Optimization of pH for Combined Bio Scouring and Bleaching with Pectinase Enzyme and Peracetic Acid

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***Abstract*—** Bioprocessing resulted in a substantial reduction in energy, water, chemicals, time, and cost requirements. This study investigates the effect of pH on the combined bioscouring and bleaching of cotton fabric using pectinase enzyme and peracetic acid. We evaluated many quality characteristics, including absorbency, whiteness index, process loss, bursting strength, and dye uptake. The results demonstrated that a pH of 7.1 is more effective in the combined bioscouring-bleaching of cotton fabrics utilizing pectinase enzyme and peracetic acid. Additional research on the improvement of cellulase effectiveness in conjunction with bio-scouring and bleaching is required.

Keywords—Pectinase Enzyme; Peracetic Acid; Bio scouring; Bleaching; Absorbency; Whiteness Index; Process Loss; Bursting Strength.

1. Introduction

Cotton is one of the most popular natural cellulosic fibers used in the textile industry. It is primarily composed of cellulose, although proteins, pectin's, waxes, ash, and minerals can be found as impurities in the cuticle and other parts of the cotton fiber [1,2], [3], [4]. The presence of these contaminants in raw cotton fibers gives them hydrophobic characteristics. Because of this hydrophobic barrier, dyes and auxiliaries are difficult to absorb and permeate into the fibers [1,2,5], [6]. In textile processing, cotton fabrics require pre-treatment before dying, which includes scouring and bleaching etc.[1,2]. Scouring is the process of eliminating natural impurities and increasing the wettability of cotton fabrics. Cotton is traditionally scoured with a boiling solution of sodium hydroxide. This technique produces a lot of effluent, requires a lot of energy, and does not meet the sustainable development goals of the textile industry [1,2], [6 7], [8], [9]. Scouring with biodegradable enzymes has proven to be a sustainable and well-accepted alternative in the textile industry [1], [7], [10]. Scouring using pectinases alone or in combination with proteases, lipases, cutinases, and cellulases has been used under smooth reaction conditions with little cellulose deterioration [1], [7]. Pectinase is a large, negatively charged, acidic glycosidic macromolecule that breaks down complex polysaccharides in plant tissues [11]. Pectin esterases, polygalacturonases, and pectin lyases are the three major enzymes utilized to break down pectin compounds. After breaking down and removing pectin, which acts as a natural binder to link non-cellulose components to the fiber cellulose core, other non-cellulose substances can be removed from cotton using surfactants and mechanical means [4,12]. Bleaching is the next stage following scouring in textile processing. It is the process of eliminating the natural color from the grey fabric. Cellulosic fibers are often bleached with hydrogen peroxide, which is a hazardous chemical, produces lot of effluents. Instead of hydrogen peroxide, peracetic acid can be an alternative bleaching agent for sustainable development of the textile industry [8], [13], [14]. Peracetic acid (PAA) is a strong oxidizing agent with excellent antimicrobial and bleaching properties [15], [16]. Previously, it was produced in situ using acetic acid anhydride and hydrogen peroxide. However, the risk of explosion during the synthesis step hindered the confirmation of peracetic acid as a bleaching agent in industry. [17]. In recent years, PAA has been synthesized by combining acetic acid and hydrogen peroxide with an acid catalyst (Eqn 1). At higher temperatures and pH, PAA spontaneously decomposes into acetic acid and oxygen (Eqn 2). Heavy metal ions can also induce decomposition, particularly when single-electron transitions are possible (Eq 3). Eqn 4 depicts the reaction that occurs when PAA is used for bleaching. The bleaching activity of PAA is mostly due to the epoxidation of double bonds seen in undesirable-colored compounds [18].

$$CH_3COOOH + H_2O \rightleftharpoons CH_3COOH + H_2O_2 \qquad (1)$$

$$2\,CH_3COOOH \xrightarrow{T > 40\,^{\circ}C,\ pH > 5} 2\,CH_3COOH + O_2 \qquad (2)$$

$$CH_3COOOH \xrightarrow{M^{x+}} CH_3COOH + O_2 + \text{other products} \qquad (3)$$

$$CH_3COOOH + \text{impurities} \longrightarrow CH_3COOH + \text{oxidised impurities} \qquad (4)$$

Figure 1: Mechanism of bleaching with peracetic acid

Table 1: Chemicals Used in this Experiment

	Chemicals Name	Supplier Name & Country	Amount
Chemicals	Conzyme SPA-5	SUNSON, China.	0.8 %
	Per acetic Acid (CH_3COOOH)	Department of ACCE, RU	6 g/l
	Felosan NOF	CHT. R. BEITLICH, Germany	1 g/l
	Soda Ash (Na_2CO_3)	TATA Chemicals Limited, India	1.0 g/l, 1.5 g/l, 2.5 g/l and 3 g/l

Several research utilized pectinase enzyme and peracetic acid for scouring and bleaching of cellulosic fabric in a separate bath. Pusic et al. demonstrated that the results of bio scouring using acid and neutral pectinases were far superior to those of alkali scouring. Thus, the intended outcome should be considered when choosing the application processes. Since there was no need to neutralize the wastewater, using neutral pectinase was advantageous from an ecological perspective [19]. Nina et al. shown the potent bleaching performance of the new bio bleaching system using enzymatically obtained acetic acid at mild process conditions, such as a neutral pH and a temperature of 65°C [20]. Some researchers were studied combined scouring-bleaching of cotton fabric with pectinase and peracetic acid [4,8,12,15]. In combined scouring-bleaching, pH is important criteria for the activation of the pectinase enzyme and peracetic acid. There was no research work carried out on the effect of pH on combined scouring-bleaching of cotton fabric. This study focused on the effect of pH on combined bio scouring bleaching of cotton fabric in an industrial scale. Water absorbency, whiteness index, process loss, bursting strength and dye uptake were considered as quality parameters of the treated fabrics.

2. Materials & Methods

The samples selected for this investigation are 100% cotton single jersey fabric with a GSM of 160 g/m^2. The chemicals that were used in this experiment are mentioned in table I.

3. Testing Methods

A. *Absorbency*

The water absorbency was measured according to DIN 53 924 (the velocity of the soaking water for textile fabrics, the method for determining the rising height) [7,8].

B. *Whiteness Index*

The scoured-bleached fabric samples were tested in a spectrophotometer (datacolor 650, USA) for determining the whiteness index in D_{65} illuminant and 10° observer setting [21], [22].

C. *Process Loss%*

Fabric process loss was calculated based on dry weight using the following equation [1],[10], [21].

$$\text{Process Loss\%} = \frac{W1 - W2}{W1} * 100$$

where W_1 and W_2 are the dry weights of the fabric before and after scouring-bleaching treatment, respectively.

D. *Bursting Strength*

Bursting strength test was carried out using a standard hydraulic diaphragm-type bursting strength tester according to the standard ASTM D 3786 [23].

E. *Dye uptake%*

Dye uptake% of the scoured-bleached fabric samples were determined according to the following equation.

$$\text{Dye Uptake \%} = = \frac{W1 - W2}{W1} * 100$$

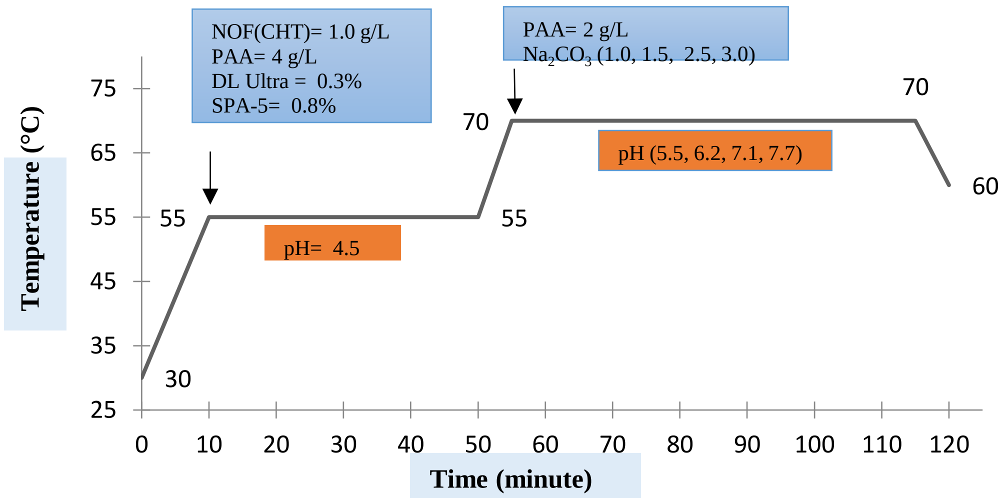

Figure 2: Working Process Curve

Table 2: Absorbency, Whiteness and Process Loss Test Results

Machine No.		M/c 01	M/c 02	M/c 03	M/c 04	M/c 05	Average Results
Machine Capacity (kg)		**10 kg**	**20 kg**	**50 kg**	**100 kg**	**300 kg**	
Absorbency (mm)	Na_2CO_3 = 1.0 g/l (pH = 5.5)	39	39	38	37	38	38.0
	Na_2CO_3 = 1.5 g/l (pH = 6.2)	40	39	38	38	39	38.8
	Na_2CO_3 = 2.5 g/l (pH = 7.1)	40	40	41	39	40	40.0
	Na_2CO_3 = 3.0 g/l (pH = 7.7)	41	40	41	41	40	40.6
Whiteness Value (CIE)	Na_2CO_3 = 1.0 g/l (pH = 5.5)	53	52	53	51	51	52
	Na_2CO_3 = 1.5 g/l (pH = 6.2)	58	56	57	56	58	57
	Na_2CO_3 = 2.5 g/l (pH = 7.1)	65	64	65	64	65	64.6
	Na_2CO_3 = 3.0 g/l (pH = 7.7)	65	65	64	65	65	64.8
Process loss (%)	Na_2CO_3 = 1.0 g/l (pH = 5.5)	8.2	8.5	8.6	8.6	8.0	8.38
	Na_2CO_3 = 1.5 g/l (pH = 6.2)	8.8	8.6	8.3	8.8	8.2	8.54
	Na_2CO_3 = 2.5 g/l (pH = 7.1)	8.5	9.0	8.9	9.0	8.7	8.82
	Na_2CO_3 = 3.0 g/l (pH = 7.7)	8.9	9.1	9.1	9.0	9.0	9.02

where W_1 is the wet weights of the fabric after scouring-bleaching treatment and W_2 is the dry weights of the fabric after scouring-bleaching treatment respectively.

Table 3: Bursting Strength and Dye Uptake Test Results

Machine No.		M/c 01	M/c 02	M/c 03	M/c 04	M/c 05	Average Results
Machine Capacity (kg)		**10 kg**	**20 kg**	**50 kg**	**100 kg**	**300 kg**	
Bursting strength (KPa)	Na_2CO_3 = 1.0 g/l (pH = 5.5)	438	436	439	440	439	438.4
	Na_2CO_3 = 1.5 g/l (pH = 6.2)	436	435	435	436	437	435.8
	Na_2CO_3 = 2.5 g/l (pH = 7.1)	436	434	435	436	437	435.6
	Na_2CO_3 = 3.0 g/l (pH = 7.7)	434	432	432	431	432	432.2
Dyes Uptake Rate (%)	Na_2CO_3 = 1.0 g/l (pH = 5.5)	106%	105	104	106	105	105.20
	Na_2CO_3 = 1.5 g/l (pH = 6.2)	103	104	102.5	103.9	103.8	103.44
	Na_2CO_3 = 2.5 g/l (pH = 7.1)	103	103	104	104.08	104	103.61
	Na_2CO_3 = 3.0 g/l (pH = 7.7)	103.2	104	103	102	103	103.04

4. Working Procedure:

At first, we weighed the fabric and added the required amount of water and fabric into the bath. Then, run the machine and increase the temperature up to 55 °C. Calculated amounts of enzymes and auxiliaries were added to the machine. Initial pH 4.5 of the bath was maintained by adding 4 g/L of peracetic acid and running the machine for 40 minutes. Sodium carbonate (1.0, 1.5, 2.5, and 3.0 g/L) was added together with 2.0 g/L of peracetic acid. The temperature was then increased to 70 °C, and the machine was run for 60 minutes. Then, decrease the temperature and drain the bath. After that, hot washing was done to remove excess chemicals from the treated fabrics. Each experiment was scaled up from a 10 kg machine to a 300 kg machine, and the average result was taken into consideration.

5. Results & Discussions:

A. *Effect of pH on Absorbency*

The ability of a fabric to retain liquids within its structure and absorb moisture affects a variety of properties, including shrinkage, water repellency, wrinkle recovery, and skin comfort. Since increasing the textile materials' absorbency is the primary goal of scouring, the scoured materials' absorbency should be assessed. The length of the sample portion that absorbs a specific amount of water or solution in a specific time is the basis for the measurement. Better printing options will arise from the higher absorbency. In table 2, the average absorbency test results showed that the fabric was able to absorb more liquids as the concentration of Na_2CO_3 increased. The elimination of non-cellulosic particles by high concentration Na_2CO_3 might have resulted in an increase in absorbency. Although, absorbency values varied between 38 and 40.6 mm, which is the perfect range for dyeing. However, a slightly basic environment is found to be best for high absorbency according to the data.

B. *Effect of pH on Fabric Whiteness*

A surface is considered white when it most closely resembles the characteristics of an ideal reflecting diffuser, which is a surface that evenly reflects light in all directions instead of absorbing or transmitting it. Whiteness is an important parameter for assessing the quality of the bleached fabrics. Table 2 revealed that the higher concertation of Na_2CO_3 raised the whiteness value. When Na_2CO_3 was present at concentrations of 1.5 g/L to 2.0 g/L, the whiteness value increased from CIE 52 to CIE 57.

However, as the concentration increased further, the whiteness value became stable at around CIE 65.

C. *Effect of pH on Process Loss*

The quantity of fabric lost during processing, such as desizing, scouring, bleaching, etc., is known as process loss. It is a crucial criterion for evaluating the scrubbed fabrics' quality. It is a general fact that with the increase in bleaching activity there should be more process loss. Previous research on the weight loss of knitted materials that were both conventionally scoured and bleached revealed that weight loss increased with increasing H_2O_2 concentration[24]. PAA forms active oxygen at pH values greater than 5, which functions as a bleaching agent. The following circumstances maximize the effectiveness of PAA as a bleaching agent for cotton: pH 6–7, 50–80 °C bleaching temperature, and 20–60 min bleaching time, depending on temperature[25]. In this study, it was observed that the weight loss rose progressively as the pH increased over the range investigated (Table 2) which supports previous research data. The process loss of the fabric ranged between 8.38-9.02%. It's also noteworthy that the 300 kg cloth sample lost less weight in an acidic medium than other samples with lower weights.

D. *Effect of pH on Bursting Strength*

The ability of a fabric to withstand bursting or rupture under pressure or force is known as its bursting strength. It serves as an indicator of the fabric's resilience to damage brought on by abruptly high localized pressure. It is a crucial factor in assessing the quality of fabrics. A previous study on a 9 gm (±2%) polyester rayon blend fabric sample, which was conventionally scoured and bleached, showed steady bursting strength of around 109 kPa at pH 5, 6 7. This focuses on the hypothesis that the mildly basic, neutral, and mildly acidic medium may not have a high impact on bursting strength. The data in Table 3 reveals a slight decrease in bursting strength as pH increased. No significant change was observed for bursting strength when different weights of the samples were compared at a certain concentration of Na_2CO_3.

E. *Effect of pH on Dye Uptake*

The quantity of dyes, expressed as a percentage of weight, that are absorbed by the fiber, yarn, or fabric during the application process is known as dye uptake. It is a crucial factor in assessing the caliber of fabrics that have been scourged and bleached. The color strength value on the fabric is observed to increase with the pH of the dye bath, and at pH 9, the dye uptake value is higher than at neutral and acidic pHs[26]. Although, the increased reaction between dye and cotton fabric in mild alkaline solution is the reason for this better uptake of dyes, more basic condition might show the opposite result. At high pH, cellulose tends to carry more hydroxyl groups which lead to an increase in negative repulsion between fabric and dye[26]. As the concentration of Na_2CO_3 rose, Table 3 showed a small decrease in the dye uptake rate. A high dye pick-up rate of 105.2% was observed at 1.0 g/L Na_2CO_3 concentration. At the remaining concentrations No statistically, significant difference was found when comparing dye uptake of different weights of the samples at a specific concentration of Na_2CO_3.

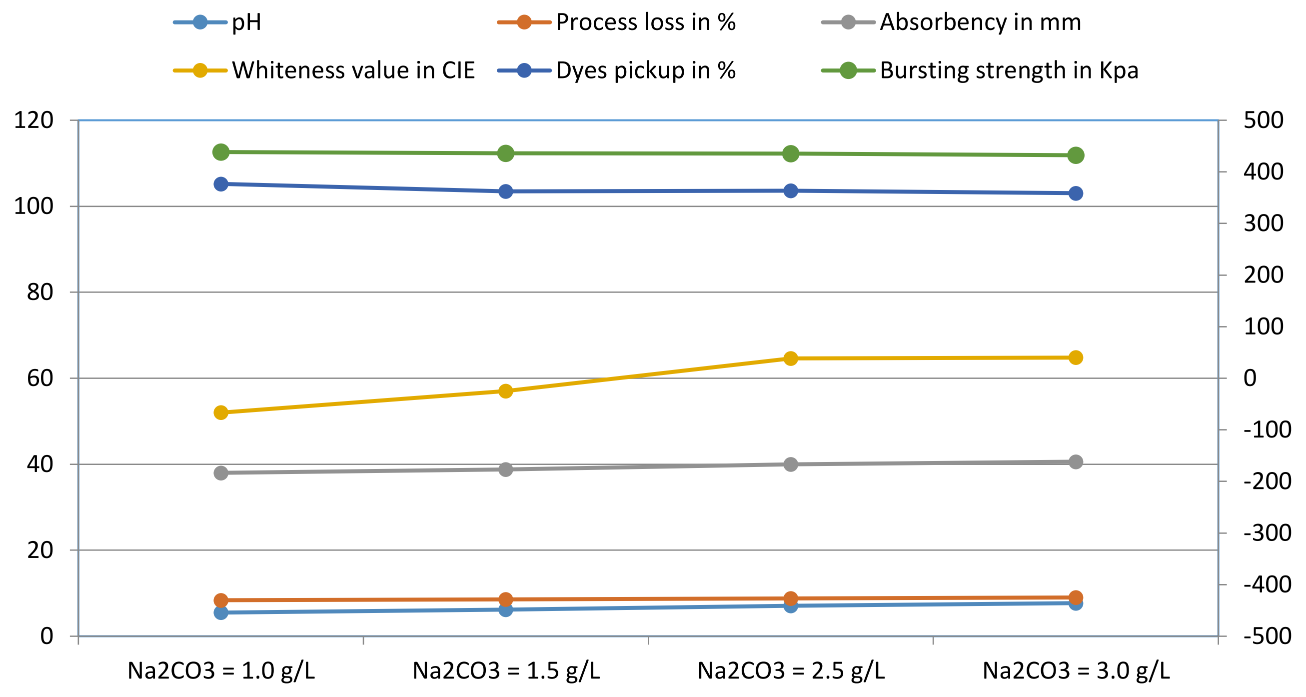

Figure 3: Absorbency, whiteness, process loss, pH, dyes uptake and bursting test according to Na_2CO_3 concentration.

6. Conclusion:

Several factors influence the combined bioscouring-bleaching of cotton fabric. The pH is a crucial factor in the combined bioscouring-bleaching process of cotton fabric using pectinase enzyme and peracetic acid. To maintain pH, Na2CO3 is employed, and experimental results show that a concentration of 2.5 g/L is suitable for bleaching following bio scouring. In the case of absorbency and whiteness, an increase in the values was observed with the increase of Na2CO3 concentration. However, the difference between the average values at pH 7.1 and 7.7 was not significant. Similarly, dye uptake of selected samples was almost the same for pH 6.2, 7.1, and 7.7. Considering process loss and bursting strength tests reveal a gradual increase and decrease in values, respectively, considering the whole analysis, pH 7.1 seemed to be the best condition for bioscouring and bleaching with PAA. Further investigation into the optimization of the cellulase activity combined bio-scouring-bleaching should be conducted.

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