APPLICATION OF ULTRASOUND TO ENZYMATIC PROCESSING OF COTTON TEXTILES

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Abstract

The combination of ultrasound with conventional enzymatic treatment of cotton offers significant advantages such as less consumption of expensive enzymes, shorter processing time, less fiber damage and better uniformity of enzymatic treatment. Our laboratory research has shown that introduction of ultrasonic energy during enzymatic treatment resulted in significant improvement in the performance of the enzyme.

Introduction

Enzymatic treatment of cotton fibers is a nontoxic, environmentally benign process, which gained wide recognition for various textile processing applications such as desizing, scouring, bleach cleanup, bio-stoning and bio-polishing (Lange 1996; Yonghua 1997). The application of enzymes in the textile industry is becoming increasingly popular because of the mild conditions of temperature and pH that are required and their capability for replacing harsh organic/inorganic chemicals. Also important is that wastewater effluent from enzymatic treatments is readily biodegradable and accordingly does not pose any environmental threat. In addition to numerous advantages provided by the use of enzymes for textile wet processing there are several shortcomings of enzymatic treatment of cotton fabric, such as more expensive processing costs, longer processing time and significant decrease in fabric strength properties. Enzymatic treatments of the cotton fabrics, like any wet processing of textiles, involves the transfer of mass from the processing liquid medium (enzyme solution) across the surface of the textile substrate. It is well known that large enzyme molecules have low diffusion rates and mostly attack external cellulose fibers in cotton yarn, resulting in excessive damage of the fibers. In general, mechanical agitation of the enzyme solution improves transport of bulky enzyme molecules toward the surface of cellulose fibers and into internal spaces of cotton yarn and provides more effective and uniform treatment of cellulose fibers throughout the cotton yarn (Hartzell and Durrant 1998). Many nontraditional techniques, such as the use of radio frequency and microwave energy, infrared heating, and ultrasound are being investigated by numerous researchers to reduce processing time, energy consumption and improve product quality. In this connection, ultrasound has been of considerable interest for textile wet processing (Thakore et. al., 1990). Investigators have shown that introduction of ultrasonic energy into the processing bath significantly accelerates physical and chemical processes (Suslic 1989). Several authors have reported that ultrasonic energy has been successfully applied in the mercerization of cotton and cotton blends, desizing and scouring of fibers and yarns (Elgal et. al., 1979), peroxide bleaching (Safonov 1984), dyeing (Oner et. al., 1995) and finishing(Simkovich and Yastrebinskii 1975). We can expect similar benefits in cases of application of ultrasonic energy to enzymatic reactions. A thorough search of available literature did not yield any information directly related to application of ultrasound energy to enzymatic treatment of natural fibers. It is also unclear how ultrasonic energy can affect very complex and sensitive structures of enzyme molecules. The objective of the present work was to study the influence of ultrasound on enzymatic treatment of cotton fabric.

Reprinted from the *Proceedings of the Beltwide Cotton Conference*
Volume 2:1369-1372 (1999)
National Cotton Council, Memphis TN

Experimental

Desized, scoured, bleached and mercerized cotton printcloth (3.2 oz./sq. yd.) was used for all tests. The samples (20 in. x 13 in.) of cotton fabric were sewn around the edges to prevent unraveling during processing. A whole cellulase enzyme (Cellusoft L) from Novo Nordisk with 0.1 M acetate buffer (pH – 4.87) was used in all trials. The enzymatic treatment of all samples was carried out in a NEARFIELD™ Acoustical Processor. This dual frequency ultrasonic reactor with thermal control capabilities was specially designed and manufactured for controlled sonication of the textile samples by Advanced Sonic Processing Systems. Three tests were carried out with different treatment parameters, such as enzyme concentration, sonication power, treatment time, and circulation rate. The temperature of the enzyme solution in the reaction chamber was maintained at $50^0C \pm 1^0C$ in all trials in all tests. After enzyme/ultrasound treatment, all samples were thoroughly rinsed in deionized water, tumble-dried and placed in an oven at 110 ^0C for six hours in weighing bottles to determine dry weight after treatment. Breaking strength in the warp and fill directions was determined in accordance with the standard method - ASTM D 5035-95 (strip test) on the Instron tester machine.

Test # 1

The treatment parameters for Test #1 are presented in the Table 1. All samples of cotton fabric were treated with enzyme solution in combination with mechanical agitation/circulation and/or sonication for 45 min. The first trial (control) was run with only buffer solution instead of enzyme solution. The objective of this trial was to determine how sonication alone could possibly affect the cotton fabric in addition to the enzyme treatment. The weight loss and breaking strength for all samples of cotton fabric in all trials (original and treated with enzyme and ultrasound) are presented in the Table 1 and Figures 1 and 2. In general, the average weight loss of the samples treated with 0.2 % of enzyme solution was in the range of 1.8 – 2.4 %. The control samples (trial # 1-1) that were treated with buffer solution under identical conditions showed only marginal weights loss – 0.18 %. The experimental data from Test #1 show that the weight loss of the sample of cotton fabric that was sonicated in addition to enzymatic treatment increased by 32.8 % (trial # 1-3 vs. trial # 1-2). It is important to note that this substantial improvement in the efficiency of the enzymatic treatment resulted from sonication of only half of the total surface area of the fabric samples. The active surface of diaphragm plates with attached ultrasound transducers was ~ 50% of the total surface. In contrast, conventional mechanical agitation/circulation of the enzyme solution (trial # 1-3 vs. trial # 1-4) provided only modest improvement in enzyme performance (up to 6.8%) in comparison with the sonication treatment. The decrease of tensile strength for all samples of cotton fabric treated with enzyme only or by combination of enzyme and sonication or circulation was comparable (trial # 1-2 – 19.1%; trial # 1-3 – 17.7%; trial # 1-4 – 18.7%). The unexpected decrease of the tensile strength (24.5 %) was observed for the samples that were treated with buffer solution only (trial # 1-1) instead of enzyme solution. It was not immediately clear what caused this decrease in strength – acidic nature of the buffer solution itself or sonication or both. To resolve this problem two separate trials were run in Test #2 – one with buffer solution plus circulation (trial # 2-5) and the other with the buffer solution plus sonication (trial # 2-1).

Test # 2

The treatment parameters in Test # 2 were also altered to make all effects more pronounced: concentration of the enzyme solution was doubled (0.4 %), sonication power was increased up to maximum generator output (15 amps.), and sonication time was increased up to 60 min. An additional trial was carried out in which three samples of cotton fabric were treated with buffer solution only and circulation (trial # 2-5). The objective of this trial was to determine how acidic buffer itself affects the strength of the cotton fabric. Also, samples of the original, untreated cotton fabric were tested for breaking strength after drying in the oven under condition similar to those for treated samples. The objective of this extra test was to determine how the drying process itself could possibly affect the strength of the cotton fabric in addition to an enzyme and/or ultrasound treatment. The results of Test #2 are presented in Table 2 and Figures 3 and 4. In general, the average weight loss of the samples treated with 0.4 % solution of enzyme was in the range of 3.14 – 3.89 %. Comparison of data from Test #1 and Test #2 shows that weight loss of samples of cotton fabric treated with double the concentration of enzyme (0.4 %) was only 65 -75 % higher then for samples treated with 0.2% enzyme. Samples that were sonicated in addition to enzymatic treatment (trial # 2-3 vs. trial # 2-2) once again showed an improvement in enzyme performance (average increase in weight loss was of 23.9 %). Detailed analysis indicated that this improvement in enzyme performance caused by sonication was much more pronounced in the case of the less concentrated (0.2 %) enzyme treatment - 32.8 % (Test #1), then in case of treatment with double concentration of enzyme (0.4 %) - only 23.9 % (Test #2). The data indicate that the maximum benefit provided by sonication of the enzyme solution occurs at relatively low concentration of enzyme. Comparison of the average weight loss of samples in the trial # 2-1 ~ 0.29 % (circulation + sonication) and in the trial # 2-5 ~ 0.07 % (circulation only) reveals that sonication processing is primarily responsible for the weight loss of samples of the cotton fabric in the absence of enzyme. This additional weight loss inflicted by sonication could be explained by more thorough removal of size, soils and other impurities that still might be present in fabric after conventional desizing, scouring and bleaching operations. Comparison of the data on breaking strengths of these samples shows that they are practically equal (trial # 2-1 ~ 88.7 lb. and trial # 2-5 ~ 85.7 lb.). The data indicate that the sonication itself does not affect the tensile strength of cotton fabric as shown in Table 2. The observed decrease in the strength for all samples in these trials (2-1 and 2-5) in comparison with the original, untreated samples (≈ 6 %) could be attributed to the influence of the acidic buffer solution at elevated temperature. Comparison of the breaking strengths of the two original, untreated samples of the cotton fabric, one of which was dried similarly to all treated samples, reveals that both samples have nearly equal breaking strength.

Test # 3

The primary objective of Test #3 was to find how enzymatic treatment of cotton fabric with various concentration of enzyme would be affected by sonication. All test parameters in Test #3 were similar to those of Test #2. The only difference was that the concentration of enzyme was varied from 1.0 g/l. to 8.0 g/l. The first trial (3-1) was run with just deionized water and sonication. The objective of this trial was to provide baseline data on the weight loss and tensile strength of textile samples under sonication condition and in the absence of enzymatic treatment. Also, for comparison purposes an additional set of original, untreated samples (trial 3-11) was dried in the oven under conditions similar to those for treated samples and tested for breaking strength. The treatment parameters and results of Test #3 are presented in Table 3 and Figure 5. Throughout all concentration ranges, the average weight loss of all samples that were treated with combination of ultrasound and enzyme was greater then for samples treated with enzyme only. This experimental data has shown that the maximum benefit provided by sonication occurs at the concentrations of enzyme of 1-3 g/liter (improvement up to 35 % compared with about 20 % for enzyme concentrations of 4 –8 g/liter). The experimental data for tensile strength of treated samples show the gradual decrease of breaking strength of the samples treated with more concentrated enzyme solution. The samples that were sonicated in addition to enzyme treatment showed slightly higher decrease in tensile strength, which is attributed to improved efficiency of enzymatic treatment under sonication conditions.

Comparison of the tensile strength of original samples (trial 3-10), original samples that were dried under identical conditions like all treated samples (trial 3-11) and original samples that were only sonicated but not treated with enzyme (trial 3-1) shows that they are essentially equal.

Discussion

The general trend that was observed in all tests indicated that the introduction of ultrasonic energy to the reaction chamber during enzymatic treatment resulted in a significant improvement in the performance of the enzyme, but it did not contribute to a decreasing the tensile strength of cotton fabric. It appears that the greatest enhancement of enzymatic treatment of cotton fabric could be achieved by combination of sonication with conventional mechanical agitation. Such a combination could provide significant economical advantage either through shorter treatment time or less concentration of enzyme or both. The observed enhancement in the enzymatic treatment of cotton fabric by introduction of ultrasonic energy in the reaction chamber may be caused by various physical and chemical phenomena resulting from interplay between ultrasound waves, enzyme molecules and liquid media. The introduction of ultrasonic energy in the reaction chamber for enzymatic treatment of the cotton fabric can cause the following effects:

- Acceleration of the diffusion rate of enzyme molecules toward the fiber surface through the border layer of liquid. The concentration of enzyme molecules in this layer is a controlling factor, which defines the overall rate of reaction.
- Improved removal of the products of enzymatic hydrolysis from the reaction zone, which accelerates the overall rate of reaction.
- Degassing expulsion of dissolved or entrapped gas or air molecules from fiber capillaries and interstices at the crossover points of fabric into liquid and removal by cavitation.

While these postulates seem reasonable, much more detailed studies need to be carried out to achieve better understanding of the mechanism of the influence of ultrasound on the enzymatic treatment of cotton fabric.

Conclusion

This research has shown that at the laboratory scale, introduction of ultrasonic energy in the reaction chamber during enzymatic treatment of cotton fabric resulted in significant improvement in enzyme efficiency, but did not contribute to decrease of tensile strength of cotton fabric. Also, it appears that ultrasound does not affect specific activity of complex structures of enzyme molecules. It was established also that the greatest improvement in the efficiency of enzymatic treatment of cotton fabric was provided by the combination of conventional mechanical agitation with sonication of the treatment solution. The experimental data indicate that the maximum benefit provided by sonication of enzyme solution occurs at relatively low concentrations of enzyme. Introduction of ultrasonic energy in the reaction chamber for enzymatic treatment of cotton fabric could provide significant economical advantage through reduced processing time, less concentration of enzyme and better uniformity of enzymatic treatment.

Disclaimer

Specific company, product, and equipment names are given to provide exact description of experimental details. Their mention does not imply recommendation or endorsement by the U.S. Department of Agriculture.

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Table 1. Treatment parameters and results of Test #1.

Trial	Enzyme Conc., g/liter	Sonic. Power, Amps.	Weight Loss, %	Breaking Strength, lb. Warp	Fill	Warp + Fill
1-1	0.0	12	0.18	39.7	31.5	71.2
1-2	2.0	0	1.77	45.0	31.3	76.3
1-3	2.0	12	2.35	45.0	32.6	77.6
1-4	2.0	12	2.20	42.9	33.8	76.7
Orig. untr. fabric				51.6	42.7	94.3

Table 2. Treatment parameters and results of Test #2.

Trial	Enzyme Conc., g/liter	Sonic. Power, Amps.	Weight Loss, %	Breaking Strength, lb. Warp	Fill	Warp + Fill
2-1	0.0	15	0.29	49.6	39.1	88.7
2-2	4.0	0	3.14	44.6	32.2	76.8
2-3	4.0	15	3.89	36.1	32.4	68.5
2-4	4.0	15	3.81	42.2	35.0	77.2
2-5	0.0	0	0.07	46.6	39.1	85.7
Orig. untr. fabric				48.6	44.1	92.8
Orig. untr. fabric (dried)				51.6	42.7	91.5

Table 3. Treatment parameters and results of Test #3.

Trial	Enzyme Conc., g/liter	Sonic. Power, Amps.	Weight Loss, %	Breaking Strength, lb. Warp	Fill	Warp + Fill
3-1	0.0	13.0	0.14	51.97	46.86	98.83
3-2-1	1.0	0.0	2.22	49.40	38.16	87.56
3-2-2	1.0	13.0	2.86	50.04	36.93	86.97
3-3-1	1.5	0.0	2.72	45.96	39.28	85.24
3-3-2	1.5	13.0	3.44	40.08	40.48	80.56
3-4-1	2.0	0.0	2.45	46.63	41.36	87.99
3-4-2	2.0	13.0	3.38	42.57	40.85	83.42
3-5-1	2.5	0.0	3.16	46.74	39.05	85.79
3-5-2	2.5	13.0	3.47	43.00	39.12	82.12
3-6-1	3.0	0.0	3.02	47.18	38.44	85.62
3-6-2	3.0	13.0	3.51	39.61	39.77	79.38
3-7-1	4.0	0.0	3.31	46.65	38.31	85.62
3-7-2	4.0	13.0	4.07	39.38	35.42	74.80
3-8-1	5.0	0.0	3.59	46.77	38.37	85.14
3-8-2	5.0	13.0	4.13	36.52	37.40	73.92
3-9-1	8.0	0	3.87	45.68	38.82	84.50
3-9-2	8.0	13.0	4.13	42.61	36.25	78.86
3-10	Orig., untr.			48.97	46.75	95.72
3-11	Orig., untr. (dried)			49.13	44.16	93.29

Figure 1. Effect of treatment conditions on weight loss of cotton printcloth in Test #1.

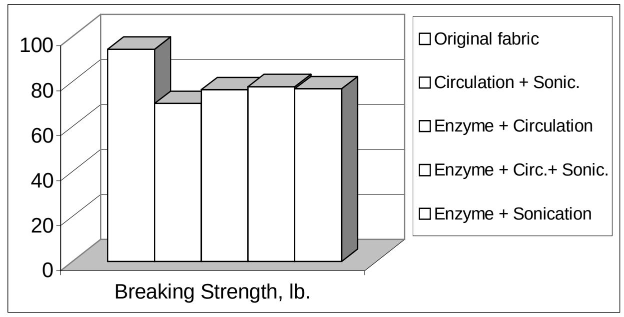

Figure 2. Effect of the treatment conditions on the breaking strength (Warp + Fill) of the cotton printcloth in Test #1.

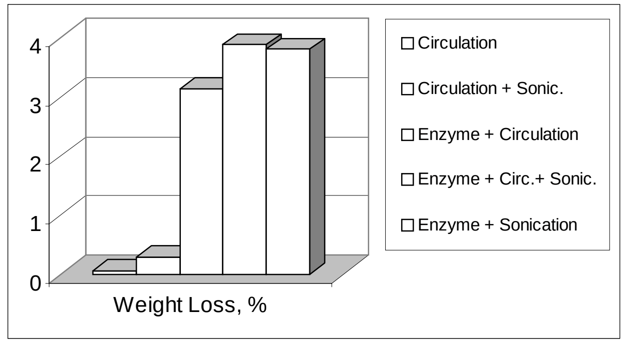

Figure 3. Effect of the treatment conditions on weight loss of cotton printcloth in Test #2.

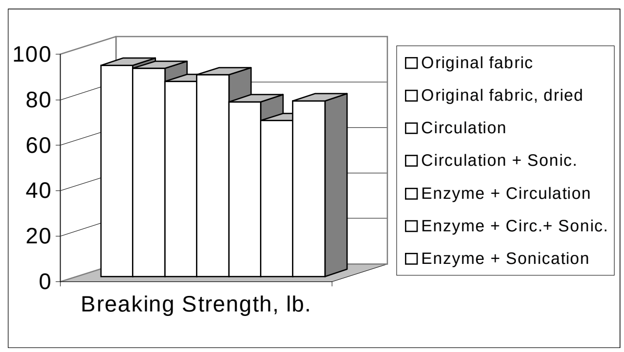

Figure 4. Effect of treatment conditions on breaking strength (Warp + Fill) of cotton printcloth in Test #2.

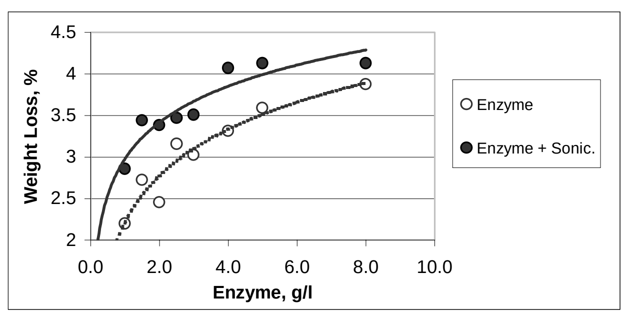

Figure 5. Effect of the treatment conditions on weight loss of cotton printcloth in Test #3.