



Evaluation of Hydrothermal Pretreatment on Lignocellulose-Based Waste Furniture Boards for Enzymatic Hydrolysis

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Abstract

Three typical waste furniture boards, including fiberboard, chipboard, and blockboard, were pretreated with conventional hydrothermal method. The responses of chemical composition, physicochemical morphology, and performances of enzymatic hydrolysis were evaluated. Results indicated the almost complete hemicellulose removal at higher pretreatment temperatures, the enhanced crystallinity index, and disordered morphology of the pretreated substrates indicated that the hydrothermal pretreatment deconstructed these boards well. However, the very low enzymatic hydrolysis (< 8% after 72 h) of the pretreated substrates showed the poor biological conversion. Three hypotheses for the weakened enzymatic hydrolysis were investigated, and results indicated that the residual adhesives and their degraded fractions were mainly responsible for poor hydrolysis. When NaOH post-pretreatment was attempted, cellulose-glucose conversion of the hydrothermally pretreated fiberboard, chipboard and blockboard can be improved to 28.5%, 24.1%, and 37.5%. Herein, the process of NaOH hydrothermal pretreatment was integrated, by which the hydrolysis of pretreated fiberboard, chipboard and blockboard was greatly promoted to 47.1%, 37.3%, and 53.8%, suggesting a possible way to pretreat these unconventional recalcitrant biomasses.

Keywords Waste furniture boards · Hydrothermal pretreatment · Post-pretreatment · Integration process · Enzyme-hydrolytic performances

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Introduction

Buoyed by a strong domestic economy, construction sector, and requirements of furniture, China's furniture industry has grown rapidly over the past 10 years and has become an important industry to produce many kinds of furniture for demands of domestic and foreign markets [1]. Lignocellulose-based materials played an important role in furniture production, in which 70% share of furniture boards were wood-based materials, and the wood consumption in furniture industry accounted for 10–15% of the total wood industry. However, approximately 6.0×10^7 t (equal to 8.5×10^7 m³) of the used furniture was annually discarded as solid waste in China [2]. The discarded furniture was accompanied by the problems of piled-up randomly without systematic recycling paths and appropriate valorizing technologies, and these issues are urgently confronting the solid waste management sector. The waste furniture was typically characterized by large volume, low recovery cost, and easy collection, and it could be valorized further through proper treatments. Therefore, turning the waste management challenge into an economic opportunity to achieve circular economy will be a consensual choice for future society [3].

The traditional disposal ways to this waste furniture were landfill and incineration [4, 5]. Landfill is a limited way owing to insufficient landfill space and other potential risks, such as the generation of leachates and soil contamination [6]. Although incineration plays an important role in solving the problem of insufficient space for landfill [7], its impact on the environment and human health cannot be ignored for the release of CO₂, CH₄, N₂O, and dioxin from incineration process [8]. Moreover, it is worth noting that the waste furniture cannot be burned directly due to the containing adhesive and paint coating, which would potentially yield a lot of harmful substances in the exhausted smoke. Overall, the disposal ways for these generated waste furniture are an urgent problem to be solved now, and how to valorize these wastes is gradually becoming a crucial issue, after all, the existing methods not only suffers a resource waste of natural lignocellulose, but also results in potential environment pollutions.

Basically, furniture boards was mainly made from some waste lignocellulosic resources, such as reed, wheat straw, and wood chip, which thereby had the conversion potential in biological route based on a sugar platform [9, 10]. The technology to convert the lignocellulosic substrates into fermentable monosaccharides, and then further convert into liquid fuels and other bio-based chemicals will be a positive way to achieve more sustainability in the waste management and waste valorization. In this process, pretreatment is a crucial step to open the nature structural recalcitrance of raw materials for the good performances on the subsequent biochemical conversion. Additionally, these furniture boards are generally manufactured at higher temperature and pressure with adding different types of adhesives. Thus, the nature recalcitrance of lignocellulosic biomass and additional adhesives will greatly intensify the pretreatment difficulty for the conversion on the sugar platform [11]. Therefore, it was necessary to select appropriate pretreatment methods to facilitate these biomasses for subsequent conversion.

Various pretreatment technologies, including physical, chemical, physicochemical, and biological method, have been widely developed for natural lignocellulosic biomass in recent years. Pretreatment made the substrates more accessible during the process of enzymatic hydrolysis through disrupted the crystal structure of cellulose and increased the available surface area [12]. Meanwhile, it was reported that the combination of two or more pretreatment methods may be more effective than separate single one [13]. Hydrothermal pretreatment is an

autohydrolysis pretreatment with water only as the reaction medium. It was considered to be environmentally friendly and can promote sugar conversion performance of lignocellulosic biomass [14]. Partial fractions of the lignocellulosic biomass, especially hemicellulose, will be firstly degraded into oligosaccharides, as the pretreatment temperature was enhanced to 150–180 °C, and lignin also started to be depolymerized [15]. It has been shown that nearly 80% hemicellulose can be removed after hydrothermal pretreatment [16]. Moreover, the structure of lignin also can be changed during hydrothermal pretreatment via depolymerization or repolymerization [17]. However, there are some potential difficulties of hydrothermally pretreating furniture boards according to their manufacturing process. The contained adhesives and paints may be melted and tightly attached on the substrates, which may affect the accessibility of enzymes. Moreover, these additives, as refractory polymer organic compounds, will probably be degraded into some small molecules with toxicity, which may inactivate the employed enzyme or microorganisms in the following biochemical process [5]. Therefore, the enzymatic hydrolysis of the hydrothermally pretreated waste furniture boards deserves evaluating more compared to the traditional natural lignocellulose.

In this work, 3 frequently used waste furniture boards (fiberboard, chipboard, and blockboard) were pretreated with hydrothermal pretreatment under various temperatures. The efficiency of hydrothermal pretreatment on these furniture boards was evaluated based on the responses of chemical composition, performances of enzymatic hydrolysis, and the changes of crystallinity and morphology. Furthermore, some possible difficulties, which may potentially affect hydrothermal pretreatment and subsequent sugar conversion, were analyzed. Based on the analysis results, the post-pretreatment method and integrated process were proposed accordingly to improve the hydrolysis. Overall, this work can provide a technical support for the valorization of this unconventional waste biomass in a possible way.

Materials and Methods

Feedstocks and Chemicals

Three waste furniture boards in real world, including fiberboard, chipboard, and blockboard, were collected from Chang'an landfill site, Chengdu, China. The main chemical composition of these 3 furniture boards is listed in Table 1. The obtained furniture boards were air-dried and milled through 20-mesh (the corresponding particle size was less than 0.850 mm). The milled substrates were stored in a plastic bag at ambient temperature for the subsequent pretreatment. The employed cellulase for enzymatic hydrolysis was Cellic® CTec2, which was gifted by Beijing branch of Novozymes in China. The protein concentration in the cocktail of Cellic® CTec2 was determined as 228.72 mg/mL.

The other related chemicals in this work, such as acetic acid, sodium acetate, glucose, and xylose, were all provided by the Cologne Chemical Company, Chengdu, China.

Hydrothermal Pretreatment

The milled waste furniture boards were presoaked in distilled water with solid-liquid ratio of 1:30 (*w/v*) for 12 h. The slurry was transferred into a 1.5-L reaction kettle for the pretreatment following the designed temperatures of 150, 180, 210, and 250 °C for 15 min, respectively. A top-mounted motor was employed for stirring with 100 rev./min during pretreatment. When a

Table 1 Chemical compositions of 3 waste furniture boards

Samples	Chemical composition (%)					
	Cellulose ^a	Hemicellulose ^b	Acid-insoluble lignin ^c	Acid-soluble lignin	Extractives	Ash
Fiberboard	31.6 ± 0.30	10.0 ± 0.35	28.8 ± 0.08	2.6 ± 0.04	9.5 ± 0.01	2.4 ± 0.01
Chipboard	28.2 ± 0.23	8.7 ± 0.42	30.3 ± 0.31	1.6 ± 0.05	5.4 ± 0.01	3.8 ± 0.09
Blockboard	29.5 ± 0.29	10.1 ± 0.42	24.0 ± 0.34	2.4 ± 0.01	5.9 ± 0.47	5.4 ± 0.13

^a Cellulose content was represented by glucan content

^b Hemicellulose content was represented by xylan content

^c Adhesive that cannot be dissolved by sulfuric acid, thereby was included in acid-insoluble lignin

batch of pretreatment was completed, the reaction kettle was cooled down to room temperature to transfer the slurry out, and 800 mL distilled water was employed to wash the reactor so that the pretreated substrate can be harvested as completely as possible. Afterwards, the slurry was filtered by filter paper in a Buchner funnel, and the solid fraction was washed further by the distilled water until pH of approximately 5.0.

Enzymatic Hydrolysis

The enzymatic hydrolysis of all samples was performed in 25-mL serum bottles with working volume of 12.0 mL. The solid loading of pretreated substrates for enzymatic hydrolysis was controlled as 2% (*w/w*, dry basis), and the acetate buffer (0.05 M, pH 5.0) was supplemented to maintain a stable pH during hydrolysis. The relatively enzyme input of Cellic® CTec2 was 20 mg protein/g cellulose to ensure enough enzyme loading and eliminate the negative effects from limitation of enzyme loading. Sixty microliters of tetracycline solution (approximately 40 mg/L) was added prior to the enzymatic hydrolysis to inhibit the growth of microorganisms that may consume the yielded glucose from cellulose hydrolysis. The bottles were shaken in an orbital shaker at 50 °C with shaking frequency of 150 rev./min, and each hydrolysis group was performed in duplicated. During the hydrolysis, approximately 500 µL of the hydrolysate was periodically sampled at 0, 4, 8, 12, 24, 48, and 72 h. The sampled hydrolysates were heated at 100 °C for 5 min to inactivate the enzymes, then centrifuged at 4 °C with 1.0×10^5 rev./min for 5 min. The supernatant was stored in the -20 °C freezer for further sugar determination.

Determination of Glucose Concentration in Enzymatic Hydrolysate

A high-performance liquid chromatography (HPLC) (1260 Infinity II, Agilent, Santa Clara, CA, USA) with a sugar column (SH1011, Shodex, Showa Denko America, Inc., New York, NY, USA) at 60 °C, in which 0.05 mol/L H₂SO₄ was employed as the mobile phase with flow rate of 0.8 mL/min, and a refractive index detector was employed at 50 °C to determine the concentration of glucose in the supernatant of enzymatic hydrolysis. The standard curve was prepared with quantitative glucose standard solution (2.0 mg/mL) and nanopure water, and 200 µL lactose solution (1.0 mg/mL) was added as an internal standard. Fifty microliters supernatant of enzymatic hydrolysis was diluted by nanopure water and the fixed 200 µL lactose solution to make sure the total volume was 1.0 mL, then 10 µL of the mixed liquid was auto-injected into HPLC for glucose determination. The standard curves were employed to calculate the concentrations of glucose in hydrolysate, and every sample analysis by HPLC was performed in duplicated.

Observation by SEM

The untreated and hydrothermally pretreated substrates were observed by a scanning electron microscope (SEM) (Sigma 500, ZEISS, Germany). The air-dried samples were mounted on aluminum stubs using carbon tape and coated with Au/Pt in the presence of argon gas. The images were captured at low vacuum at 20 kV and taken at $\times 2000$ magnifications [18].

Determination on the Degree of Crystallinity

Untreated and hydrothermally pretreated samples were analyzed by X-ray diffraction (XRD), in which the samples were scanned using Cu-K $_{\alpha}$ radiation ($k=0.1540$ nm) at an accelerating voltage of 40 kV and a current of 40 mA. Scans were collected from 10° to 50° with a step interval of 0.02° . The degree of crystallinity can be relatively expressed by the percentage crystallinity index (CrI, %). CrI calculation was described by the following equation (Eq. 1).

$$\text{CrI (\%)} = \frac{I_{002} - I_{am}}{I_{002}} \times 100 \quad (1)$$

where I_{002} is the maximum intensity of 2θ close to 22° and I_{am} corresponds to the minimum intensity of 2θ close to 18° [19].

Results and Discussion

Evaluation of Performances of Hydrothermal Pretreatment on Waste Furniture Boards

It is well known that the hydrothermal process with high temperature and high pressure provides a supercritical condition, by which some typical responses on the main compositions of lignocellulosic biomass can be observed after pretreatment [20]. The hemicellulose fraction is extremely susceptible with the increased pretreatment temperature; thus, it will be firstly degraded into oligosaccharides, and then degradation products akin to catalysts further promotes the hemicellulose degradation [15, 21]. Meanwhile, lignin fraction was reported to condense with other degradation products to form pseudo-lignin at high temperatures (especially higher than 210°C), which will lead to an increase in lignin content [22, 23]. To check the pretreatment performances on these unconventional feedstocks of waste furniture boards, they were hydrothermally pretreated using the increased temperatures from 150 to 250°C to check the typical responses on the main fractions.

After hydrothermal pretreatment, the main chemical compositions of the recovered solids are displayed in Fig. 1. As expected, the contained hemicellulose was degraded more seriously with the increased temperatures. It was noticed that the hemicellulose content of fiberboard, chipboard, and blockboard was relatively increased to 12.6%, 13.6%, and 13.6%, respectively, as the hydrothermal pretreatment was performed at

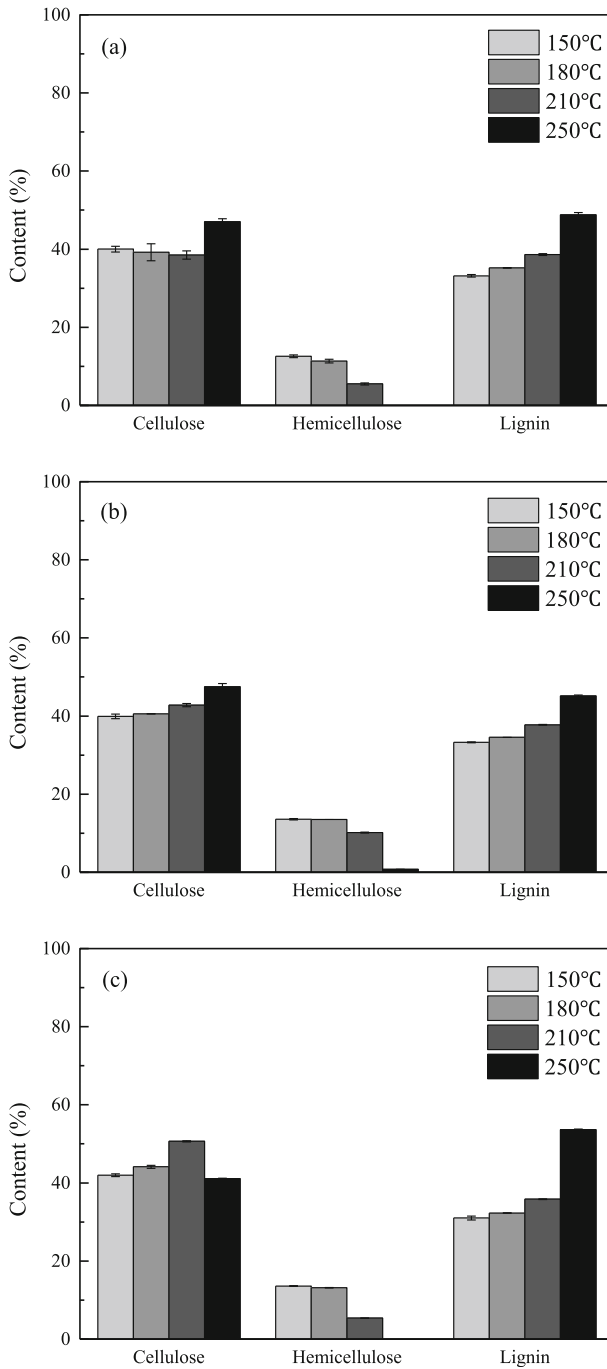


Fig. 1 Chemical composition of these 3 hydrothermally pretreated furniture boards under designed temperatures (a: fiberboard; b: chipboard; c: blockboard)

150 °C. When the pretreatment temperature was increased, the relative hemicellulose content of these 3 boards was reduced significantly, even to zero by the pretreatment at 250 °C, indicating the enhanced pretreatment temperature achieved the promoted hemicellulose removal. These results on the hemicellulose fraction were basically similar to the hydrothermal pretreatment on other conventional lignocelluloses, such as hardwood (poplar branches and grapevine pruning), softwood (pine sawdust), and sunflower oil cake, in which almost no hemicellulose could be detected at very high pretreatment temperature of 220 °C for 15 min [24, 25].

Unlike the degradation of hemicellulose with the increased pretreatment temperature, an increase of lignin content to 48.8%, 45.2%, and 53.6% after hydrothermal pretreatment can be observed on the fiberboard, chipboard, and blockboard, respectively (Fig. 1). In previous studies, the lignin content of other biomasses, sunflower oil cake and bamboo, was increased by 16.0% and 12.5%, respectively, through hydrothermal pretreatment [25, 26]. It was reported that condensation reaction and hemicellulose degradation led to a relative increase of lignin content and hydrothermal pretreatment was a redistribution process for lignin rather than the direct removal process [27, 28].

The cellulose fraction was more stable during hydrothermal pretreatment than hemicellulose and lignin fraction. It was attributed to the highly resistant nature of the cellulose microfibrils [16, 29]. As shown in Fig. 1, the cellulose content of the hydrothermally pretreated fiberboard varied from 38.5 to 47.0% compared with 31.6% of the untreated fiberboard. Similarly, the cellulose content of chipboard and blockboard varied in the range of 39.9–47.5% and 41.1–50.7% after pretreatment, which all typically higher than their corresponding untreated raw boards. These results were comparable to the hydrothermal pretreatment on the various feedstocks, such as rapeseed straw, oil palm biomass, and mixed hardwood [15, 27, 30]. Besides, hydrothermal pretreatment can remove the amorphous fraction of hemicellulose while it showed less effects on the disruption of the highly crystalline cellulose. The X-ray diffraction diagrams of these 3 raw substrates and its corresponding pretreated substrates were displayed in Supplementary file (Fig. S1), and the crystallinity index (CrI) was calculated correspondingly. The initial CrI of fiberboard, chipboard, and blockboard was 43.6%, 46.2%, and 47.1%, respectively, which was correspondingly increased to 45.7%, 50.3%, and 50.4% after the hydrothermal pretreatment at 180 °C. It was hard to say the increase on CrI after hydrothermal pretreatment at 180 °C was significant compared with their corresponding raw furniture boards; however, it was reported that the significant increase on CrI (approximately 13%) can be observed under the optimized hydrothermal pretreatment on corn cob [19]. Moreover, the similar observations also happened on the other pretreatment technologies for lignocellulosic biomass, such as alkali pretreatment, and microwave irradiation, which was due to the solubilization or removal of the amorphous hemicellulose by pretreatment [31]. Besides, some obvious cracks and chaotic arrangement of the physical morphology on fibers in the hydrothermally pretreated substrates can be captured, especially in the images of pretreated fiberboard (Fig. S2).

According to the investigations on the responses of main fractions (cellulose, hemicellulose, and lignin) before/after pretreatment, it could be easily found that their changes were similar to the conventional lignocellulosic biomass by hydrothermal pretreatment. Thus, it can be believed that the artificial process, such as the hot-process and the

adhesive addition, did not negatively affect the composition responses, indicating that the pretreatment was well performed to deconstruct these boards. In previous investigations, the hydrothermally pretreated corncobs displayed a good performance on enzymatic hydrolysis with the glucose recovery of 73.1% [19]. A maximal glucose yield of 37.3 g/100 g raw material was observed in the enzymatic hydrolysis of hydrothermally pretreated sugarcane bagasse, in which approximately 90% glucose was liberated from cellulose [32]. In this work, the structural barriers of raw materials were well opened. Theoretically, these 3 hydrothermally pretreated furniture boards should achieve the potentials of good performances on enzymatic hydrolysis. However, it was surprising that the cellulose-glucose conversions were all lower than 8%, even though 72 h for hydrolysis was employed on the pretreated substrates with the highest severities of 250 °C (see Fig. 2). Obviously, the hydrothermal pretreatment did not give a satisfactory performance on the sugar release from these as same as the reported natural lignocellulose, which motivated us to elucidate what really happens on the hydrolysis of these lignocellulose-based furniture boards after hydrothermal pretreatment.

Understanding the Poor Enzymatic Hydrolysis Performance of the Hydrothermally Pretreated Furniture Boards

As stated above, the poor performances of enzymatic hydrolysis unexpectedly happened on these 3 furniture boards after hydrothermal pretreatment. Hot-pressed and additional adhesive are generally artificially involved in the manufacturing process of these furniture boards, and these process may differentiate the pretreatment performances compared with the natural lignocelluloses, potentially triggering the difficulty for the subsequent biochemical process after hydrothermal pretreatment [5]. Herein, three possibilities were proposed for the poor enzymatic hydrolysis of the hydrothermally pretreated substrates: (1) the relatively higher lignin content after hydrothermal pretreatment may trigger irreversible adsorption to the cellulase, which potentially reduce the available enzyme in the actual enzymatic hydrolysis [33]; (2) the contained adhesives or paints will be melted at high temperatures, which probably can tightly attach on the cellulose surface, and restrict cellulase accessibility; (3) the contained adhesives will be decomposed/converted into potential toxicants during hydrothermal pretreatment, which will be some inhibitors to cellulase activity. To better understand what really happened in enzymatic hydrolysis using these type substrates, these 3 possibilities were investigated correspondingly based on the hydrothermally pretreated blockboard (180 °C for 15 min).

The adsorption of cellulase by lignin is generally believed as a spontaneous process, and the residual lignin in the hydrothermally pretreated substrates provided a significant obstacle on the enzymatic hydrolysis. Previous studies indicated that some exogenous proteins or surfactants were successfully employed to prevent the nonproductive adsorption of cellulase from the residual lignin [17, 33]. Addition of bovine serum albumin (BSA) improved the glucose yield by 40% for dilute acid pretreated corn stover due to alleviate the non-productive adsorption of cellulase by lignin [34]. Therefore, 1% (*w/v*) BSA solution was attempted to quantitatively add into the acetic acid-sodium acetate suspension to occupy the adsorption sites of lignin in advance. After 1.5-h adsorption, the cellulase was supplemented for enzymatic hydrolysis. Based on this way for the investigation, the cellulose-glucose conversion of

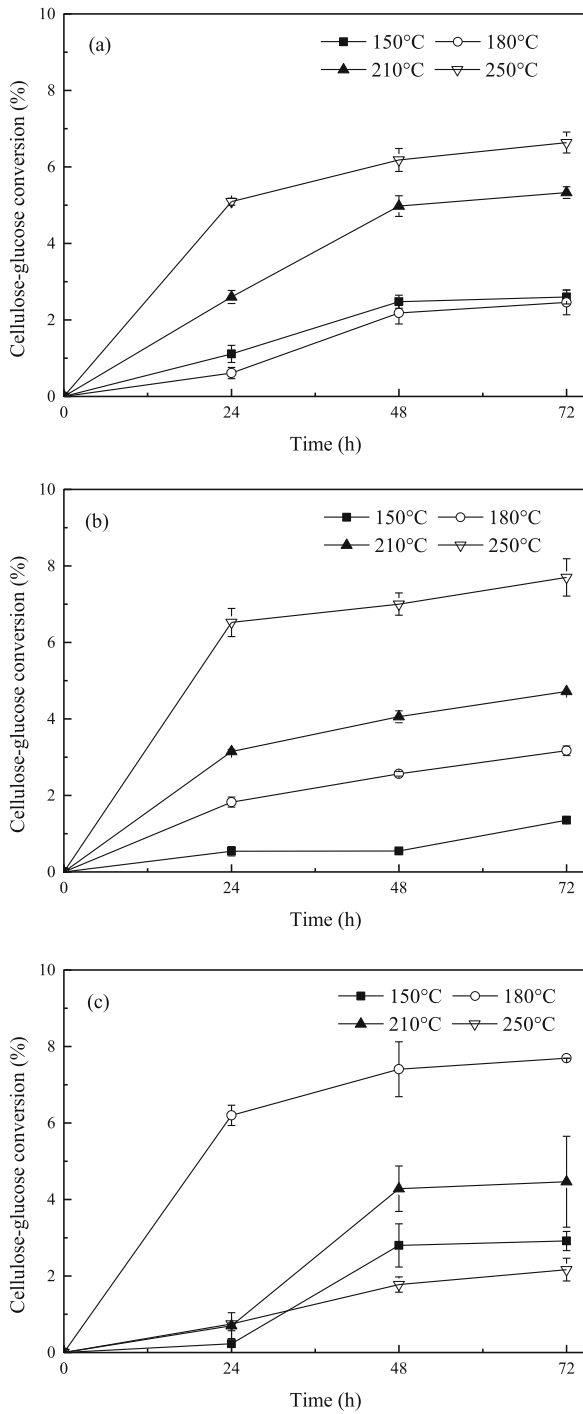


Fig. 2 Performances of cellulose-glucose conversion of hydrothermally pretreated furniture boards (a fiberboard; b chipboard; c blockboard)

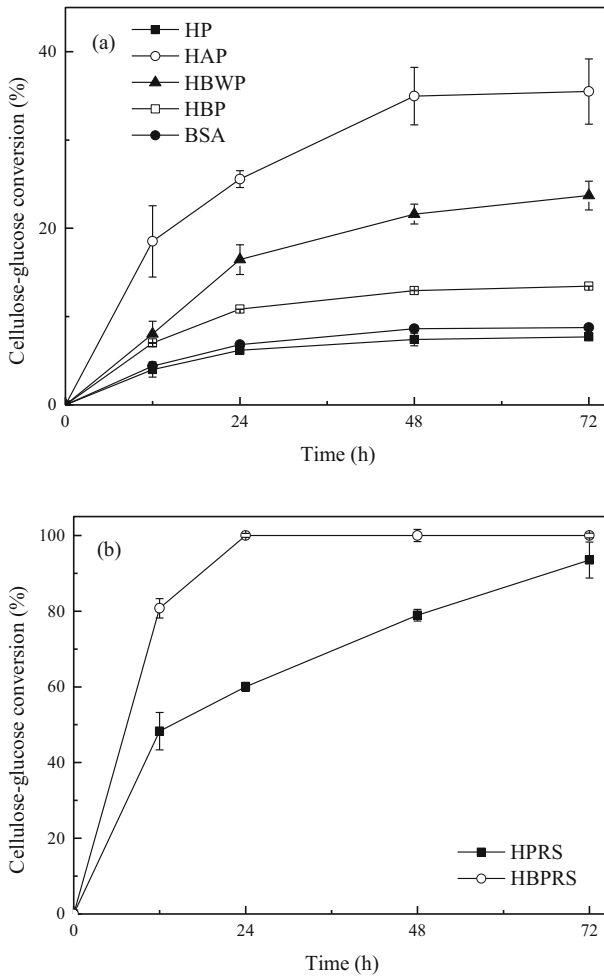


Fig. 3 The cellulose-glucose conversion with some possible ways to promote the hydrolysis performances. **a** The hydrothermally pretreated blockboard (180 °C, 15 min). **b** The hydrothermally pretreated rice straw (180 °C, 15 min). The abbreviation of HP referred to hydrothermal pretreatment; HAP referred to NaOH post-pretreatment at 120 °C for 60 min on the basis of HP; HBP referred to the post-pretreatment by ball-milling for 24 h on the basis of HP; HBWP referred to substrates were washed by distilled water on the basis of HBP; BSA referred to the addition of 1% (w/v) BSA ahead of enzymatic hydrolysis; HPRS referred to the hydrothermal pretreatment on rice straw at 180 °C for 15 min as a contrast; HBPRS referred to the post-pretreatment by ball-milling for 24 h on the basis of HPRS

hydrothermally pretreated blockboard was slightly promoted to 8.8% with BSA comparing to that of no-BSA added substrate (7.7%) (see Fig. 3a). Apparently, it could be deduced that the non-productive adsorption by the residual lignin will not to be the dominant possibility to weaken the enzymatic hydrolysis.

In consideration of the second possibility, ball-milling, an effective mechanical method, was employed as a post-pretreatment to destroy the structure barriers from adhesives (the detailed protocol see the Supplementary file) [35]. Based on Fig. 3a, it could be observed that the cellulose-glucose conversion of post-pretreatment by ball-milling can be promoted to 13.5% compared to 7.7% of the original pretreated blockboard. However, when the typical natural

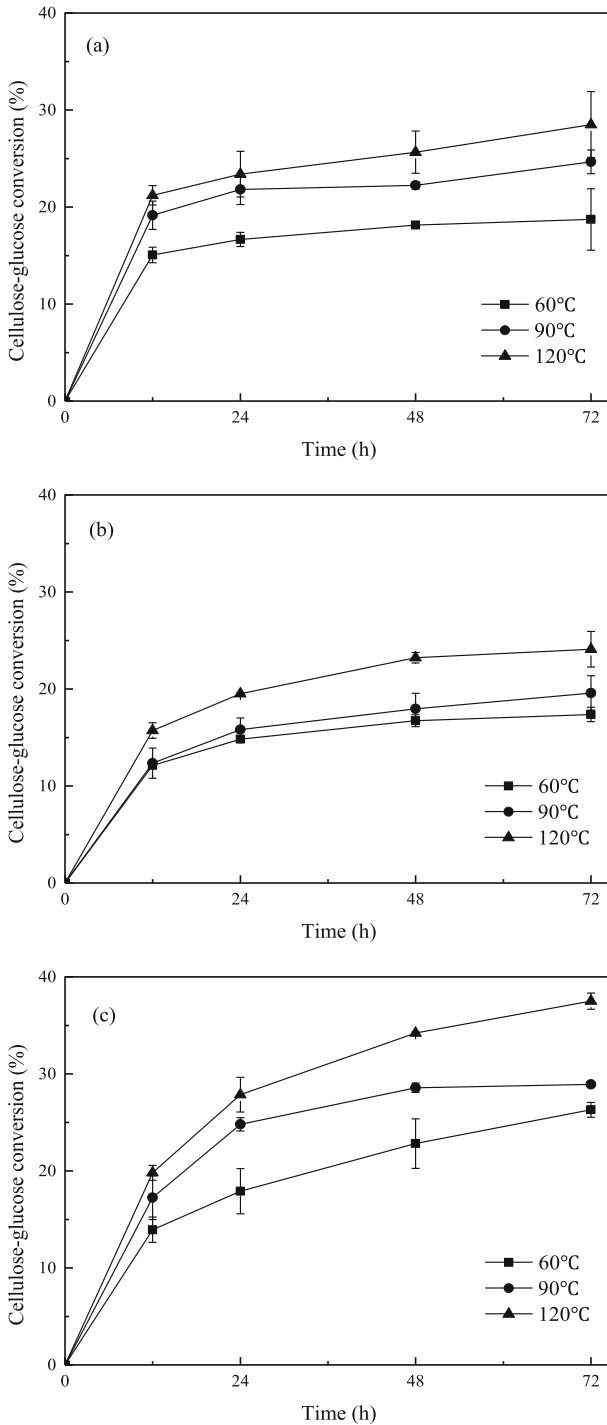


Fig. 4 The cellulose-glucose conversion of NaOH post-pretreatment (60 min) with different temperatures on the basis of hydrothermal pretreatment (180 °C for 15 min). **a** Fiberboard. **b** Chipboard. **c** Blockboard

lignocellulosic biomass of rice straw was hydrothermally pretreated and the post-pretreatment by ball-milling was also performed, the cellulose-glucose conversion was promoted from 93.5% (HPRS) to 100% (HBPRS) (see Fig. 3b). By contrast, the promotion by the post-pretreatment of ball-milling on blockboard and rice straw were almost similar, suggesting that the possibility of the weakened the enzymatic hydrolysis by the attached adhesives can be basically excluded, and the promotion by the post-pretreatment of ball-milling was mainly attributed to the particle size reduction [35]. Furthermore, the water-washing process after the post-pretreatment of ball-milling (HBWP) can greatly promoted the cellulose-glucose conversion to 23.7%, partially implying the weakened enzymatic hydrolysis may be due to some potential inhibitors, which can be partially liberated from the pretreated solid and removed by the water-washing process.

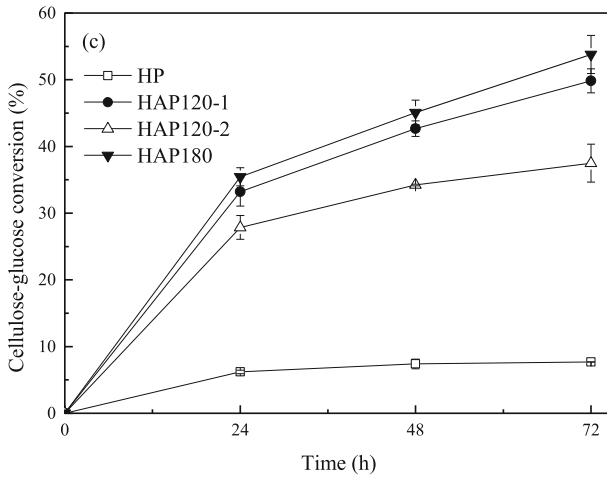
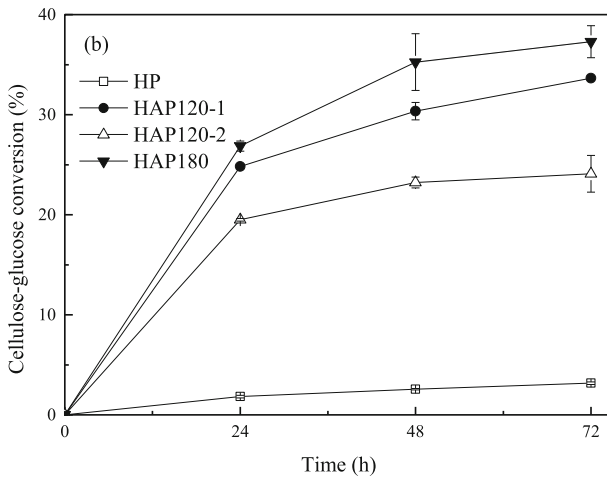
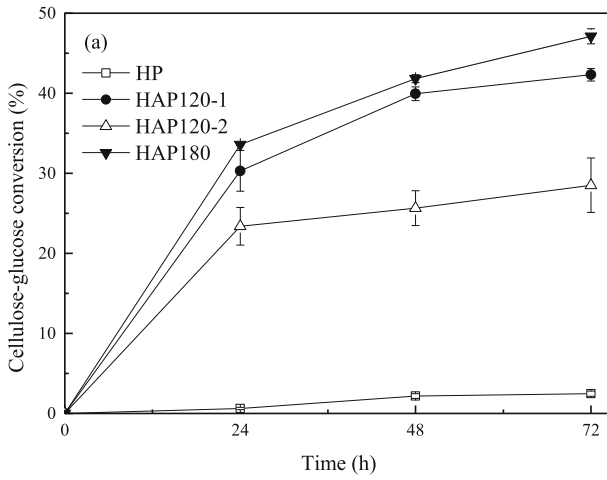
Alkali can be employed to dissolve the adhesives in furniture boards, such as phenolic resin; moreover, it also has been proven as one of the best methods for detoxification and delignification [36, 37]. It was reported that wheat straw pretreated with 4% NaOH solution at 121 °C achieved a good cellulose-glucose conversion (87.2%) compared with sulfuric acid and single hydrothermal pretreatment [38]. Therefore, hydrothermally pretreated blockboards were post-pretreated with 4% (*w/v*) NaOH (the detailed protocol in the Supplementary file). It was noticeable that the cellulose-glucose conversion was promoted to 35.5% by the NaOH post-pretreatment (HAP), which was 3.6 times higher than that of the hydrothermally pretreated blockboard (HP). In addition, it is worth noting that the derived liquid of NaOH post-pretreatment was dark brown, also suggesting that the containing adhesives and potential inhibitors could be greatly removed from the recovered solid [39]. To further prove this speculation, a typical adhesive, phenolic resin, was directly added into 4% NaOH with high purity water as a comparison. Their absorbance spectra harvesting from ultraviolet spectrophotometer displayed significant difference from 190 to 500 nm (see Fig. S3), which can directly prove that the adhesive fraction in these waste boards can be removed by NaOH, and these adhesives may be greatly responsible for the unsatisfactory enzymatic hydrolysis.

Based on the discussion above, the poor enzymatic hydrolysis of these hydrothermally pretreated substrates was greatly related to the adhesives and their degraded products, and the alkaline post-pretreatment can be suggested a possible way to promote the enzymatic hydrolysis of hydrothermally pretreated furniture boards.

Integrating a Feasible Way to Pretreat Waste Furniture Boards

As aforementioned, the alkaline (NaOH) post-pretreatment can promote enzymatic hydrolysis of the hydrothermally pretreated furniture boards, which offered a possible way to pretreat these unconventional solid wastes. In this process, hydrothermal pretreatment firstly removed the typical hemicellulose fraction, and NaOH post-pretreatment liberated partly lignin and removed potential toxicants. When these 3 furniture boards were

Fig. 5 Cellulose-glucose conversion of the integrated mode of NaOH hydrothermal pretreatment. **a** Fiberboard. **b** Chipboard. **c** Blockboard. HP referred to the hydrothermal pretreatment at 180 °C for 15 min; HAP120-1 referred to the integrated mode of NaOH hydrothermal pretreatment at 120 °C for 60 min; HAP120-2 referred to the separated hydrothermal pretreatment (180 °C, 15 min) followed by NaOH post-pretreatment (120 °C, 60 min); HAP180 referred to the integrated NaOH hydrothermal pretreatment at 180 °C for 15 min



checked for NaOH post-pretreatment with the increased temperatures. It could be found that the final cellulose-glucose conversion will be promoted with the temperature for post-pretreatment (see Fig. 4), in which the promotion at 120 °C was 3.32, 2.13, and 3.87 times compared with the highest cellulose-glucose conversion of hydrothermally pretreated fiberboard, chipboard, and blockboard in Fig. 2 (6.6%, 7.7%, and 7.7%, respectively). Thus, it can be deduced that the post-pretreatment by NaOH should be effective to improve the enzymatic hydrolysis of typical furniture boards that were hydrothermally pretreated. However, the different performance of these substrates may be attributed to the original biomass for these boards and the employed adhesives. For example, fiberboard was made of soft fibers, while blockboard was mainly made of sawdust or wood chips, therefore, much more adhesives were demanded to ensure the compactness and strength of these furniture boards [40, 41].

Although hydrothermal pretreatment followed by NaOH post-pretreatment had some promotions on the hydrolytic performances, there were still some limitations in the actual process, such as requirement of solid-liquid separation. Moreover, the final cellulose-glucose conversion was still in a relatively lower level. Thus, an integration of hydrothermal pretreatment and the NaOH post-pretreatment was considered to simplify the whole process and improve the conversion efficiency. Herein, the NaOH hydrothermal pretreatment was proposed, which has been reported to pretreat some traditional lignocelluloses for enzymatic hydrolysis and ethanol fermentation [42–44]. In this work, the integrated mode of NaOH hydrothermal pretreatment at 120 °C (HAP120-1) was investigated with the separated hydrothermal pretreatment and NaOH post-pretreatment (HAP120-2) as a comparison. After pretreatment with the integrated mode of HAP120-1, the cellulose-glucose conversion can be further increased to 42.3%, 33.6% and 49.8% compared with the separated mode (HAP120-2) of 28.5%, 24.1%, and 37.5% for fiberboard, chipboard, and blockboard (see Fig. 5). In this comparison, the employed temperature of 120 °C was consulted from the NaOH post-pretreatment; however, the cellulose-glucose conversion can be greatly promoted to 47.1%, 37.3%, and 53.8% once the integrated mode of NaOH hydrothermal pretreatment was performed at higher temperature (180 °C). By contrast, the cellulose-glucose conversion was respectively improved by 7.1-, 4.8-, and 7.0-fold than the highest cellulose-glucose conversion of the hydrothermally pretreated fiberboard, chipboard, and blockboard. Apparently, the integrated mode of NaOH hydrothermal pretreatment can be potentially extended to these unconventional recalcitrant biomass wastes. Of course, the conditions, such as the pretreatment temperature and the holding duration, for the integrated mode of NaOH hydrothermal pretreatment still need more investigations and optimization, after all, the final conversion was not high enough to make a competition with other traditional biomass.

Conclusions

Based on the responses of chemical composition and physical morphology of 3 furniture boards after pretreatment, hydrothermal pretreatment can deconstruct these feedstocks

well. However, poor enzymatic hydrolysis of these hydrothermally pretreated substrates indicated the difficulty of the following biological conversion. Post-pretreatment by NaOH promoted enzymatic hydrolysis, suggesting that the residual adhesives in the pretreated substrates were a great possibility to weaken hydrolytic performances. NaOH post-pretreatment was suggested to be integrated into hydrothermal pretreatment, which can greatly improve the subsequent enzymatic hydrolysis; however, more investigations should be performed on the integrated NaOH hydrothermal pretreatment to make conversion of these unconventional lignocellulose more competitive.

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Compliance with Ethical Standards

Conflict of Interest The authors declare that they have no conflict of interest.

Ethical Approval This article does not contain any studies with human participants or animals performed by any of the authors.

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