

Research Article

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Study on the Comparison of the Effects of pH Buffer in Single Stage Preparatory Process with that of Conventional for CVC Woven Fabric

Halim AFMF^{1*} and Zhou W²

¹Department of Textile Engineering, World University of Bangladesh, Bangladesh ²School of Materials and Textile Engineering, Zhejiang Sci-Tech University, China

Abstract

To minimize water and energy consumption, it has become necessary to combine several textile processing stages in order to reduce the number of operations, process time and cost. In this project work an attempt was made to combine the pre-treatment process with optimal quantities of chemicals in single bath by varying different pH with the help of different buffers and comparison was made with conventional process. It has found that buffered system at pH 11 for 60 min shows the best result in respect to wicking test, drop test, immersion test, weight loss%, whiteness index, yellowness index. Then all the pre-treated samples were dyed with reactive dyes by varying the depth of shade. After dyeing it has been found that buffered system at pH 11 for 60 min can be selected for any depth of shades and buffered system at pH 11 for 45 min is suitable for dark shade. This kind of a one-bath process is shorter, consuming less energy and hence being less expensive. The combined processes also lead to substantial saving in water energy and time along with chemicals.

Keywords: Buffer; Single bath dyeing; pH; DSB process; Conventional dyeing

Introduction

Pre-treatment is an important sequence in chemical processing of cotton fabric and comprises of several operations:

- a) Designing, i.e., removal of sizing materials
- b) Scouring to remove fats and waxes
- c) Oxidative treatment for destroying natural coloring matter.

A good pre-treatment is always essential for successful production of dyeing, printing, and finishing of fabrics. In the conventional preparation, the designing, scouring and bleaching processes are carried out separately at high temperatures, requiring the use of large amount of thermal energy. Now-a-days all efforts in the field of pre-treatment processes of textiles are directed towards shortening and simplification of the treatment. In order to minimize energy consumption it has become necessary to combine several pre-treatment stages by reducing number of operations or by shortening the reaction time. The conventional threestage designing, scouring and bleaching operations are exceptionally intensive in water, energy and chemicals which ultimately influence the process cost. At the same time, limited availability of water is also a challenge. It is clear that the pre-treatment process, i.e., designing, scouring and bleaching, involves more water usage [1-6]. Also, about 55 to 60% energy consumed in the textile industry is used in various pre-treatment stages. The high consumption of water and energy for textile pre-treatment sector emphasizes the urge for improvement of process, i.e., to adopt cost effective practice to reduce the use of scarce resources like water and energy. Hence, the logical approach to combine designing, scouring and bleaching operations to save energy and water was put forward by many researchers. In this work, we take further steps to challenge the conventional preparatory and the dyeing processes. The aim of the work was to ensure efficient reduction in water consumption, time and thermal energy consumption in the pre-treatment sector. In our work we tried to overcome the conventional pre-treatment process which is carried out at several bath processes. To fulfil the objective, an unprocessed grey fabric was subjected to combined pre-treatment process which was carried out using all the conventional chemicals in one bath. We carried out this at different pH. We used some buffers to adjust the pH [7-9]. After that several test for pre-treatment was carried out and further we dyed the fabric to check the dyeing efficiency of a grey fabric after various steps of pre-treatment. Thus we compare the one bath process with the convention one. We also check the pH stability in the bath and the effect of buffered and unbuffered system on the pre-treatment process. We also should notice that this can tremendously save water, chemicals and energy required for processing a fabric for making it ready for dyeing. Thus, efforts were made towards not only reducing the water and minimize the intermediate washings but also to minimize the chemicals wherever possible to make existing process, more sustainable and environment friendly [10-13].

Materials

Following materials are used (Table 1).

Chemicals used

The following chemicals are used in the pre-treatment (Tables 2-4).

Methodology

The process sequence is well explained in the following flowchart (Figure 1).

Test on pre-treated sample

Weight loss (%): With a considerable removal of impurities during pre-treatment, weight loss (%) gives an indication of the efficiency. Loss is fabric weight after pre-treatment indicates the removal of impurities.

*Corresponding author: Halim AFMF, Lecturer, Department of Textile Engineering, World University of Bangladesh, Bangladesh, Tel: 013065738219; E-mail: Fahadrabby314@gmail.com

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Fabrics composition	Fabrics type	Fabric construction	warp count	Weft coun	t EPI	PPI	GSM
CVC Woven Fabric	Sized Fabric	Plain (1 × 1)	36	29	56	44	107
Table 1: Material used.							
Chemical Name		Function		Form		Manufacture	!
sodium carbonate (Na ₂ CO ₃)		Used as buffer		Granular	Merck Speci	Merck Specialities Private Limited. India	
Sodium bicarbonate (NaHCO ₃)		Used as buffer		Granular	Merck Speci	Merck Specialities Private Limited. India	
Sodium hydrogen phosphate (NaHP	O ₄)	Used as buffer		Granular	Merck Speci	alities Private	Limited. India
Borax (Na ₂ B ₄ O ₇ .10H ₂ O)		Used as buffer		Granular	Merck Speci	Merck Specialities Private Limited. India	
sodium Hydroxide (NaOH)	Used as sco oil into wate	Used as scouring agent. The function of alkali is to convert the oil into water soluble fatty acid and soap. Alkali carries out the saponification reaction		Flakes	Loba Chen	Loba Chemie Pvt. Ltd Mumbai, India	
Hydrogen peroxide (H_2O_2)	Used as bleach	Used as bleaching agent. The perhydroxy ions (HO ₂) formed during reaction are the actual bleaching agents.		Liquid	Samuda S	bec Chem Lim	iited. Dhaka
Sodium silicate(Na ₂ SiO ₃)	Used as perc	Used as peroxide stabilizer which control the breakdown of H ₂ O ₂		Granular	Loba Chen	nie Pvt. Ltd Mu	umbai, India
Wetting agent (Samwet BLF)	It reduces	s the surface tension between	water and fabrics	Viscous liqui	d Samuda s	Samuda spec Chem Limited. Dhaka	
Sequestering agent (Nanoquest CI	B) l	t is used to remove the water h	hardness	Liquid		Nano Chem	
Detergent		It is used to cleaning the sub	ostrate	Viscous liqui	Samuda Spec Chem Limited. Dhal		ited. Dhaka
Acetic Acid		Used for neutralization		Liquid	Samuda Spec Chem Limited. Dha		ited. Dhaka
Enzyme (Rucolase HCH)	Desizing age then more sl	Desizing agent. It breakdown the starch into soluble dextrin and then more slowly convert this to reucing substances and sugar, such as maltose		Liquid	Spectra Dy	e Chem (Pvt) Pabna	Ltd Paksey,
		Table 2: Chemicals use	ed for pre-treatment.				
Chemical Name	Function Form Manufa		anufacure				

Chemical Name	Function	Form	Manufacure
Glauber's salt (Na ₂ SO ₄ .10H ₂ O)	Used as electrolytes. It increases exhaustion rate of the dying process.	Granular	Merck Specialities Private Limited Worli, Mumbai, India
Sodium carbonate (Na ₂ CO ₃)	It creates proper pH in dye bath and performs as the dye-fixing agent.	Granular	Merck Specialities Private Limited Worli, Mumbai, India
Sodium hydroxide (NaOH)	It creates proper pH in dye bath and performs as the dye-fixing agent.	Granular	Loba Chemie Pvt. Ltd, Mumbai, India
Wetting agent (Samwet BLF)	It reduces the surface tension between water and fabric.	Viscous liquid	Samuda Spec Chem Limited, Dhaka
Sequestering agent (Nanoquest CIB)	It is used to remove water hardness.	Liquid	Nano Chem

Table 3: Ch	nemicals	used	for read	ctive	dyeing.
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Particulars	Specification
Name	Corazol Red 3BSN
Туре	Bifunctional (VS and MCT based reactive group)
Fixation Temperature	60°C
Reactivity	Medium
Company	Imperial Allied Chemicals LTD
Origin	China

Table 4: Specification of Reactive Dye.

The weight of fabrics is taken separately and after pre-treatment the weight loss is expressed as a percentage.

 $Weight Loss = \frac{Weight before pretreatment - Weight after pretreatment}{Weight before pretreatment} \times 100\%$

Tegewa test: Preparation of tegewa solution: For making this solution potassium iodides 10 g of KI (100%) in 100 mL of water is added with 0.6358 g of iodine (100%) and shake this well to dissolve iodine crystals. Then make it up to 800 mL using distilled water and finally make it up to 1000 mL with ethanol. To test the presence of sizing material i.e., starch based sizing material in the fabric iodine test is conducted. In which the fabric is spotted or treated with Iodine solution-Iodine dissolved in an aqueous solution of potassium iodide, which reacts with the starch and produce a bluish-purple-black color. If there is no starch the color may be brownish-yellowish. [14,15] (Figure 2).

Drop test (AATCC test method TS-018): For the drop test, the sample is placed over the top of a beaker so that the centre is unsupported. The time starts from the moment a drop of water touches the surface of the sample to the moment the water drop is completely

absorbed by the sample. This is taken by a stopwatch; the longest will be the time, the lower the absorbency. The drop test is carried out using a 0.1% direct dye solution in order to make the drop more visible. In addition to the time taken for the liquid to be absorbed by the fabric sample, the scouring efficiency is also evaluated by assessing the evenness of scouring from the shape of the absorbed area on the fabric. This gives an indication of the behaviour of the prepared sample towards the absorption of chemical solution is subsequent stages of processing. Standard time for absorption of the drop should be 0.5-0.8 second. Although less than 1 second is acceptable [16].

Assessment

- If the drop is noncircular and small then the fabric is scoured uneven and incomplete.
- If the drop is circular and small then the fabric is even but incomplete scoured.
- If the drop is circular and big then the fabric is complete and evenly scoured [17].

Immersion test: A sample of 1 cm \times 1 cm dimensions is cut. A distilled water solution of 500 mL in a beaker is taken. The cut fabric sample is dropped into the solution. Right when the sample touches the solution surface, the immersion time is immediately started recording until the sample reached to the bottom surface of the solution. Standard immersion time should be 5 seconds. Maximum limit is up to 10 seconds for acceptance [18].

Wicking/column test: A fabric sample is cut to the dimensions of 18 cm \times 5 cm. for carried out this test, 0.1% direct dye solution is

The process sequence of our task is given below Sized Fabric Pretreatment DSB process Conventional process Unbuffered system Buffered system pH 9 pH 10 pH 11 Testing & Dyeing me on treated samples Tests done on Dyeing treated samples 0.5% Shade (Red) Wicking WI & YI 1.5% Shade (Red) Weight loss% Test done on Wetting Strengtl loss% time dyed samples K/S value *DSB-Desizing, Scouring & Bleaching, Figure 1: Methodology flowchart.



prepared and taken in a beaker. A mark is drawn 1 cm above from the bottom of the sample. This sample is hung from a support by immersing the 1 cm portion of the fabric only in the dye solution, making sure the support doesn't move. Once immersed, 5 minute duration is given, after which the point up to which the colored solution has been absorbed is measured. This is known as the wicking height.

Tensile strength

Principle: A fabric test specimen of specified dimensions is extended at a constant rate until it ruptures. The maximum force and the elongation at maximum force and, if required, the force at rupture and the elongation at rupture are recorded. Grab and Strip method are followed [19,20].

Procedure: The samples are individually supported as defined in the standard by clamping of the specimen, Slack Mountain. The application is to pull to the pre tensioning force, then the test is started at a continuous increasing extension (constant rate of Extension) applying a force longitudinally to a test piece of a 20 cm in length and 5 cm in width until the specimen fails. The result is taken from the machine (Figure 3).

Measurement of whiteness index, yellowness index by spectrophotometer: This type of instruments measures the reflectance spectra of the sample, rather than analyze the incoming light in

Pretension - 2.00N	Jaw Scheme- T27
Break detection -10%	Rate of Extension - 300 mm/min

colorimetric terms. Since the reflectance spectrum is a physical property of the sample, calculation of colorimetric data for different illuminants and observers can be easily performed through software [21].

Determination of whiteness index (WI): Whiteness is defined as a measure of how closely a surface matches the properties of a perfect reflecting diffuser, i.e., an ideal reflecting surface that neither absorbs nor transmits light, but reflects it at equal intensities in all directions. For the purposes of this standard, the color of such a surface is known as preferred white. The two most commonly used methods for computing a whiteness index are CIE Whiteness Index and Ganz-Griesser Whiteness Index [22-25].

Determination of yellowness index (YI): Yellowness is defined as the attribute by which an object color is judged to depart from a preferred white toward yellow. By this definition the object is clearly yellow, although the levels of luminosity are high it is recognized as yellow and not as white. Evaluation of yellowness is very important because most of the material even after extensive bleaching show certain amount of degree of yellowness.

Test on dyed sample

K/S value: Strength of any colorant (dyestuff/pigment) is related to its absorption property. The reflectance is measured and not absorbance. It is known that when reflectance is more, absorbance is less and when reflectance is less, absorbance is more. Kubelka-Munk theory gives us the following relation between reflectance and absorbance:

$K/S = [\{(1-R)^2/2R\}].$

Where R is the reflectance, K is absorbance and S is the scattering. K/S vs. Wavelength curve is always characteristics of every colorant. In our project, the treated fabrics are dyed using the same recipe, dyestuffs and dyeing process and the K/S value and color differences are calculated using spectrophotometer [26]. Color differences and variations in color strength between differently pre-treated samples will indicate the acceptability of dyeing's. When assessing spectrophotometric data of the dyeing's, the conventionally pre-treated fabric is considered as the "standards" and the other samples are considered as the "batch" [27-29].

Results and Discussions

Conventionally pre-treated sample: Following list of test names and test results are given below in the following table (Table 5).

Tegewa rating of pre-treated samples: It is apparent from the graph that pH 11 has higher tegewa rating which means more size material removed in this system. In this system disodium hydrogen phosphate was used as a buffer which has some cleaning power. As a cleaning agent it removes some size materials present in the fabric thus showing higher tegewa rating (Table 6 and Figure 4).

Weight loss% of pre-treated samples: From this graph it can be easily concluded that maximum weight loss% was found for unbuffered at 60 min and buffered (pH 11) at 45 and 60 min system, which is almost nearer to conventional system. The reason is that for pH 11 disodium hydrogen phosphates was used as a buffer which have also

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Test names	Test results
Tegewa rating	7
Weight loss%	12.87%
Immersion time	2.11s
Wicking height	42mm
Strength loss%	14.69%
Whiteness index	66.93
Yellowness index	6.26
K/S value for 0.5% shade	0.7964
K/S value for 1.5% shade	2.5928

Table 5: Test results for conventionally treated sample.

Treated sample	Treated time (min.)	Tegewa rating
рН 9	30	6
	45	6
	60	6.5
	30	6.5
pH 10	45	6.5
	60	7
pH 11	30	7
	45	7
	60	7.5
	30	6.5
Unbuffered	45	6.5
	60	7

Table 6: Tegewa rating for different pre-treated samples.



some cleaning power thus it removes more amount of starch material and results in more weight loss% (Table 7 and Figure 5).

Immersion time of pre-treated samples: It is apparent from the graph that the Immersion time is decreased with the increased of treatment time. The fibre does not become absorbent until the size material and impurities are removed significantly. pH 11 buffered system have lowest immersion time. This is happened because the size material and impurities content present in the fabric were removed very well since we know disodium hydrogen phosphate acts as a cleaning agent. Due to this reason the fabric become more absorbent (Table 8 and Figure 6).

Wicking height of pre-treated samples: It is apparent from the graph that wicking height is increased with the increased of treatment time, pH 11 shows better wicking height. The size material and impurities present in the fabric were removed very well in this buffered system. The fabric become more absorbent and also increased its capillary or wicking action. Thus, the wicking height is higher (Table 9 and Figure 7).

Treated sample	Treated time (min.)	Tegewa rating
рН 9	30	11.25
	45	11.48
	60	11.93
	30	11.92
pH 10	45	12.23
	60	12.48
pH 11	30	12.37
	45	12.71
	60	13
	30	12.13
Unbuffered	45	12.45
	60	12.82

Table 7: Weight loss% for different treated samples.



Figure 5: Graphical representation of weight loss% of differently treated DSB samples and comparison with the conventional.

Treated sample	Treated time (min.)	Tegewa rating
рН 9	30	2.61
	45	2.58
	60	2.54
pH 10	30	2.52
	45	2.37
	60	2.19
pH 11	30	2.2
	45	2.13
	60	2.08
	30	2.33
Unbuffered	45	2.21
	60	2.14

Table 8: Immersion time for different treated samples.



Strength loss% of pre-treated samples: It is apparent from the graph that strength loss% of conventional and DSB by buffered system at pH 11 for 60 min and without buffered system for 45 and 60 min

Treated sample	Treated time (min.)	Tegewa rating
рН 9	30	38
	45	39
	60	40
	30	39
pH 10	45	40
	60	41
рН 11	30	41
	45	42
	60	44
	30	40
Unbuffered	45	41
	60	42

Table 9: Wicking height for different treated samples.



Treated sample	Treated time (min.)	Tegewa rating
рН 9	30	8.09
	45	8.73
	60	9.41
	30	10.16
pH 10	45	12.52
	60	13.23
pH 11	30	11.74
	45	13.62
	60	14.87
	30	12.09
Unbuffered	45	14.86
	60	15.38

Table 10: Strength loss% for different treated samples.

show more or less similar result. The bath was in higher alkaline condition than others which results in cellulose degradation in the system. Another reason for higher strength loss, at higher pH the liberation of HO_2 - ion is so rapid and it becomes unstable with the formation of molecular oxygen. The rate of decomposition is very high and the unutilized HO_2 - may damage the fibre, lowering the DP and reducing the tensile strength (Table 10 and Figure 8).

Whiteness index of pre-treated samples: It is shown from the graph that, the whiteness is more in pH 11 than the others. The whiteness is occurred due to breakdown of H_2O_2 . The H_2O_2 became highly unstable in the pH 10.8-11. Below pH 10.5 H_2O_2 does not produce any whitening effect and above 11.5, H_2O_2 converted into HO_2 - and reaction become difficult to control. pH 11 have higher whitening effect because the optimum pH for bleaching was around in this range and within this range, there is a moderate concentration of per hydroxyl ions. The buffer used for pH 11 which was disodium hydrogen phosphate has some own bleaching effect. This buffer also increases the rate of H_2O_2 decomposition. Thus produce more active oxygen which was responsible for the bleaching effect (Table 11 and Figure 9).

Yellowness index of pre-treated samples: It is apparent from the graph that the yellowness is decreased with the increased of treatment time. The yellowness decreases the most for pH 11. We can say that the yellowness reflects the reverse of whiteness that we have found in earlier graph (Table 12 and Figure 10).

K/S value of pre-treated samples (0.5% shade): Different K/S value of pre-treated is show in the below Table with apparent graph (Table 13 and Figure 11).

K/S value of pre-treated samples (1.5% shades): We know that, the rate of dye take up or exhaustion % is depends on the pre-treatment. If the pre-treatment is better, then the exhaustion % will also be better. K/S value of pH 11 buffered systems is better. Since the pre-treatment





Treated sample	Treated time (min.)	Tegewa rating
рН 9	30	58.06
	45	58.91
	60	59.83
	30	62.42
pH 10	45	64.39
	60	65.17
	30	64.76
pH 11	45	66.87
	60	68.08
Unbuffered	30	63.47
	45	64.86
	60	65.92

Table 11: Whiteness index for different treated samples.



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Tegewa rating

2 0924

2.0981

2.1148

2.2374 2.2678 2.3287 2.3913 2.5362 2.6047 2.2792 2.4173 2.4826

Treated sample	Treated time (min.)	Tegewa rating
рН 9	30	10.43
	45	10.22
	60	10.09
	30	9.11
pH 10	45	8.78
	60	9.67
	30	7.08
pH 11	45	6.56
	60	6.01
	30	7.84
Unbuffered	45	7.21
	60	6.77





Figure 10: Graphical representation of yellowness index of differently treated DSB samples and comparison with the conventional.

Treated sample	Treated time (min.)	Tegewa rating
рН 9	30	0.6592
	45	0.6657
	60	0.6758
	30	0.6857
pH 10	45	0.6921
	60	0.7013
рН 11	30	0.7264
	45	0.7583
	60	0.8027
	30	0.7018
Unbuffered	45	0.7269
	60	0.7612

Table 13: K/S value for different treated samples.



of pH 11 buffer systems was better so the rate of dye take up is also better thus increase the K/S value (Table 14 and Figure 12).

Water, energy and time consumption for conventional process: The Water, Energy and Time consumption for Conventional process is shown in the following Table 15.

		30	
	pH 10	45	
		60	
		30	
	pH 11	45	
		60	
		30	
	Unbuffered	45	
		60	
	Table 14: K	/S value for different treated sa	mples
	3		
-	2.5		
	o 2		
	215		

Treated sample

pH 9



Treated time (min.)

30

45

60

Figure 12: Graphical representation of K/S value (1.5% shade) of differently treated DSB samples and comparison with the conventional.

Steps in the process	Water consumption (L)	Energy consumption (Kcal)	Time required (Min)
Desizing	50	2800	30
Cold wash	50	0	5
Hot wash	50	2800	10
Neutralization	50	1800	15
Scouring	50	3800	45
Cold wash	50	0	5
Hot wash	50	2800	10
Bleaching	50	3800	45
Cold wash	50	0	5
Hot wash	50	2800	10
Neutralization	50	1800	15
Dyeing	50	1800	45
Dye Fixing Treatment	20	1120	30
cold wash	50	0	5
Total	670	25320	275

Table 15: Value for conventional process.

Steps in the process	Water consumption (L)	Energy consumption (Kcal)	Time Required (min)
combined pre- treatment	50	3800	30/45/60
Dyeing	50	1800	45
Dye Fixing treatment	20	1120	30
cold wash	50	0	5
Total	170	6720	110/125/140

Table 16: Value for combined process.

Water, energy and time consumption for combined process: The amount of energy transferred during a chemical reaction (either from the burning of a fuel or a chemical reaction in solution) can be calculated using the equation (Table 16):

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• Q=mc ΔT

Where:

Q=the heat energy transferred (joule, J), m=the mass of the liquid being heated (grams, g), c=the specific heat capacity of the liquid (4.186 joule per gram degree Celsius, J/g°C), Δ T=the change in temperature of the liquid (degree Celsius, °C). From the above graph it can conclude that the combined process is much economical with respect to conventional process in all aspects of water, energy and time consumption (Figure 13, 13a and 13b).

Key Findings

- In case of dark shade, DSB at pH 11 for 45 min can be selected.
- In case of any depth of shades, DSB at pH 11 for 60 min can be selected.
- In conventional process more chemical is used but in case of combined process less chemical is needed compared to the conventional one. In combined process less water, less thermal energy is needed compared to conventional process.







- In case of single bath process, if the treated time of the sample is increased with controlled pH then better result was found in case of whiteness and dye take up rate compared to the conventional process.
- The conventional process has at least two bath processes but in single stage preparatory process, the pre-treatment is done in a single bath which results shortening and simplification of the process.

Conclusion

In this project work, an attempt was taken to replace the conventional pre-treatment system by single bath with adjusting pH by using buffer. In one bath preparatory process, buffered pH 11 for 60 min shows better result and buffered pH 11 for 45 min and unbuffered sample (60 min) shows imminent performance in compare with conventional in respect to weight loss%, immersion time, wicking height, whiteness index and K/S value of dyed sample. This kind of a one-bath process is shorter, consuming less energy and hence being less expensive. The combined processes also lead to substantial saving in water energy and time along with chemicals.

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