Appl Biochem Biotechnol (2012) 166:744–752
DOI 10.1007/s12010-011-9463-0

Recycling of Cellulosic Fibers by Enzymatic Process

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Received: 1 October 2011 / Accepted: 15 November 2011 /
Published online: 13 December 2011

Abstract In this research, enzymatic treatment as an environmental friendly process has been used for recycling process of old cellulosic wastes such as cotton, viscose, and lyocell. Cellulase hydrolyses cellulosic chains and shortens cellulosic fibers. This study investigates to detect the optimum enzyme concentration and time of treatments for suitable changes of length and weight loss. The main purposes of this article are shortening of cellulosic fibers and evaluating of enzymatic treatment in different kind of cellulosic fibers. According to the data of experiments, with the increase of enzyme concentration and the treatment time, the length and weight loss percentage of the cellulosic fibers has been decreased. The length and weight loss percentage of treated viscose is more than that of lyocell and cotton fibers. Optimized condition, reaction time, and enzyme concentration have been determined by mean length of treated cellulosic samples. Suitable longitudinal distribution of fiber for papermaking industries is in the range of 0 to 4 mm. Optimum enzyme concentration and treatment time for recycling cotton, lyocell, and viscose fibers are 2% and 48 h for cotton and lyocell and 0.5% and 48 h for viscose, respectively. According to the data of experiment, the length of treated fibers is appropriate for its usage as a raw material in papermaking industries.

Keywords Recycling · Cellulosic fiber · Cellulose · Enzyme · Weight loss · Waste · Fiber shortening

Introduction

Environmental concerns have fueled a desire to reduce landfill usage and to decrease the harvest of trees for the production of new paper. The efficient utilization of this process

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could make a significant contribution to the solving of present environmental and ecological problems; these processes have a great role in decreasing the energy and fuel consumption, pollution, and expenses [1].

The objective of the experimental case study research is cellulose waste fibers in paper sheets. The importance of secondary fibers as a raw material in paper production is increasing, which has led to the introduction of recycled fibers to higher paper grades. Some of the chemical process takes place in existing environment proceeds slowly (if any other factor does not influence them). The catalysts are needed to enhance the rate of these kinds of reaction in an existing cell. In biological systems, the catalysts are enzymes [2].

They accelerate the chemical reactions, and in contrary to the old opinions, they are never alive. During the time of reaction, some changes take place in enzymes like any other catalysts, but after the reaction, they obtain their initial structure so they are able to catalyze the next reaction, and it can be repeated. That means a small quantity of enzyme is able to react with a large amount of substrate. Enzymes are being used increasingly in textile processing, mainly in the finishing of cellulosic fabrics.

In some studies, the effects of cellulase enzymes on cotton fabrics have been evaluated [3, 4]. Dadashian [5] investigated about the effect of enzymatic treatment on fine structure of cellulosic fibers.

Rosenberg et al. [6] investigated about the effect of enzymatic pretreatment on the properties of cellulose dissolved in aqueous 7.6% NaOH and in an ionic liquid.

The results of the investigation carried out by Buschle-Diller et al. show the effect of cellulase on the pore structure of bead cellulose.

In this study, an enzymatic treatment with cellulases from *Trichoderma viride* was investigated due to its effect on the pore structure of different types of bead cellulose [7].

In enzymatic processes, control of pH and temperature is very important. In most of the time, specific buffer has been used in order to keep pH in a constant value.

The main purposes of this study are shortening of cellulosic fibers and, after that, evaluating of optimum enzymatic treatment in different kinds of cellulosic fibers according to the mean length of enzyme-treated samples. Suitable longitudinal distribution of fiber for papermaking industries is in the range of 0 to 4 mm.

In this study, many factors such as weight loss, moisture regain, and microscopic structure of treated samples have been measured.

The results show that the amount of reduction in weight and length of treated fibers is appropriate for its usage in papermaking process.

Experimental

Materials

The cellulase enzyme (Cello soft L.) and buffer solution (acetate buffer) have been provided by NOVOZYME Company and Merck Company, respectively.

The cotton fiber waste has been provided by Natanz Spinning Company, which has a count number of 8 deniers and diameter of 20 μm. The longitudinal distribution of cotton fiber is in the range of 18–24 mm.

The viscose and lyocell fibers wastes have been provided by Lenzing Company in Austria which have a diameter of 30 μm and count number of 10 deniers. The longitudinal distribution of lyocell and viscose fiber are in the range of 14–16 and 13–15 mm, respectively.

Enzymatic Treatments

Washing Process and Enzymatic Process

Different kinds of cellulose fiber wastes have been washed by soap solution in 60 °C for 15 to 20 min.

After the washing process, the enzyme was applied to the fiber wastes in a static bath without any liquor movement or agitation of the samples at 40 °C by using 0.1 M acetate buffer (pH=4.8) and different enzyme concentrations in the range of 0.5% to 2% in 24 to 72 h. In all experiments, the ratio of liquid to gram of sample is 1:8 (L:R: 1:8).

The residue of each sample was washed by distilled water till it was free from enzyme and then dried at 100 °C in the oven.

Testing and Analysis

In this study, optical projection microscope and Philips scanning electronic microscope have been used to measure fiber length and evaluate the surface morphology of treated and untreated cellulosic samples, respectively.

All of the samples have been conditioned at atmospheric condition (temperature, 25 °C and humidity, 65%) for 24 h, after that, their weights have been measured.

The weight loss percentage of the samples was measured by weighing cellulosic samples before and after enzymatic treatments.

The weight loss percentage was defined as follows:

$$\text{Weight loss percentage } (\%) = (A_2 - A_1)/A_1 \times 100$$

where A_1 and A_2 stand for the dry weight and conditioned weight of the samples before and after enzymatic treatment, respectively.

The moisture regain of the samples was measured by gravimetric method. The moisture regain was defined as follows:

$$\text{Moisture regain } (\%) = (W_2 - W_1)/W_1 \times 100$$

where W_1 and W_2 stand for the dry weight and conditioned weight of the samples, respectively. The FTIR spectra were obtained by using a Nicolet Magna IR spectrophotometer equipped with a microscope.

Results and Discussion

Weight Loss

In Fig. 1, the effect of enzymatic treatment on weight loss percentage of cellulose fiber wastes has been evaluated according to the time and enzyme concentration.

According to the data in the figure, with the increase of enzyme concentration and enzymatic treatment time, weight loss percentage of cellulosic samples has been increased.

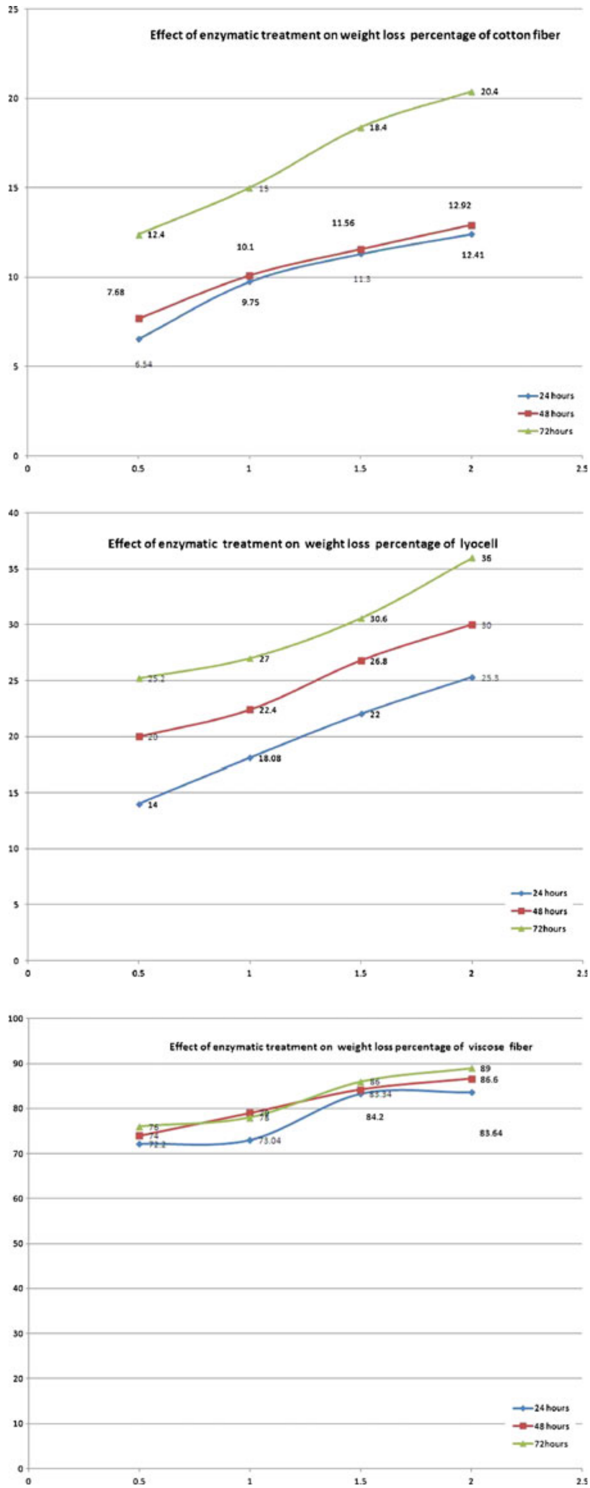

Fig. 1 Effect of enzymatic treatment on weight loss of cellulosic fiber

The results show that treated viscose fiber has more weight loss percentage compared with cotton and lyocell fiber, which is related to the lower crystallinity of viscose fiber compared with other cellulosic fibers.

Mean Length

Mean length of untreated and treated samples has been measured by projection optical microscope in a scale of ×400.

In Fig. 2, the effect of enzymatic treatment on mean length of cellulosic fiber wastes has been evaluated according to the time and enzyme concentration.

According to the data in the figure, with the increase of enzyme concentration and enzymatic reaction time, mean length of cellulosic samples has been decreased.

The results show that the mean length of viscose is lower than lyocell and cotton fiber, which is related to the lower crystallinity of viscose fiber compared with other cellulosic fibers.

Fiber length more than 2 mm have been used as long fiber, and fiber length less than 2 mm have been used as short fiber in papermaking industries and other related industries. Obtained fibers have a longitudinal distribution in the range of 0 to 4 mm in optimum enzyme concentration.

Moisture Regain

The moisture regain of the samples was measured by gravimetric method. The moisture regain was defined as follows:

$$\text{Moisture regain } (\%) = (W_2 - W_1)/W_1 \times 100$$

Moisture regain of treated and untreated cellulosic sample has been shown in Table 1. According to the data in Table 1, the downward rate of moisture regain in viscose fiber waste is higher than lyocell and cotton fiber wastes in optimized enzymatic treatment.

Microscopic Structure and IR Spectroscopy

Electronic microscopic photographs have been used to evaluate macrostructure of untreated and enzyme-treated samples. In Fig. 3, scanning electronic microscopic photographs of untreated and enzyme-treated cellulosic fiber wastes in optimum condition have been shown in a scale of ×690. According to these macroscopic photographs, destruction of cotton samples is in a longitudinal direction, and destruction of lyocell and viscose samples is in a cross direction.

FTIR Spectroscopy

In order to show the effects of enzymatic treatments on the fine structure of cellulose samples, FTIR spectra of 650–4,000 cm^{-1} have been recorded. The spectra of enzyme-treated and untreated cotton, viscose, and lyocell fiber wastes have been shown in Figs. 4, 5, and 6.

According to the data in these figures, there is a marked difference between the spectra of treated and untreated cellulosic fiber wastes.

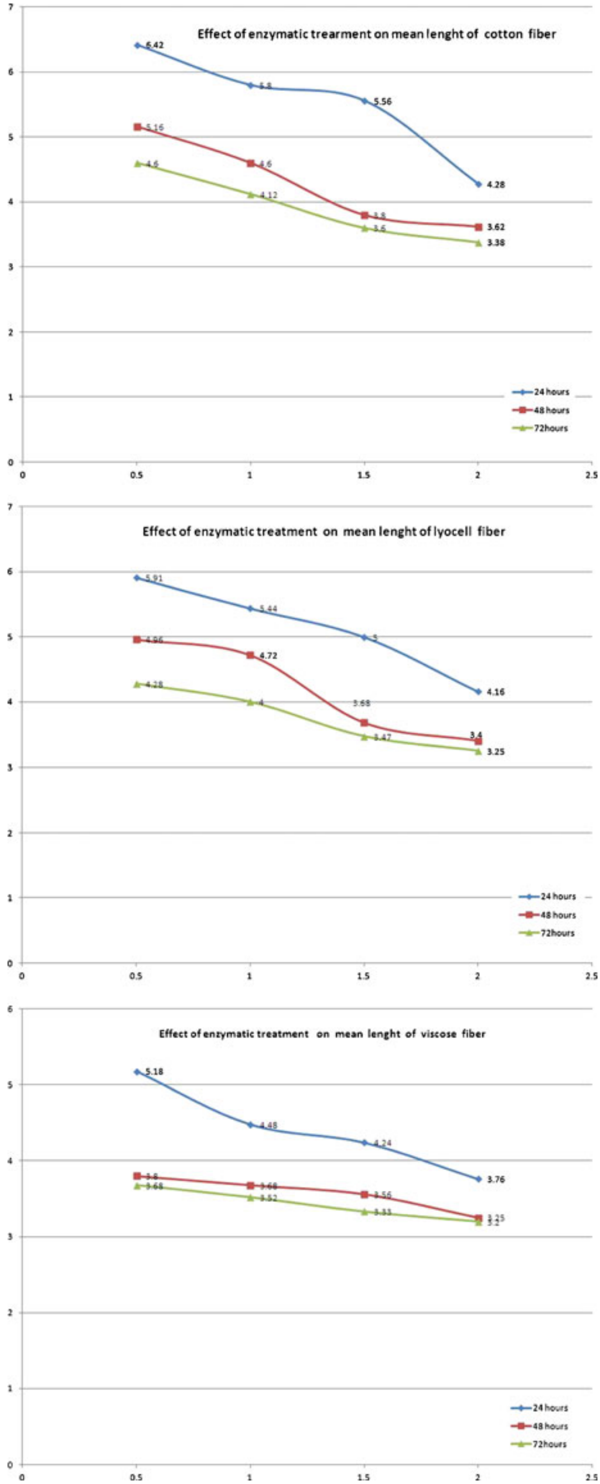

Fig. 2 Effect of enzymatic treatment on mean length of cellulosic fiber

Table 1 Moisture regain of cellulosic fiber wastes after and before enzymatic treatment in optimized condition

Type of sample	Untreated sample (%)	Treated sample (%)	Downward rate (%)
Cotton	4.8	4	16.66
Lyocell	5	2.94	41.2
Viscose	6	2.22	63

A broad absorption peak near 3,350 cm^{-1} was found for all cellulosic materials. This has been assigned to the vibrations due to the stretching of the O–H group.

The intensity and frequency of this band are closely related to the hydrogen bonding system.

According to the comparisons that have been carried out between untreated and treated cotton, viscose, and lyocell fibers, the region of stretched OH groups in enzyme-treated lyocell and viscose is wider than untreated lyocell and viscose, which means higher hydrogen bonding exists.

Conclusion

In this study, enzymatic treatments have been used to modify cellulosic wastes such as cotton, lyocell, and viscose.

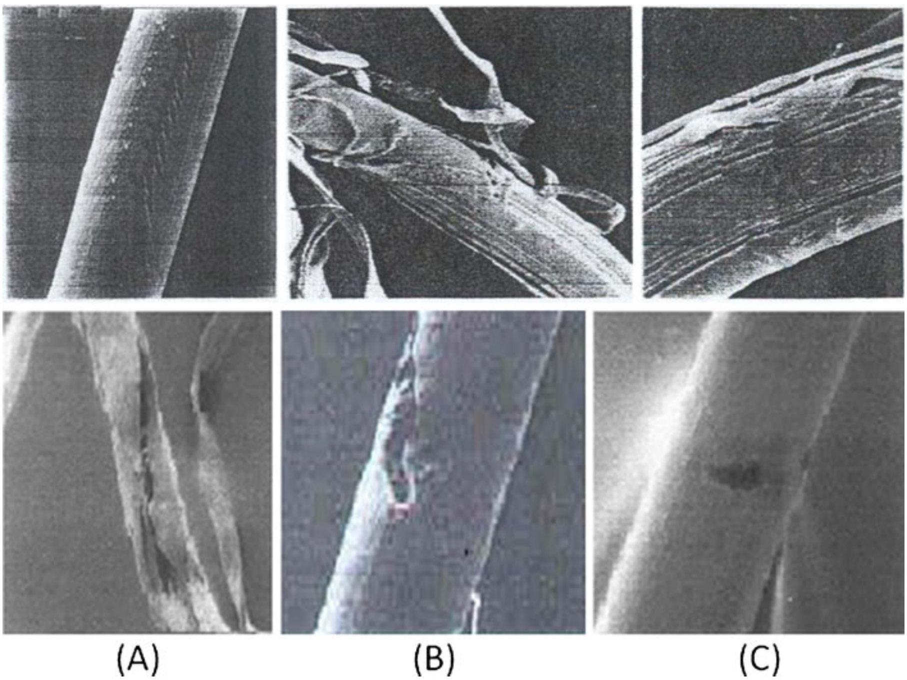

Fig. 3 SEM photograph of enzyme-treated and untreated cellulosic fiber wastes in optimized enzyme concentration. **a** Cotton. **b** Lyocell. **c** Viscose

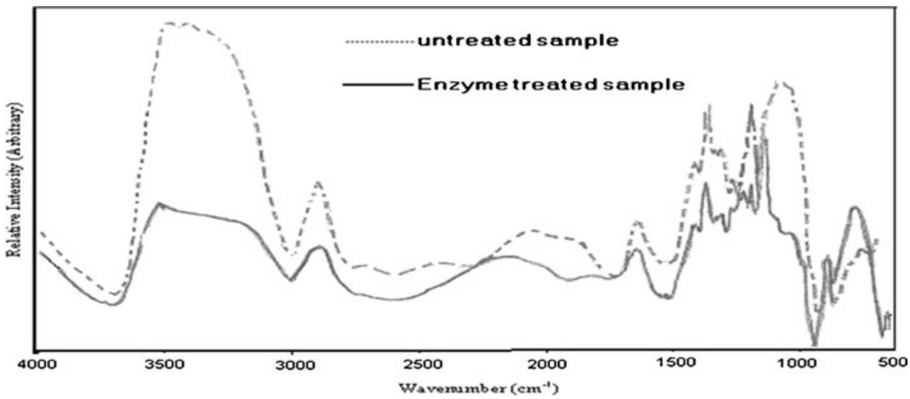

Fig. 4 FTIR spectra of enzyme-treated and untreated lyocell

The results of the investigation have revealed that enzymatic treatment has an effect on the cotton, viscose, and lyocell fiber wastes.

Enzymatic treatment causes to shorten cellulosic fiber wastes by hydrolysis of cellulosic chains. Under the same condition, viscose fiber length is shorter than lyocell and cotton fiber, and weight loss percentage of viscose fiber is more than lyocell and cotton fiber.

This kind of treatment causes to decrease moisture regain percentage in cellulosic fiber wastes; moisture regain percentage of viscose and lyocell enzyme-treated samples decreases more than that of cotton.

According to the IR spectra of treated and untreated cellulosic fiber wastes, enzyme-treated cotton, viscose, and lyocell fiber wastes have more hydrogen bonds than the untreated samples.

Optimum condition, reaction time, and enzyme concentration have been determined by mean length of treated cellulosic samples. (Suitable longitudinal distribution of fiber for papermaking industries is in the range of 0 to 4 mm.)

Optimum enzyme concentration and treatment time for recycling cotton, lyocell, and viscose fibers are 2% and 48 h for cotton and lyocell and 0.5% and 48 h for viscose, respectively.

According to the data of the experiment, the length of treated fibers is appropriate for its usage as a raw material in papermaking industries.

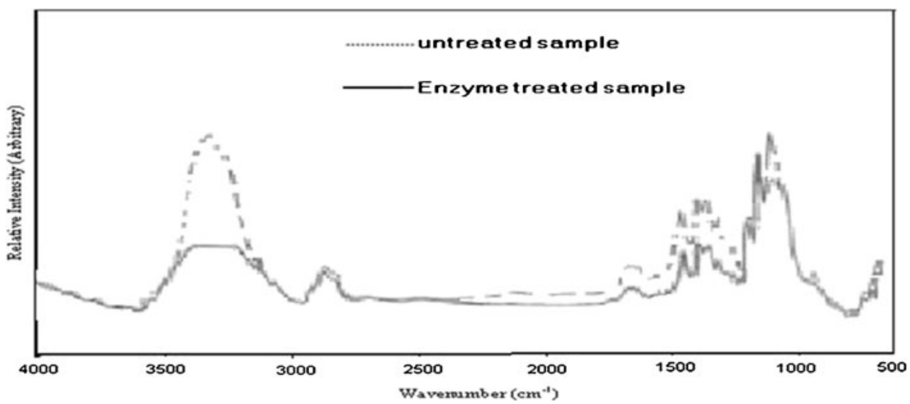

Fig. 5 FTIR spectra of enzyme-treated and untreated cotton

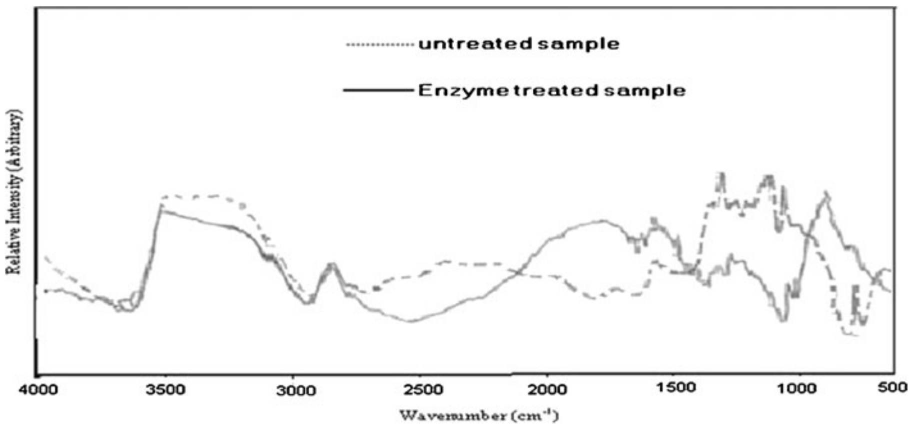

Fig. 6 FTIR spectra of treated and untreated viscose fibers

Acknowledgement The authors appreciate Dr. Ali Reza Khosravi and the reviewers of the journal for cooperating in revising and editing the article.

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