Ultrasonic-assisted pretreatment of cotton textiles

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Abstract: Ultrasound waves are being successfully used in many industrial and medical fields and it is only natural that this form of energy may find application in the textile industry. Use of ultrasound in the textile wet processing has been found to have some advantages as it alters the fibre structure to increase adsorption and also raises diffusion coefficient of the chemical molecules and the dye particles in the aqueous solutions. In the present study, influence of ultrasound on desizing, bioscouring, peroxide bleaching and the Solomatic bleaching of cotton material of 136 gsm has been studied. Various objective parameters like the desizing efficiency, absorbency, loss in weight and tenacity with and without ultrasound were studied and the results compared to determine the possible advantages of the ultrasound treatment. Better desizing and relatively higher absorbency and whiteness in scouring and bleaching are realised under the influence of ultrasound and its use is especially advantageous in the Solomatic bleaching.

Introduction

The non-cellulosic constituents (impurities) of cotton fibers are commonly removed by scouring with a caustic soda solution at temperatures both near and above the boiling point of the scouring liquor. The alkaline scouring treatment emulsifies the waxes and breaks down pectin and proteins into water-soluble or water emulsifiable products, that are later washed off the cotton materials.

This treatment is very effective for almost complete removal of all the impurities associated with cellulose of cotton, except the colouring matter but this process is energy, chemical and water intensive and above all, the effluent is ecologically undesirable due to its high alkalinity, BOD and COD values.

In this context, these drawbacks encourage the alternatives and bioscouring of cotton products with suitable enzymes. The use of enzymes in the textile wet-treatments has already been established for a number of processes.

The enzyme amylase has been used for centuries to remove the starch size from the cotton fabrics. Cellulase enzyme is used to remove loose fibers from the surface of the cotton fabrics to impart a smooth and bright finish, commonly termed as 'biopolishing'.

The production of 'aged' denim jeans with cellulase enzyme with or without stone washing is increasingly practiced on about a billion pairs of jeans that are produced annually.

Removal of residual hydrogen peroxide after bleaching is now done by the enzyme oxidase and has greatly replaced use of sodium bisulphite. The increasing use of enzymes in the industry has also been facilitated because the effluent is relatively eco-friendly and its treatment is cost effective.

Nature and function of Enzymes

Enzymes are high molecular weight proteins that are produced by living organisms. These are composed of about 200 to 250 amino acids that catalyse (i.e. lower the activation energy) of many organic reactions without being consumed in the process. Enzymes activity can, however, be reduced or even completely destroyed (denatured) by high temperatures, extremes of pH and high concentration of electrolytes that destroy their 3-dimensional structures. Heavy metal ions and oxidizing and reducing agents also deactivate the enzymes. Certain enzymes, however, require bivalent metal cations as activators to stabilize the structure of the enzyme-substrate complex.

Enzymes or biocatalysts are very specific in their reactions and there is a different enzyme for each part of a series of reactions like those occurring in the vegetable and animal life processes. The specificity of function of enzymes is often compared with the lock and key system, but actually their functioning is much more complex than what this simple analogy might suggest. Enzymes themselves are biodegradable and are converted into harmless substances in the effluent.

Bioscouring with Enzymes

A great deal of work has been carried out during the last 15 years to study the effect of different enzymes for removing the non-cellulosic constituents from cotton, linen and lignocelluloses in the paper industry and their effects on the properties of the substrates.

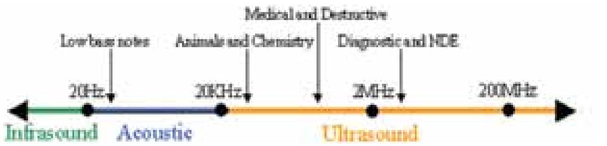
The most commercially successful enzyme in this respect is 'pectinase' and some studies on it are reviewed below.

Pectinase on Cellulose: Pectin appears to act as a matrix or cement, that stabilizes the cuticle and primary wall of the fibers. Digestion of the pectin by the pectinase enzyme loosens the matrix and then the unbound waxes and proteins become emulsifiable in a hot water scouring. The treatment improves absorbency and also whiteness of the cotton.[1, 2, 3]

During the usual treatment time of about 30 minutes or so, about 30% of the pectin is digested, but that is enough to destabilize its structure and to release the non-cellulosic products for removal by subsequent emulsification. Comparison of sorption and dye uptake of the pectinase-treated and the conventional alkali scoured ones indicates a slightly higher sorption of water by the former but there is no appreciable difference in the values of K/S.[3]

Use of Ultrasound in textiles

In order to improve efficiency and to reduce time of processing of the bioscouring and other enzyme induced procedures, simultaneous application of ultrasound has been suggested. Earlier work on use of enzymes and bioscouring and application of ultrasound in textile processing is briefly reviewed below.



Ultrasound Waves: Ultrasound is a cyclic sound pressure with a frequency greater than the upper limit of human hearing. The human hearing sound waves range between 16Hz to 16 KHz while the ultrasound waves range between 20 KHz to 500 MHz. Ultrasonic vibration travel in the form of a wave, similar to the way light travels. However, unlike the light waves that can travel in a vacuum, the ultrasound requires an elastic medium such as a liquid or a solid. The range of sound waves and their uses are briefly shown below.

Mechanism of ultrasonic energy: When a liquid, in which a gas is dissolved, is irradiated by the strong ultrasonic waves, many tiny bubbles appear. The bubbles repeatedly expand and contract according to the pressure oscillation of an ultrasonic wave, but some bubbles collapse violently at the contraction phase of an ultrasonic wave. On collapsing of the bubbles, energy is released and temperature and pressure around the bubbles increase to about 5000 K and 300 atmospheres in the respective order.[8]

Effect of Ultrasound Waves on the dyeing systems: Following changes appear to take place when a dyeing system is exposed to the ultrasonic energy:[9,10,13,14]

- Increase in the swelling of fibers in water.
- Increase in the diffusion coefficient of the dye molecules.
- Increase in the fibre/dye partition coefficient.
- Improvement in the movement of dye molecules to fibre surface.
- Disintegration of aggregates of the dye particles into smaller units or in the molecular forms.
- Decrease in the glass transition temperature of fibers.

Effect of Ultrasound on the Bioscouring Process: The cavitation phenomenon has the following significant effects on the bioprocessing applications: [11,12]

- Effect of cavitation is several hundred times greater in heterogeneous (e.g. all textile wet processing) than in homogeneous systems.
- In the aqueous media maximum effects of cavitation occur at 50°C, which is the almost the optimum temperature for many enzymatic bioprocessing applications.

Aims of the study

The commonly practiced scouring of cotton textiles involves highly alkaline chemicals, like caustic soda and sodium silicate. These chemicals remove the non-cellulosic impurities from the cotton, but cause a heavy loss of weight of the material. Furthermore, use of these chemicals result in high COD, BOD and TDS in the waste water (effluent). Recently a new enzymatic scouring process known as 'Bio-Scouring' has been introduced that makes the fibers well absorbent for subsequent bleaching and dyeing with a relatively lower loss in weight of the material. Bioscouring also reduces environmental burden by reducing BOD and COD values in the effluent and there is a substantial saving in the cost of the effluent treatment and total energy in the scouring process.

Materials and methods

Cotton Fabric:

$\frac{20 \times 20}{60 \times 58}$ 136 gsm

All the untreated and the treated materials were conditioned at 65% R.H. for 24 hours before weighing and conducting other experiments.

Sonicator: Frequency of the sonicator was maintained at 35 kHz and its volume was 28 liters.

Reagents: The desizing was carried out with Bactosol PHC. HC (liquid) of Clariant and Bioscouring was done with Scourozyme L of Novozymes.

Recipes and procedures for desizing, bioscouring and bleaching: The experimental work was carried out to compare the normal processing with that performed under the influence of the ultrasound energy for desizing, bioscouring and bleaching of the cotton material, both by the exhaust and the pad-batch methods. The recipes for these are given below.

Desizing in the Long Liquor or the Exhaust Process:

Bactosol PHC HC liq 0.3g/l
Hostapal UH (wetting agent) 1.0g/l
Sirrix 2UD (seq. agent) 0.5g/l
Liquor Ratio = 1:10
pH 5.5 and temperature 70°C.

Samples were treated for different times of 10, 15, 20, 25 and 30 minutes. This was followed by washing at 95°C for 5 minutes.

Desizing by the Pad-Batch Process:

Bactosol PHC HC (liquid) 0.5g/l
Hostapal UH (wetting agent) 3.0g/l
Sirrix 2UD (Seq. agent) 1.0g/l
pH 5.5 and Pick-up 90%

Samples were padded in the above liquor and treated for 15, 30, 45 and 60 minutes followed by washing at 95°C for 5 minutes.

Bioscouring

Scourzyme L (Pectinase Enyzyme) 0.5g/l
Hostapal UH (wetting agent) 3.0g/l
Sirrix 2UD (Sequestering agent) 1.0g/l
pH 9.0 and pick-up 90%

The material was treated with Scourzyme L and the wetting agent for 20 minutes at 55°C and then after adding the sequestering agent, temperature of the bath was raised to 85°C. After 10 minutes, the bath is dropped and the material is washed thrice with water at 95°C.

Bleaching

Hydrogen Peroxide (35%) 7.0%
Sirrix Antox (seq. agent) 1.0%
NaOH(solid) 2.0 g/l
Hostapal UH (wetting agent) 1.0%
Stabilizer Sifa 1.0%
pH 9.0 and liquor pick up 90%.

The scoured fabric was treated at 95°C for 45minutes as per the above recipe and then washed at boil and rinsed with cold water.

Bioscouring and Peroxide Bleaching

Scourzyme L 6.0 g/l
H_2O_2 (35%) 7.0 g/l
NaOH(solid) 2.0 g/l
Hostapal UH (wetting agent) 1.0%
Stabilizer Sifa 1.0%
Sirrix Antox (seq. agent) 1.0%

The desized fabric was treated with Scourzyme L and Hostapal UH at 55°C for 15minutes followed by addition of Sirrix Antox, H_2O_2, NaOH and peroxide stabilizer. Temperature was then raised to 90°C and the material treated for 30 minutes at this temperature. The bleaching treatment was followed by washing with water at 95°C.

Results of the experiments

Desizing by the Exhaust Process: The desizing by exhaust process with and without ultrasonic energy is explained in table 1:

Table 1

Desizing Time (minutes)	Without Ultrasonic Energy				With Ultrasonic Energy			
	Wt. b/f desizing	wt. a/f desizing (gm)	wt. loss (%)	Tegewa Value	wt. b/f desizing (gm)	wt. a/f desizing (gm)	wt. loss (%)	Tegewa Value
10		4.17	5.86	2	5.85	5.47	6.40	3
15	5.85	5.48	6.30	3-4	6.60	6.23	6.70	4-5
20	5.70	5.33	6.50	4	6.58	6.13	6.80	6
25	6.52	6.09	6.60	5	4.27	3.97	7.00	7
30	5.50	5.33	7.3	6-7	6.52	6.00	7.9	8-9

Desizing by the Pad-Batch Process: The desizing by Pad-Batch process is explained in table 2:

Table 2

Desizing Time (minutes)	without Ultrasonic Energy				with Ultrasonic Energy			
	Wt. b/f desizing	wt. a/f desizing (gm)	wt. loss (%)	Tegewa Value	wt. b/f desizing (gm)	wt. a/f desizing (gm)	wt. loss (%)	Tegewa Value
15	4.26	4.02	5.63	2	4.39	4.12	6.15	3
30	2.75	2.58	6.1	3	5.70	5.32	6.6	4-5
45	6.52	6.09	6.6	4	6.65	6.19	7.0	6
60	5.50	5.09	7.4	5	5.7	5.26	7.7	6.5

Bio-Scouring: Table 3 shows bio scouring with and without ultrasound energy.

Table 3

Without Ultrasonic Energy				With Ultrasonic Energy			
Weight loss			Capillary (cm)	Weight loss			Capillary (cm)
wt. of desized fabric	wt. of scoured fabric	% wt. loss		wt. of desized fabric	wt. of scoured fabric	% wt. loss	
5.80	5.60	3.45	4.0	5.67	5.45	3.8	4.5
5.67	5.47	3.52	4.5	6.30	6.06	3.8	4.5
5.80	5.60	3.45	4.0	5.85	5.62	3.9	4.7

Peroxide Bleaching of Scoured Fabric: Table 4 shows peroxide bleaching of scoured fabric with and without ultrasound energy.

Table 4									
Without Ultrasonic Energy					With Ultrasonic Energy				
Weight loss			Capillary (cm)	White-ness	Weight loss			Capillary (cm)	White -ness
wt. of scoured fabric	wt. of bleached fabric	% wt. loss			wt. of scoured fabric	wt. of bleached fabric	% wt. loss		
5.5	5.45	0.9	4.5	61	6.0	5.93	1.2	5.5	72
5.05	5.0	1.0	4.5	59	5.10	5.03	1.3	5.5	71
5.10	5.04	1.1	4.5	60	5.05	4.99	1.4	5.5	70

Combined Bioscouring and Peroxide Bleaching: Table 5 shows combined bioscouring and peroxide bleaching with and without ultrasound energy.

Table 5									
Without Ultrasonic Energy					With Ultrasonic Energy				
Weight loss			Capillary (cm)	White -ness	Weight loss			Capillary (cm)	White -ness
wt. of scoured fabric	wt. of bleached fabric	% wt. loss			wt. of scoured fabric	wt. of bleached fabric	% wt. loss		
5.5	5.45	0.9	4.5	61	6.0	5.93	1.2	5.5	72
5.05	5.0	1.0	4.5	59	5.10	5.03	1.3	5.5	71
5.10	5.04	1.1	4.5	60	5.05	4.99	1.4	5.5	70

Tenacity of the Treated Fabrics: Trials were also carried out to compare tenacities of the fabrics processed with and without ultrasound to determine any excessive damage caused on exposure to the ultrasound energy. The results are tabulated below. These tests were carried out as per ASTMD 2261.

Table 6							
Parameter	Greige fabric	Desized fabric		Bio- scoured fabric		Solomatic Bleached fabric	
		Without US	With US	Without US	With US	Without US	With US
Tear strength (Kg)	3.59	3.05	2.75	1.84	1.68	1.61	1.58

Discussion of the results

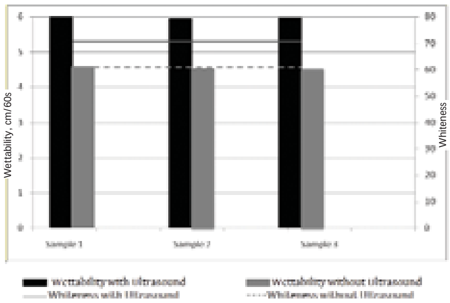
Exhaust Desizing with and without Ultrasound: Comparing the results of desizing by the exhaust method with and without ultrasound, there is a negligible difference in the weight-loss, but a considerable increase in the Tegewa values (the desizing measure) of the ultrasound-exposed fabric. This indicates that starch has been more completely hydrolysed on the ultrasound exposure but the broken down particles have not been completely removed from the fabric during the subsequent washing off stage.

Desizing by the Pad-Batch Process: On comparing desizing values after 30 minutes treatment with the exhaust and 60 minutes treatment with the pad-batch processes, it is observed that loss in weight is almost the same both with and without the ultrasound treatments. However, the Tegewa values in the pad-batch method are significantly lower than those obtained with the exhaust process. This indicates that contact of the desizing enzymes with starch on the fabric is slower due to the material being in the padded state. However, the Tegewa value is relatively higher with ultrasound due to the higher kinetic energy imparted to the enzymes molecules.

Bioscouring: In this study, both the loss in weight and wettability increase on exposure to the ultrasound waves but these increases are not significantly high and there appears to be not much advantage in using ultrasound. These results, however, appear to be in conflict with some previous studies15 and need further investigation.

Peroxide Bleaching of Scoured Fabrics: There is a very small loss in weight with hydrogen peroxide bleaching of the scoured material but still it is 30% more with the ultrasound treatment. The wettability and whiteness of the fabric bleached with ultrasound are 22% and 18% higher respectively over the normal bleached fabric and these are important advantages that may lead to economy in the use of chemicals.

Combined Bioscouring and Peroxide Bleaching: The results are graphically shown below for a ready reference. From the above figure and the tabulated results in section 4.5, it can be observed that both the whiteness and absorbency are improved by processing with the ultrasound and their values increase by 18% and 22% respectively over the normal method. However, there is a greater loss (30%) in the weight of the material due the more energetic ultrasound treatment.



Tenacities of Desized, Bioscoured and Solomatic-Bleached Fabrics with and without Ultrasound: Some interesting results were found in this study to show the effect of ultrasound on the tenacities of fabrics at different stages of processing. Firstly there was a loss of strength of about 15% on desizing even on desizing without ultrasound and this can be attributed to the binding effect of starch on the fibres in the sized yarn of the fabric. However, desizing with ultrasound caused a further reduction in tenacity by 9.5%. In bioscouring, the loss in tenacity is 7% more on exposure to the ultrasound energy but surprisingly there is almost no loss in strength (only 2%) in the Solomatic bleaching with ultrasound as compared with the normal process. This is a significant finding because large quantities of the cotton fabrics are Solomatically bleached these days and treatment with ultrasound treatment gives important advantages of better whiteness and higher absorbency.

Conclusions: In the work presented above, efforts have been made to have an overall view of pretreatment processes for cotton textiles with or without the ultrasound energy. It has been learnt that desizing by the exhaust process with the ultrasound gives a 30% higher Tegewa value or the desizing efficiency and this is an important technical advantage. However, desizing by the pad-batch method under influence of ultrasound does not provide such a great advantage. Similarly, there appears to be no gain on bioscouring with ultrasound but this result needs to be checked further. Peroxide bleaching of the desized and scoured fabric with ultrasound has definite advantages of higher whiteness and absorbency but is accompanied by 18% greater loss in weight. Solomatic or combined scouring and peroxide bleaching process with ultrasound is promising because it improves both whiteness and absorbency of cotton without any appreciable loss in strength although there is a small (only about 0.1%) increase in loss in weight of the material as compared with the normal method.

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