

Alkaline Peroxide Pretreatment of Waste Lignocellulosic Sawdust for Total Reducing Sugars

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Abstract – The surge in the oil prices, increasing global population, climate change, and waste management problems are the major issues which have led to the development of biofuels from lignocellulosic wastes. Cellulosic or second generation (2G) bioethanol is produced from lignocellulosic biomass via pretreatment, hydrolysis, and fermentation. Pretreatment of lignocellulose is of considerable interest due to its influence on the technical, economic and environmental sustainability of cellulosic ethanol production. In this study, furniture waste sawdust was subjected to alkaline peroxide (H_2O_2) for the production of reducing sugars. Sawdust was pretreated at different concentrations from 1-3% H_2O_2 (v/v) loadings at a pH of 11.5 for a residence time of 15-240 min at 50, 75 and 90 °C. Optimum pretreatment conditions, such as time of reaction, operating temperature, and concentration of H_2O_2 , were varied and evaluated on the basis of the amount of total reducing sugars produced. It was found that the changes in the amount of lignin directly affected the yield of reducing sugars. A maximum of 50% reduction in the lignin composition was obtained, which yielded a maximum of 75.3% total reducing sugars yield and 3.76 g/L of glucose. At optimum pretreatment conditions of 2% H_2O_2 loading at 75 °C for 150 min, 3.46 g/L glucose concentration with a 69.26% total reducing sugars yield was obtained after 48 hr. of the hydrolysis process. Pretreatment resulted in lowering of crystallinity and distortion of the sawdust after the pretreatment, which was further confirmed by XRD and SEM results.

Key words: Alkaline peroxide, Biomass, Enzymatic hydrolysis, Lignocellulose, Pretreatment, Sawdust

1. Introduction

It is no exaggeration to say that ethanol from lignocellulosic biomass is the most promising fuel for the coming future. Currently, it is very important to expedite the industrial production of second-generation biofuels to replace other non-renewable fuels with bioethanol. The surge in the oil prices, increasing global population, climate change, and waste management problems are the major issues which have led to the development of biofuels from the lignocellulosic wastes [1]. Bioenergy from the present lignocellulosic waste, i.e., carbohydrates and lignin, will not only lessen its dependence on fossil fuels but will also offset the energy security concerns. Currently, most of the countries are promoting renewable energy from the biologically derived products to stimulate a bio-based economy [2-4]. High octane number, better combustion efficiency, good antiknock value, and most importantly, upon combustion, reduction in the emissions of the greenhouse gases makes ethanol a

better substituent for gasoline. Lignocellulosic biomass is an easily available low-cost raw material produced from various agricultural and forest activities, and municipal and industrial wastes. Bioethanol from the lignocellulosic wastes also avoids the food versus fuel debates, which makes lignocellulosic biomass materials as potential bioenergy feedstock [5].

Lignocellulose is a 3-D polymeric complex structure consisting of cellulose, hemicellulose, and lignin. Cellulose is a polymer chain of D-glucose units which are bundled together in the form of fibrils. Hemicellulose is amorphous, highly branched, lower molecular weight carbohydrate which acts a bridge between cellulose and lignin [6]. Thirdly, lignin is a non-carbohydrate polymer present in plant cells walls which offers impermeability and resistance to the different chemical and microbial attack [7]. The composition of the constituents varies according to the area, soil, climatic conditions etc. The composition of few lignocellulosic materials is summarized in Table 1 [8-17].

So, an efficient pretreatment process is necessary to unlock the complex lignocellulosic structure for the extraction of cellulose and to remove hemicellulose and lignin. Pretreatment serves many purposes. It decreases cellulose crystallinity and increases the accessible surface area for enzymatic hydrolysis. It also increases the digestibility of cellulose with the minimum degradation caused due to pretreatment

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Table 1. Composition of few lignocellulosic materials

Component	Composition							Ref.
	Glucan	Xylan	Arbinan	Galactan	Mannan	Lignin	Ash	
Kanlow Switchgrass	33.6	24.7	3.9	1.3	0.1	25.6(K)	1.7	[16]
switchgrass	31.99	17.9	1.87	1.73	N.D.	21.37	N.D.	[11]
switchgrass	39.5±1.8	20.3±1.4	2.1±0.0	2.6±0.0	N.D.	21.8±0.2	4.1±0.1	[16]
switchgrass	30.0±3.6	18.4±2.1	N.D.	N.D.	0.3±0.09	8.6	N.D.	[15]
corn stover	41.6±0.2	27.7±0.4	3.3±0.0	N.D.	N.D.	20.2±0.2	4.6±0.0	[8]
corn stover	38.5±0.1	21.6±0.2	2.6±0.03	N.D.	0.5±0.02	4.2	N.D.	[15]
corn stover	36.2	20.1	3	1.45	N.D.	21.2	1.56	[10]
Big bluestem	37.6±0.1	19.9±0.1	2.4±0.02	N.D.	0.4±0.01	6.9	N.D.	[15]
Mixed Hardwood	42.8±0.5	13.4±0.1	0.6±0.01	N.D.	1.8±0.01	16.1	N.D.	[15]
Mixed perennial grasses	36.0±0.3	21.5±0.1	2.5±0.03	N.D.	0.5±0.04	7	N.D.	[15]
Spruce	49.76	5.4	N.D.	N.D.	10.35	30.60(K)	N.D.	[9]
Spruce	45.4	3.8	1.3	3.2	10.3	31.5	N.D.	[14]
Coastal bermuda grass	25.59	15.88	1.95	1.46	N.D.	19.33	N.D.	[12]
Yellow poplar wood	65.9	N.D.	N.D.	N.D.	N.D.	23.5	N.D.	[17]

*N.D. – Not Determined

(K) – Klason Lignin only

severity. Further pretreatment eliminates the formation of toxic compounds, which can inhibit the hydrolysis yield and increase the solubility of the feedstock by the activation of cellulose [11,18].

Although there are number of pretreatment processes such as thermo-chemical, mechanical, and biological pretreatments, none of them proved satisfactory in regard to its sustainability. Dilute acid pretreatment with H_2SO_4 and HCl, etc. is simpler and has proved effective in hydrolyzing hemicellulose but requires more energy and corrosion-resistant reactors, which makes the process more expensive [3]. Although hot water pretreatment does not generate any inhibitory compounds like hydroxymethylfurfural (HMF) and acetic acid, its unfeasibility with soft woods limits its use. Ozone pretreatment is effective for the reduction in the lignin content with no formation of the inhibitory compounds, but a large amount of ozone gas is required, which makes it a quite expensive process. Alkaline pretreatment through sodium hydroxide has proven effective in hydrolyzing hemicellulose and altering the lignin structure at the expense of longer reaction times [12,19]. The effectiveness of the pretreatment varies from method to method and according to the feedstock used. Alkaline pretreatment has an advantage over the other treatment methods due to its effectiveness even at mild temperature.

H_2O_2 is considered an environmentally benign chemical that is even effective at lower temperature ranges and at lower loadings. H_2O_2 is an oxidizing agent that has been studied by many researchers [12,19-21]. Banerjee et al. and Qi et al. have examined the alkaline peroxide treatment on grass stovers and wheat straw [13,22].

These considerations prompted us to study the feasibility of alkaline peroxide H_2O_2 on waste furniture sawdust for the production of reduction sugars [23-25]. The solubility of the pretreated sawdust, which is an important parameter during enzymatic hydrolysis, was also determined. Higher solubility of the raw materials would help in the easy percolation of the enzymes for the better hydrolysis process and in increasing the reducing sugars yield.

2. Materials and methods

2-1. Materials and Reagents

Waste furniture sawdust was collected from a local furniture making company in Seoul. It was screened through 40 mesh size and then dried at 60 °C for 12h. H_2O_2 , NaOH, ethanol and, toluene was purchased from sigma Duksan Pure Chemical Co., Ltd. Cellulase Onozuka Y-NC (Enzyme *Aspergillus niger*) was purchased from Yakult Co., Ltd., Japan.

2-2. Alkaline Peroxide Pretreatment

Before every pretreatment process, sawdust was subjected to toluene: ethanol (2:1 (v/v)) in the soxhlet apparatus for the extractive removal process. Extractive free sawdust was then washed and dried at 60 °C. A sample of 4 gm of extractive free sawdust was stirred with alkaline hydrogen peroxide. pH of 11.5 was adjusted with 0.5 M NaOH. The solid to liquid ratio was maintained at 1:25(w/v). The pretreatment was conducted at three different temperatures (50, 75, and 90 °C) for 4.5 hours at 150 rpm in a water thermostat. The oxidative delignification was carried out by 1, 2, and 3% H_2O_2 in 250 ml bottles. The insoluble pulp was filtered and washed with distilled water until the pH reached 7. The pretreated insoluble pulp was then dried at 70 °C for 2 hr. and stored in a desiccator for further hydrolysis process.

2-3. Saccharification Process

2 ml of commercially available enzyme from *Aspergillus Niger* was added to 0.05 M solution of sodium acetate and an acetic buffer at a pH of 4.8. The cellulase activity of enzyme was assayed to be 0.84 filter paper unit (FPU) by using Whatman filter paper No. 1 according to the procedure described by Ghose [26]. A unit activity is defined as the amount of enzyme that produces 1.0 μ mol of reducing sugar/min from the feed used. The reactions were carried out in 50

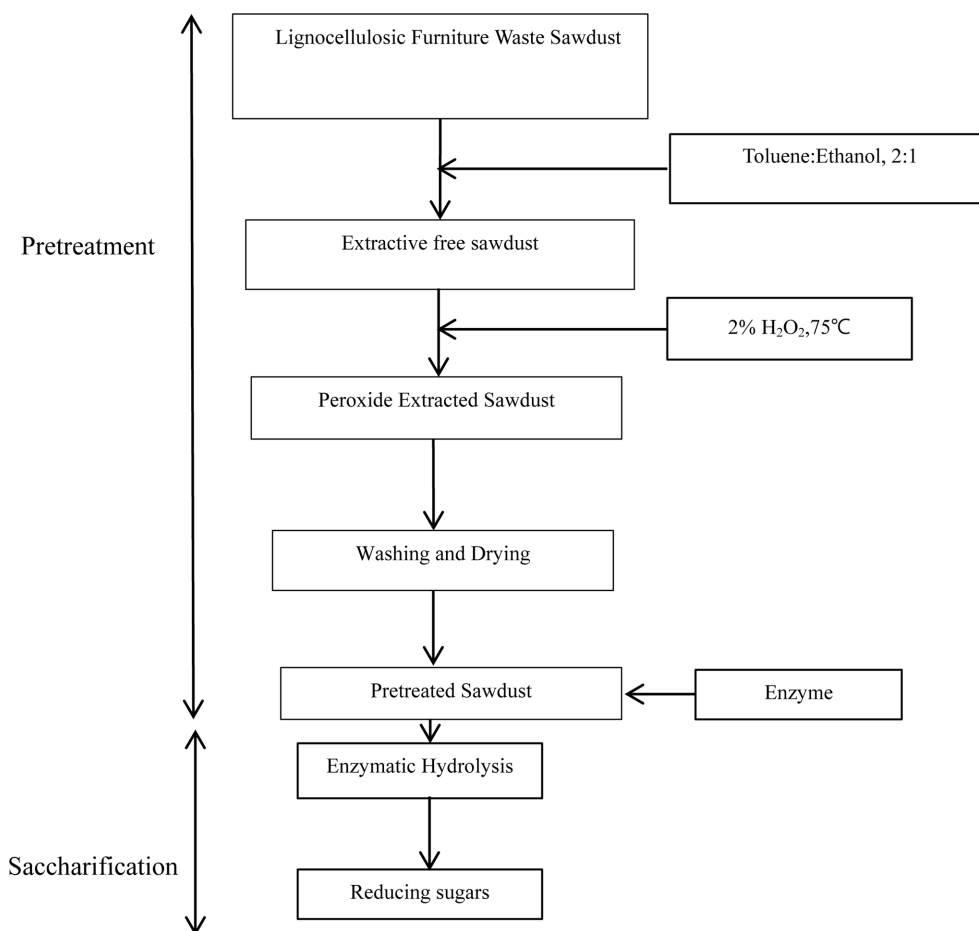


Fig. 1. Flow diagram of the process for the production of reducing sugars.

ml falcon tubes at 150 rpm in a water thermostat at 50 °C. Tubes were taken out after a particular time interval to check the change in the glucose concentration and total reducing sugars yield. After the incubation period, 3 ml of alkaline 3,5-dinitrosalicylic acid (DNS) was added and boiled to deactivate the enzymes. The tubes were then cooled, and the solution was filtered with 0.2 µm Whatman nylon filters for sugars analysis. The complete flow diagram of the experimental process is shown in Fig. 1.

2-4. Analytical Procedures

Klason lignin was determined according to NREL Laboratory Analytical Procedures [27]. Total reducing sugars were analyzed by the dinitrosalicylic acid (DNS) method [28]. Filter paper activity (FPase, units of $\text{mg}^{-1} \text{mL}^{-1}$) was assayed at a pH of 4.8 and 50 °C [29]. Solubility was determined by mixing 1 gm of pretreated sawdust in 100 ml of the distilled water for 10 hr. at 60 rpm and at 25 °C. Solubility was calculated according to the following equation:

$$\text{Solubility (\%)} = (M(g) - R(g)) / M(g) \times 100 \quad (1)$$

where M is the dried mass of the mixed pretreated sample and R is the dried mass of the insoluble sample remaining after filtration.

The crystallinity of raw and pretreated samples was characterized

through X-ray diffraction (XRD). Analyses were conducted using an XRD-6000 (Shimadzu, Japan) with Cu K α radiation of $\lambda = 1.541 \text{ \AA}$ between $2\theta = 10^\circ$ to 40° at 40.0 kV and 30.0 mA with a scanning speed of 4.0 (deg/min). To obtain more insight into pretreatment effects, surface morphology was conducted using JSM-6701F FESEM (JEOL, Japan). Samples were platinum coated by sputter coater before the microscopy.

3. Results and Discussion

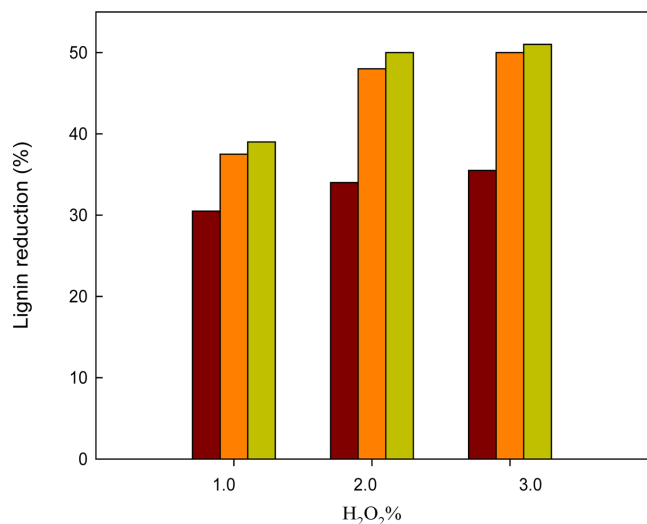
3-1. Pretreatment of Waste Lignocellulosic Sawdust

The percentage composition of untreated and pretreated furniture waste sawdust after each pretreatment attack is shown in Table 2. Untreated sawdust contains 39.02% cellulose, 26.98% hemicellulose, and 28% lignin. The concentration of cellulose increased after each pretreatment with a significant decrease in the hemicellulose and lignin composition. The content of cellulose increased from 39.02% to 75.2% after pretreatment at 75 °C. Maximum decrease from 26.98% to 6.39% in amount of hemicellulose and 50% decrease in the lignin composition were obtained.

The change in the lignin composition was studied at different temperatures to determine the optimum operating temperature condition

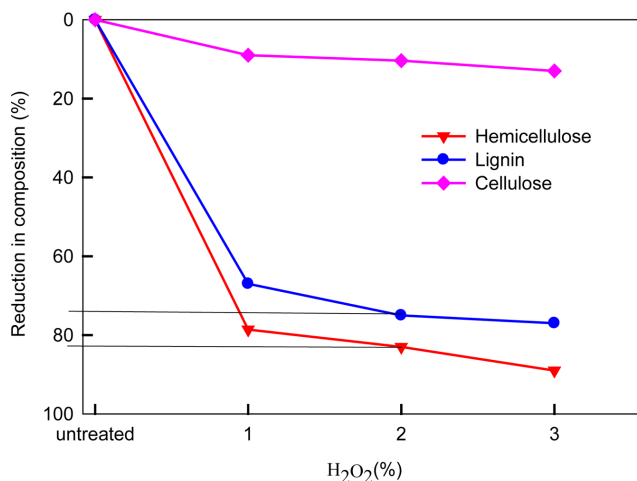
Table 2. Chemical composition (wt%) of untreated (raw) and pretreated sawdust

H ₂ O ₂ Loading (%)	Cellulose	Hemicellulose	Lignin
Untreated Sawdust	39.02	26.98	28.00
1.0	66.91	10.59	17.50
2.0	71.52	9.44	14.56
3.0	75.20	6.36	14.00

**Fig. 2. The effect of H₂O₂ concentration on the lignin reduction at different temperatures: 50 °C (dark brown); 75 °C (orange); 90 °C (green).**

for the pretreatment. A pretreatment process should be optimized to facilitate fast enzymatic hydrolysis at lower enzyme loading [30]. Lignin, which acts as a physical barrier for the enzymatic hydrolysis, has to be removed beforehand for reducing the pretreatment severity and for proper hydrolysis process with lesser amount of the enzyme consumption. Fig. 2 shows the amount of lignin removal with the change in both temperature and concentration of H₂O₂. Lignin removal increases with the increase in temperature. A considerable amount of lignin reduction was obtained between 50 °C and 75 °C, and 1% H₂O₂ and 2% H₂O₂. Increase in the temperature is believed to break the lignin shield, which makes easy accessibility of the carbohydrates. Further increase in temperature and concentration does not offer significant increase in the lignin removal.

Fig. 3 shows the effect of pretreatment on the amount of the cellulose, hemicellulose and lignin as a function as a function of H₂O₂ loading. Although there was an increase in the composition of cellulose, the amount of cellulose remaining for the further enzymatic process decreased due the pretreatment severity. It is very important to optimize the operating conditions like temperature, reaction time, and concentration with respect to cellulose degradation, which directly affects the reducing sugars yield. It has been found that the actual amount of reduction in cellulose, hemicellulose, and lignin with 1% H₂O₂ attack at 75 °C was 4.6%, 78%, and 65.22%, respectively. A maximum of 8.8% reduction in actual cellulose amount occurred with 3% attack. This pretreatment showed good signs of minimum degradation for cellulose with maximum removal of 88.84%

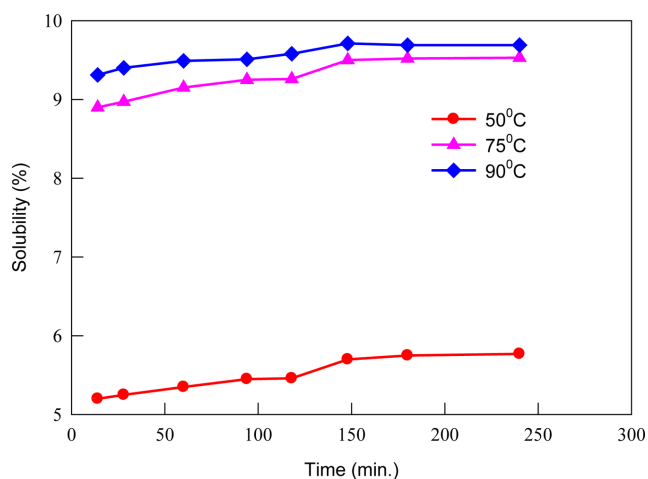
**Fig. 3. The pretreatment effect of increasing H₂O₂ loading on the reduction in the actual amount of the components.**

hemicellulose and 76.35% lignin.

Banerjee et al. have shown that the process is effective on grass stoves, which yielded 95% monomeric glucose at 0.5 g/g biomass H₂O₂ loading [22]. Qi et al. examined the alkaline peroxide treatment on the wheat straw with a significant reduction in the amount of lignin from 13.49% before pretreatment to 4.59% after pretreatment [13].

3-2. Solubility of Pretreated Sawdust

Solubility of the pretreated sawdust was determined with respect to the pretreatment time at 50, 75, and 90 °C as shown in Fig. 4. Solubility of the pretreated sawdust increased with increase in

**Fig. 4. Effect of pretreatment time and temperature on the solubility of the sawdust.**

pretreatment time owing to the longer contact in the reaction media [31]. Maximum solubility was achieved at 150 min of the pretreatment time, after which it remained constant. Increase in temperature showed a noticeable increase in solubility of the pretreated sawdust. Maximum solubility of 10.2% was achieved at 90 °C. The optimum conditions of the pretreatment were determined based on the reducing sugars yield.

3-3. Enzymatic Saccharification

Enzymatic hydrolysis of both untreated and pretreated furniture waste sawdust at three different temperatures 50, 75, and 90 °C was carried out to determine the optimum conditions for alkaline peroxide pretreatment as shown in Fig. 5(a-c), respectively. The hydrolysis process was conducted for 72 hours, glucose concentration and total reducing sugars yield were analyzed at different time intervals. A quick conversion of the pretreated sawdust occurred in the first 24 h followed by a little increase in the reducing sugars production after 48 hours due to slow enzymatic hydrolysis. Continuous increases in the H₂O₂ dosage also led to the increase in the reducing sugars yield.

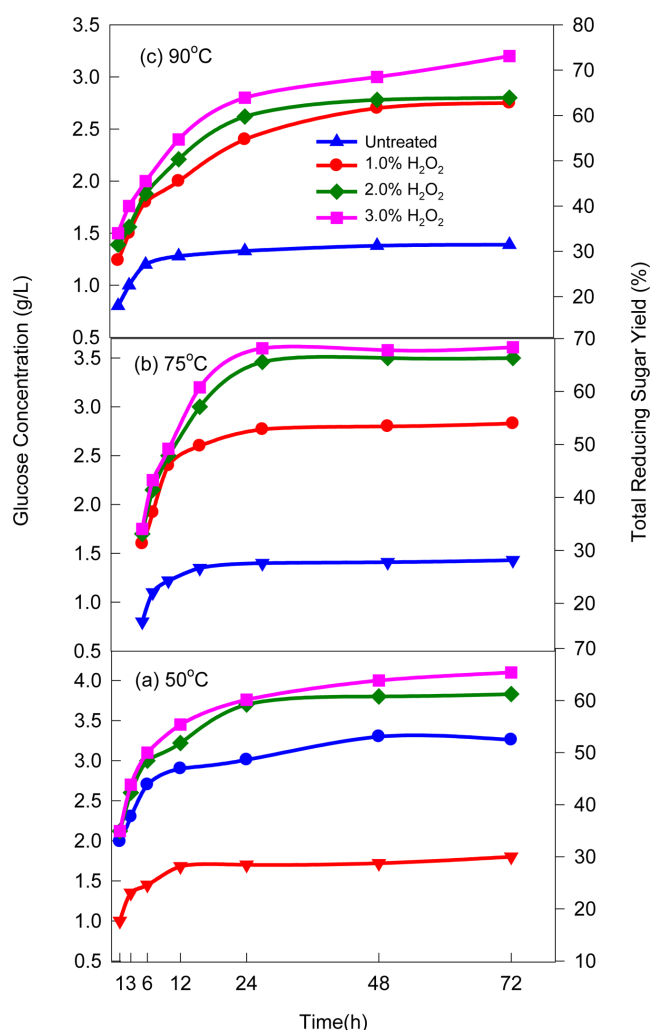


Fig. 5. Effects of H₂O₂ loading and pretreatment temperature on the glucose concentration and total reducing sugar yield with 0.5 g/l enzyme loading.

At 90°C, the reducing sugars yield increased from 62.1% at 1% H₂O₂ loading to 75.3% at 3% H₂O₂ loading along with glucose concentration also increased from 3.01 g/L to 3.76 g/L, respectively. This increase was due to the removal of lignin and due to the weakening of the hydrogen bonding, which made cellulose easily accessible for better enzymatic activity. As shown in the figures, the increase in the pretreatment temperature from 75 °C to 90 °C did not work well in enhancing the reducing sugars yield even though there was decrease in the lignin reduction. For enzymatic hydrolysis process, pretreatment with 2% H₂O₂ loading at 75 °C for 150 min was considered optimum, which produced 3.46 g/L glucose concentration with a 69.26% total reducing sugars yield. The conditions found were suitable with the lesser amount of cellulose degradation and more lignin removal. Banerjee et al. showed that the process is effective on grass stoves, which yielded 95% monomeric glucose at 0.5g/g biomass H₂O₂ loading [22].

3-4. Sawdust Crystallinity

Reduction in the sawdust crystallinity after the pretreatment can significantly increase the hydrolysis efficiency. Analysis of the untreated sawdust (Fig. 6a) and the pretreated sawdust (Fig. 6b) was performed through XRD. The peaks were normalized for the better understanding of the pretreatment effect [18,30]. Reduction in the peak heights at 2θ = 22.4, 15.8, and 34.6 were observed, which signifies the reduction in the crystallinity. The peak broadening and peak shifting at 2θ = 15.8 towards the lower angles clearly indicate the weakening of the hydrogen bonding and increase in the intermolecular distance of the cellulosic units.

3-5. SEM Imaging

The morphology of the untreated and alkaline peroxide pretreated sawdust samples was studied by SEM imaging (Fig. 7 (a-c)). Images showed significant changes on the fiber surfaces, as shown in Fig. 7. Fig. 7(a) shows the surface of the extracted free sawdust, and Fig.

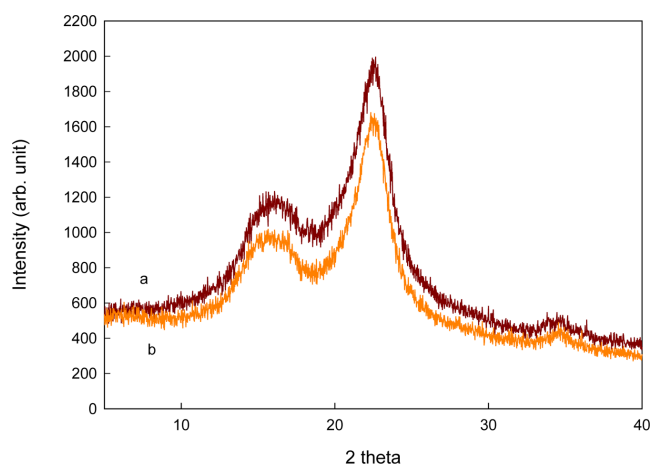
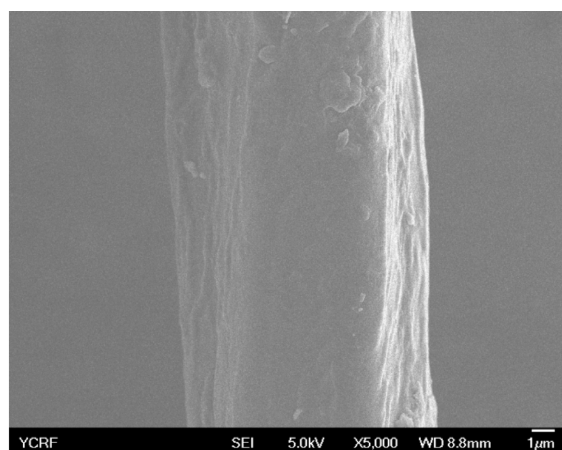
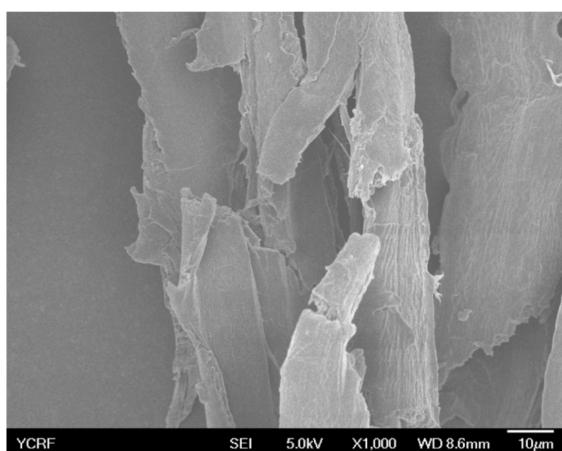


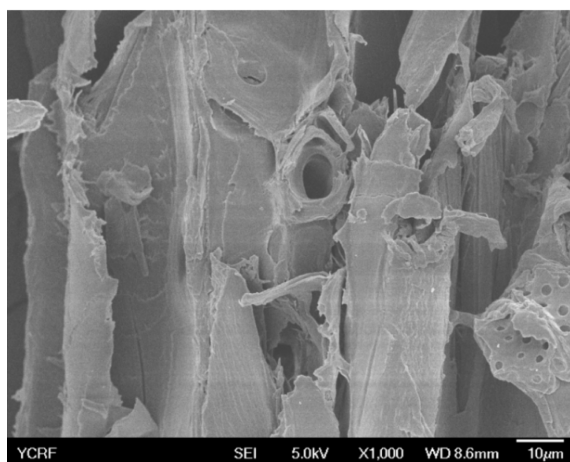
Fig. 6. XRD of untreated sawdust (a) and pretreated sawdust (b) after alkaline peroxide attack at optimized conditions [18] [30].



(a)



(b)



(c)

Fig. 7. SEM images showing surface of (a) untreated extracted sawdust, (b) and (c) after pretreatment sawdust at the optimized pretreatment conditions.

Fig. 7(b) and Fig. 7(c) present the images of the deformed structure after pretreatment. Untreated extracted free sawdust has a smooth, intact, and even surface, which after the pretreatment got completely distorted with the formation of holes. This shows the breaking of the strong 1-4 glycosidic bonding and reduction in the crystallinity due

to the swelling of lignocellulosic structure by the alkaline attack. The increase in the surface area of the fragmented surface after the pretreatment was suitable for the hydrolysis process.

4. Conclusion

The alkaline peroxide pretreatment process for furniture waste sawdust was investigated for the production of reducing sugar. This pretreatment has shown positive results for removal of lignin and hemicellulose with minimum degradation of cellulose. Solubility of the pretreated substrate also increased with increase in the alkaline peroxide loading by the activation of cellulose. Sawdust was pretreated at different concentrations from 1-3% H_2O_2 (v/v) loadings at a pH of 11.5 for a residence time of 15-240 min at 50, 75 and 90 °C. Optimum pretreatment conditions such as time of reaction, operating temperature, and concentration of H_2O_2 were varied and evaluated on the basis of the amount of total reducing sugars produced. It was found that the changes in the amount of lignin directly affected the yield of reducing sugars. A maximum of 50% reduction in the lignin composition was obtained, which yielded a maximum of 75.3% total reducing sugars yield and 3.76 g/L of glucose. At optimum pretreatment conditions of 2% H_2O_2 loading at 75 °C for 150 min, 3.46 g/L glucose concentration with a 69.26% total reducing sugars yield was obtained after 48 hr. of the hydrolysis process. The XRD and SEM study of untreated and pretreated sawdust confirms the lowering of crystallinity and distortion of the sawdust after the pretreatment, which was further confirmed by XRD and SEM results.

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