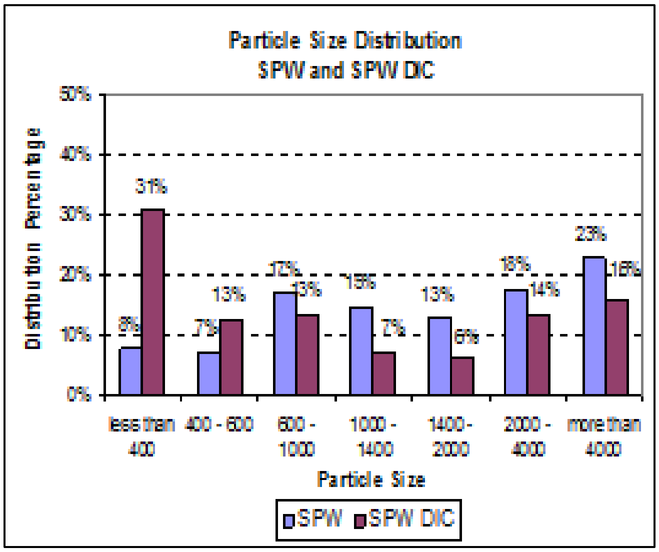
RESULTS AND DISCUSSION

The particle size distribution and composition of starch, cellulose, and residue of SPW are presented in Fig. 2. The particle size distribution of the materials was almost homogenously distributed within the 600 to 4,000 μm range, while the composition was approximately 70 to 75% total polysaccharides (starch, hemicelluloses, and cellulose).

The particle size distribution of the raw SPW (Fig. 2a) and DIC treated SPW (Fig. 2b) demonstrated that the DIC treatment increased the percentage of low particle size materials. The raw SPW contained 60% of particle size ≤ 2000 μm. After the DIC treatment, the percentage of particle size less than 400 μm increased from 8% to 31% and the percentage of particle size of ≤ 2000 µm increased to 70%. This was due to the decrease in size for the other particles. The decrease in size of the DIC-treated material was expected due to the thermal effects caused by DIC and the subtraction of material surface as caused by the acidic condition. These factors resulted in an overall lower size of particles (Maache-Rezzoug *et al.* 2011).

The composition analyses of the SPW indicated that the smaller particle size contained higher amounts of starch and cellulose (Fig. 2b). The highest amounts, about 60% starch and 15% cellulose, were detected at particle size 200 µm. The starch and cellulose contents of the SPW were comparable with other starchy lignocellulosic biomasses, such as wheat straw and wool fiber (Sun *et al.* 2005; Xu *et al.* 2006).

(a)



(b)

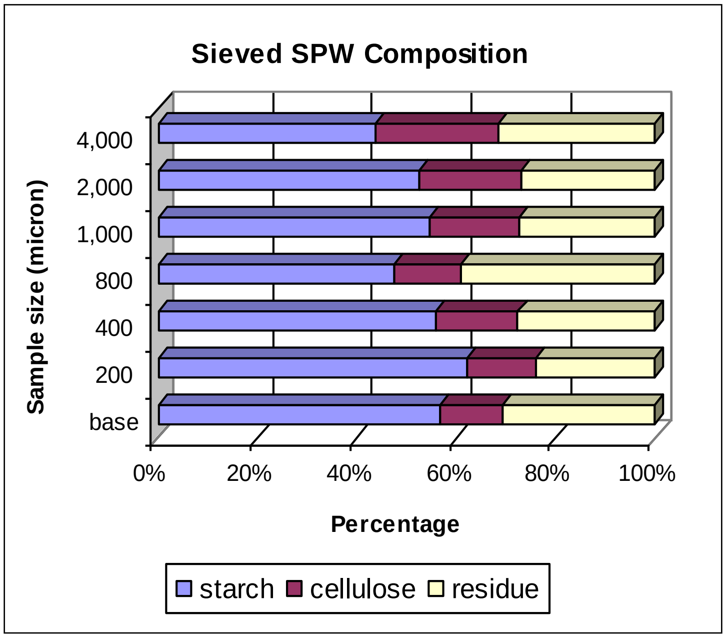


Fig. 2. Plot (a) shows particle size distribution (SPW raw and SPW DIC) and plot (b) shows the composition of total polysaccharides for raw SPW.

As mentioned earlier, the central composite design was utilized to determine the glucose formation from SPW treated by autohydrolysis and DIC technology. The experimental results on glucose formation (mg/g) and percentage glucose formation in various experimental conditions are presented in Table 2.

Table 2. CCD Experimental Design in its Coded and Actual Level with its Respective Glucose Response

No	Coded form			Y (Glucose production, mg/g)		
	X_1	X_2	X_3	Observed value	Predicted value	(%)
1	0	0	0	215.296	242.974	26.91
2	0	0	0	254.236	242.974	31.78
3	0	0	0	257.882	242.974	32.24
4	0	0	0	200.169	242.974	25.02
5	0	0	0	280.743	242.974	35.09
6	0	0	0	244.758	242.974	30.59
7	0	0	0	229.715	242.974	28.71
8	0	0	0	268.499	242.974	33.56
9	-1	-1	-1	24.284	38.7745	3.04
10	1	-1	-1	59.079	91.9375	7.38
11	-1	1	-1	220.389	197.232	27.55
12	1	1	-1	292.442	259.843	36.56
13	-1	-1	1	44.114	45.7688	5.51
14	1	-1	1	55.953	48.1659	6.99
15	-1	1	1	381.732	317.93	47.72
16	1	1	1	220.111	252.226	27.51
17	-1.682	0	0	39.378	112.677	4.92
18	1.682	0	0	196.880	167.342	24.61
19	0	-1.682	0	17.634	24.3193	2.20
20	0	1.682	0	357.293	394.369	44.66
21	0	0	-1.682	38.988	75.1794	4.87
22	0	0	1.682	132.296	139.866	16.54

Table 3. Analysis of Variance of Glucose for Fitted Model

Source	Sum of Squares	Df	Mean Square	F-value	P-Value
X_1:Pressure	3607.16	1	3607.16	1.66	0.2217
X_2:Acid	165298.	1	165298.	76.13	0.0000
X_3:Time	5050.95	1	5050.95	2.33	0.1531
X_1^2	20529.9	1	20529.9	9.45	0.0096
X_1X_2	2318.87	1	2318.87	1.07	0.3218
X_1X_3	8232.37	1	8232.37	3.79	0.0753
X_2^2	2190.07	1	2190.07	1.01	0.3351
X_2X_3	653.556	1	653.556	0.30	0.5933
X_3^2	35528.9	1	35528.9	16.36	0.0016
Total error	26056.6	12	2171.38		
Lack-of-Fit	20831	5	41662	476	0.056
Pure Error	4373	5	874.7		

$R^2 = 90.24$ %; $R^2(adj)$ = 82.92%

Cells with grey color indicate significant values.

It was observed that about 30% of the total polysaccharides were converted into glucose at the central point’s combination (pressure 0.275 MPa, acid concentration 0.06 M, and time 3.25 min). The highest glucose formation, approximately 48%, occurred at 0.275 MPa, 0.08 M, and 4.42 min. This exposed that the glucose yield depended on low severity DIC combinations.

The second order polynomial response surface model (Eq. 1) was fitted to each of the response variables (X). Regression analysis and ANOVA were used to fit the model and to determine the statistical significance of the term. The degree of significance of each factor was determined by the estimated regression coefficients of the quadratic polynomial model for the response surface. The degrees of significance and the regression coefficient of determination (R^2) are presented in Table 3. In addition, the R^2 *(adj)* value was estimated in order to determine model accuracy. As shown in Table 3, the variable with the most significant effect on the glucose yield was the linear term of acid concentration. Additionally, the quadratic term of pressure and time had a significant effect on the extraction glucose yield. The regression consideration of the *p*-value had a confidence level of $\alpha = 0.05$.

The regression coefficient of determination, R^2, is the proportion of variation in the response characteristic. The R^2 value should not be less than 0.80 for a well fit model, as lower R^2 values shows the inability of the model to explain the relation between variables (Hossain *et al.* 2015). Further, a large value of R^2 does not always imply the adequacy of a model. Therefore, it is important to predict the R^2 *(adj)* value of a model, which is more capable of evaluating the model adequacy (Davoudpour *et al.* 2015). Moreover, the term ‘lack of fit’ is an indication of the failure of a model. A significant ‘lack of fit’ for a model indicates the response predictor of the model is discarded. The results obtained from the ANOVA and regression analysis indicated the R^2 and R^2 *(adj)* values are 0.9024 and 0.8292, respectively (Table 3). The higher R^2 and R^2 *(adj)* values (< 0.80 and 0.90, respectively) indicated that non-significant terms were not added to the model. Moreover, an insignificant ‘lack of fit’ of the model (Table 3) for the studied variable indicated that the model was sufficiently accurate for predicting the relevant responses. The comparison between the actual values and the predicted value obtained from the regression model displayed that the polynomial model was in agreement with the experimental data (Table 2). Thus, the model utilized in this study was able to determine the optimal operating condition for the selective glucose production from SPW treated with DIC-assisted *in situ* autohydrolysis.

The Pareto chart shows that the main factor, acid concentration (B), and the quadratic factors of pressure (A) and time (C) were statistically significant for the glucose production in DIC pretreatment process (Fig. 3). The acid concentration had the largest effect that resulted in a positive effect to the yield of glucose. The quadratic factors of pressure and time had a significant negative effect on the glucose production. The magnitude of the increments in glucose yield for the main factors and quadratic factors is presented in Fig. 4. As can be seen in Fig. 4, the glucose production slightly increased with pressure and time until a certain level and then decreased thereafter. The glucose production significantly increased with acid concentration (Fig. 4a).

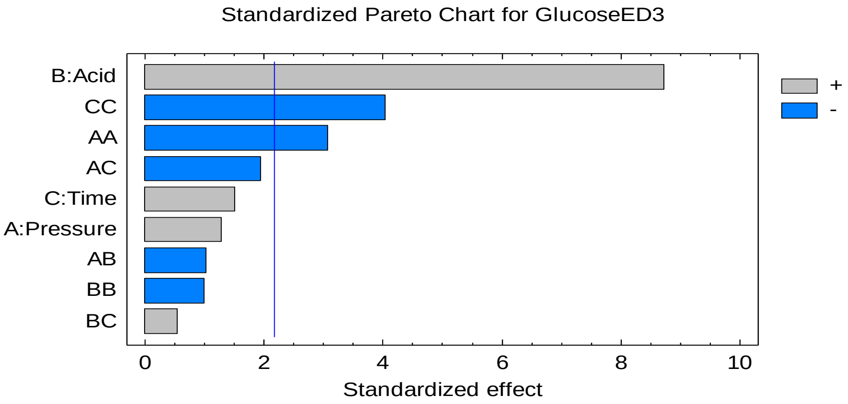


Fig. 3. Pareto chart for the effect of treatment factor on glucose response

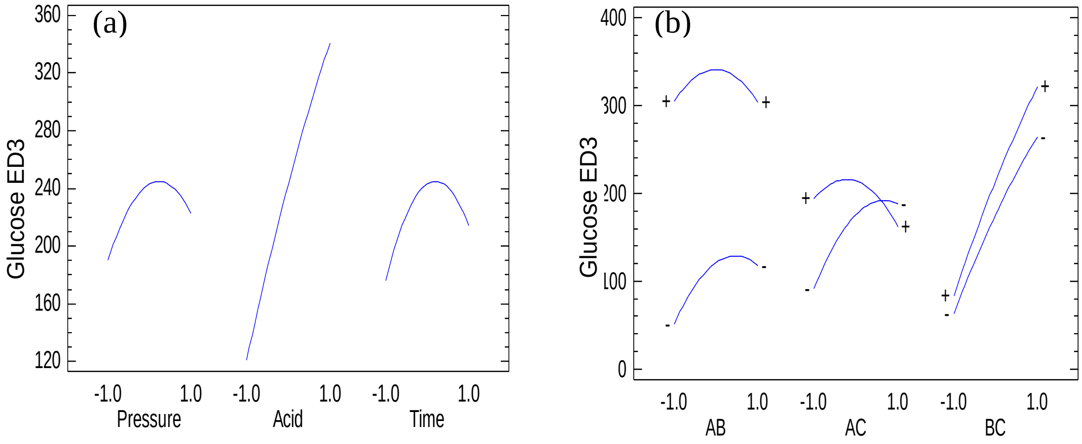


Fig. 4. Factor influences on the glucose prodcution from SPW treated with DIC-assisted autohydrolysis. (a) Main effects, and (b) Interaction effects

The relationship between dependent and independent variables and their effect on the glucose yield from SPW is illustrated in three dimensions by the response surface plots. The data were generated by keeping one variable at their respective intermediate level and varying the other two variables within the studied experimental range. The response surface was visualized using the glucose yield in relation to the pressure, acid concentration, and time in Fig. 5.

Figure 5a denotes the contour and response surface plot of the glucose yield from SPW as a function of pressure and acid concentration at a time of 3.25 min. Pressure had a negligible effect on the glucose yield. The acid concentration showed a positive effect, while the interaction between pressure and acid concentration had an insignificant effect on the glucose production from SPW treated with the DIC method followed by autohydrolysis (Table 3). However, the significant quadratic effect of pressure indicated that there was an influence due to pressure on the glucose production from SPW. Figure 5b shows the contour and surface plot for the interaction effects of pressure and time at a constant acid concentration of 0.06 M.

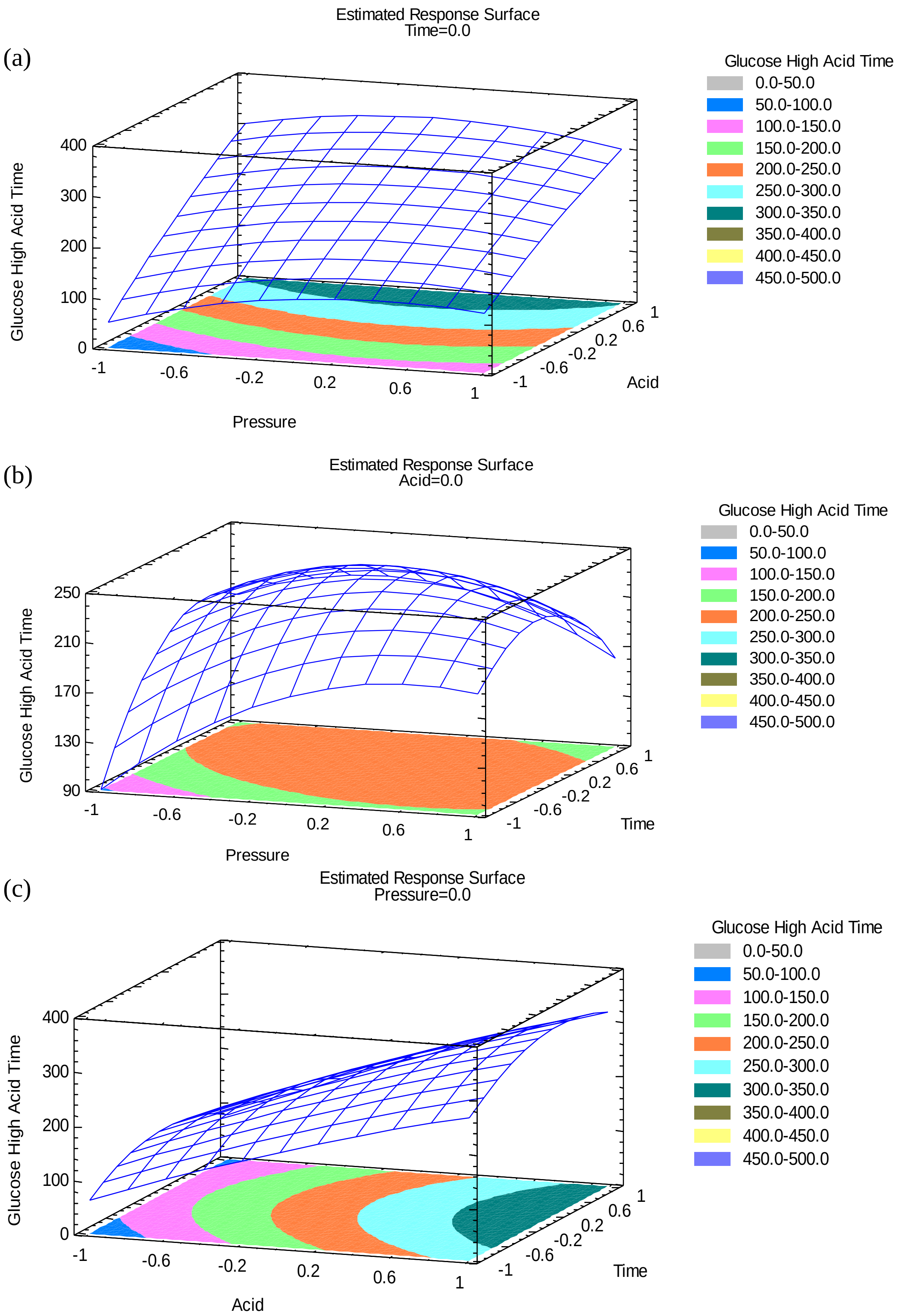


Fig. 5. Estimated response surface and contour plot for the glucose production from SPW subjected to DIC-assisted autohydrolysis. (a) Pressure *vs*. acid, (b) pressure *vs*. time, and (c) acid *vs*. time

The pressure increase from 0.089 M to 0.3 M increased the glucose yield, and thereafter the glucose yield decreased with further increased pressure. However, the insignificant interaction effect between pressure and time revealed that the increase of pressure and time over the studied range might not increase the glucose yield. Figure 5c shows the influence of the acid concentration and time at a constant pressure (0.275 MPa) on the glucose yield from SPW treated with DIC-assisted autohydrolysis. The acid concentration time showed a linear effect on the glucose yield, wherein time had a negligible influence on the glucose yield from SPW. The insignificant interaction between acid concentration and time (Table 3) indicated that the increased acid concentration at higher extraction times might not increase glucose yield. Based on the study, the optimized experimental conditions for the maximum glucose production (48.21%) from SPW subjected to DIC-assisted autohydrolysis were determined using the statistical software Statgraphics and were indicated to be a pressure 0.1 MPa, an acid concentration of 0.1 M, and a time of 4 min.

The mechanism of starch conversion to glucose that was suggested involved two steps of conversion before glucose could be obtained as a final product (Kumar *et al*. 2009). Because two conversion steps means a longer time requirement, the results for lower treatment severity may suggest that an increase in treatment time will increase the glucose yield and, subsequently, there is a correlation between acid and treatment time. This combination was utilized during higher treatment severity to further improve the yield of glucose. At higher treatment severity, certain treatment conditions, such as acid concentration, were high enough to influence the two conversion steps. Together with longer treatment time, a higher yield of glucose can be expected. The enzymatic conversions of starch into lower DP are also affected by the composition of starch (amylose:amylopectin ratio) (Sun *et al*. 2005). In the case of crude sago starch, several granular starches remained intact after the high severity DIC treatment combination, which is presented in Fig. **6**6.

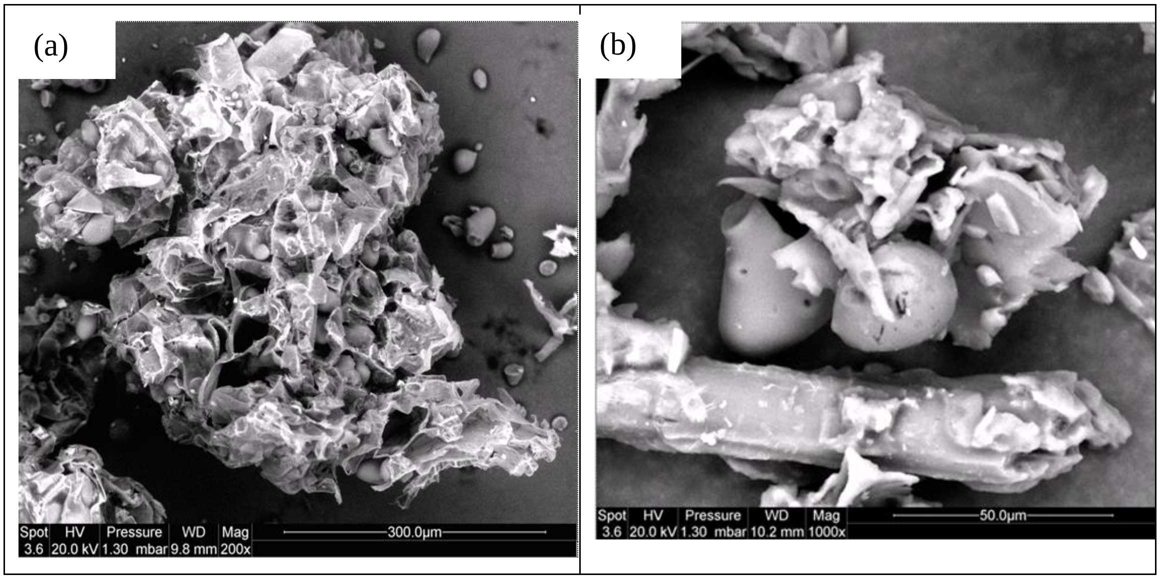


Fig. 6. ESEM of (a) crude starch and (b) SPW treated with DIC-assisted autohydrolysis at optimized experimental conditions

This result suggested that the starch granules may behave like a resistant starch, which is a similar type of starch used in a human diet. This may indicate the existence of resistant starch granules in native sago starch. However, the SEM micrograph of the treated SPW showed the existence of new granular materials. Presumably, this was lignin being deposited on the surface of cellular materials upon exposure of the SPW to high temperatures.

CONCLUSIONS

1. The composition analyses of SPW showed that smaller particle sizes contained higher amounts of starch and cellulose. The highest amounts, about 60% for starch and 15% for cellulose, were detected at particle size 200 μm.
2. The highest glucose formation, about 48%, was detected at a pressure of 0.275 MPa, an acid concentration of 0.08 M, and a time of 4.42 min, which indicated that the glucose yield depended on low severity DIC combinations.
3. The regression analysis demonstrated that the second order polynomial response surface model fit the experimental data.
4. The variables with the most significant effects on the glucose yield were the linear term acid concentration, followed by the quadratic term of pressure and time.
5. The optimized experimental conditions for the maximum glucose production (48.21%) from SPW subjected to DIC-assisted autohydrolysis were determined to be a pressure of 0.1 MPa, an acid concentration of 0.1 M, and a time of 4 min.
6. The SEM micrograph of the treated SPW showed the existence of new granular materials, which was presumably lignin being deposited on the surface of cellular materials upon exposure of the SPW to high temperatures.

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