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RESEARCH ARTICLE

Coconut husk to reducing sugar conversion using combined ultrasound and surfactant aided subcritical water

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OBJECTIVES The first purpose of this study was to investigate the effect of operating variables and surfactant concentration in subcritical water after ultrasonic process on the sugar-producing yield from coconut husk. The second purpose was to obtain the optimum operating condition of the subcritical water process. METHODS The sonication before subcritical water process was done by dispersing 40 mesh coconut husk powder in water at 60°C, and 35 kHz. The effect of sonication time was studied by comparing the material crystallinity and composition after being treated for 30 minutes. In this research, the optimization was done by using a Box-Behnken response surface methodology (RSM) experimental design with 3 factors (temperature, time, and surfactant concentration). The designed lower and upper levels were 130°C and 170°C, 40, and 80 minutes, as well as 1 and 3% (w). RESULTS The results showed that the quadratic response surface model predicted the maximum reducing sugar yield to be 12.0%, which was achieved at the optimum condition of 170°C, 77.5 minutes, and 2.3% SDS surfactant addition. CONCLUSIONS The experiment run at the obtained optimum condition resulted in a reducing sugar yield of 11.7%, which was close to that obtained from the model prediction.

KEYWORDS sonication; subcritical water; surfactant; lignocellulose; box behnken.

1. INTRODUCTION

For centuries, the vast majority of the energy used in the world has come from fossil fuels processed in petroleum industries. However, the continuously increasing consumption of fossil fuels had produced large amounts of carbon dioxide that exacerbates global warming. It is therefore necessary to look for alternative industrial raw materials and green methods to produce energy (Edenhofer and Kalkuhl 2011; Escobar et al. 2009). Biofuel is considered to be a viable alternative to fossil fuels in terms of both environmental and economic considerations.

Lignocellulosic biomass can be used as an alternative energy source to reduce dependency on fossil fuel. Despite that, lignocellulose biomass can be converted into a variety of environmentally friendly chemical products (Zhang et al. 2015). Coconut husk is one of the abundant lignocellulosic biomass that contains cellulose by 26.72% and hemicellulose by 17.73% (Sangian et al. 2015) . This high content of cellulose and hemicellulose, makes the coconut husk potential to be converted into reducing sugars and fermented into biofuels as a substitute for fossil energy (Sangian et al. 2015). However, sugar production from coconut husk is challenged by its recalcitrant because of its high lignin content, which is 41.19% (Muharja et al. 2018).

Subcritical water hydrolysis (SWH) technology has been identified as a viable alternative for breaking down the lignocellulosic structure of biomasses (Abaide et al. 2019). This technology has the potential to convert cellulose and hemicellulose into sugar and products (Vedovatto et al. 2021). Subcritical water is essentially liquid water in the boiling point temperature range to the critical point (100–374°C) and pressure higher than its vapor saturation pressure (Prado et al. 2014). The process is categorized as an environmentally friendly lignocellulosic pretreatment process because it only

TABLE 1. Level of parameters in the box-behnken design.

Parameter	Level		
	Low level (-1)	Medium level (0)	High Level (+1)
Temperature (°C)	130	150	170
Time (minute)	40	60	80
Surfactant	1%	2%	3%

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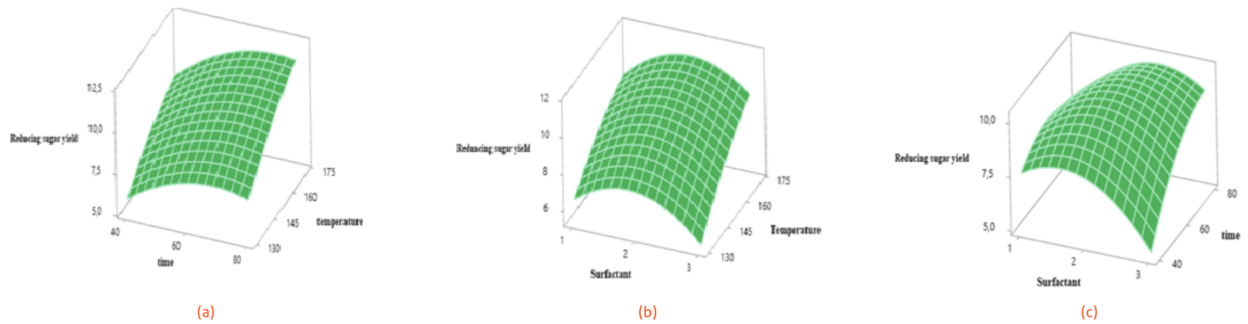


FIGURE 1. Response surface plot: (a) reducing sugar yield vs temperature, and time; (b) reducing sugar yield vs temperature, and surfactant concentration; (c)reducing sugar yield vs time, and surfactant.

TABLE 2. Chemical composition of coconut husk before and after pretreatment.

Composition (%)	Before	After
Cellulose	36.1031	39.7320
Hemicellulose	21.6300	20.3660
Lignin	34.2655	30.0040
Ash	0.1326	0.2724

uses water as a solvent, does not produce solvent residues, and exhibits less sugar-lowering when compared to other methods (Prado et al. 2016). Despite that, the subcritical water process has also been reported to be efficient in obtaining reducing sugar (Liang et al. 2017).

The lignocellulose degradation process in subcritical water can be increased with the assistance of ultrasonic waves (sonication methods). Ultrasonic is an emerging new technology that is potential as an alternative pretreatment technology. The cavitational effects of ultrasonic waves can damage the structure between cellulose, lignin, and hemicellulose, making the production of reducing sugar easier (Bussemaker and Zhang 2013; Wongsorn et al. 2010). Hapsari et al. (2015) reported that pretreatment using ultrasound had enlarged the surface area of bagasse and changed the structure of the cellulose to be more amorphous. Moreover, most of the hemicellulose content was degraded into sugar and the length of processing time could increase the conversion of cellulose. The increase in lignin loss percentage was reported by the work of Yin et al. (2014) who compared the sugar production using supercritical CO_2 (SCCO2) and its combination method with sonication on corn cobs and corn stalks. However, this method has the disadvantage of producing adverse derivate products such as furfural and phenolic compounds that act as inhibitors in subsequent processes that cause the yield of reduced sugars to decrease (Jönsson and Martín 2016).

A promising way to overcome the difficulty in the lignocellulose conversion into high-reducing sugar through subcritical water is to add surfactant to the process. Surfactants have hydrophilic and hydrophobic properties that can reduce surface tension between two liquid phases during the process. In the enzymatic hydrolysis process, the hydrophobic properties improve cellulose conversion by reducing the sugar by blocking non-productive adsorption performed by lignin (Qing et al. 2010). Muharja et al. (2019) reported that the addition of surfactants to the subcritical water process resulted in greater sugar yield than when added to the enzymatic process. The authors reported that SDS surpasses the two other used surfactants (PEG and Tween 80) in increasing the reducing sugar yield of the subcritical water process.

Despite the potential in producing reducing sugar, there is still no information about the optimum condition concerning this sugar production through a combined process of ultrasonication and subcritical water technology. Therefore, this study focuses on obtaining the optimum operating conditions for the subcritical water process in producing reducing sugar from ultrasonic preprocessed coconut husk. Despite that, the effect of the combination of ultrasound and surfactant aided subcritical water technology on reducing sugar yield as well as on the solid characteristics will be elucidated in this work.

2. MATERIALS AND METHODS

2.1 Materials

Coconut husk was obtained in Manado, North Sulawesi, Indonesia. It was washed using tap water and then dried under the sunlight. The material was milled and screened to obtain a particle size of 40 mesh. The chemicals used were surfactants Sodium Dodecyl Sulphate or SDS (> 98% Sigma, Aldrich China), 3,5-dinitrosalicylic acid (> 98%, Sigma Aldrich, USA), sodium hydroxide (> 99%, Sigma Aldrich, USA), sodium potassium tartrate (99-102%, Merck, Germany), sodium metabisulfite (> 99%, Sigma Aldrich, USA).

2.2 Procedures

2.2.1 Ultrasound process

The ultrasonic process was carried out using a bath-type reactor (Ultrasonic Cleaner Bath Elma LC-20H, Germany). The operating frequency and power of the ultrasonic bath were 35 kHz and 100 W. In each of the experiment runs, 150 mL beaker glass that contained 6 g coconut husk was dispersed in 120 mL deionized water. The suspension was sonicated at 600°C for 30 minutes.

2.2.2 Subcritical water process

Subcritical water was carried out using a high-pressure stainless-steel reactor. The process was run under batch mode. The batch reactor configuration is the same as that used in the previous work (Ju et al. 2011). The experimental system includes a reactor, a high-pressure carbon dioxide (CO_2) tube tank, valve pressure regulator, heater, temperature controller, and pressure gauge. The suspension

TABLE 3. Design experiment box behnken.

Std order	run order	Temperature (Level)	Time (Level)	Surfactant (Level)	Reducing sugar yield (%)
1	3	-1	-1	0	5.8644
2	1	1	-1	0	9.1203
3	11	-1	1	0	7.5376
4	8	1	1	0	11.8014
5	12	-1	0	-1	6.4620
6	5	1	0	-1	9.6233
7	13	-1	0	1	5.5992
8	10	1	0	1	10.1591
9	2	0	-1	-1	7.6708
10	3	0	1	-1	7.4997
11	6	0	-1	1	5.0936
12	15	0	1	1	9.2779
13	9	0	0	0	10.2917
14	7	0	0	0	9.4794
15	4	0	0	0	10.0626

TABLE 4. Anova of experimental results of box behnken design (BBD).

Source	Glucose regression model calculation				
	DF	Adj SS	Adj MS	F-Value	P-Value
Model	9	57.1206	6.3467	77.55	0.000
Linear	3	38.9881	12.996	158.8	0.000
Temperature (β_1)	1	29.8329	29.8329	3646	0.000
Time (β_2)	1	8.9924	8.9924	109.9	0.000
Surfactant (β_3)	1	0.1628	0.1628	1.99	0.217
Square	3	12.4963	4.1654	50.9	0.000
temperature*temperature (β_{11})	1	0.5893	0.5893	7.2	0.044
time*time (β_{22})	1	3.566	3.566	43.57	0.001
surfactant*surfactant (β_{33})	1	9.5856	9.5856	117.1	0.000
2-Way Interaction	3	5.6362	1.8787	22.96	0.002
temperature*time (β_{12})	1	0.2609	0.2609	3.19	0.134
temperature*surfactant (β_{13})	1	0.5024	0.5024	6.14	0.056
time*surfactant (β_{23})	1	4.8728	4.8728	59.54	0.001
Error	5	0.4092	0.0818		
Lack-of-Fit	3	0.0488	0.0163	0.09	0.959
Pure Error	2	0.3604	0.1802		
Total	14	57.5297			

from the sonication bath was added to the reactor. Afterward, CO_2 was supplied to the reactor until the reactor pressure reached 60 bar. The CO_2 gas was chosen instead of N_2 because as the CO_2 is solubilized in high-pressure water, it would form carbonic acid which could act as a hydrolysis catalyst (Gurgel et al. 2014). The reactor temperature was then set according to the run variable. The reaction time is set as zero as soon as the desired temperature has been reached. At the end of the reaction, the reactor was immediately cooled down to 30°C by immersing it in cold water and the pressure was released instantaneously using the ball valve. The extracted sample was then filtered using filter paper, and the solid residue was then washed with deionized water. It was then dried in the oven at 60°C for 2 days until constant weight and stored for analysis.

2.2.3 Design of experiments

Subcritical water process variables (temperature, time, and surfactant concentration) combinations were designed using response surface methodology (RSM) to obtained maximum reducing sugar yield. The experiments were conducted based on three factors Box–Behnken design. The experimental variables were studied at two levels (–1, 0, and +1). The lower level was at 130°C, 40 minutes, and 1% (w) SDS, while the upper one was at 170°C, 80 minutes, and 3% (w) SDS, and the medium level was at 150°C, 60 minutes, and 2% (w) SDS. The level of parameters in the design Box-Behnken and the randomized run combinations are represented in Table 1 and Table 2.

TABLE 5. Model summary statistics.

S	R-sq	R-sq(adj)	R-sq(pred)
0.282219	99.29%	98.01%	97.23%

TABLE 6. Predicted and Actual Yield with Optimum Value of Each Variable.

Variable s			Yield sugar (%)		Error%
Temperature (°C)	Time (min)	Surfactant (%)	Predicted	Actual	
170	77.5758	2.3737	12.0611	11.7004	2.9906

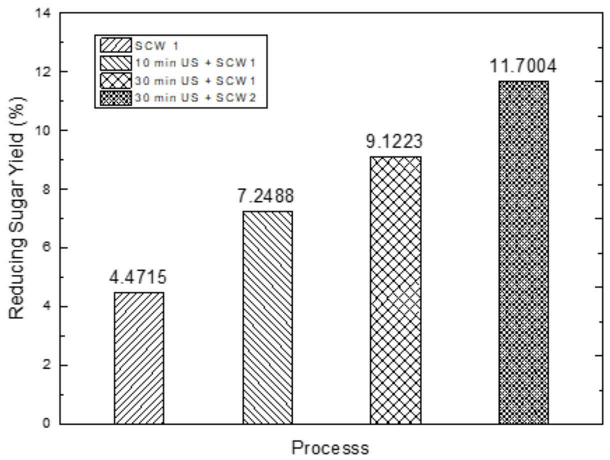


FIGURE 2. Comparison of reducing sugar yield (US = ultrasonication at 60°C; SCW 1 = subcritical water process at 150°C and 60 minutes; SCW 2 = subcritical water process at 170°C, 77.5758 minutes and 2.3730% SDS).

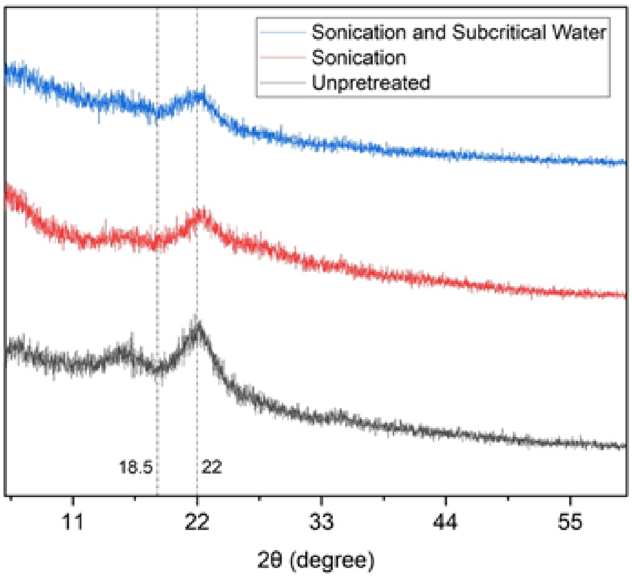


FIGURE 3. XRD pattern of coconut husk: a) unpretreated (black); b) after 30 min sonication (red) c) after sonication & subcrtical water (blue).

2.2.4 Analytical methods

The solid residues and liquid fractions were thoroughly examined. To assess the concentration of TRS, a Vis-Spectrophotometer (CECIL 1001, Cambridge, UK) was used to quantify the liquid fraction from SCW (Miller 1959). The structure and morphology of the solid fraction following SCW pretreatment were investigated using the Chesson method, Scanning Electron Microscopy (SEM), Fourier Transform Infrared Spectroscopy (FTIR), and X-Ray Diffraction (XRD) assay. SEM images were performed using the scanning electron microscope EVO®LS 10 (Carl Zeiss Micro Imaging GmbH, Göttingen, Germany). Fourier transform infrared (FTIR) spectroscopy (FT/IR MODEL 4200 JASCO, Tokyo, Japan, and Nicolet iS 10 FT-IR spectrometer, Waltham, Massachusetts, USA) is used to characterize the chemical bonds of solid components. X'Pert PRO XRD (PANanalytical BV, Netherlands) was used for measuring the diffractogram of the samples involving the used radiation from Cu Kα, with 40 kV and 30mA electric current. The rate was 2 degrees per minute using a scanning angle 2θ of 5–60. The crystallinity index (CrI) values were calculated using the diffractogram using the following formula 1:

$$CrI(\%) = \frac{I_{002} - I_{am}}{I_{002}} x100 \quad (1)$$

Where I_{002} is the highest intensity for lattice diffraction and I_{am} is amorphous diffraction intensity. Those values of intensity were obtained after subtraction of the background signal.

3. RESULTS AND DISCUSSION

3.1 Effect of sonication on chemical composition

Table 2 describes the differences in lignocellulose composition before and after the ultrasonic process. As seen on the table, lignin composition decreased after this pretreatment, indicating that the pretreatment using ultrasonic has slightly removed lignin and increased cellulose composition. The hemicellulose content of the sample decreased from 21.6300% to 20.3660% after pretreatment. The percentage decrease was 5.83%. This decrease indicates that the pretreatment not only causes degradation of lignin, but also depolymerization of hemicellulose (Saha and Cotta 2008). This is because hemicellulose is a short, shapeless polymer chain that is more readily soluble in water (Gírio et al. 2010). Correspondingly, the study of coconut husk by (Subhedar and Gogate 2014) showed that sonication can reduce lignin in coconut husk by 80% using ultrasonic-assisted alkaline pretreatment. In this work, the lignin content percentage decrease was only 12.43%. The results of the lignocellulosic content differ from the literature in this regard because it is influenced by the pretreatment condition used. In the literature, the ultrasonic device used was a probe sonicator while in this work a bath-type sonicator was used.

3.2 Optimization of the subcritical water

Surface methodology response based on Box-Behnken Design (BBD) is used to optimize operating parameters in the process of subcritical water. The method employs a quadratic relationship between the response variable and factors as seen in Equation 2.

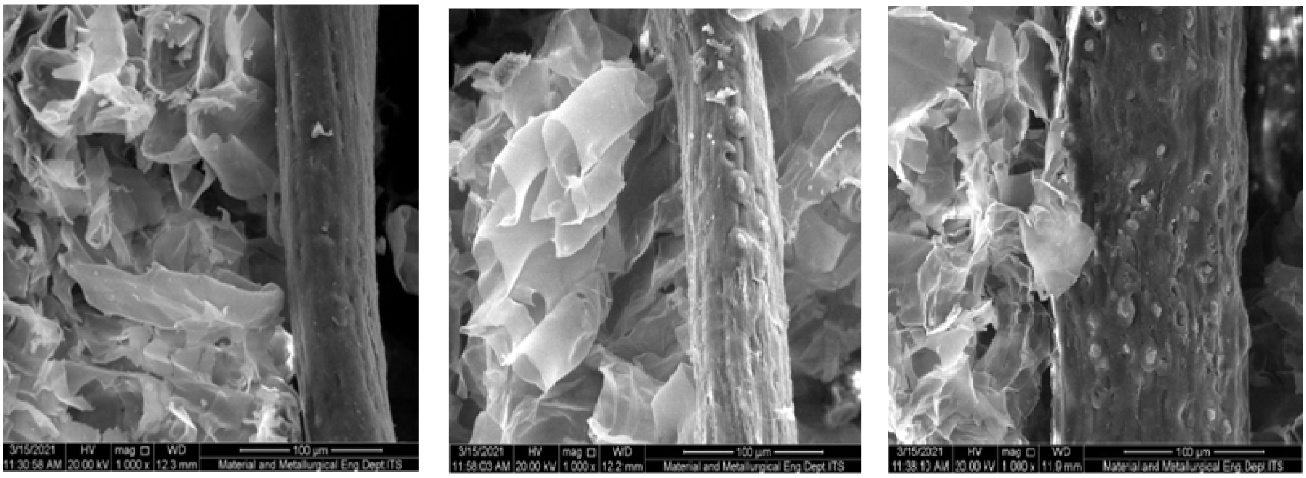
$$Y_M = \beta_0 + \sum_i \beta_i X_i + \sum_i \sum_{j,j \neq i} \beta_{ij} X_{ij} + \sum_i \beta_{ii} X_{ii}^2 \quad (2)$$

Where Y_M = reducing-sugar yield obtained from the model (%), β_0 = equation constant, β_i, β_{ij} and β_{ii} are coefficients for single, interaction, and quadratic effects of the designed experiment factors. In this study, the response variable is the reducing sugar yield which is calculated according to the following formula shown in Equation 3.

$$Y = \frac{C_{RS} \times V}{M_{CCH}} 100\% \quad (3)$$

Where Y= reducing-sugar yield calculated from the experiment results (%), CRS = concentration of reducing sugar obtained from colorimetry measurement (g/L), V= slurry volume (L); MCCH = initial mass coconut coir (g).

The randomized run combinations design Box Behnken are represented in Table 3 and Table 4 presents the ANOVA



(a) (b) (c)

FIGURE 4. SEM analysis with 1000x on coconut husk: (a) Raw coconut husk, (b) 30 minutes sonication, (c) sonication and subcritical water.

TABLE 7. Crystalline index of original and treated coconut husk.

Sample	I002		IAM		CrI(%)
	2θ	Int	2θ	Int	
Raw coconut husk	22.9897	155.1980	19.0625	50.2223	67.6400%
Sonication 30 min	23.2069	85.3355	18.5445	21.1493	75.2163%
Sonication and subcritical water + 2% SDS	22.9061	72.3527	18.7116	17.9992	75.1230%

of quadratic model regression of the experimental results. Based on the table, the model can represent the experimental result as the p-value of the model is less than 0.05. Factors that are more influential on the yield response of reducing sugar are temperature and operating time, this can be seen from the P-value, where a lower P-value indicates that the factor is more significant (Pan et al. 2006). The surfactant concentration has no linear effect on the response, indicated by the p-value higher than 0.05. However, it has a strong quadratic effect and interaction effect with temperature and time as the p-values on those parameters are higher than 0.05. These ANOVA results prove that the yield of sugar in the subcritical water process is influenced by the operating conditions of temperature, reaction time, and surfactant concentration where reaction time is the most influential variable. This is similarly explained in a previous study by (Cardenas-Toro et al. 2014) that a long residence time increases the formation of sugar. Testing the suitability of the model was done by using the lack-of-fit test to give meaning to the suitability of the selected model. From the results of the analysis of the data table above, it is found that the lack-of-fit is the p-value of 0.959 which is greater than 0.05 (P-value > 0.05). This indicates that the model made is appropriate. The Model summary and statistics are given in Table 5. The R-squared values (R^2) of the model is 99.29%, which indicates that the model adequately represents the real relationship between the variables considered. This also means that 99.29% of the variability could be explained by this model and about only 0.71% of the total variation cannot be explained by this model. The R-square value is very close to the R-adjusted value which is 98.01%. The RSM method was used to determine the optimum level of each variable for maximum response and to investigate the interaction between the important reaction parameters. Figure 1a showed the interaction between two variables (time and temperature) and their effects on the response variable (yield sugar). It shows that time

TABLE 8. FTIR Absorption bands for raw, 30 min sonication processed, sonication and subcritical water processed coconut husk.

Wave Number (cm^{-1})			Vibration	Functional group	Biomass component
Raw coconut husk	Sonication	Sonication and Subcritical water			
3342.47	3340.65	3305.09	O-H (Stretch)	Phenolic, alcoholic, carboxylic	Lignin
2927.03		2919.74	C-H (Stretch)	CH_2, CH_3	Lignin, Cellulose
1605.44	1612.82	1601.4	C=C (Stretch)	Aromatic ring	Lignin
1509.26	1510.48	1513.05	C=C (stretch)	Aromatic ring	Lignin
1424.79			C–H deformation		Celullose
1241.77	1243.57	1231.45	C-O-C, C–O (stretch)	Lignin, polyssaccharides	Hemicellulose, cellulose, lignin
1024.4	1029.97	1028.09	C–O (stretch)	Lignin, polyssaccharides	Hemicellulose, cellulose, lignin
523.45		507.58	-	-	-
472.32		468.32	-	-	-

and temperature of subcritical water were found to have a significant effect on the reducing sugar yield where reducing sugar yield increases with a further increase in these two variables. Figure 1b shows the effect of temperature and surfactant concentration on the reducing sugar yield where the the closing operation condition to the middle level of the temperature and surfactant operating conditions, the higher the yield of sugar produced. These show that the operating condition variable affects the sugar yield. Based on Figure 1c, it can be seen that the optimum surface for yield is in the middle. This indicates that the closer the condition to the middle of the operating time and surfactant concentration, the higher the yield of sugar produced.

Table 6 presents the optimization result of the subcritical water process condition which was carried out using Minitab 19 software. The optimum result is at 170°C, 77.5758 minutes, and a surfactant concentration of 2.3738%. The predicted reducing sugar yield at this condition is 12.0611%. An experiment was carried out to validate the optimum conditions and the yield of reducing sugar obtained was 11.7004%, which did not differ much from the predicted value (< 5%).

3.3 Effect of coconut husk processing on the reducing sugar yield

The reducing sugar concentration produced after ultrasonic, subcritical water, and their combination were analyzed using the DNS (Dinitrosalicylic acid) method (Sangian and Widjaja 2017). Figure 2 shows the yield of reducing sugar obtained from several process conditions. The SCW 1 process stands for subcritical water process carried out at a temperature of 150°C for 60 minutes without sonication. The 10 min US + SCW1 process is 10 minutes of sonication followed by subcritical water at 150°C and 60 minutes. The 30 min US + SCW1 process is for 30 minutes sonication followed by subcritical water at 150°C and 60 minutes. The 30 min US + SCW2 for 30 minutes sonication was followed with subcritical water at 170°C, 77 minutes, and a surfactant concentration of 2.373%. Figure 2 explains the difference in yield of reducing sugar produced with different variations in operation. In addition, the sugar yield increased from 4.4715% to 7.2488% this was due to the addition of ultrasonic pretreatment which opened the lignocellulosic structure of coconut coir thereby increasing the sugar yield. The highest yield of reducing sugar is the result of optimization with operating conditions when the temperature is 170°C for 77.5758 minutes with a surfactant concentration of 2.373%, which is 11.7004%. Muharja et al. (2018) research found subcritical water operating conditions at 80 bar, 150 °C, 60 minutes with a sugar yield of 14.71%, while the Prado et al. (2014) study found sugar yield of 11.70% at operating conditions of 208 °C, 200 bar, 30 min. This result could be caused by an increase in temperature, an increase in the ionization constant of water at high temperatures, where the concentration of H^+ and OH^- increases, thus facilitating the hydrolysis of cellulose and hemicellulose into monosaccharides (Zhu et al. 2011). The increase in the concentration of reducing sugars can also be caused by the formation of surfactant micelles in subcritical water conditions. The hydrophilic groups of micelles can facilitate cellulose and hemicellulose to dissolve in subcritical water. This performance is instantly supported by hydrophobic interactions that reduce the lignin component (Chang et al. 2016). This is also similarly explained by a previous study, (Cardenas-Toro et al. 2014). Cardenas-Toro et al. (2014) that a long residence time in subcritical water can increase the formation of sugar.

3.4 Sample characterization

The effect of Combined Ultrasound and Surfactant Aided Subcritical Water Technology was studied to improve the reducing sugar yield. Therefore, it is very important to investigate the structural changes that occur after treatment using SEM Analysis (Scanning Electron Microscopy), FTIR (Fourier Transform Infrared Spectroscopy), and XRD (X-Ray Diffraction).

3.4.1 XRD (X-Ray Diffraction)

XRD method was used to determine the crystallinity index of lignocellulosic coconut husk before pretreatment and after ultrasonic and subcritical water pretreatment. Changes in the values of the crystallinity index (CrI) indicate the effect of the process. Table 7 and figure 3 describe the crystallinity index value (CrI) of coconut husk solids that have not been pretreated with solids that have been pretreated with ultrasonic and solids that have been treated using a combination of ultrasonic and subcritical water. The crystal index value (CrI) of real coconut husk solids is 67.640%, which is lower than that of coconut husk that has been pretreated with sonication for 30 minutes, which is 75.2163%. The increase in crystal index value after sonication is caused by the reduction of amorphous lignocellulosic materials such as some hemicellulose and lignin has been lost, so that only crystalline cellulose remains (Subhedar and Gogate 2014) . The crystallinity index value (CrI) of coconut husk solids that had been pretreated using a combination of sonication and subcritical water was 75.1230%, which is lower than using the sonication process only. This decrease in crystallinity indicates that the process can cut the cellulose chains of break down the intramolecular and intermolecular hydrogen bonds. This degradation causes changes in the crystalline and amorphous regions. This process occurs repeatedly until the degradation of cellulose is achieved according to the operating conditions (Zhao et al. 2009). The results of XRD analysis showed that ultrasonic pretreatment and subcritical water had reduced lignin by destroying its amorphous structure which was indicated by an increase in the crystallinity value of coconut husk.

3.4.2 SEM analysis (Scanning Electron Microscopy).

SEM is used to compare the morphological changes of coconut husk before and after ultrasonic pretreatment, and the morphological changes of coconut husk after subcritical water treatment using surfactants. Figure 4a is a SEM result for coconut husk that has not been pretreated. Coconut husk structure before being treated looks long, smooth, still organized, tight, and strong. This is because cellulose is still firmly encased with hemicellulose and lignin. Figure 4b shows the results of SEM analysis for the coconut coir after pretreatment with sonication at 600°C for 30 minutes. Figure 4b shows some parts of the coir were damaged which are shown by rough and blister formation on the surface due to the cavitation effect. The bubble collapses during cavitation and releases a large amount of energy that can damage the coconut

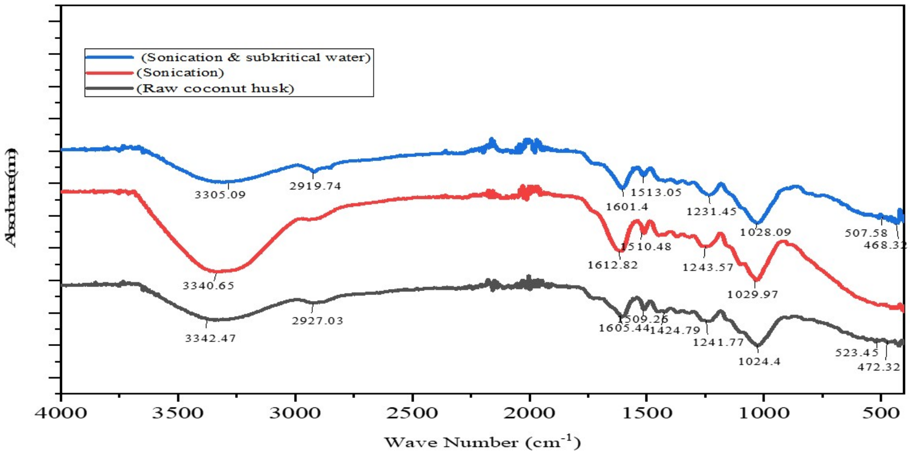


FIGURE 5. FTIR spectra of coconut husk: a) Raw coconut husk, b) 30 minutes sonication, c) sonication and subcritical water

coir. Figure 4c shows the structure of coconut husk after being processed with ultrasonication and subcritical water with surfactant addition. The figure shows a more damaged surface which is shown by a rougher surface with many blisters and holes. This indicates that the lignocellulosic complex compounds have been destroyed (Sangian and Widjaja 2017). This structural change plays a role in producing high sugar in the subcritical water hydrolysis process, because the crystallinity has decreased. These structural changes identify that sonication and subcritical water pretreatment efficiently destroy lignocellulosic cell walls

3.4.3 FTIR (Fourier Transform Infrared Spectroscopy)

Figure 5 shows the FTIR spectra of treated and untreated coconut coir. The spectra show almost similar bands as explained in Table 8. A strong and broad hydrogen hydrogen-bonded (O-H) stretching absorption present in phenolic, alcoholic, and carboxyl functional groups around 3342.47 cm^{-1}, has the same intensity and shape for all of the treatments tested. The band at around 2927 cm^{-1}, which corresponds to the C-H stretch of the CH_2, CH_3 functional group, presents in unpretreated, and combined sonication and surfactant aided SCW treated coir (Jiang et al. 2013). This indicated that the lignocellulose O–H and C–H bonds were reduced after the initial treatment was applied (Muharja et al. 2020). Vibration peaks in the range 1605.44-1601.4 and 1509.26-1513.05 indicate the presence of vibrations in the aromatic ring of lignin. Vibration in the ranges 1241.77-1231.45 cm^{-1} and 1024.4-1028.09 cm^{-1} in the sample showed the formation of C-O crystalline bonds and stretching cellulose and asymmetrical hemicellulose C-O-C (Xu et al. 2013). Changes in spectra after treatment indicate the presence of a delignification process. There is dissolving of hemicellulose and cellulose. FTIR analysis showed the presence of delignification and solubilization of hemicellulose and cellulose after pre-treatment.

4. CONCLUSIONS

The optimization of the subcritical water process has been carried out using the Box-Behnken design to determine the optimum process parameters that provide high yields of reducing sugars. The optimum operating conditions for the subcritical water process were found at a temperature of 170 °C for 77.1717 minutes with a surfactant concentration of 2.353%. The operating conditions that dominantly affect reducing sugar yield are temperature and time. Physicochemical characterization using SEM, XRD, and FTIR, had been carried out and the results revealed some changes between coconut husk before and after being processed using sonication, and subcritical water.

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