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Bulletin of Chemical Reaction Engineering & Catalysis, 16 (3) 2021, 565-570

Research Article

The Influence of H_2O_2 on The Photocatalytic Pretreatment of Cellulose for 5-Hydroxymethyl Furfural (5-HMF) Production

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Received: 8th February 2021; Revised: 3rd May 2021; Accepted: 6th May 2021
Available online: 10h May 2021; Published regularly: September 2021

Abstract

Photocatalysis has been widely known as a simple green technology to be applied in the synthesis and degradation process of organic molecules. An application of photocatalysis in a biomass pretreatment for a 5-hydroxymethylfurfural (5-HMF) production was investigated in this study. The results have revealed that photocatalysis, applied during pretreatment, facilitates the breakdown of cellulose. The presence of oxidizing agent (H_2O_2) in the ratios to cellulose of 11:1, 18:1, and 37:1 mol.mol^{-1} has been investigated for its effect on the production of 5-HMF. The optimum conditions obtained for the pretreatment process was the presence of H_2O_2 at 37:1 mol.mol^{-1}, which was followed by the process of evaporation of the remaining H_2O_2 after pretreatment. The 5-HMF yield from the hydrolysis process involving pretreatment was 13.07%, while the yield from the process without pretreatment was 9.79%. The application of the pretreatment has succeeded in increasing the 5-HMF yield by 25.09%. The progress in the pretreatment was also marked by the presence of the carboxyl groups in the pretreated samples which were observed by the Fourier Transforms Infrared spectroscopy (FTIR).

Keywords: Photocatalytic Pretreatment; TiO_2; Cellulose; Microwave-Assisted Conversion; 5-Hydroxymethyl furfural (5-HMF).

How to Cite: M.I. Syafi'i, K. Nueangnoraj, S. Boonyarattanakalin (2021). The Influence of H_2O_2 on The Photocatalytic Pretreatment of Cellulose for 5-Hydroxymethyl Furfural (5-HMF) Production. *Bulletin of Chemical Reaction Engineering & Catalysis*, 16(3), 565-570 (doi:10.9767/bcrec.16.3.10311.565-570)

Permalink/DOI: https://doi.org/10.9767/bcrec.16.3.10311.565-570

1. Introduction

Cellulose is a major component of biomass that potentially provides a rich source of sugars needed for the production of versatile chemicals, such as 5-hydroxymethyl furfural (5-HMF) [1–7]. Edible cellulose, such as starch, has been successfully transformed to 5-HMF [8]. Nevertheless, the work has been recently criticized due to increasing food prices all over the world. Non-edible cellulose, such as wood waste, grass, and other sources, has become alternative renewable resources for chemical transformation into various chemicals. One of the major obstacles is the high crystallinity of cellulose which makes it difficult for such transformation to occur [9–13].

Biomass pretreatment becomes an important requirement to soften crystalline cellulose [9]. The use of microwaves, electric heaters, ovens, and burners with the presence of chemicals, such as acids and alkalis, has facilitated break-

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ing strong hydrogen bonds in cellulose. However, the process often leads to high costs, and even less environmentally friendly [14]. On the other hand, pretreatment is a supporting process of the whole production process, so the process should be simple and economical.

Photocatalysis is one of the economical and simple green technologies and is widely used in the process of synthesis and degradation of organic molecules [15]. Photocatalysis work by utilizing ultraviolet (UV) light to activate the catalyst which then initiates radical reactions. Water is usually used as a solvent, as well as a source of radical hydroxyl ions, which then can be accelerated by the presence of oxidizers, such as oxygen and H_2O_2 [16].

The utilization of photocatalysis in biomass pretreatment has been studied currently. TiO_2 is an important photocatalyst that is widely used because it is inexpensive, inert, stable, and easily recycled [17]. The use of TiO_2 and H_2O_2 under UV radiation has depolymerized husks into various types of valuable chemicals [16]. Starch, whose molecules are easily accessed by solid catalysts, has been successfully depolymerized for the production of bioethanol [18]. Also, the biomass with the more complicated cellulose components than starch is successfully depolymerized for the production of bioethanol. The process is able to reduce the reaction time of the saccharification process in producing sugar up to 80%, which is from 24 h to 0.5 h. Moreover, the process is likely superior to the alkaline process [19].

The use of TiO_2 anatase in the pretreatment process would provide added value, especially if the continuous process also utilizes the same catalyst. Previous literature reported that TiO_2 could convert fructose which is the part of biomass that is easiest to be transformed to 5-HMF directly through hydration [20,21]. Cellulose, the part of biomass that is difficult to be broken down, can also be converted by TiO_2 into 5-HMF through depolymerization, hydrolysis, isomerization, and hydration [8]. Therefore, if the TiO_2 is employed in the pretreatment process and shows good performance, 5-HMF yield produced is expected to increase.

This research studied the performance of photocatalytic pretreatment of cellulose as biomass under H_2O_2. TiO_2 was employed as a photocatalyst in the pretreatment step as well as in the transformation of biomass into 5-HMF. Water and H_2O_2 were used as a green solvent and oxidizing agent respectively. UV light served as a photon source. Furthermore, the pretreatment performance was observed by Fourier-Transform Infra-Red (FTIR) spectroscopy to determine changes in chemical bonds, High-Performance Liquid Chromatography (HPLC) to determine the yields of sugar and 5-HMF produced.

2. Materials and Methods

2.1 Materials

Cellulose (microcrystalline powder, 20 μm) and TiO_2 anatase (powder, ~325 mesh) were purchased from Sigma-Aldrich. H_2O_2 30% (analysis grade) was purchased from Merck.

2.2 Photocatalytic Pretreatment

Samples for pretreatment were prepared by mixing cellulose (1 g), TiO_2 (0.4 g), and a solution of H_2O_2 in water (20 mL). The amounts of H_2O_2 (30%) in the ratio of moles to cellulose ($mol.mol^{-1}$) were varied as the following: 11:1, 18:1, or 37:1. Samples were placed under UV

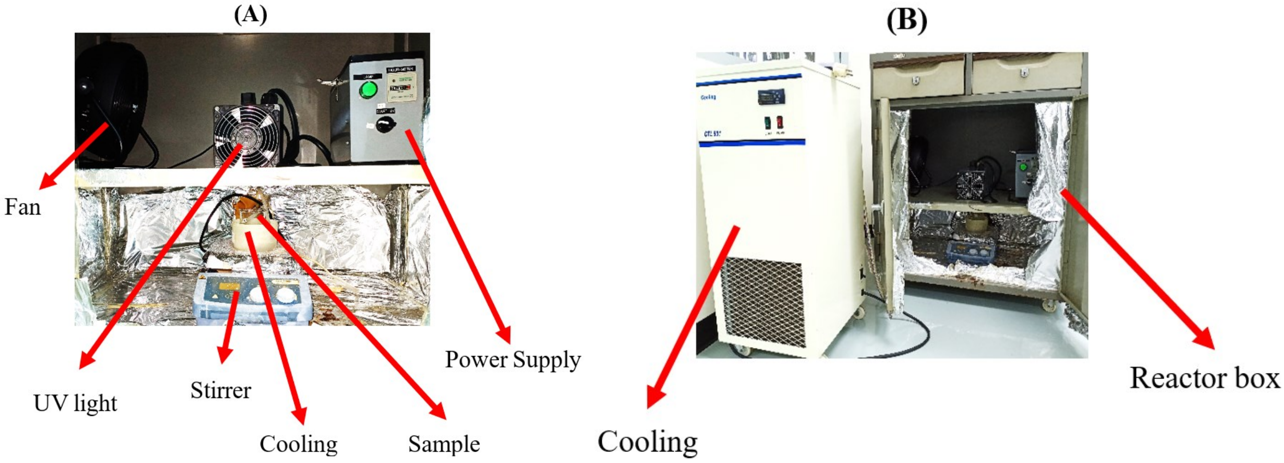

Figure 1. UV reactor design for photocatalytic pretreatment with views from inside (A) and outside (B) the reactor box.

light (mercury UV lamp, 400 watts) for 1 h as shown in Figure 1. The remaining solution was evaporated by drying process at 80 °C for 2 days.

2.3 Microwave-Assisted Biomass Conversion to 5-HMF

The pretreated samples were transferred into Teflon vessels followed by the addition of water (15 mL). The mixtures in closed vessels were placed in a microwave reactor (Milestone, flexiWAVE, Italy). The microwave treatment was performed under the maximal power of 1,000 watt, reaction temperature of 220 °C (20 °C increments), and reaction time of 15 minutes. On the other hand, samples without pretreatment as controls were also prepared. These preparations included mixing cellulose (1 g), TiO_2 (0.4 g), and a solution of H_2O_2 in water (15 mL), or water only (15 mL).

2.4 Separation of Crude 5-HMF

Separation of crude 5-HMF was carried out by washing and filtering. The reaction product mixtures were filtered and washed three times using deionized (DI) water and centrifugation (5000 rpm, 15 min). The remaining solids were dried at 80 °C for 1 day, while the solutions obtained were analyzed by high-performance liquid chromatography (HPLC).

2.5 Analysis and Characterization

The cellulose consumption was calculated according to Equation (1).

$$Cellulose\ consumption\ (\%) = \frac{(remaining\ solids\ (g) - catalyst\ (g))}{initial\ cellulose\ (g)} x100\% \quad (1)$$

The 5-HMF analysis was carried out using HPLC (Agilent 1260 series) equipped with an Ultraviolet (UV) Detector at a wavelength of 284 nm. The injection volume was 20 µL. Hydrophobic column (Luna® 10 µm C18 100 Å, LC Column 250 4.6 mm) was used at 40 °C. Water and methanol mixed (90:10, v/v) with a flow rate of 1 ml.min^{-1} was used as the mobile phase. The 5-HMF yield was calculated according to Equation (2).

$$5-HMF\ yield\ (\%) = \frac{5-HMF\ product\ (mol) \times 162.14\left(\frac{g}{mol}\right)}{initial\ cellulose\ (g)} x100\% \quad (2)$$

where 162.14 g.mol^{-1} is a cellulose molecular mass. The glucose analysis was carried out using HPLC (Agilent 1260 series) equipped with a Refractive Index Detector (RID). The injection volume was 100 µL. A carbohydrate column (Transgenomic CARBOSEP CHO682, LEAD column, CHO-99-9854, USA) was used at 80 °C. Water with a flow rate of 0.4 mL.min^{-1} was used as the mobile phase. The glucose yield was calculated according to Equation (3).

$$Glucose\ yield\ (\%) = \frac{glucose\ in\ product\ (g)}{initial\ cellulose\ (g)} x100\% \quad (3)$$

Pretreated and non-pretreated dry sample powders were analyzed by an attenuated total reflectance-Fourier transforms infrared spectroscopy (ATR-FTIR, Nucolet iS5) with an iD7 ATR accessory with a diamond crystal. The FTIR spectra were recorded from 400 to 4,000 cm^{-1} with 32 scans at a resolution of 8 cm^{-1} in transmittance mode.

Table 1. The influence of pretreatment and H_2O_2 on microwave-assisted cellulose conversion to 5-HMF.

No.	Pretreatment (No/Yes)	H_2O_2:Cellulose (mol.mol^{-1})	Microwave-Assisted Cellulose Conversion		
			5-HMF Yield (%)	Glucose Yield (%)	Cellulose Consumption (%)
1	No	0:1^{NT}	4.93±0.42	18.99±0.65	29.30±0.46
2	No	0:1	7.70±0.92	9.43±0.37	29.37±0.71
3	No	11:1	9.79±1.28	25.91±0.71	80.13±1.29
4	No	18:1	8.52±1.45	8.51±1.45	90.76±1.26
5	No	37:1	0.66±0.59	3.69±0.53	99.37±0.90
6	Yes^{NE}	37:1	0.00±0.00	0.00±0.00	100.00±0.00
7	Yes^{E}	11:1	11.33±0.52	23.93±0.82	58.67±0.80
8	Yes^{E}	18:1	12.02±0.30	27.45±1.30	62.57±2.60
9	Yes^{E}	37:1	13.07±0.30	28.94±1.22	70.36±2.10

^{NE} without evaporation process after pretreatment; ^{E} with evaporation process after pretreatment; ^{NT} without TiO_2 as a catalyst

3. Results and Discussion

Initially, the study began with testing the effect of the presence of catalysts on samples that were not undergone the pretreatment process. Table 1 (No. 1-2) shows that the value of cellulose consumptions between the samples with the presence of the catalyst, and without the presence of a catalyst were similar. The results show that the catalyst has no major impact on the overall cellulose consumption. The high heat (220 °C) from the microwave might be very influential in the process. The high heat could weaken the hydrogen bonds in cellulose [22]. The heat could even hydrolyze cellulose to smaller saccharide molecules, such as glucose and oligosaccharides. At high temperatures, water is easily ionized into hydrogen ions (H^+) and hydroxyl ions (OH^-) which can promote the hydrolysis of cellulose. After that, glucose was hydrated into 5-HMF by the Lewis acid site on the catalyst. That is why the value of glucose produced by samples without the presence of a catalyst was higher than the value of glucose produced by samples in the presence of a catalyst, while the 5-HMF value is the opposite. Thus, TiO_2 as a catalyst had the more effect in the process of converting glucose into 5-HMF, although the amount of 5-HMF produced was still low compared with its cellulose consumption value.

Due to the difficulty of the cellulose depolymerizing, the use of H_2O_2 as an oxidizing agent to facilitate the breakdown of cellulose was investigated. Previously, Zhang *et al.* [23] revealed that cellulose oxidation could reduce the molecular weight of cellulose, and modify its functional groups to be soluble in water. Further decomposition of H_2O_2 produced oxygen and water that are not harmful to the environment, so thus this process constitutes green chemistry.

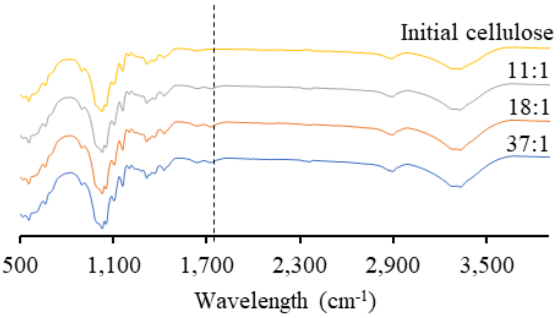

Figure 2. FTIR spectra of the cellulose with the different mole ratios of H_2O_2: initial cellulose applied during pretreatment process (11:1 mol.mol^{-1}, 18:1 mol.mol^{-1}, and 37:1 mol.mol^{-1}).

The results of the utilization of H_2O_2 in the microwave facilitated biomass conversion for samples without pretreatment are shown in Table 1 (No. 2-5). As the mole ratio of H_2O_2 increased from 0:1 to 37:1 mol.mol^{-1}, the value of cellulose consumption also increased. However, glucose and 5-HMF values decreased due to an increase in H_2O_2 from 18:1 to 37:1 mol.mol^{-1}. Ideally, an increase in cellulose consumption should be followed by an increase in glucose and 5-HMF yield. The presence of residual H_2O_2 may further oxidize glucose into smaller molecules, such as organic acids. At the utilization of 37:1 mol.mol^{-1}, which was the highest mole ratio, the aroma of organic acids was observed. The color of the reaction solution was bright yellow, which was not the same as the color of the other reaction solutions that were golden brown. As a result, 11:1 mol.mol^{-1} was identified as a suitable mole ratio of H_2O_2 for samples without pretreatment.

Cellulose pretreatment is an important method to depolymerize cellulose at the early state, so that cellulose is easily converted into desired products through a catalysis process. In this study, photocatalysis was chosen as a pretreatment process to achieve green conditions. The effects of H_2O_2 as pretreatment methods were investigated. The pretreatment by H_2O_2 at 37:1 mol.mol^{-1} resulted in high cellulose consumptions, but low glucose and 5-HMF as shown in Table 1 (No. 6). As mentioned above, this disparity might be caused by the further oxidization of cellulose to organic acids. This shortcoming could be overcome by the evaporation of H_2O_2 from the pretreated sample. As shown in Table 1 (No. 7-9), although the cellulose consumption decreased due to the H_2O_2 evaporation, the amounts of glucose and 5-HMF increased. For pretreated samples with H_2O_2 at 11:1, 18:1, and 37:1 mol.mol^{-1} followed by evaporation, the increases in cellulose consumption, glucose yield, and 5-HMF yield were in line with the increase in the mole ratio of H_2O_2. The highest 5-HMF yield is 13.07% at the presence of H_2O_2 37:1 mol.mol^{-1}. Thus, it was determined as the optimum mole ratio of H_2O_2 for this pretreatment.

The influences of the H_2O_2 mole ratio during pretreatment were further examined by FTIR. In this case, the pretreated samples without employing TiO_2 were chosen as the controlled samples for this observation. The presence of a large amount of TiO_2, which is 40% of the total cellulose, is an obstacle to producing clear FTIR spectra. At the very least, previous literature has reported that degradation of organic molecules has occurred more effectively under UV/TiO_2/H_2O_2 than UV/H_2O_2 [24]. Thus, the IR curves generated from these controlled samples can be an early indication for pretreated samples under TiO_2/H_2O_2/UV.

As shown in FTIR spectra (Figure 2), the presence of H_2O_2 in the pretreatment positively affected the formation of a peak at 1,730 cm^{-1}, which is a carboxyl group. In a similar fashion to the starch oxidation by H_2O_2 studied by Zhang *et al.* [23], the change in the FTIR indicates that the cellulose molecules were broken down. If H_2O_2 oxidizes the hydroxyl groups on C2 and/or C3 of anhydrous glucose units into carboxyl groups, the glycosidic bonds at C1 are weakened. Consequently, more depolymerization of the cellulose chains takes place. If this oxidation occurs at C6, it would result in weaker intermolecular hydrogen bonds among the cellulose molecule [25]. Overall, these would cause cellulose to be more amorphous and more susceptible to further chemical conversions into the desired product.

4. Conclusions

The optimum mole ratio of H_2O_2 obtained for photocatalytic pretreatment of cellulose in 5-HMF production was 37:1 $mol.mol^{-1}$, which was followed by the process of evaporation of the remaining H_2O_2 after pretreatment. The presence of residual H_2O_2 could reduce the productivity of 5-HMF because 5-HMF may be further oxidized to smaller molecules. The 5-HMF yield produced from the optimum conditions is 13.07%, while the 5-HMF yield produced from non-pretreated cellulose is 9.79%. In other words, pretreatment has succeeded in increasing the 5-HMF yield by 25.09% on controls. The improvement occurs because cellulose was depolymerized early by the presence of H_2O_2 under UV light so that the structure of cellulose is softened resulting in the more susceptible cellulose for further chemical conversions into 5-HMF.

Acknowledgment

This work was supported by the Thailand Research Fund (RSA6280106). We thank Center of Scientific Equipment for Advanced Research, Thammasat University (TUCSEAR) for supporting facilities and equipment.

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