Recovery of glucose and polyester from textile waste by enzymatic hydrolysis

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Abstract

In order to recover glucose and polyester from textile waste, the enzymatic hydrolysis of alkali pretreated textile waste was investigated. The influence of key factors related to hydrolysis process was evaluated, including substrate loading ratio, temperature, cellulase dosage, as well as the supplementation of β- glucosidase. Results showed that increasing substrate loading from 1% to 7% had a negative effect on glucose recovery yield and the significant inhibitive effect was observed at over 3% substrate loading. Substrate loading at 3% was selected based on glucose yield and hydrolysis efficiency. The optimal temperature for enzymatic hydrolysis was 50 °C and significant reduction was observed over 60 °C. More than 50 IU/g β-glucosidase did not contribute to the increase of glucose recovery yield. Therefore, the optimum enzymatic hydrolysis condition was using 20 FPU/g cellulase and 50 IU/g β-glucosidase at 50 °C, based on the criterion for minimizing enzyme dosage and maximizing glucose recovery. The maximum glucose yield of 90% was achieved and the glucose production was 606 mg/g textile waste after 96 h hydrolysis.

Keywords: textile waste, enzymatic hydrolysis, glucose recovery, polyester recycling.

Introduction

It was reported that the worldwide consumption of textile fibers was over 96 million tonnes in 2015 [1]. Currently, the main textile waste management methods were recycling by donation, combustion with energy recovery and landfilling [2]. Around 64.5% of textile waste ends up in landfills in the United States in 2015 [2]. In Hong Kong, the amount of textile waste generated is 306 tonnes per day [3]. Most of textile waste is disposed at landfills, up to 95.7% [3]. Textile waste disposed at landfills is a waste of valuable materials and also exerts more pressure on the limited landfill space. Therefore, it is necessary to find an environmentally and economically sustainable process to recycle textile waste.

According to Fiber year book (2016), the annual consumption of cotton fibers accounted for 39% of total textile fibers [1]. Cotton content in textile waste is mainly considered as an alternative source for the production of renewable energy, and has been investigated as feedstock in the process of bioethanol biorefinery [4-6] and biogas production [4]. For cotton-based ethanol production, efficient saccharification is of great importance and contributes to higher concentration of ethanol. Relatively lower enzyme input in hydrolysis was also suggested in previous techno-economic evaluations [7-8].

However, previous researches mainly focused on the pretreatment of textile waste. Several pretreatment methods have been investigated, including NaOH and urea [9], ionic liquids [5] and phosphoric acid [10]. The optimization of enzymatic hydrolysis of textile waste has not been reported yet.

In this work, we aim to investigate the optimal conditions for enzymatic hydrolysis and recycling textile waste with the lowest energy and enzyme input. The key factors related to hydrolysis were investigated in this study, such as substrate loading and temperature. Regarding enzyme input, the dosages of cellulase and β-glucosidase were also investigated.

Materials and methods

Substrate and enzyme

Textile waste blending of cotton and polyester (PET) by 60/40 from H&M plants in China was employed as raw material. The crude textile fabrics were grinded into small pieces (size approximate 1×1 cm^2) by the grinder WSM-D200X160 (OMS Machinery Co. Ltd., Zhongshan, China).

Two types of commercial enzymes were used in this study, cellulase Celluclast 1.5L (Novozymes, China) and β-glucosidase (Sunson, China). The activities of cellulase and β-glucosidase were 75 FPU/ml and 9,000 IU/ml, respectively.

Textile pretreatment

Textile waste was pretreated by soaking in NaOH/urea solution at -20 °C for 6 h. The pretreated samples were washed by tap water, and dried in oven for overnight.

Enzymatic hydrolysis

Textile fabrics were enzymatically hydrolyzed in 50 mM sodium citrate buffer (pH 5.1) in 250 ml Duran bottles with 100 ml working volume. Different substrate loadings (w/v%), cellulase and β-glucosidase dosages were used. The hydrolysis was performed at 50 °C in water bath at 350 rpm stirring for 96 h. Samples were taken at 0 h, 9 h, 12 h, 24 h, 48 h, 72 h and 96 h in hydrolysis process. After centrifugation at 10,000 rpm for 3 min, the supernatant was collected for sugar analysis. All hydrolysis experiments were carried out in duplicate. The glucose yield was calculated using Eq.1.

$$\text{Glucose yield} = \frac{\text{Amount of glucose released}}{\text{Initial amount of cellulose} \times 1.11} \times 100\% \qquad \text{Eq.1}$$

Analytical methods

Amount of glucose was analyzed by HPLC (Waters, Milford, USA), which was equipped with RI detector (Waters 2414). Glucose was analyzed on an Aminex HPX-87H column (Bio-Rad, USA) at

60 °C with 0.6 mL/ min eluent of 5 mM sulfuric acid.

Scanning Electron Microscopy (SEM) Detection

The textile substrates were detected by scanning electron microscopy (SEM). Images of textile structure before and after enzymatic hydrolysis were taken at a magnification of 100, voltage 20 kV using a Germany SEM (Carl Zeiss EVO 10).

Statistical analysis

All data were processed by IBM SPSS software (version 22.0), and the hydrolysis results at different conditions were compared using one-way ANOVA (Analysis of variance). The significant differences among the different conditions were determined by Duncan multiple range test and significant P-value was obtained after statistical analysis.

Results and discussion

Different substrate loading

The enzymatic hydrolysis efficiency of cellulose highly depends on temperature, substrate loading, enzyme dosage and the structural features of substrate [11]. Firstly, in order to assess the optimal substrate loading for the subsequent enzymatic hydrolysis, hydrolysis was conducted at different substrate loadings, ranging from 1% to 7% in 250 mL Duran bottles. Fig.1a showed the hydrolysis profiles of the pretreated textile fibers at different substrate loadings with 30 FPU/g cellulase and 300 IU/g β-glucosidase. Hydrolysis results indicated that the increase of substrate loading from 1% to 7% reduced the glucose yield (96 h) of pretreated cotton/ PET 60/40 from 90% to 78%. The similar results have been reported in previous work with different substrates [12]. Hydrolysis rate at different substrate loadings could keep at the same level in the first 24 h. With time prolonging, compared with 1% substrate loading, hydrolysis rate of 3%, 5% and 7% substrate loadings decreased by 0% ($p > 0.05$), 5% ($p < 0.05$) and 12% ($p < 0.05$), respectively. The significant reduction was observed at substrate loadings over 5%, based on the ANOVA analysis. These results suggested that increasing substrate loading had a negative effect on the conversion of cellulose to glucose. It could be explained by the significant product inhibitive effect. As shown in Fig.1b, higher glucose concentration was obtained when increasing solid concentration from 1% to 7%. Over 3% substrate loading in 250 ml Duran bottles, glucose concentration started to limit the effective conversion during hydrolysis process. This was because glucose had a strong inhibition effect on cellulase adsorption [13]. As enzyme adsorption was crucial to hydrolysis of insoluble substrates [14], this led to lower glucose recovery yield at higher solid loading (5% and 7%). Therefore, in order to digest cotton as much as possible, it could be concluded that 3% was the optimal substrate loading based on the hydrolysis rate and final glucose yield.

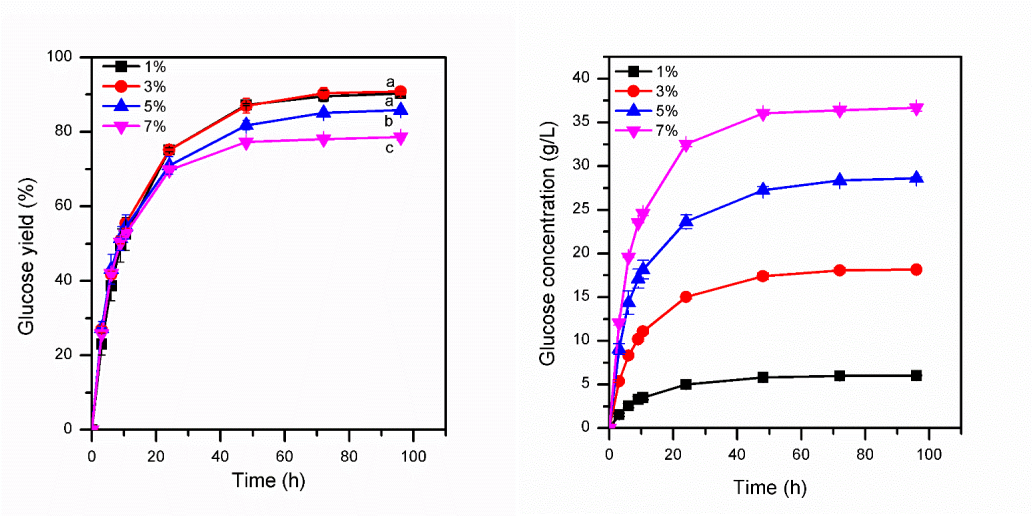


Fig.1 Enzymatic hydrolysis of alkali pretreated textile fibers at different substrate loadings, a) glucose yield profile; b) glucose concentration profile.

Temperature

Temperature is the key factor of enzymatic hydrolysis. The optimal incubation temperature is necessary to reduce energy input and to recover the highest amount of glucose from textile waste. Therefore, different temperatures, ranging from 40 ^{o}C to 65 ^{o}C, were investigated at 3% substrate loading with 30 FPU/g cellulase and 300 IU/g β-glucosidase. After 96 h, the final glucose yields at different temperatures were shown in Fig.2. It was found that higher glucose yields were obtained at 45 ^{o}C -55 ^{o}C and there was no significant difference ($p > 0.05$) in glucose recovery yield under these conditions. The maximum glucose yield of 90% was achieved at 50 ^{o}C. It could be noted that higher temperatures over 60 ^{o}C inhibited the conversion from cellulose to glucose. The significant inhibitive effect was observed at 60 ^{o}C ($p < 0.05$) and 65 ^{o}C ($p < 0.05$), and the huge decrease of glucose yield was found at 65 ^{o}C, from 90% to 22%. It was due to the fact that cellulase activity was damaged at high temperatures. The similar results have been reported in previous studies, and more than 90% of enzyme activity was lost after 48 h incubation at 60 ^{o}C [15]. The significant decrease ($p < 0.05$) of glucose yield was also observed at 40 ^{o}C. It could be explained that lower temperature had a negative effect on cellulase activity and resulted in the loss of glucose yield. Based on the above results, it could be concluded that the optimum temperature condition for the cellulase used was 50 ^{o}C.

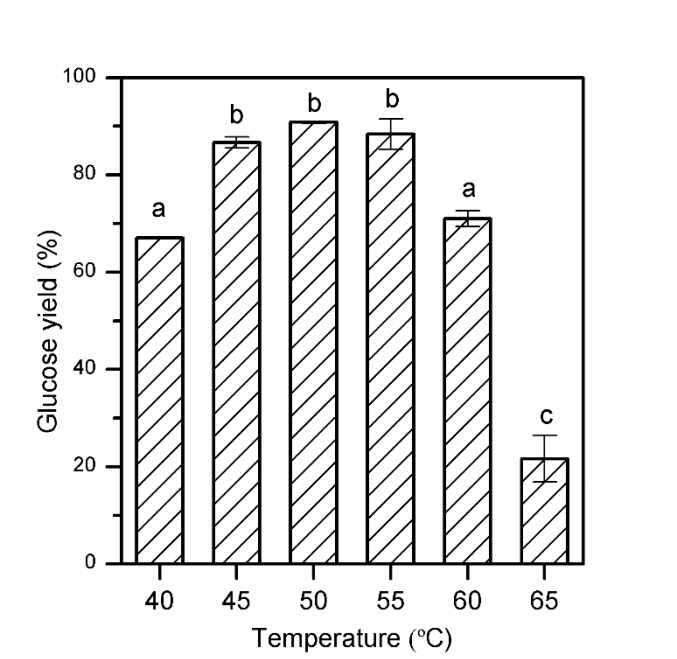


Fig.2 Glucose yields after 96 h enzymatic hydrolysis of alkali pretreated textile fibers at different temperatures.

Cellulase dosage

In order to use the amount of cellulase at a relatively low level, various cellulase dosages ranging from 10 to 40 FPU/g substrate were investigated at substrate loading of 3%. As presented in Fig.3, the glucose yield was enhanced from 61% to 82% when cellulase dosages increased from 10 to 40 FPU/g. This is because more enzyme accessible sites on textile fibers were occupied with the increase of cellulase dosage [12]. Based on the ANOVA results, the hydrolysis yield increased significantly ($p < 0.05$) when increasing cellulase dosage from 10 to 20 FPU/g. There was no significant difference ($p > 0.05$) when comparing the results obtained from 20 and 30 FPU/g. A slight improvement ($p < 0.05$) was observed with further increased cellulase dosage to 40 FPU/g. It could be concluded that the cellulase dosage of 20 FPU/g substrate was selected for sebsequent researches.

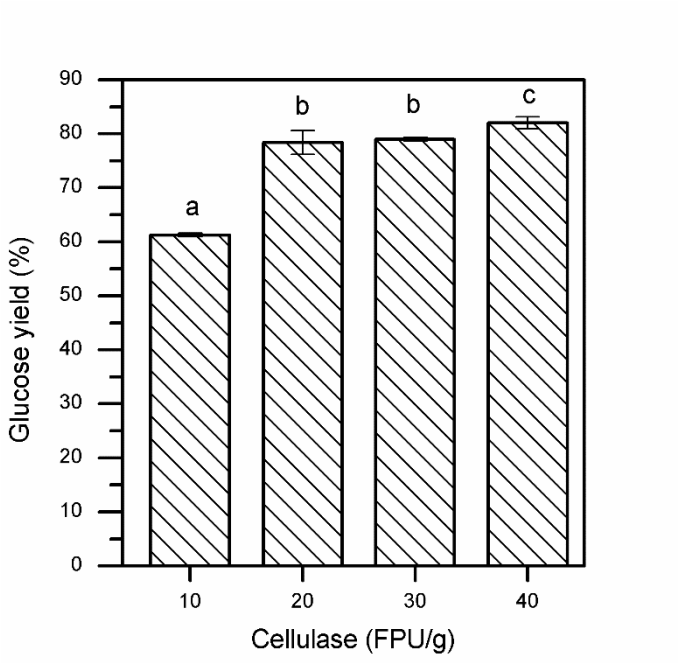


Fig.3 Glucose yields after 96 h enzymatic hydrolysis of alkali pretreated textile fibers at different cellulase dosages.

β-glucosidase supplementation

The accumulation of cellobiose was found during hydrolysis process [16] and strongly inhibit cellulase action [13, 14, 16-17], which resulted in less effective cellulose hydrolysis to glucose. In order to avoid inhibition of cellobiose, additional β-glucosidase was added to hydrolyze cellobiose to glucose. Moreover, the minimum required β-glucosidase input was important to the reduction of hydrolysis cost. Therefore, different β-glucosidase dosages were investigated, ranging from 0 to 400 IU/g substrate with the fixed cellulase dosage of 20 FPU/g substrate. As presented in Fig. 4, there was a significant improvement ($p < 0.05$) in hydrolysis yield from 78% to 90% with the supplementation of 50 IU/g β-glucosidase. The results indicated that the addition of β-glucosidase can enhance glucose yield significantly ($p < 0.05$). Further increasing β-glucosidase dosage over 50 IU/g substrate could not result in the improvement of hydrolysis conversion ($p > 0.05$) to a higher level. Therefore, the optimal β-glucosidase dosage was selected as 50 IU/g substrate.

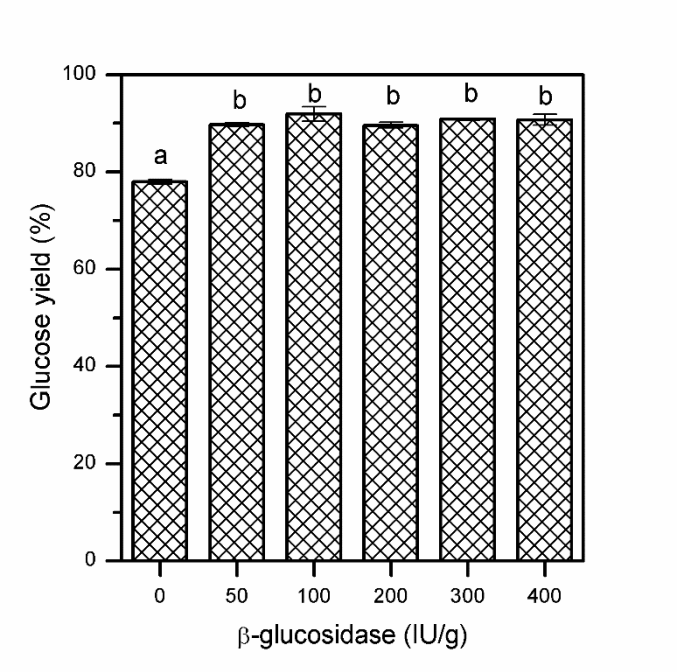
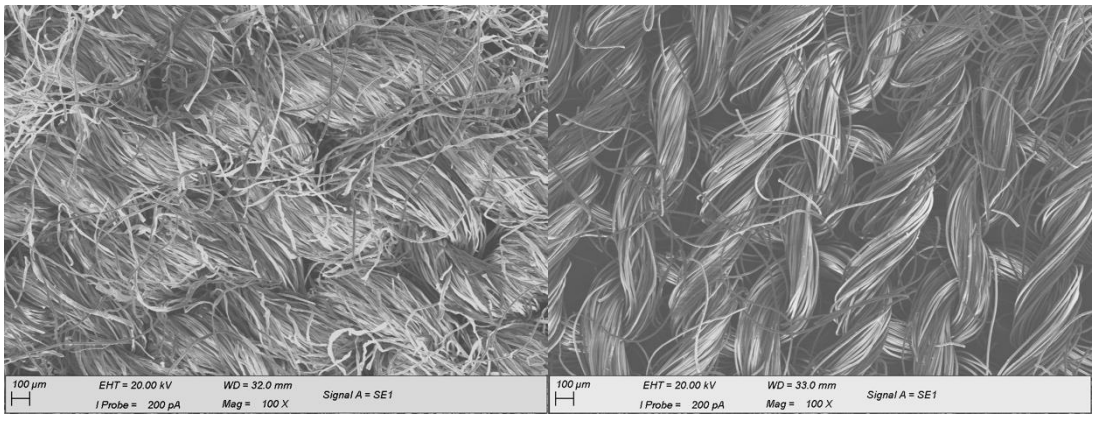


Fig.4 Glucose yields after 96 h enzymatic hydrolysis of alkali pretreated textile fibers at different β-glucosidase dosages.

Changes of surface morphology

The morphological change of textile substrate (pretreated cotton/PET 60/40) was detected by SEM. According to the SEM images of pretreated cotton/PET 60/40, the dramatic changes of surface morphology can be observed on textile after hydrolysis. As presented in fig.5a, pretreated textile fibers before enzymatic hydrolysis was in a compact binding structure. After enzymatic hydrolysis (fig.5b), the binding was obviously loosed with less fibers. Many small holes among fibers were observed. It is because in enzymatic hydrolysis, most cellulosic fibers were digested to soluble sugars and non-biodegradable PET fibers were left in fabrics. These images revealed the removal of cotton from textile waste by enzymatic hydrolysis.



(a) (b)

Fig.5 SEM pictures of textile substrate (pretreated 60/40) before (a) and after (b) enzymatic hydrolysis.

Conclusions

Recovery of glucose and polyester from textile waste by enzymatic hydrolysis was investigated in this study. Substrate concentration, temperature, cellulase dosage and β-glucosidase dosage were optimized to reduce the hydrolysis cost. The maximum glucose recovery of 90% was obtained with 20 FPU/g of cellulase dosage and 50 IU/g of β-glucosidase dosage at 3% (w/v) solid loading. In conclusion, a novel bioprocess using textile wastes to recover glucose by biological methods was developed.

Acknowledgements

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