



Effective conversion of waste polyester–cotton textile to ethanol and recovery of polyester by alkaline pretreatment

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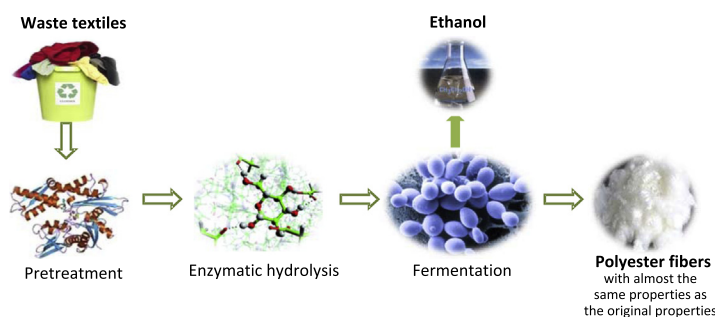
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HIGHLIGHTS

- A cotton–polyester waste textile was pretreated with alkaline solvent at various conditions.
- Pretreatment increased the glucose and ethanol yields from the textile.
- The properties of recovered polyester evaluated by FTIR, DSC and viscosity analyses.
- The pretreatment resulted in a minor change on the properties of polyester.

GRAPHICAL ABSTRACT



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ABSTRACT

The majority of textiles typically contain a biodegradable part that is cellulose and a non-biodegradable part which is a polyester. In this study, alkali pretreatment was evaluated for improvement of ethanol production from the cellulose part of a polyester–cotton textile and recovery of the polyester. The pretreatment was conducted by different alkali solutions of NaOH (12 wt%), NaOH/urea (7/12 wt%), NaOH/thiourea (9.5/4.5 wt%) and NaOH/urea/thiourea (8/8/6.4 wt%) at -20 , 0 , 23 , and 100 °C for 1 h. All of the pretreatments resulted in improvement of enzymatic hydrolysis yield to over 88%, while it was only 46.3% for the untreated textile. The best hydrolysis results were observed by the pretreatments at the reduced temperatures (-20 and 0 °C). The maximum yield of ethanol production from the textile by simultaneous saccharification and fermentation was 70%, obtained after the pretreatment with NaOH/urea at -20 °C whereas it was only 36% for the untreated textile. The polyester part of the textile was recovered after the hydrolysis of the cellulosic part and its properties were studied by FTIR, Differential Scanning Calorimetry (DSC), and viscosity measurements and compared with the untreated polyester used in the textile. The results showed that the alkaline pretreatment followed by hydrolysis resulted in recovery of 98% of the polyester without significant change in its properties.

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1. Introduction

The global consumption of fibers has increased along with the population growth and general increase of the living standards.

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In 2011, the worldwide fiber consumption exceeded 85 million tonnes, consisting around 40% and 60% cellulosic and man-made fibers, respectively [1,2]. The fibers are used for production of apparel, home textiles, and industrial products, and these textiles are appeared as wastes after a certain time [3]. The current management of waste textile mainly comprises incineration and land-filling, while these wastes have a high potential to serve as an alternative feedstock for production of biological products via bio-conversion of the cellulosic part [4–6]. Furthermore, the non-cellu-

losic part of the textiles, which remained after the bioconversion as a purified value-added product, is currently degraded through the disposal methods which cause environmental problems [7,8], despite their potential for recycling and reuse.

Similar to lignocelluloses, waste textiles have a cellulosic part that can be converted to glucose through hydrolysis and consequently fermented to ethanol [4–6]. However, the structure and composition of waste textile and lignocelluloses are totally different. Cellulosic part of waste textile is accompanied by a man-made fiber, e.g., polyester, while it is covered by hemicellulose and lignin in the lignocelluloses [4,9]. Even though, hydrolysis of cellulose in waste textile, in contrast to lignocelluloses, is not inhibited by lignin part, the higher crystallinity of cellulose is the main obstacle in bioconversion of waste textile [4,10,11]. Therefore, using a proper pretreatment prior to hydrolysis is essential for efficient hydrolysis of cellulosic part of waste textile [4,5,12].

The bioconversion of waste textile has recently been considered through pretreatments with N-methylmorpholine-N-oxide (NMMO), NaOH, and phosphoric acid, in order to enhance the rate of cellulose hydrolysis [4,5,13].

Alkaline pretreatment has been previously applied for enhancement of ethanol and biogas production from lignocelluloses materials. This pretreatment is one of the most effective and low cost pretreatments in which the crystallinity of cellulose decreases through dissolution and consequent regeneration [14–17]. The reduction of hemicellulose and lignin contents is among the important features of the process for enhancement of ethanol and biogas production from soft and hardwood. Moreover, addition of urea, thiourea, or a mixture of urea and thiourea has been demonstrated to enhance the dissolution of cellulose in the NaOH solution. These components ensnare the free water and prevent the reassociation of cellulose molecules [15,18,19]. In addition, it has been shown that even higher yields could be obtained through alkaline pretreatment with addition of urea, thiourea, or combination of them [14–16].

To the best of our knowledge, the alkaline pretreatment with urea, thiourea, or combination of them have not been evaluated for bioconversion of waste textile. In addition, the effect of the pretreatments on changing the properties of non-cellulosic part of waste textile has not been considered in previous studies.

In this study, the alkaline pretreatments using NaOH, NaOH/urea, NaOH/thiourea and NaOH/urea/thiourea aqueous solutions at different temperatures were evaluated for improvement of the enzymatic hydrolysis and ethanol production from polyester–cotton textile. In addition, the properties of recovered polyester as a value-added product were compared with the untreated polyester.

2. Material and methods

2.1. Materials

The waste textile used in this study was a white 40/60 polyester/cotton blend (Poya Baft factory, Isfahan, Iran), in which its polyester part was polyethylene terephthalate produced by Polyacryl, Iran. The textile solid content was $98 \pm 1.3\%$ as determined by drying at 110°C until a constant weight. The textile was cut into small pieces (approximately $3 \times 3\text{ cm}^2$) and milled by a whirring-blade grinder (KSM2, Braun) to less than 1 mm particles before subjecting to the experiments. Enzymatic hydrolysis was conducted using two enzymes of β -glucosidase (Novozym 188, Novozyme, Denmark) and cellulase (Celluclast 1.5 L, Novozyme, Denmark). The activity of β -glucosidase was 190 IU/ml as measured by Ximenes et al. method [20], while the activity of cellulase was 52.5 FPU/ml measured according to Adney and Baker procedure [21].

2.2. Alkaline pretreatment

Aqueous alkaline mixtures of NaOH (12 wt%) [5], NaOH/urea (7/12 wt%) [22], NaOH/thiourea (9.5/4.5 wt%) [23], and NaOH/urea/thiourea (8/8/6.5) [24] were used for pretreatment of waste textiles. The pretreatments were conducted by mixing 5 g of waste textile with 95 g alkaline solution at different temperatures of -20 , 0 , 23 , and 100°C for 1 h. The mixture was mixed every 10 min using a glass rod. Subsequently, the pretreated textile samples were washed with distilled water to obtain pH 7. The washed samples were air-dried for one day.

2.3. Enzymatic hydrolysis

The treated and untreated waste textiles were subjected to 72 h enzymatic hydrolysis at 45°C and pH 4.8 (in 50 mM sodium citrate buffer supplemented with 0.5 g/l sodium azide) with 3% (w/v) solid (substrates) loading, using 30 FPU cellulase and 60 IU β -glucosidase per gram of cellulose. The yield of enzymatic hydrolysis is defined as (grams of glucose released)/(grams of initial cellulose $\times 1.111$). The dehydration factor (1.111) is employed to consider the water addition to the cellulose chains [25]. After 72 h enzymatic hydrolysis, the remaining solid (mainly contains polyester) was separated through sieving.

2.4. Simultaneous saccharification and fermentation (SSF)

The treated and untreated waste textiles were subjected to simultaneous saccharification and fermentation (SSF). The substrates were suspended in 50 mM sodium citrate buffer (pH 5) with solid loading of 5% (w/v), supplemented with 5 g/l yeast extract, 7.5 g/l $(\text{NH}_4)_2\text{SO}_4$, 3.5 g/l K_2HPO_4 , 0.75 g/l $\text{MgSO}_4 \cdot 7\text{H}_2\text{O}$, and 1 g/l $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$. The mixture was then autoclaved at 121°C for 20 min. An amount of $1.0 \pm 0.1\text{ g/l}$ *Saccharomyces cerevisiae* (base on dry biomass) (Golmayeh, Iranmayeh, Iran) and 15 FPU cellulase and 30 IU β -glucosidase per gram cellulose were then added to the suspension. It was then incubated at 36°C for 72 h under anaerobic conditions [26]. The yield of ethanol production was defined as (grams of produced ethanol)/(grams of initial cellulose $\times 0.51 \times 1.111$) [25].

2.5. Analytical methods

The concentration of ethanol produced in SSF was determined by high-performance liquid chromatography (HPLC) equipped with RI detector (Jasco International co., Tokyo, Japan). Ethanol was separated on Aminex HPX-87H column (Bio-Rad, Hercules, CA) at 60°C using 0.6 ml/min eluent of 5 mM sulfuric acid.

Cellulose content of original waste textile was determined by following the standard method of the National Renewable Energy Laboratory (NREL, Denver, CO, USA) [27]. The percentage of separation polyester and cotton by enzymatic hydrolysis was defined as:

$$\text{Separation (\%)} = (W1 - W2)/W1 \times 100 \quad (1)$$

where $W1$ and $W2$ are the weight of cotton residue in treated samples before and after hydrolysis, respectively.

The glucose content of the hydrolysates was measured using glucose (HK) assay kit (Pars azmone, Iran).

2.6. Polyester characterizations

The chemical properties of polyester were collected by FTIR spectra (Jasco 680 plus FTIR spectrometer, Japan). The infrared spectra recorded between $400\text{--}2000\text{ cm}^{-1}$ with a 4 cm^{-1} resolution and summations over 32 scans.

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