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RESEARCH ARTICLE

Factors influencing hydrothermal pretreatment of banana peel for high recovery of pectin oligosaccharides (POS)

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ABSTRACT - Utilizing banana peels creates opportunities for technological advancement in the direction of production strategies for food ingredients. Banana peel is an inexpensive by-product that can be processed into oligosaccharides with significant added value that has been reported to benefit food, animal feed and human health. Recently, pectin oligosaccharides (POS) have been the main research subject on banana peel polysaccharides. Hence, the present research study was conducted to identify the best pretreatment conditions particularly for hydrothermal pretreatment with respect to temperature, pH and substrate ratio in order to produce high POS. Prior to the pretreatment, the banana peel hydrolysate was characterized to measure the C, N,, H and S elements by a CHNS elemental analyzer as well as the cell wall's morphology using FESEM. Next, the hydrothermal pretreatment method started by varying the temperature with three different values of 105°C, 120°C, and 135°C by using an autoclave followed by pH varied at (3, 4, and 5), and lastly different substrate ratio (1:5, 1:10, and 1:15 of solid: liquid ratio, w/v). After that, DNS method was chosen to determine the amount of reducing sugar i.e. POS in each sample. The study's findings suggest that the highest amount of reducing sugar POS was achieved by using UV/Vis spectroscopy at an absorbance of 540 nm were at a temperature of 135°C, pH of 5, and a substrate ratio of 1:15 (w/v). Furthermore, the CHNS elemental analyzer shows the percentage of C, H, N and S element from the banana peel whereby the carbon has the highest percentage also, the morphology of raw and pretreated banana peel has been compared and revealed. In conclusion, the study has successfully recovered high POS from banana peel hydrolysate via hydrothermal pretreatment whereby the highest POS measured was 44.60 mg/mL and the increment of yield calculated was about 418%.

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1.0 INTRODUCTION

Banana plant produces one of the largest fruit plantations in the world. In Asia, 62.48 million tonnes bananas produced recorded in 2018 [1]. Bananas are a tropical fruit that are accessible all year long and among the most popular fruit in the world [2]. Banana production in Malaysia reached its peak in 2017 at 350,493 tonnes. However, its output fell from 331,255 tonnes in 2018 to 325,447 tonnes in 2019 [3]. Banana farms generate a lot of waste both at the farming site such as pseudo stem, leaves, peel and inflorescences and at the processing sites like rachis and rejected bananas where the fruit is packaged [4]. Consequently, each year about 39.9 million tons of banana peel are produced [1].The lignocellulosic biomass derived from these crops which is banana peels are one of the great importance and high volume, which represent approximately 35% of the weight of these crops [5]

Principal elements that make up lignocellulosic biomass (LCB) are cellulose, hemicellulose, and lignin that come primarily from uncultivated plants. Uncultivated plants, such as grasses, agricultural residues, and woody biomass, are the primary sources of lignocellulosic biomass (LCB) [6]. These plants have evolved to thrive in their natural environments without human intervention, resulting in a higher proportion of lignocellulosic components compared to cultivated plants [7]. It is complex matrix that is mostly composed of the proteins, phenolic polymers, and polysaccharides that made up the woody cell walls of plants [8]. A dense framework made of hemicellulose, and lignin surrounds cellulose, which is a carbohydrate polymer, in the complicated spatial structure of LCB [9]. Banana peels contain more pectin than other fruits, according to certain studies [10]. It has been suggested that POS represent a novel class of prebiotics with potential wellness benefits, such as antioxidant activity and the promotion of bifidogenic flora [11]. Pectin, a polysaccharide primarily composed of a partially methyl esterified homogalacturonan backbone, is the source of POS [12]. Periodically, regions of alternating L-rhamnose and D-galacturonic acid residues, which are attached to L-arabino and D-galactooligosaccharides, break up homogalacturonan [13]. POS are soluble dietary fibers that have a number of advantageous physiological effects on the gastrointestinal tract, such as reducing the amount of glucose absorbed, slowing down the transit time through the gut, increasing the bulk of the feces, and lowering cholesterol [14].

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Pretreatment is an essential step in the process of turning biomass into oligosaccharides [15]. Particularly targeting hemicellulose, pretreatment releases its constituent oligosaccharides, monomeric sugars, and other products [16]. When it comes to environmental, energy, and economic considerations, hydrothermal pre-treatment (HTP) is a very effective method that uses water as a solvent under extreme pressure and temperature conditions [17]. In HTP, moist feedstocks are heated to a high temperature for a predetermined amount of time, either by themselves or in the presence of chemical additives. HTP often takes place at an acidic pH, which is either brought on by organic acids released from the biomass or by the addition of additional acids [18]. Hemicelluloses are hydrolysed in HTP, particularly when a low starting pH is used, although most of the cellulose and lignin will still be present in the processed solid biomass [17].

Hence, pretreatment particularly hydrothermal is a crucial step in order to ensure the openness of cell wall to release sugar i.e. POS. A high recovery of POS from banana peel hydrolysate can be enhanced via appropriate hydrothermal pretreatment conditions. Previous study demonstrated the effectiveness of hydrothermal treatment in extracting pectin from grapefruit peel, optimizing conditions such as temperature, time, and pH [19]. Yet, there are limited research has been reported on the hydrothermal on banana peel. Uncommonly, a biomass conversion from banana peel to POS has not been extensively explored when compared to xylo-oligosaccharides (XOS). Consequently, the present study was intended to shed a light on the hydrothermal pretreatment conditions.

Pectin has also been found in the pits of fruits like plums and peaches. Research has looked into ways to take pectin and POS out of these by-products of fruit [20] In addition, beet pulp, a by-product of sugar beet processing, is another biomass used for extracting pectin and POS. Studies have investigated methods to maximize the recovery of these compounds from beet pulp [21].

2.0 METHODS AND MATERIAL

2.1 Materials

The banana residues (peels) were collected at the pisang goreng stall in Gambang, Pahang. Analytical grade of chemicals such as sulphuric acid 97-99% (H_2SO_4) (Cas No. 7664-93-9), sodium potassium tartrate tetrahydrate (CAS No. 6381-59-5), sodium hydroxide (CAS No. 1310-73-2), and 3,5-dinitrosalicylic acid (CAS No. 609-99-4) were purchased from Sigma-Aldrich, China.

2.2 Substrate Preparation

Targeted lignocellulosic biomass which is the ripe banana peel at stage 7 of ripening (yellow with brown spots, according to the color scale of Von Loesecke) [22] was separated from the fruits. About 500 g of banana peels were weighed and the sample was washed by using deionized water. Next, the sample was cut into small pieces and was dried in an oven at 70°C for 48 h. After that, the dried small banana peel was ground by using a Waring grinder (Model CB15VE; Made from United States) until it turns into a small fine powder fine powder (<150 μm) [23].

2.3 Hydrothermal Pretreatment

After the banana peel has turned into a fine powder, the ground banana peel was undergoing a hydrothermal pretreatment producing banana peel hydrolysate. This hydrothermal pretreatment was conducted using Hirayama Hv-110 autoclave (Hirayama MFG. Corp. Japan) with temperature varied at 105°C, 120°C, and 135°C for 60 min. A sulphuric acid (1 M concentration) was used in order to vary the pH of sample at pH 3, 4, and 5. Then, distilled water was added to each sample of banana peel, resulting in a mass or consistency load of 1:5, 1:10, and 1:15 (solid: liquid ratio, w/v). The parameters used for this study shown in Table 1. The initial concentration of substrate (banana peel hydrolysate before treatment) was measured at 8.603 mg/ mL.

Table 1. Parameters and ranges for hydrothermal pretreatment of banana peel

Parameters	Min	Mid	Max
Temperature (°C)	105	120	135
pH	3	4	5
Substrate ratio (w/v)	1:5	1:10	1:15

2.4 CHNS analysis

Elemental analysis (CHNS) of the non-treated banana peel powder were carried out by using the LECO TruSpec Micro CHNS (USA) elemental analyzer. The analysis of carbon, hydrogen and nitrogen components was determined in this procedure.

2.5 Reducing Sugar Analysis

The DNS method was used to determine the amount of reducing sugar present in each sample of banana peels [24]. DNS reagent was prepared by starting with 10 g of 3,5-dinitrosalicylic acid (DNS) and 800 mL of 0.5 M NaOH was added to the alkaline solution. By using a hot plate stirrer, the mixture was heated and stirred until it fully mixed. In a distinct beaker, 300 g of sodium potassium tartrate tetrahydrate also called Rochelle salt were added into 125 mL of distilled water and was gently heated [25]. Finally, DNS and salt solutions were mixed and stirred to dissolve the reagents. The mixture was allowed to cool to room temperature. The volume of the mixture was then made up to 1.0 L with distilled water in a volumetric flask. Next, 0.2 mL of the hydrolysate was combined with 2 mL of DNS solution in a test tube. The test tube was placed in boiling water for 10 min and then immediately cooled to room temperature using a container of ice and water before a UV/Vis spectroscopic examination by using Shimadzu Thermo scientific UV-visible spectrophotometer (Japan) at 540 nm absorbance was performed on it.

2.6 FESEM Analysis

The samples of raw banana peel powder and pretreated banana peel were dried in a desiccator for 48 h to eliminate moisture. The banana peel surface was examined using a cold field emission scanning electron microscope (cold FE-SEM, Model SU-8220, Hitachi Co. Ltd., Tokyo, Japan). By using FE-SEM, the surface and morphology of the sample were studied.

3.0 RESULTS AND DISCUSSION

3.1 Characterization of Banana Peel

Carbon, hydrogen, and oxygen atoms make up the majority of sugar molecules; the quantity of carbon atoms in a sugar molecule varies depending on the kind of sugar. Other elements, like sulfur or nitrogen, may also be present in some sugar molecules, but they are usually not present in large quantities and do not always signify a higher sugar content. Thus, the CHNS analysis result is displayed in Table 2. The carbon component shows the highest percentage of the other three components, hydrogen, nitrogen, and sulphur.

These values were in good comparison with the previous studies [23], by which the CHNS elemental analysis for banana peels resulted in values of 37.14%, 7.24%, 2.03%, and 0.0022% for carbon, hydrogen, nitrogen, and sulfur, respectively. Indicating that carbon element in banana peel is the highest compared to the other three elements revealing the sugar molecules present in this banana peel powder. These results obtained a good comparison with earlier research conducted by Fernandes et al., (2013); Mohd Taib et al., (2014) which noted that the expected elemental percentage range in a lignocellulosic biomass are as follows: carbon to be between 35-54%, hydrogen 4.5-9%, oxygen 40-48%, nitrogen less than 3% and sulfur less than 1%.

Table 2. CHNS analysis of banana peel powder

No.	Parameter	Results (%)
1.	Carbon	46.547
2.	Hydrogen	6.700
3.	Nitrogen	1.236
4.	Sulphur	0.193

3.2 Effect of Temperature

The POS concentrations of all temperatures i.e. 105 ℃, 120 ℃, and 135 ℃ is displayed in Figure 1. The POS recovery of the hydrolysate of banana peels at three different temperatures treated with sulfuric acid at pH 4 and a substrate ratio of 1:10 (w/v) contains 2.40 to 14.13 mg/mL of POS concentration. This demonstrates that sulphuric acid, (H_2SO_4) pretreatment of banana peels result in a mixture of sugars. It is evident that the maximum sugar recovery occurred at 135℃ by which the POS recovery was 14.13 mg/mL. On the other hand, hydrothermal pretreatment that was conducted at 105 ℃ and 120 ℃ produced POS at 2.4 mg/mL and 7.28 mg/mL respectively. The rate of hydrolysis can be accelerated by higher temperatures, which resulted in a more rapid and comprehensive conversion of polysaccharides into sugars. High temperatures, however, can also promote the formation of inhibitory byproducts and the breakdown of sugar [27] as opposed to 105 ℃ and 120 ℃. The ideal temperature range typically falls below 140°C, where POS yield is maximized while minimizing degradation [28].

Similar patterns have been found for lemon peel, orange peels and sugar beet pulp, with the highest oligogalacturonides, OGalA solubilization at the highest temperatures, where high temperature was needed for the optimum extraction of oligosaccharides [29]. Correspondingly, pretreated Italian green pepper waste at a high temperature, 180 °C, resulted in an increased of sugar recovery from 25.42% to 44.76% where it is the maximum recovery value of sugar at the studied operating conditions [30]. Additionally, increasing temperature also raising the H^+ and OH^- ions whereby this autohydrolysis process facilitates the release of sugar compounds. The concentration of H^+ and hydroxide ions rises with temperature due to an increase in water ionization. By rupturing the glycosidic bonds in cellulose and hemicellulose, these ions can speed up the hydrolysis processes [31]. In addition, the rate of hydrolysis and the stability of the sugars produced are significantly impacted by the temperature of the reaction [32]. Partially degradable lignin, a complex and resistant polymer found in lignocellulosic biomass, can occur at higher temperatures. Due to this reduction in lignin content, more cellulose and hemicellulose may be prone to hydrolysis [33].

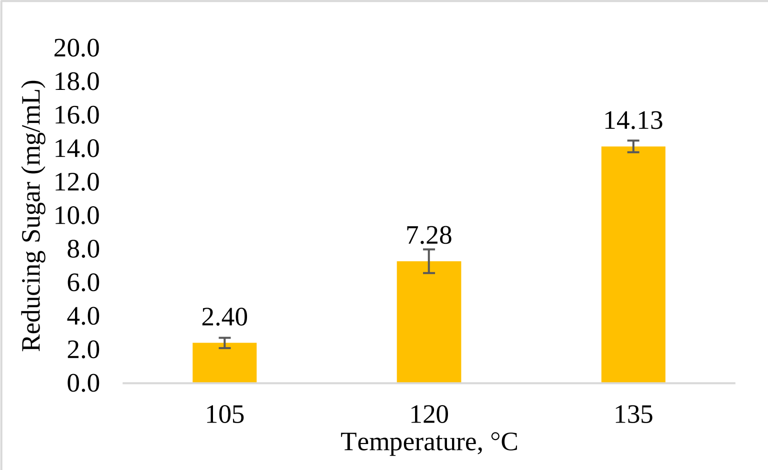

Figure 1. POS recovery at pH 4 with substrate ratio of 1:10 (w/v) at different temperature.

3.3 Effect of pH

The corresponding POS concentrations for each pH are displayed in Figure 2. The POS recovery from banana peel contained 6.62 to 18.59 mg/mL of POS at three different pH whereby at a temperature of 135 °C and a substrate ratio of 1:10 (w/v) was kept constant. According to the results obtained, pH 5 showed the highest recovery of POS content i.e. 18.59 mg/mL; than pH 3 and pH 4, by which POS produced at 6.62 mg/mL and 14.13 mg/mL respectively. Such results can be justified whereby a high pH could dissolve and hydrolyze the oligosaccharides in the lignocellulosic biomass more efficient than the low pH. This is because by using moderate amount of sulphuric acid to control the pH may increase the POS recovery where the lignin was disintegrated during this pretreatment process [34]. This was attributed to the effect of pH on the degradation of components.

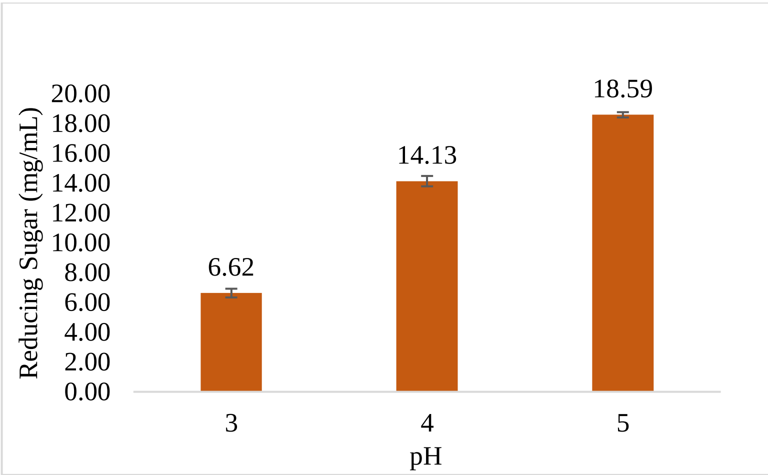

Figure 2. POS recovery at 135 °C with substrate ratio of 1:10 (w/v) at different pH.

Even though the acidic pH could give a high amount of POS recovery, but a lower pH or acidic condition could lead to a problem of corrosion for the apparatus [23]. Hydrogen ions, which are frequently produced by adding acid, catalyze the hydrolysis of hemicellulose chains in low pH processes, which leads to the formation of sugar oligomers and monomers that can be further broken down [35]. These procedures yield a solid fraction primarily composed of cellulose and lignin, as well as a liquid product rich in reducing sugar. Also, this result shows that the hydrothermal pretreatment with pre-adjustment pH has wide applicability for banana peel hydrolysate.

From the previous study by [37], the total reducing sugar content increased gradually with pH between 3.0 and 5.0. It was 3.84% at pH 3.0 and then it increased to 15.21% at pH 5.0. The total reducing sugar content in the hydrolysate remained unchanged after the pH was above 5 [38]. The reaction may have reached equilibrium at a higher pH or run into inhibitory effects that stopped it from changing any further in terms of the concentration of reducing sugar [39], [40]. The reducing sugar i.e., glucose oligomer content increased from 3.33% to 5.09% at pH 3.0 to 3.5, respectively; however, there was a smaller increase in the glucose oligomer content between pH 3.5 and 4.0, which was 5.49%. Concluding that the total reducing sugar content and glucose oligomers increased with increasing pH [41]. Therefore, pH 5 can be concluded as the best pH for the production of POS.

3.4 *Effect of Banana Peel to Liquid Ratio*

POS recovery from banana peel hydrolysate was obtained between 15.72 to 44.60 mg/mL when the three-substrate ratio i.e. 1:5, 1:10, and 1:15 (w/v) were tested at pH 5 and 135 °C as shown in Figure 3. In comparison to substrate ratios of 1:5 and 1:10 (w/v), the observation indicates that POS recovery is the highest for substrate ratio of 1:15 (w/v). The POS recovery was 15.72 mg/mL at a substrate ratio of 1:5, and 18.59 mg/mL at a substrate ratio of 1:10. The concentration of pectin oligosaccharides was 44.60 mg/mL at a substrate ratio of 1:15 (w/v).

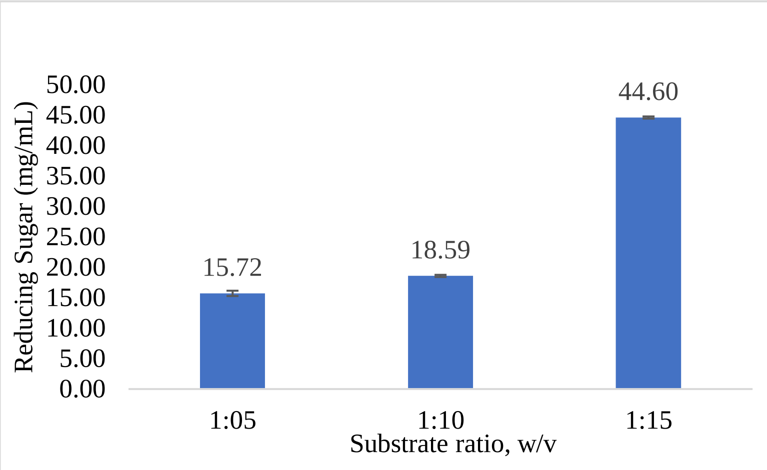

Figure 3. POS recovery at 135 °C with pH of 5 at different substrate ratio (w/v).

According to the He et al., 2013, the 1:15 (w/v) of substrate ratio in 45 mL mixture formed a loose mixture, makes it easier to mix thoroughly. This condition leads to the increasing of POS recovery than substrate ratio of 1:5 and 1:10 (w/v) since there is more water molecule to interact with in order to break the lignin bonds with cellulose and hemicellulose. The increase in POS concentration may also be due to the low amount of banana peel, thus it is less saturated providing a large surface area for contact with the banana peel where POS recovery is more efficient [43]. Reducing sugar concentration per unit volume may be higher when using less banana peel [44]. The higher observed concentration of reducing sugars can be attributed to the fact that the sugar released into the solution is concentrated in a smaller volume of hydrolysis solution [45]. Hence, the higher the substrate ratio (1:15, w/v) gave a higher POS recovery.

As observed from other research, pretreated pineapple waste at high values of solid-to-liquid ratio w/v, a maximum concentration of 25 mg/mL of reducing sugar was obtained Sepúlveda et al., (2018). It is an evident that the solid-to-liquid ratio significantly influences the sugar recovery after hydrothermal pretreatment, with higher solid loadings generally leads to a high sugar yield. Similarly, research observed from Ren et al., (2020), the reducing sugar yield increased significantly with the increase in solid-to-liquid ratio from 1:5 to 1:15, and the maximal reducing sugar yield reached 170.2 mg/mL respectively, at a solid-to-liquid ratio of 1:15. Hence, these finding are in a line, as the parameters of solid-liquid ratio increase, the reducing sugar also increase.On the contrary, the total yield of reducing sugar i.e., galactose and glucose could be decreased with further increasing biomass (solid) loading as sugars was limited by mass transfer and substrate accessibility [48], [49].

3.5 *FESEM Analysis*

The FESEM analysis had shown microstructure of the untreated and pretreated banana peel sample. It is apparent that the structure of banana peels has altered with nearly all of the sugars and hemicelluloses removed. Figure 4a shows the structure of raw banana peel before the pretreatment process where it contains cells arranged regularly and compactly. The surface layer of raw banana peel showed compact and rigid structure of highly ordered fibers which restrains the recovery of POS. On the contrary, Figure 4b indicates the surface of banana peel after the HTP whereby autoclaving pre-treated banana peel showed irregular cracks with pores of different sizes and disruption of fibers structure. The pre-treatment of the banana peel sample at pH 5, a substrate ratio of 1:15, and assisted autoclaving at 135 °C may have increased the amount of fiber bundles because it partially broke down the hemicellulose and lignin structure. The ability to view the fibrillar organization of the cell wall surface in minute detail is one of the benefits of the FESEM technique.

a) b)

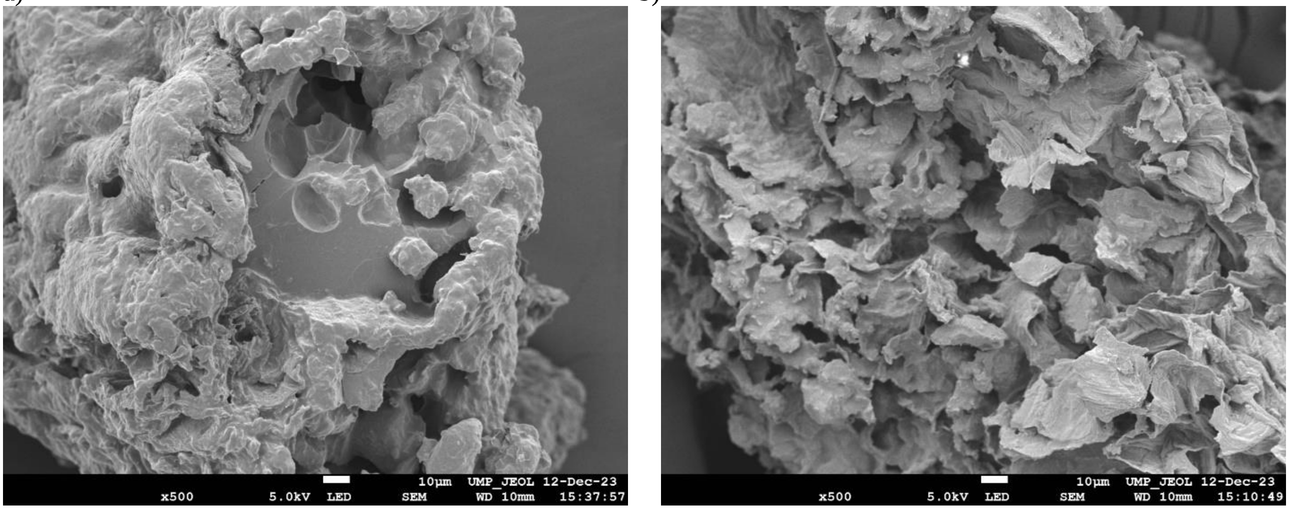

Figure 4. Morphology of raw banana peel before pretreatment (a) and after pretreatment (b) under 500x magnification.

The morphology of the biomass before and after hydrothermal pretreatment showing that the particle is partially destroyed while some lignin has dissolved and depolymerized during the pretreatment [50]. Effect of the hydrothermal pretreatment in the biomass also increase the specific surface area and total pore volume of the treated samples. Other studies have been reported that the hydrothermal pretreatments used in the study expose the sugarcane bagasse fiber bundles and partially remove the surface lignin layer [51]. This effect is most likely the result of pretreatment-induced lignin redeposition and partial delignification of sugarcane bagasse samples. Previous research justified the results obtain for the untreated and pretreated lignocellulosic biomass (banana peel) from the FESEM analysis in this study.

4.0 CONCLUSION

The study on the investigation of hydrothermal pretreatment conditions of lignocellulosic biomass particularly for banana peel to recover high POS was successfully achieved whereby the highest POS measured was 44.60 mg/mL at 135 °C, pH of 5, and a substrate ratio of 1:15 (w/v). This can be proven from the morphology of cell wall before and after hydrothermal pretreatment of banana peel whereby the destroyed layer of cell wall and cracks on the banana peel surface can be seen and thus disrupting the lignin layer that causes the lignin and hemicelluloses to be eliminated. In addition, this indicates that the specific hydrothermal pretreatment parameters, including temperature, pH, and substrate ratio, significantly influence the POS recovery.

As for the future perspective, although hydrothermal pretreatment on the banana peel hydrolysate shows a high value of reducing sugar recovery, pectin oligosaccharides, it requires further optimization and the parameters chosen should be further study. This include the characterization of the banana peel hydrolysate and the pretreatment conditions. On the other hand, quantifying or precisely identifying POS is not possible with the common reducing sugar analysis, although it is helpful for a preliminary evaluation of the reducing sugar content. More detailed analyses are required in order to completely validate and optimize conditions for POS production. A more thorough assessment of the hydrolysis process will be possible thanks to these techniques, which will yield precise data on the amount and caliber of POS. The improvement of POS recovery from banana peel hydrolysate could be done by varying more parameters like residence time, temperature, pH and biomass to water ratio in order to find the optimal conditions for the reducing sugar recovery. Additionally, by combining different types of pretreatment methods, this approach may enhance the reducing sugar release.

5.0 CONFLICT OF INTEREST

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper

6.0 AUTHORS CONTRIBUTION

Z. Nur Irdina Izzati (Investigation; Formal analysis; Visualisation; Writing - original draft)

M.S. Mohd Shafiq (Conceptualization; Resources; Supervision; Writing - review & editing; Funding acquisition)

J. Shariza (Resources; Writing - review & editing; Project administration)

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