



# Enzymatic saccharification coupling with polyester recovery from cotton-based waste textiles by phosphoric acid pretreatment



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## HIGHLIGHTS

- ▶ H<sub>3</sub>PO<sub>4</sub> pretreatment for waste textiles was applied for sugar and polyester recovery.
- ▶ H<sub>3</sub>PO<sub>4</sub> concentration, temperature, time, ratio of textiles: H<sub>3</sub>PO<sub>4</sub> were investigated.
- ▶ 100% polyester was recovered by enhancing H<sub>3</sub>PO<sub>4</sub> concentration, time and temperature.
- ▶ Maximum of 79.2% sugar recovery can be achieved by enzymatic saccharification.
- ▶ The process was technically feasible and potentially profitable to recover polyester and sugar.

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## ABSTRACT

In order to recycle the cotton-based waste textiles, a novel process was designed for pretreating waste textiles with phosphoric acid to recover polyester and fermentable sugar. The effects of pretreatment conditions including, phosphoric acid concentration, pretreatment temperature, time, and ratio of textiles and phosphoric acid were thoroughly investigated. Results indicated the mentioned four factors had significant influences on sugar and polyester recovery. Almost complete polyester recovery was achieved by enhancing phosphoric acid concentration, temperature and pretreatment time or reducing the ratio of textiles and phosphoric acid. However, these behaviors decreased the sugar recovery seriously. 100% polyester recovery with a maximum sugar recovery of 79.2% was achieved at the optimized conditions (85% phosphoric acid, 50 °C, 7 h, and the ratio of 1:15). According to the technical and cost-benefit analysis, it was technically feasible and potentially profitable to recover polyester and sugar from waste textiles by phosphoric acid pretreatment.

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## 1. Introduction

It was reported that the consumption of global fiber was  $7.05 \times 10^7$  ton in 2009, in which the proportion of cotton fiber and polyester fiber amounted for 40% and 45.2%, respectively (Engelhardt, 2010). At the end of their lifetime, only few part of these fibers can be reused or recycled due to the complex component of waste textiles, and most of them are disposed of in landfills or subjected to incineration (Wang, 2010). Thus, waste textiles have been regarded as one of the major solid wastes in the world. (<http://www.epa.gov/osw/conserve/materials/textiles.htm>).

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Similarly, the cotton-based waste textiles are mainly derived from the cotton, in which the cellulose fibers can be regarded as an alternative renewable biomass source for refining bioethanol, biogas or other bio-based products via various pretreatments and subsequent biological processes (Jeihanipour et al., 2010b; Jeihanipour and Taherzadeh, 2009; Kuo et al., 2010). There are some special advantages of the cotton-based waste textiles for biorefineries comparing to lignocellulosic materials. The cellulose content in different cotton-based textiles are generally in the range of 50–100% (Hong et al., 2012; Jeihanipour et al., 2010a; Vasconcelos and Cavaco-Paulo, 2006), which is higher than that of the typical lignocellulosic materials. Moreover, the contents of lignin and hemicellulose in the waste textiles are almost zero, and the hindrance from lignin and hemicellulose to the subsequent biological processes therefore can be potentially weakened, even can be neglected (Zhang et al., 2007). Thus, the processes for delignification and hemicellulose removal can be potentially avoided in the pretreatment stream (Koo et al., 2011; Kumar et al., 2009). However, the conversion of cotton-based waste textiles for the

mentioned bio-based materials also encounters some difficulties from the high-ordered crystalline structure of cotton fibers. The well constructed inter- and intra-molecule hydrogen bonds make the fibers uneasily accessible to enzymes for sugar production (Jeihanipour and Taherzadeh, 2009). Moreover, the well-organized construction of the textiles of polyester and cotton fibers from the blending process also limits the enzymatic hydrolysis. In addition, the existence of polyester may potentially absorb the enzymes, which also negatively affects the efficiency of the enzymatic hydrolysis (Vasconcelos and Cavaco-Paulo, 2006). Therefore, it is necessary to develop some pretreatment methods to improve the enzymatic hydrolysis of the cotton-based waste textiles.

Currently, some chemical methods, including ionic liquids (Hong et al., 2012; Jeihanipour et al., 2010a; Jeihanipour et al., 2010b) and NaOH/urea (Ioelovich and Morag, 2011; Jeihanipour and Taherzadeh, 2009; Wang et al., 2008) have been developed to pretreat the waste textiles for the cellulose decrystallization. For the ionic liquids pretreatment, it was characterized by the broad liquid regions, high thermal stabilities and negligible vapor pressure, and it thereby was designed as the efficient solvents for pretreating the cellulosic materials. However, the high viscosity and the potential toxicity to subsequent bio-degradation made it inapplicable up to now (Holm and Lassi, 2011; Hong et al., 2012; Jeihanipour et al., 2010b). NaOH can penetrate the amorphous area of the cellulose, and disrupt the neighboring crystalline regions. Thus, NaOH can swell and even dissolve cellulose in a certain concentration. However, NaOH could not dissolve cellulose completely based on the results in references (Isogai and Atalla, 1998; Wang et al., 2008). The non-cellulose part of polyester in the waste textiles thereby could not be separated purely by NaOH pretreatment. In addition, the high corrosivity of NaOH to the equipments was another key negative issue for its application. Therefore, it is necessary to seek some nontoxic and non-corrosive chemicals, which can be used for waste textiles pretreatment to facilitate polyester recovery and subsequent enzymatic saccharification.

Phosphoric acid can be employed as a solvent for crystalline cellulose dissolution and subsequent regeneration, which has been investigated for at least 80 years (Mosier et al., 2005). It was characterized by non-corrosivity and non-toxicity and safety compared to the mentioned chemicals for the pretreatment of textiles (Zhang et al., 2010). Moreover, it can dissolve crystalline cellulose at atmospheric pressure with moderate temperature. The polyester thereby can be separated from the cotton-based waste textiles. The cellulose can be regenerated by diluting the phosphoric acid, by which the degree of polymerization and crystallinity was correspondingly decreased, and the specific surface area of cellulose was also improved remarkably (Zhang et al., 2010; Zhang et al., 2009; Zhang et al., 2006). Hence, the phosphoric acid pretreatment may be potentially beneficial to the separation of polyester from waste textiles and the subsequent enzymatic saccharification of regenerated cellulose.

Previous researches mostly focused on the micro-crystalline cellulose dissolution using phosphoric acid to investigate the effects of temperature, agitation, and pretreatment duration (Kumar et al., 2001; Zhang et al., 2006). The molecular changes, the dissolution mechanism and kinetics were also reported (Zhang et al., 2009). The obtained results from the mentioned work could offer some necessary references to utilize the cotton part of the waste textiles for fermentable sugar production. However, the results cannot be directly consulted for the pretreatment of waste textiles, especially, when the polyester recovery is considered together. Thus, the aims of this work are to investigate the possibility of phosphoric acid pretreatment on cotton-based waste textiles for sugar and polyester recovery. Meanwhile, the key factors related to the phosphoric acid pretreatment such as the acid concentration, the ratio of textiles and phosphoric acid, the pretreatment

time and temperature are also investigated to clarify their effects on polyester recovery and subsequent enzymatic hydrolysis.

## 2. Methods

### 2.1. Substrate and enzymes

20 pairs of used jeans, collected from the campus of Sichuan Agricultural University, were employed for this work. The used jeans were washed and dried before they were cut into small pieces (approximately  $0.5 \times 0.5 \text{ cm}^2$ ) and mixed thoroughly. The mixed pieces were afterwards milled with a lab-scale hammer crusher (WXJ100, Wuxi Zhongyin Machinofacture Co., Ltd., Jiangsu).

Two enzymes, cellulase from *Trichoderma reesei* (Spezyme-CP, Gencencor, Palo Alto, CA) and cellobiase from *Aspergillus niger* (Novzymes188, Bagsværd, Danmark), were employed for enzymatic hydrolysis. The activity of enzymes was 86 FPU/mL and 300 CBU/mL, respectively.

### 2.2. Phosphoric acid pretreatment

The pretreatment was performed in 250 mL Erlenmeyer flasks with 5.0 g milled sample. Based on experimental design, the commercial phosphoric acid (purity of 85%), purchased from Chengdu Kelong Chemical Reagent Factory, China, was diluted to various concentrations for pretreatment. The diluted phosphoric acid was introduced to the Erlenmeyer flasks and mixed with the milled samples. The mixture was shaken in an incubator at various temperatures for different durations at 130 rpm. The pretreatment was ceased through rapid dilution with more than 400 mL deionized water by a thorough agitation. Meanwhile, the cellulose can be regenerated in this dilution process.

The crude polyester in pretreatment was separated using a 30 mesh sieve, and washed afterwards using 250 mL deionized water to remove the regenerated cellulose on the crude polyester. The regenerated cellulose suspension was separated with filter paper by a vacuum filter. The separated cellulose afterwards was washed 3–5 times with 300 ml tap water. Both of the washed polyester and regenerated cellulose were oven-dried for 10 h at 65 °C. The dried samples were weighted respectively to calculate the cellulose and polyester recovery after the pretreatment. The pretreatment was repeated twice for each run.

### 2.3. Enzymatic hydrolysis

The regenerated cellulose was enzymatically hydrolyzed in 50 mM sodium citrate buffer (pH 4.8) in 150 mL Erlenmeyer flasks with 50 mL work volume. The substrate consistency for enzymatic hydrolysis was controlled at 1.0% (w/v) with the cellulose loading of 7.5 FPU/g regenerated cellulose and cellobiase loading of 15 CBU/g regenerated cellulose. The hydrolysis was performed at 50 °C in an air-bath shaker at 130 rpm for 96 h. 1 mL samples were withdrawn periodically and centrifuged at 4 °C with 8000 rpm for 6 min. The supernatants were collected and kept in  $-4 \text{ }^\circ\text{C}$  till for sugar analysis.

Overall, the flowchart for the sugar and polyester recovery from the waste textiles using phosphoric acid pretreatment was designed in Fig. 1.

### 2.4. Analytical methods

The cellulose content of original sample was determined according to the protocol in the "International Standard ISO 1833-11:2006 Textiles Quantitative chemical analysis-Part 11: mixtures of cellulose and polyester fibers (sulfuric acid method)".

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