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Impact of surfactant-aided subcritical water pretreatment process conditions on the reducing sugar production from oil palm empty fruit bunch

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Impact of surfactant-aided subcritical water pretreatment process conditions on the reducing sugar production from oil palm empty fruit bunch

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Abstract. Oil palm empty fruit bunch (OPEFB), waste produced from the palm oil mills, has not been intensely utilized while its abundant availability in tropical countries such as Indonesia. It is one of the potential lignocellulosic material sources which can be utilized to produce sugar. The subcritical water process has been known as a promising lignocellulose-to-sugar conversion process. This study examined the effects of temperature, surfactant types, and concentrations on the sugar yield from OPEFB through the subcritical water process. In this research, the subcritical water process was conducted at a varied temperature of 140-180 °C, constant pressure and time of 60 bar, and 60 minutes, respectively. The types of surfactants studied were non-ionic, anionic, and cationic (Tween 80, SDS, and CTAB). Reducing sugar concentration analyses and solid characterizations using SEM, XRD, FTIR, and TGA were carried out in this research to evaluate the impact of the subcritical water process variables. The experiments showed that Tween 80 addition to the subcritical water process produced significant sugar yields at temperature 140 °C. The addition of CTAB to the subcritical water process shows adverse effects, while that of SDS showed support in the sugar production yields. This may be caused by hydrophobic and hydrophilic interactions between functional groups of the lignin unit and the hydrophobic and ionic groups of the surfactants. The subcritical water process run at a temperature of 180 °C assisted by SDS is the best scenario to produce the highest reducing sugar yield of 4.034%.

1. Introduction

Oil palm empty fruit bunches (OPEFB), a waste from palm oil mills, have not been fully utilized, while their availability is abundant in tropical countries such as Indonesia. Oil palm empty fruit bunches are one of the solid waste biomass of oil palm plantations which contain cellulose of 23.9-25.1%, hemicellulose of 23.5%, lignin of 7.4%, and 2.05% of oil [1]. In fact, OPEFB is one source of lignocellulosic material that has the potential to be processed sequentially to produce sugar through acid and enzymatic hydrolysis. In addition to having high cellulose and hemicellulose content, OPEFB waste also contains a significant lignin component. In the biofuel such as bioethanol and biohydrogen production process, the high lignin content inhibits substrate penetration of enzymes and microbial growth, which causes a decrease in sugar and biofuel yields [2].

Using chemical methods, pretreatment technology to reduce lignin content and cellulose crystallinity and increase sugar yield has been carried out [3,4]. Acid or alkaline solutions are often used in the pretreatment process because of their low cost and simplicity. However, another problem arises the need to neutralize the reaction medium after the process produces wastewater [5]. One of the pretreatment

technologies currently being developed is the subcritical water (SCW) process, which has shown destroying lignin efficiently while protecting cellulose and hemicellulose. However, the subcritical water process has also been recognized as a promising sugar-producing process.

The production process through subcritical water still has shortcomings that immediately must be addressed because it produces derivative products in high severity conditions that can inhibit further processing [6,7]. Several studies aimed at increasing the sugar yield have been carried out, but in this research, the innovation is by adding surfactants to the SCW process. Surfactants have unique characteristics, namely hydrophilic and hydrophobic groups that can reduce the surface tension between the two liquid phases during the process so that surfactants can bind to lignin components and their degradation products, which causes an increase in the conversion of polysaccharides into sugars [8].

With these properties, the addition of surfactants (non-ionic, anionic, and cationic) in the SCW process is expected to help reduce lignin and its degradation products during the subcritical water process and assist the dissolution of hemicellulose in water to become monomeric sugars. Several researchers have conducted research and showed the positive effect of surfactants addition. The addition of Polysorbate 80 (Tween 80), sodium dodecyl sulfate (SDS), and hexadecyltrimethylammonium bromide (CTAB) each increased the sugar yield in the pretreatment using alkali, acid, and hydrothermal. [9–11]. Although research on the effect of adding surfactants on the pretreatment process has been carried out, in several previous studies, the addition of anionic (SDS), cationic (CTAB), and non-ionic (Tween 80) surfactants in subcritical water processes involving OPEFB has never been studied and discussed in detail. Therefore, this study aimed to compare the effect of surfactants' addition in the SCW process to the sugar produced in SCW to study the interaction effect of the addition with the SCW process condition.

2. Materials and method

2.1. Materials

Oil palm empty fruit bunches are obtained from plantation waste oil palm in Pasaman, West Sumatra, Indonesia. The Empty Fruit Bunch was sundried, milled, and screened to obtain 150 mesh OPEFB powder. The surfactants used in this research were hexadecyltrimethylammonium bromide (>98%, Sigma Aldrich, USA), Sodium dodecyl sulfate (>98% Sigma, Aldrich China), and Tween 80 (>98%, Merck, Germany). Citric acid monohydrate (99.5 - 100.5%, Merck, Germany) and trisodium citrate dihydrate (99-101.0%, Merck, Germany), 3,5- dinitro salicylic acid (>98%, Sigma Aldrich, USA), sodium hydroxide (>99%, Merck, Germany), sodium metabisulfite (>99%, Sigma Aldrich, USA), and potassium sodium tartrate tetrahydrate (99-102%, Merck, Germany). This research also uses high purity carbon dioxide (CO_2) (PT. Samator, Sidoarjo, Indonesia) as a gas compressor.

2.2. Subcritical Water Experiment

Based on sample weight, six grams of OPEFB, 120 mL deionized water, and surfactant (1-3% w/w) were fed into the reactor. The compressing gas CO_2 was flowed into the reactor until the desired pressure was achieved. Afterward, the temperature was set and kept constant using a temperature controller device. The SCW process was carried out in batch mode for 60 minutes, where time zero was set as the desired temperature had been reached. Finally, the reactor is cooled to room temperature by immersing in cold water. Samples, treated with SCW, were filtered using filter paper (Whatman, UK) to separate the solids and the filtrate. The solids were dried in an oven at 60 °C for two days. All of the experiment was conducted in duplicate.

2.3. Experiment Design and Statistical analysis

The experiment was designed using general full factorial using three factors and three levels. There were 27 runs, and each was carried out in duplicate. Determine the significance level of surfactant addition to the SCW process by using Analysis of Variance (ANOVA) on Minitab 16 statistical software (Minitab Inc., ITS Surabaya, Indonesia). The significance level (α) used is 0.05. The reducing sugar yield is calculated by the following equation [12]:

$$RSY = \frac{mass\ of\ reducing\ sugar\ obtained\ (g)}{mass\ of\ initial\ OPEFB\ (g)}\ x\ 100\% \quad (1)$$

2.4. Chemical Analysis and Characterization of native and pretreated OPEFB

In this study, the reducing sugar concentration analysis was performed by colorimetry method using DNS (3,5-dinitro salicylic Acid) reagent [13]. The absorbances of the samples were measured using UV-Vis Spectrophotometer (Cecil 1100, UK). Sample characterization for heat treatment was observed using Thermogravimetric Analysis (TGA) (TGA/DSC1, Mettler Toledo, Columbus, USA). All solids that have been treated with SCW and those that have not been treated with SCW were characterized by X-ray diffraction (XRD) and Scanning Electron Microscopy (SEM). XRD analysis was performed using X'Pert PRO, Netherlands. The analysis used radiation from Cu Kα, with 40 kV and 30mA electric current. The rate used was 2 degrees per minute using scanning angle 2θ of 10-60°. The crystallinity index (CrI) values were calculated using data from XRD analysis according to the already described procedures [14]. The formula for crystallinity index (CrI) is as follows [15]:

$$Crl\ (\%) = \frac{I_{002} - I_{am}}{I_{002}}\ x\ 100 \quad (2)$$

Where I_{002} is the highest peak intensity of the crystal fraction I_{am} is defined as the low-intensity peak in the amorphous region. SEM images were used to visualize the morphological differences of the solids. Solid morphology was performed using SEM Evo MA 19 (Carl Zeiss, UK). Functional group differences between the OPEFB prior and following the subcritical water process were examined through the result of Fourier Transform Infrared Red (FTIR) was used to determine the difference in functional groups between OPEFB before and before the subcritical water process (FTIR MODEL 4200 JASCO, Tokyo, Japan, and Nicolet iS 10 FT-IR Spectrometer, Waltham, MA, USA).

3. Result and discussion

3.1. Composition of Oil Palm Empty Fruit Bunch (OPEFB)

Analysis of the chemical content of OPEFB fiber using the AOAC [16] method was carried out by the nutrition laboratory of Airlangga University, Surabaya, Indonesia. The high content of holocellulose (cellulose and hemicellulose) from OPEFB indicates that these carbohydrates' cellulose and hemicellulose content has the potential to be a source of sugar that is ready for further processing. Table 1 shows that the values of OPEFB content in this study are close to the analysis results in other studies. The differences in content levels are very slight because the varieties of oil palm used are different.

Table 1. Proximate analysis of the native OPEFB.

Carbohydrate %	Protein %	Oil %	*Water* %	Ash %	Reference
77.92	5.42	3.35	5.36	7.95	[17]
83,04	3.19	2.14	5.18	6.45	[18]
75.58	4.95	2.05	5,73	11.69	This Study

3.2. Subcritical Water of OPEFB

The OPEFB process with a subcritical reactor aims to destroy lignin because the lignin component in OPEFB can inhibit the sugar production from cellulose and hemicellulose. In the enzymatic hydrolysis process, the destruction of the lignin structure, cellulose, and hemicellulose contained in lignocellulose will be more easily accessible by enzymes so that the hydrolysis process will run more easily and a larger reducing sugar will be produced [19]. However, in this paper, the subcritical water process was focused on sugar production. In this process, water's physical and chemical properties change drastically, such as decreasing the dielectric constant and density, while the ionization constant increases

with increasing temperature. In this condition, water decomposes into H_3O^+ and OH^-, which makes the water can be used as an organic solvent to hydrolyze OPEFB [20].

The addition of SDS significantly affected the concentration of reducing sugar (p<0.05) and reached the optimum at 180 °C at a concentration of 3%. Under these conditions, the accessibility of water for hydrolysis of hemicellulose and cellulose is increasing. SDS surfactants can help remove lignin through the hydrophobic interaction mechanism between the lignin fraction and the hydrophobic portion of SDS surfactants in water systems. Firstly, cellulose and hemicellulose compounds are hydrolyzed into monomeric sugars [2]. Afterward, SDS forms micelles consisting of a liquid-forming core surrounded by a hydrophilic surface layer composed of a sulfate molecule head group [21]. The hydrophobic side of SDS interacts with the hydrophobic side of lignin consisting of phenyl, CH_2, and CH_3 through ether bonds [22]. Furthermore, the hydrophilic side of SDS, which is a sulfate group, can attract and dissolve glucose and xylose [23].

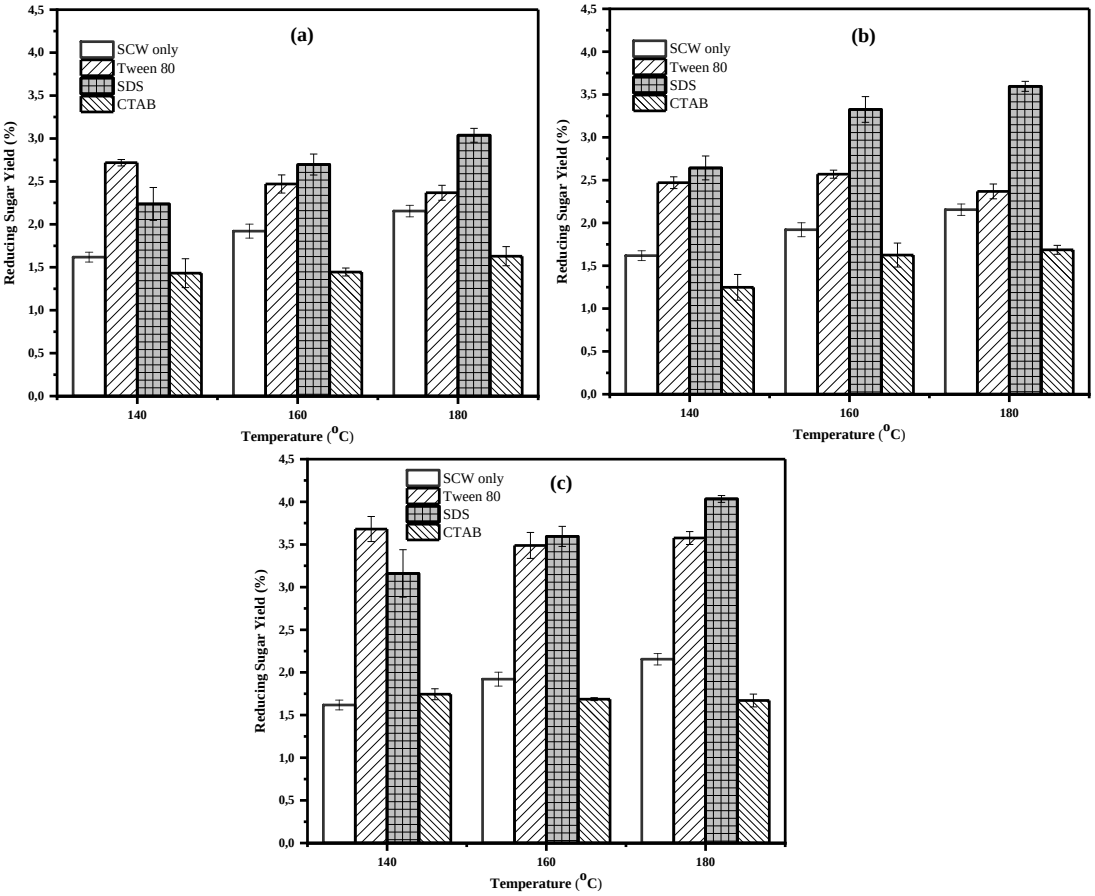

Figure 1. Sugar yields from the effect of temperature during the subcritical process of water on the surfactant concentration (a) 1%, (b) 2%, and (c) 3%.

The effect of the Tween 80 addition also showed that the sugar obtained was increased compared to that without surfactant. (see figure 1. a and c) that at a temperature of 140 °C, the sugar concentration produced is higher than 160 °C and 180 °C. Under these conditions, it can be assumed that an increase in temperature of more than 140°C causes the surfactant molecules to be degraded to become inactive [24]. The solubility of non-ionic surfactants depends on the balance between the capacity of the hydrophilic groups to attract water. Nonionic surfactants tend to cloud over when the temperature rises. The CTAB surfactant addition on the subcritical process resulted in an adverse effect on reducing sugar production, even when compared to the subcritical water process without surfactant addition at all temperature variations. This is because it has reached the cloud point ≥140. In addition, with the addition of CTAB, the glucose concentration was lower than without the addition of surfactant. There was a further reaction possibility that made the pH of the solution tend to be lower than in SDS due to the

presence of H^+ ions released at high-temperature heating. The element that causes a sour taste is H^+ ions; if the concentration of hydrogen ions (acidity) increases, the pH will decrease, and the HMF content will be higher. This is caused by an increase in heating temperature resulting in a faster rate of HMF formation.

3.2.1. Effect Surfactant in Subcritical Water Process.

Sugar production in subcritical water processes increases with temperature. This happens because of the water autocatalytic at high temperatures and for a long time [7]. The addition of surfactants (non-ionic, anionic, and cationic) was carried out in the subcritical pretreatment process to help to increase lignin removal.

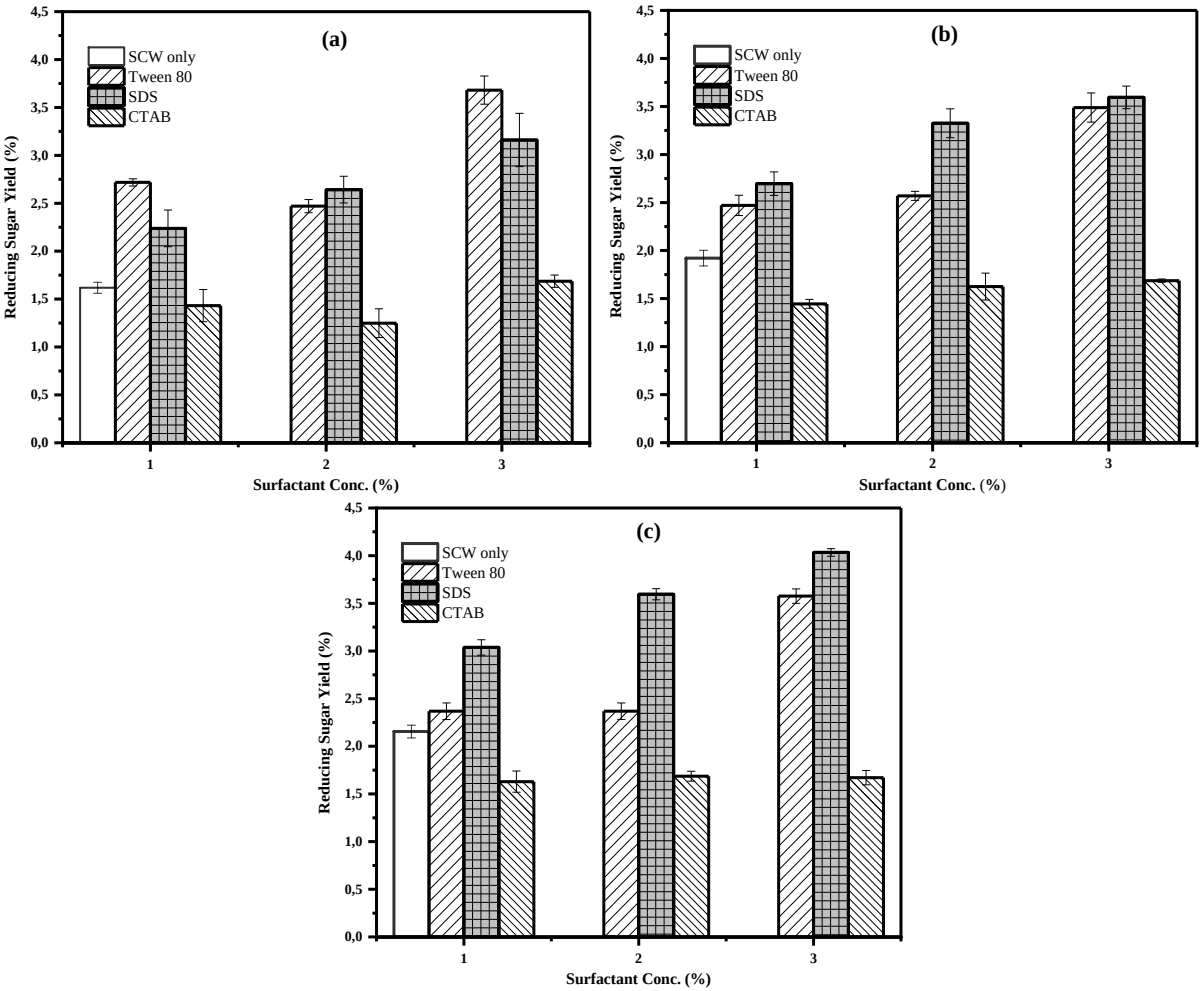

Figure 2. Sugar yield from the Effect of surfactant concentration during the subcritical process of water on the temperature (a) 140 °C, (b) 160 °C, and (c) 180 °C.

The addition of SDS and Tween 80 affects the yield of reducing sugar. Sugar production increases as the temperature get higher. This is due to the autocatalytic effect of water along with water conditions [7]. The adsorption of anionic surfactants increases with the increasing concentration of added surfactants. When the surfactant concentration increases, interactions will occur between surfactant molecules [25]. In non-ionic surfactants (Tween 80), when the surfactant concentration increases, micelles are formed, and micelles are formed due to the hydrophobic interactions between the adsorbed monomers gather at the liquid interface [26].

Table 2 shows that the addition of Tween 80 to the subcritical water process shows that the surfactant concentration factor significantly affects sugar yield ($p<0.05$). The sugar obtained increased compared to without surfactant and then decreased with increasing temperature of more than 140 °C in all concentration variations.

Table 2. The summary of two-way ANOVA for the effect of temperature and surfactant concentration on reducing sugar yield in each of the surfactant types.

Surfactant Type	Analysis of Variance	Factor	
		Temperature	**Surfactant Conc.**
Tween 80	F-value	1.949	17.476
	P-value	0.256	0.011
	Std Error (SE)	0.194	
SDS	F-value	144.172	169.438
	P-value	0.0002	0.0001
	Std Error (SE)	0.016	
CTAB	F-value	9.744	0.481
	P-value	0.029	0.650
	Std Error (SE)	0.041	

The temperature factor did not significantly affect the sugar yield ($p<0.05$) because the solubility of ether-type surfactants in water depends on the formation of bonds between water and the O-atoms of the ether. The solubility of Tween 80 decreases when unwanted micelles occur due to hydrogen bonds being destroyed by excessive heating [27]. Under these conditions, it can be predicted that the cloud point is exceeded. The solubility of Tween 80 decreases drastically above the cloud point temperature [28,29]. The addition of SDS to the subcritical water process showed that both factors (surfactant concentration and temperature) had a significant impact ($p<0.05$). In contrast, the addition of CTAB to the subcritical water process had no significant effect on the surfactant concentration factor ($p>0.05$), and the factor temperature had a significant impact on sugar yield ($p<0.05$).

Tabel 3. Reducing sugar yield with optimum value of each variable.

Variables				Reducing Sugar Yield (%)
Temperature (ºC)	Time (min)	Surfactant Type	Surfactant Conc. (%)	
180	60	SDS	3	4.034

The optimal conditions for the subcritical water process with the highest reducing sugar yield can be determined based on the experiment results entered into the DOE software with a general complete factorial design. Table 3 shows the maximum reducing sugar yield data from research and modeling tests by the Minitab 16 software.

3.3. Solid characterization.

3.3.1. TGA Analysis. Thermal Gravimetric Analysis was conducted to determine the characteristics of OPEFB degradation based on the pyrolysis method. The heat degradation of the material is strongly influenced by its chemical composition, which means that the composition of cellulose, hemicellulose, and lignin of OPEFB contributes to the heat characteristics of OPEFB. The TG (thermogravimetric) graph is shown as a decrease in mass with increasing temperature. DTG (derivative thermogravimetry) graph is a decrease in the TG graph to produce dm/dt or the rate of decrease in mass with temperature. The area bounded by the DTG line shows the magnitude of the mass reduction that occurs when the highest mass decomposition is indicated by the highest peak. In figure 3.a. The TGA curve of OPEFB fiber shows four steps of degradation, that is (a) evaporation of water at a temperature $\leq$105 °C, (b) hemicellulose at a temperature of 200–250 °C (c) cellulose at a temperature of 250–350 °C and (c) lignin at a temperature of 350–600 °C[30].

The results of TGA analysis showed that the cellulose contents of SCW-treated OPEFB without and with SDS were higher than that of the unpretreated one. Meanwhile, hemicellulose decreased after subcritical water pretreatment. However, the lignin content increased after subcritical water pretreatment, and it is assumed that pseudo-lignin occurs in the subcritical water pretreatment process. This phenomenon has been reported in studies using the biomass of hardwood, softwood, and herbal energy plants [31]. Several studies have hypothesized that polysaccharide degradation products such as Furfural and 5-HMF also contribute to increased pretreatment lignin content [23,24]. Pseudo-lignin can occur in all types of pretreatment due to low pH and high-temperature conditions such as hot water and hydrothermal pretreatment. In the hydrothermal pretreatment of straw and bagasse, the pseudo-lignin formation also occurs [32,33].

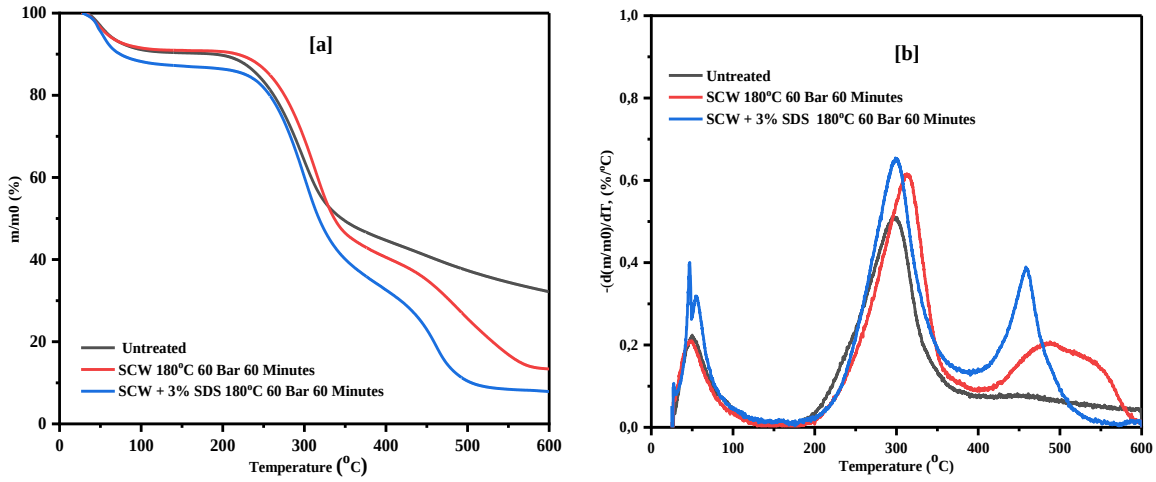

Figure 3. (a) TGA curves of OPEFB samples before and after SCW pretreatment.
(b) DTG curves of OPEFB samples before and after SCW pretreatment.

Table 4. The lignocellulosic composition of OPEFB by analyzed using TGA.

Component	Untreated (%)	SCW Only [180°C] (%)	SCW+ SDS [180°C] (%)
Cellulose	34.586	40.376	42.419
Hemicellulose	6.185	4.041	4.404
Lignin	17.123	32.854	31.998
Micellaneous	42.105	22.729	21.179

Sipponen [35] conducted a study on pseudo-lignin on the severity impact of hot water pretreatment on lignin from wheat straw, and their study showed that pseudo-lignin occurs in the high-severity temperature range of 170-200 °C. In addition, with atomic power microscopy, pseudo-lignin formation occurs at 200 °C of compressed water pretreated with Hinoki Cypress. During the cooling process, the pseudo-lignin formation can also occur. This is called cooling-induced pseudo-lignin (CIPL) [34]. This is in agreement with several other studies that reported that only small amounts of pseudo-lignin were detected from hot water pretreatment at 170 °C for less than 90.37 min. Bauer et al. was also reported that the increased probability of the increase in the pretreatment steam explosion also caused the pseudo lignin formation to be taken up late [35]. The increase in biomass can also be met through carbonization or pyrolysis, in which the biomass is thermally degraded [36]. This phenomenon occurred in this study because subcritical water was used at a temperature of 180 °C, 60 minutes in the initial treatment, causing the lignin presentation to increase.

3.3.2. XRD Analysis. X-Ray Diffraction is used to analyze the crystallinity index and determine the role of the addition of surfactants on changes in cellulose during the subcritical water process. The peak of the crystalline region is located at 22° while the contribution of the amorphous region of the substrate is

determined at 18°, which is similar to the characteristics of native cellulose [37]. And the CrI values were calculated using equation (2) [14]. Table 5 shows the CrI value of the original substrate and the substrate after subcritical water treatment added to the surfactant. The CrI value of the original substrate (53.06%) was lower than the substrate that was treated with Subcritical water and added surfactant. This was due to the presence of lignin, which was still dependent on cellulose and had an irregular structure. As shown in figure 4, the CrI value of the substrate slightly increased to 58.25% after subcritical water pretreatment at 180 °C for 60 minutes.

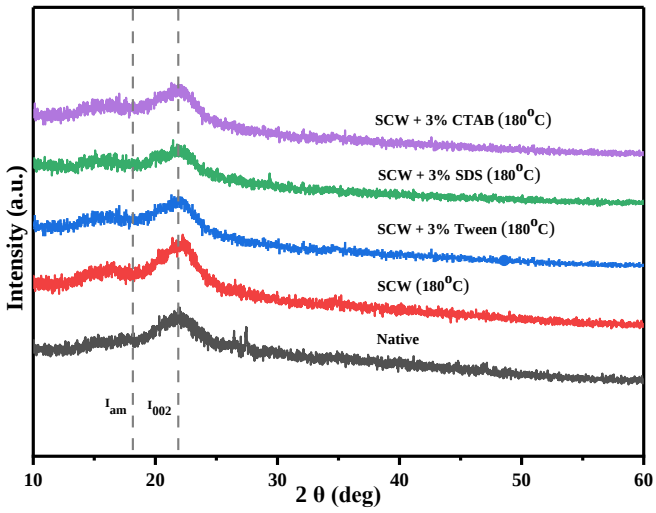

Figure 4. X-ray diffraction pattern on the native and the substrate after subcritical water processing with the addition of surfactants.

Table 5. The crystallinity index of native and SCW pretreated OPEFB.

Treatment	I_{002}		I_{am}		Crl (%)
	2θ	Int	2θ	Int	
Native	22.66	181.09	19.06	85.00	53.06
SCW 180 °C	22.82	239.52	18.78	99.99	58.25
SCW + 3% Tween 180 °C	22.84	215.46	18.96	99.66	53.75
SCW + 3% SDS 180 °C	22.89	120.79	18.51	43.43	64.05
SCW + 3% CTAB 180 °C	22.89	179.10	18.56	86.29	51.82

Investigation of the role of the addition of surfactants on changes in cellulose during the subcritical water process was shown by the Crl value, which was analyzed using XRD. Table 5 shows an increase in the Crl value when the surfactant is added. With the addition of SDS surfactant, the Crl value was higher than the Crl value of the Tween 80 and CTAB surfactants. The Crl value on SCW only substrate was 58.25% and increased to 64.05% on 3% SCW+SDS. This revealed that the addition of surfactants, especially SDS, could extract lignin and convert cellulose and hemicellulose into monomeric sugars during SCW. This result is similar to the previous study, where the use of tween filler and dilute ammonia in bagasse pretreatment showed that the Crl value increased significantly from 4% to 92.11% after the addition of surfactant [10]. Research on the addition of Tween-80 to improve enzymatic saccharification of biomass and ethanol production showed that the Crl value increased 13% from 34.98% - 60.24% [38]. This is the same as the research conducted by Pandey and Negi regarding the study of acid and base treatment with the help of surfactants on pine leaves. Jin [38] reported that the crystallinity index of steam-exploded pretreatment for OPEFB biomass increased by 13% from 53.27% to 60.24%. Mohan [39] also reported that the crystallinity value increased 15.28% from 50.55% to 65.83%.

3.3.3. SEM Analysis. Scanning Electron microscopy was used to determine the morphological changes of OPEFB before and before being treated with subcritical water with the addition of several types of surfactants. OPEFB fibers that were not treated showed rigid and regular fibrils, while significant morphological changes occurred in OPEFB fibers that had been treated with subcritical processes. In

addition, judging from the initial structure, OPEFB fibers that have been treated with SCW look separated and fully exposed. On the other hand, the untreated OPEFB fiber appeared slightly rougher than the SCW treated OPEFB fiber surface, which appeared smoother.

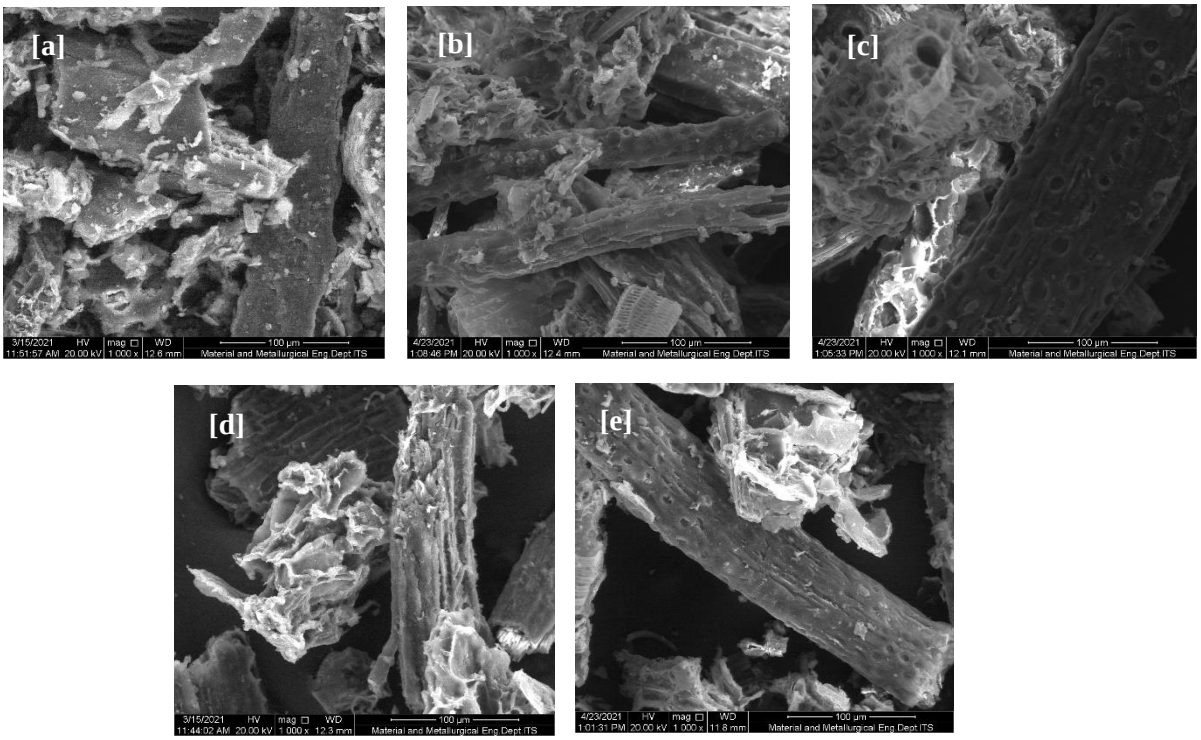

Figure 5. SEM images OPEFB 1000x (a) Substrate native, (b) Substrates treated with SCW only (c) SCW+3% Tween (d) SCW+3% SDS and (e) SCW+3% CTAB.

Figure 5 (a) depicts the structure of OPEFB before being treated shows that the surface of the original OPEFB is protected by a non-porous lignin cell wall that looks smooth and still quite tight. Figure 5 (b) After subcritical conditions at 180 °C, 60 bar shows a coarse, hollow porosity anomaly and looks more damaged than before subcritical treatment [40]. Figure 5 (c) treatment of subcritical water and the addition of 3% Tween shows a hollow OPEFB surface. Figure 5 (d) after subcritical treatment and addition of 3% SDS surfactant shows a wide and coarse, hollow, and amorphous porosity anomaly. These changes play a role in helping the accessibility of enzymes that will produce high sugar in the enzymatic hydrolysis process. However, after subcritical examination, OPEFB swelled and ruptured. These results indicate that the lignocellulose complex compounds have been destroyed. For example, a study conducted by Sangian and Widjaja [40] using coconut coir material showed that the morphology of the substrate after subcritical treatment showed a visible difference on the surface of coconut coir without subcritical treatment. While in figure 5 (e), subcritical water treatment and the addition of CTAB show that the surface of OPEFB is smoother and still denser than the addition of Tween and SDS. Figure 5 (b-c) shows subcritical water conditions at 180 °C, 60 bar, and 60 minutes.

3.3.4. FTIR Analysis. Fourier Transformed Infrared spectroscopy is used to analyze the structure of the substrates treated by chemical methods. FTIR spectra in figure 6 show the changes in chemical composition due to subcritical water on OPEFB. All the wavenumbers obtained from FTIR spectra show almost similar characteristics, as explained in table 6. The main band wave numbers for native lignocellulosic are shown in table 6, and these results are relatively comparable with other findings [41,42]. The peak of vibration in the range of 3293.60-3334.71 cm^{-1} is due to the stretching of O-H bonds of phenolic, alcoholic, and carboxylic functional groups. To determine the extent of damage to native cellulose, bands at 2917.68 to 2918.32 cm^{-1} (C–H stretching), 1578.18 to 1595.05 cm^{-1} (C=C

aromatic stretching), spectrum between 1425 cm^{-1} vibrations in the C-H deformation (on cellulose), which most likely represent cellulose related transmission.

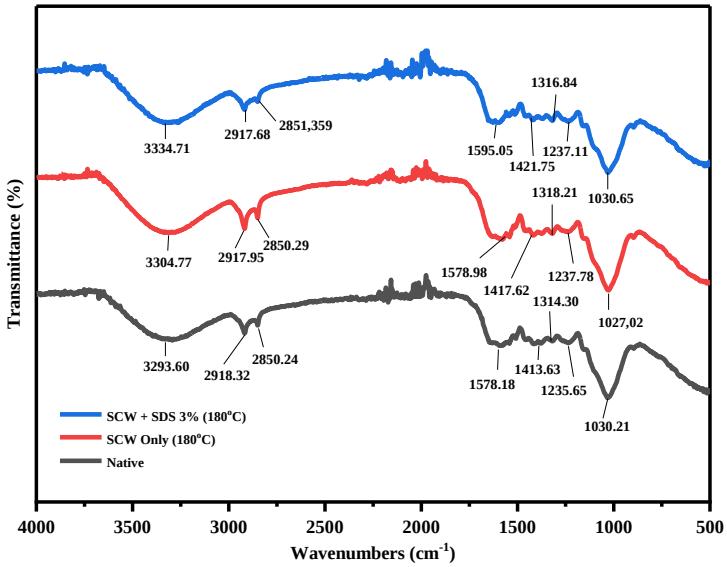

Figure 6. FTIR spectrum on the native, SCW-treated, and SCW+SDS-treated substrates (180 °C, 60 bar, 60 min).

The SCW-treated substrate showed absorption corresponding to OH stretching at 3304.77 cm^{-1}, similar to that of the original substrate; C–H symmetrical stretch at 2917.95 cm^{-1}, which is close after SCW + SDS pretreatment of 2917.68. After SCW+SDS pretreatment, absorption bands were observed at 3334.71, 2917.68, 1595.05, 1421.75, 1316.84, 1237.11, and 1030.65 cm^{-1}. As can be seen in figure 6. OPEFB before and after being treated with subcritical water and with the addition of SDS surfactant showed a comparable chemical FTIR spectrum, vibrations from 1314.30 to 1318.84 cm^{-1} by the CH_2 wagging functional group associated with cellulose. The curve on the sample that has been treated with subcritical water and the addition of SDS surfactant has wider valleys or peaks than the native. This indicates that cellulose is increasing.

Table 6. Wavenumber of Fourier Transformed Infrared spectroscopy absorption band for native and treated OPEFB.

Wavenumber (cm^{-1})			Bond absorptions
Native	SCW Only	SCW+SDS	
3293.60	3304.77	3334.71	O-H stretch (cellulose)[a]
2918.32	2917.95	2917.68	C-H stretch (cellulose)[a]
2850.24	2850.29	2851.36	
1578.18	1578.98	1595.05	C=C aromatic stretch (lignin)[b]
1413.63	1417.62	1421.75	C-H deformation (cellulose)[a]
1314.30	1318.21	1316.84	CH_2 wagging (Cellulose)[b]
1235.65	1237.79	1237.11	C-C, and C-O stretch (lignin)[b]
1030.21	1027.02	1030.65	C=O, C-H, C-O-C, C-O deformation or stretching (Cellulose)[a]

[a] [42] ; [b] [43]

4. Conclusion

The results showed that the addition of surfactants in subcritical water was better than without the addition of surfactants (except CTAB). The results showed that the sugar production with the addition of SDS surfactant in the subcritical water process was 87% better than that without surfactant. The maximum sugar yield (4.034%) was obtained by adding 3% SDS surfactant at 180 °C in the subcritical water process. These results were evidenced by the results of solids characterization using SEM, which showed that subcritical initial treatment (60 bar, 180 °C, 60 minutes) with SDS surfactant achieved

extensive, coarse, hollow, and amorphous changes in porosity structural anomalies. FTIR shows changes in functional groups, and XRD shows changes in crystallinity. The CrI value of the treated SCW+SDS solid increased significantly compared to the subcritical water only and SCW+3% CTAB, indicating that the substrate was converted to a more crystalline structure. The decrease in hemicellulose composition and increased cellulose composition before and before the subcritical process was observed using TGA. The SDS-assisted subcritical water is the best combination scenario that produces reducing sugars and prevents the formation of degradation products. The addition of Tween 80 and SDS to the subcritical water process gave rise to the attractiveness of the process for commercial purposes.

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