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Investigation of mechanical properties of bio-finished regenerated bamboo fabrics using 2³ 3¹ mixed level factorial design

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K E Y W O R D S	A B S T R A C T
Regenerated	Pilling resistance of Regenerated Bamboo (100%) and Regenerated Bamboo-Cotton
Bamboo	(50:50) blended, woven fabrics was poor (grade 3 to 1.5). There are different techniques available that may help to improve the pilling resistance of fabrics. Bio
Cotton	polishing is one of the effective ways to control the pilling of cellulosic knitted fabric
Singeing	however very few studies have been conducted on woven fabrics. Therefore, this study
Pilling	improvement of pilling of regenerated bamboo and bamboo/cotton woven fabrics.
Bio-Polishing	Effect of bio-polishing agent (acid cellulase) on Regenerated Bamboo and
Factorial Design	Regenerated Bamboo-Cotton woven fabrics was investigated using 23 31 mixed-level factorial design. Four factors were studied; Fabric weave, blend ratio, pretreatment and concentration of acid cellulase were selected. Influence of Fabric weave, blend ratio and pretreatment was examined at two levels, whereas concentration of acid cellulose was tested at three levels. The influence of the individual factors and their interactions on pilling resistance, tensile strength, tear strength and Berger whiteness were analyzed using the software Design-Expert 8.13. The outcome of this study showed that biopolishing improved the pilling resistance of Regenerated Bamboo and Regenerated Bamboo-Cotton blended woven fabrics and did not affect tear and tensile strengths and whiteness of the plain and satin fabrics, significantly.

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1. Introduction

Bamboo is rekindled cellulosic fiber. Its structure is round, shiny and lustrous. It is sterilized and gives subtle feel to skin. Bamboo fiber is hygroscopic, has good air permeability and moisture vapor transmission properties which makes it ideal for clothing [1-5]. The cellulose fibres obtained from bamboo pulp is called Regenerated cellulose and they possess somewhat same properties as of bamboo. Regenerated Bamboo is also a cellulosic

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material however it has low pilling resistance, low wrinkle resistance and poor dimensional stability [6].

Bio-polishing is an enzymatic process applied to cellulosic fabrics for smooth appearance and soft handle. Application of enzymes also eludes use of conventional chemicals that produce lethal effluents [7-11].

Textile industry is conversant with two types of cellulase; Neutral cellulase and Acid cellulase. Acid

cellulase is a harsh chemical than Neutral cellulase and it not only makes fabrics appearance unacceptable but may lead to loss of strength and weight of the fabrics [12-18]. Treatment of cellulosic fabrics by cellulase can be distributed into two main phases. In the first phase, cellulase acts on the cellulosic fabric and improves fabric appearance, softness and pilling resistance. Second phase treatment is for denim fabrics as a substitute of stone washing [1-21].

In view of the importance of bio-polishing, numerous investigations have been conducted on enzymes to improve the pilling resistance, appearance and handle of the fabrics. In one of the studies, use of acid-cellulase (0.006% to 0.008% concentration) reduced pill formation on 100% regenerated, knitted fabric [16] and along with that pilling resistance, appearance and handle of the fabric also improved; however, loss of weight and strength were reported after bio-polishing. It was also reported that there was significant loss of weight, tear strength and tensile strength of the Cotton, Linen, Ramie, and Viscose Rayon fabrics due to the enzymatic hydrolysis [13, 22].

Statistical techniques such as ANOVA (analysis of variance), factorial design, desirability, response surface methodology etc. are employed in many researches to design experiments and optimised Textile processes [4, 23-27]. In one of the researches [26] Box-Behnken design (BBD) was applied to optimized the enzymatic hydrolysis of cotton waste. A BBD model was consisted of 15 run, 3-factors and 3-levels, three replicates and the center point; and it was used to determine the finest cellulose particles.

Pilling resistance of 100% Regenerated bamboo and if blended with cotton, is poor. Many chemical finishes are available which improve the pilling resistance of textile materials (Naeem F 2019) and use of enzymes is one of the options. In this investigation, enzymes were used to improve the pilling resistance of the regenerated bamboo woven fabrics. Therefore, this study aims to statistically examine the influence of different concentration of enzymes on pilling resistance of plain and satin woven fabrics made from Regenerated Bamboo. In the proposed experimental and statistical investigation, Bio-polishing of Regenerated Bamboo and Regenerated Bamboo-Cotton (50:50) woven fabrics was carried out with acid cellulase by Exhaust method and Experimental design technique respectively. Effect of bio polishing on pilling resistance, tensile strength, tear strength and Berger whiteness was studied. Selected properties of 100% Regenerated Bamboo (Rayon) and 50:50 Regenerated Bamboo-Cotton fabrics were evaluated after two of the pretreatment methods; one followed singeing process and the other was carried out without doing singeing. These two methods were analyzed statistically because screening experiments of this study exhibited poor pilling resistance of fabrics for both process routes either followed singeing or not [29].

This study used mixed level factorial design to identify the effect of bio-polishing agent on singed and non-singed regenerated bamboo woven fabrics, which was not applied in previous investigations.

2. Materials and Method

2.1 Fabric Manufacturing

Bamboo yarn used in this study was regenerated cellulose (Rayon) which is produced from bamboo pulp. Unlike natural cellulose which has Cellulose I in their crystalline region, regenerated cellulose like bamboo has Cellulose II structure. This difference in structures is due to the use of NaOH in the regeneration process which changes Cellulose I in to Cellulose II [28]. 30's Ne yarn count was used in the construction of 100% regenerated bamboo and 50:50 bamboo: cotton fabrics. Plain and 5 harness satin weaves were formed in both materials. Thread count of all the fabrics was kept at 76 Ends per inch (EPI) and 68 Picks per inch (PPI). Materials and weaves selection were based on the requirement of the fabrics for the manufacturing of home textiles. Woven fabrics pretreatment followed the process routes mentioned below.

SIGNED [29]



2.2 Chemicals Application Procedures

Bactosol GETU from Archroma was procured as biopolishing agent. It is an Acid cellulase which is a composed mixture of selected Iso-cellulase.

Fabrics were treated with Bactosol GETU of 1%, 2.5% and 5% separately at 55°C in laundrometer for 45 minutes. The liquor ratio was 1:20. pH of the solution was 6. After cellulase treatment for forty five (45) minutes, fabrics were thoroughly rinsed with water and then dipped into an alkaline solution of pH 9 for 2 minutes. After water rinsing, fabrics were line dried. Application of the Bactosol GETU on the selected materials was carried out according to the guidelines provided by Archroma. The MSD of Bactosol GETU suggested that it should not be allowed to enter drains, water courses or the soil.

2.3 Evaluation of Fabric Properties

Standard test methods were employed to assess the properties of the fabrics. Pilling of the fabrics was tested by ISO 12945-2, ISO 13934-2 Grab method selected for Tensile strength measurement, Tear strength by ISO 13937-1 ELMENDROF and Berger whiteness was evaluated by AATCC 110. Number of samples for each test were taken as mentioned in the standard test procedure. For pilling test, 3 specimens were tested. For tensile and tear strengths measurement, five specimens each in warp and weft were evaluated. Similarly, five specimens were tested for berger whiteness. Every Test was performed twice on the fabrics, finished at two different times using freshly prepared liquors so that repeatability can be verified; coefficient of variance (CV) between the two replicates was within +/- 5%. Temperature and Relative Humidity conditions of the testing laboratory were set according to the testing conditions.

Pilling grades according to Pilling Scale (Modified Martindale ISO 12945-2) [30] were given to all samples using standard replicas in a dark room.

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2.4 Experimental Design

In the experimental design, process factors (or variables) are needed to be varied in order to perceive the changes that may have on the response variables. DOE is a statistical planning professional technique for experiments. Its application scrutinized the results obtained to yield persuasive and objective conclusions. In DOE, the objectives of the experiments are firstly determined followed by the selection of the process variables, which are desired to be investigated. An efficient experimental design, which provides detailed experimental plan before the start of the investigation, maximizes the extent of "information" that can be acquired for a given experimental work [29].

23 31 mixed level factorial design was applied to examine the effect of the following factors; Weave (A), Blend Ratio (B), Pretreatment (C) and Bio-polishing agent (D). Experimental model is shown in Table 1. A 23 31 mixed level factorial design is shown in Table 2. 24 trials with two replications were run according to the experimental design. All experiments performed randomly. Results were obtained using software Design-Expert 8.13. In this research, Responses were Pilling resistance, Tensile and tear strengths and Berger whiteness. Yarn count, weave structures, yarn densities and pretreatment parameters were kept constant.

Table 1

Factors and respective levels used in 2³ 3¹ factorial design

Fac	tor NAME	LEVEL					
		(-)		(+)			
А	Weave	Plain	-	Satin			
В	Blend ratio	100% Regenerated Bamboo	-	50:50 Regenerated Bamboo:Cotton			
С	Pretreatment	Singed	-	Non-Singed			
D	Bactosol GETU	1%	2.5%	5%			

Run	A: Weave	B:	Blend ratio	C:	Pretreatment	D:	Bactosol GETU
		%					%
1	Plain	100% Regenerated Bam	boo	Singed			5
2	Satin	50:50 Regenerated Bam	boo:Cotton	Singed			5
3	Satin	100% Regenerated Bam	boo	Singed			1
4	Plain	50:50 Regenerated Bam	boo:Cotton	Singed			1
5	Plain	50:50 Regenerated Bam	boo:Cotton	Non-Sing	ed		2.5
6	Plain	100% Regenerated Bam	boo	Non-Sing	ed		2.5
7	Satin	50:50 Regenerated Bam	boo:Cotton	Singed			2.5
8	Satin	100% Regenerated Bam	boo	Non-Sing	ed		5
9	Satin	100% Regenerated Bam	boo	Non-Sing	ed		1
10	Plain	100% Regenerated Bam	boo	Singed			1
11	Plain	100% Regenerated Bam	boo	Singed			2.5
12	Satin	50:50 Regenerated Bam	boo:Cotton	Singed			1
13	Satin	50:50 Regenerated Bam	boo:Cotton	Non-Sing	ed		5
14	Plain	100% Regenerated Bam	boo	Non-Sing	ed		1
15	Satin	100% Regenerated Bam	boo	Singed			5
16	Plain	50:50 Regenerated Bam	boo:Cotton	Non-Sing	ed		1
17	Satin	50:50 Regenerated Bam	boo:Cotton	Non-Sing	ed		1
18	Plain	50:50 Regenerated Bam	boo:Cotton	Singed			5
19	Satin	100% Regenerated Bam	boo	Non-Sing	ed		2.5
20	Plain	50:50 Regenerated Bam	boo:Cotton	Non-Sing	ed		5

Experimental run order of 2³ 3¹ Factorial Design

Table 2

3. Results and Discussion

Reaction mechanism of cellulase on cellulose material has been described in many studies. Cellulase initially forms complex with water and cellulose polymeric chains; and then endo-glucanases, beta-glucanases or cellobiohydolase get adsorbed on the fibres of cellulose based fabrics. After absorption, B (1-4) link of cellulose be hydrolysed and enzymes detach themselves from the same surface area of the fabric however remain available to perform same reaction repeatedly. Endo glucanases of cellulase unlocks further fibres on the fabric surface effectively, to be reacted by beta glucanases and cellobiohydolase which eventually form water soluble fragments. This whole synergic mechanism removes fibrils from the surface of the cellulosic fabrics however reduces strength either [18, 29]. This reaction mechanism is shown in Fig. 1R.





ANOVA (Analysis of variance) of Pilling resistance, weft tensile strength, warp tear strength, Berger whiteness are shown, see Table 3 to Table 6. The model of the warp tensile strength was not significant so it is not discussed. Furthermore, the effect of the Bactosol GETU on weft tear strength was the same as on the tear strength in warp direction; therefore, it is also not discussed separately. Effect of bio-polishing on pilling resistance, tensile strength in weft direction, tear strength in warp direction and Berger whiteness are shown in Fig.s 1(a-d), 2(a–f), 3(a–f) and 4(a-d) respectively. Effect of fabric weave on pilling resistance, tensile strength and tear strength after bio-polishing was same for all samples whether singed or not singed, therefore results will be discussed for those non- singed.

3.1 Pilling Resistance

Analysis of variance (ANOVA) for Pilling Resistance is shown in Table 03. The model of the pilling resistance is significant with Prob > F < 0.0001. Predicted R² of 0.8616 is in sound agreement with the Adjusted R² of 0.8987 which states that the model of pilling resistance accounted most of the variability.

Table 3 showed that the Fabric weave and Bactosol GETU are the significant factors. The interaction (AD) of these terms is also significant.

Figs. 1(a to d) presented the interaction plots of Fabric weave (A) and Bactosol GETU (D). Interaction plots of 100% Regenerated Bamboo in Figs. 1(a and b) showed the effect of various concentration of Bactosol GETU on non-singed and singed, plain and satin fabrics respectively. The trend in the test results is the same. It is evident from the results that for a plain fabric the maximum improvement in the pilling resistance showed at 5% of Bactosol GETU, which is pilling grade 4 and

Table 3

6	0 0	10	M	E 1	1 .	
Source	Sum of	dr	Mean	F-value	p-value	
Model	2.541	5	0.508	34.7	< 0.0001	Significant
A-Weave	1.798	1	1.798	122.8	< 0.0001	
D-Bactosol GETU	0.698	2	0.349	23.82	< 0.0001	
AD	0.255	2	0.128	8.707	0.0035	
Residual	0.205	14	0.015			
Cor Total	2.746	19				
Std. Dev.	0.121		R²		0.92533895	
Mean	1.565		Adjusted	R²	0.89867429	
C.V. %	7.731		Predicted	R²	0.86162503	
			Adeq Prec	ision	15.3779935	

Analysis of variance (ANOVA) for Pilling Resistance

above. However, the pilling resistance of 100% Regenerated Bamboo fabric at 1% Bactosol GETU is better that 2.5% of Bactosol GETU.

In 100% Regenerated Bamboo satin fabric, pilling resistance improved by increasing the concentration of Bactosol GETU (see Fig. 1a and 1b), however, the value is low if compared with the value achieved in plain fabric. This is because of the structure of the satin fabric which accounts for more pilling on the surface contrary to the plain fabric.

Figs. 1c and 1d exhibited the interaction plots of 50/50 Regenerated Bamboo- Cotton singed, plain and satin fabrics. The trend in the test results is the same as discussed for 100% Regenerated Bamboo fabrics.

Pilling resistance indicates that the acid enzyme significantly improved the pilling resistance of the treated samples. In fact, Bactosol GETU showed more activity at 5% and 1% than 2.5%. Due to the excellent bio-polishing effect, most of the protruding fibers were removed. It reduced pill formation and rating is 4 which means slight surface fuzzing.

There was less pill formation due to the treatment of Bactosol GETU. Lower concentration of Cellulase shown good results however if the concentration of the Cellulase is increased it is found that it slows its action on the fabrics. This is due to the activity of the cellulase enzyme.



Fig. 1. Interaction plot of Fabric Weave and Bactosol GETU on pilling resistance of 100% Regenerated Bamboo (Non-Singed) (A), Singed (B), 50:50 Regenerated Bamboo:Cotton (Non-Singed) (C), Singed (D)

3.2 Weft Tensile Strength

Analysis of variance (ANOVA) for weft tensile strength is presented in Table 04. The model for the Weft tensile strength is significant with Prob > F < 0.0001. The Predicted R² of 0.6599 is in reasonable agreement with the Adjusted R² of 0.7092 which indicates that the fabric weave design is the only significant factor.

The effect of fabric weave is plotted in Fig. 2 (a to f) for 100% Regenerated Bamboo and blend of 50% Regenerated Bamboo and 50% Cotton.

Tensile strength in the weft direction of 100% Regenerated Bamboo plain and satin remained the same for against all the concentrations of Bactosol GETU, selected for this investigation. It means that the variation in the concentration of Bactosol GETU didn't significantly alter tensile strength of Regenerated Bamboo plain and satin fabrics in weft direction; however, change in the weave design affected their strength. Weft tensile strength of plain fabric was higher than that of satin fabric. It is owing to the highest value of cover factor of plain structure (see Fig. 2a to 2c and Fig. 2 (d to f). By decreasing the interlacement between warps and wefts such as 4/1 satin, strength decreased i.e. 170 N while of 1/1 plain weave was 220N.

Similarly, weft tensile strength of 50/50 Regenerated Bamboo-Cotton plain and satin fabrics shown the same trend as discussed above for 100% Bamboo fabrics. It can be seen in Fig. 2 (d to f) as well.

Table 4

Source	Sum of	df	Mean	F-value	p-value	
Model	10.1	1	10.1	47.33	< 0.0001	Significant
A-Weave	10.1	1	10.1	47.33	< 0.0001	
Residual	3.842	18	0.213			
Cor Total	13.95	19				
Std. Dev.	0.462		R	²	0.72449592	
Mean	13.9		Adjus	ted RÂ ²	0.70919014	
C.V. %	3.323		Predic	ted RÂ ²	0.65987151	
			Adeq F	Precision	9.72982893	

Analysis of variance (ANOVA) for Weft Tensile Strength



Fig..2 Effect of Fabric Weave on Weft Tensile Strength for 100% Regenerated Bamboo (Non-Singed) at 1% Bactosol GETU
(A) 2.5% Bactosol GETU (B), at 5% Bactosol GETU (C), for 50:50 Regenerated Bamboo:Cotton (Non-Singed) at 1% Bactosol GETU (D), at 2.5% Bactosol GETU (E), at 5% Bactosol GETU (F)

3.3 Warp Tear Strength

ANOVA (Analysis of variance) for warp tear strength is given in Table 5. The Prob > F is < 0.0001 suggested that the model for the weft tear strength is significant. Predicted R² of 0.7200 was in good agreement with Adjusted R² of 0.7739. Fabric weave and blend ratio were the significant model terms. This indicates that the different concentrations of bio-polishing agent did not significantly alter the tear strength of the both fabrics type.

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Effect of Weave design on tear strength of both fabrics type are shown in Figs. 3 (a to f). Figs. 3 (a to c) depict the warp tear strength of 100% Regenerated Bamboo whereas, Fig. 3 (d to f) show the tear strength of 50/50 Regenerated Bamboo-Cotton. Results shown that there is no significant differences in warp tear strength of the plain and satin fabrics at different concentrations of Bactosol GETU. Tear strength is the resistance of the fabric against the force required to transmit the tear, once it is commenced. Force requires to tear 100% Regenerated Bamboo plain woven is approx. 15N whereas for satin fabric it is 19N. Similarly for 50:50 Regenerated Bamboo-Cotton, it is approx. 12N for plain and 15N for stain. It is due to the differences in the values of the cover factors of Plain and Satin weaves. In Satin, yarns float and offer higher resistance to the tearing in contrast to the plain woven

Table 5

Analysis of variance (ANOVA) for Warp Tear Strength

structure in which there is no floating to resist the tear. Results presented in Figs. 3 (a to f) shown that the warp tear strength of 100% Regenerated Bamboo is higher than 50:50 Regenerated Bamboo-Cotton due to higher strength of 100% Regenerated Bamboo compared to its blend with 100% Cotton.

Source	Sum of	df	Mean	F-value	p-value	
Model	0.534	2	0.267	33.72	< 0.0001	significant
A-Weave	0.321	1	0.321	40.5	< 0.0001	
B-Blend ratio	0.213	1	0.213	26.94	< 0.0001	
Residual	0.135	17	0.008			
Cor Total	0.669	19				
Std. Dev.	0.089		R²		0.79866083	
Mean	2.722		Adjusted R.	²	0.77497387	
C.V. %	3.269		Predicted R	²	0.72132987	
			Adeq Precis	sion	13.3412188	



Fig. 3 Effect of Fabric Weave on Warp Tear Strength for 100% Regenerated Bamboo (Non-Singed) at 1% Bactosol GETU (A), at 2.5% Bactosol GETU (B), at 5% Bactosol GETU (C), for Regenerated Bamboo: Cotton (Non-Singed) at 1% Bactosol GETU (D), at 2.5% Bactosol GETU (E), at 5% Bactosol GETU (F)

3.4 Berger Whiteness

ANOVA (Analysis of variance) for Berger whiteness is given in Table 6. The model for Berger whiteness is significant having Prob > F < 0.0001. The Predicted R² of 0.8200 is in good agreement with the Adjusted R² of 0.8546. Statistical analysis of data revealed that fabric weave and pre-treatment are significant factors for Berger whiteness.

Effect of weave on fabrics singed and non-singed, constructed of Regenerated Bamboo and Regenerated Bamboo-Cotton is illustrated in Figs. 4 (a to d). As it can be comprehended from Table 6 that the factor Bactosol GETU is insignificant, therefore the effect of fabric weave; singed and non-singed are shown in Figs. 4(a to d) at 5% Bactosol GETU. Berger whiteness of the fabric sample/samples not singed is better than those singed in both weave types because in singeing fabric is in contact with flame to burn protruded fibres which adversely reduces the whiteness of the fabric. Besides, Berger whiteness of stain fabrics is better than plain fabrics. In sample fabrics having satin weave, the weft thread floats over and under as per construction which increased light reflection therefore value of Berger whiteness is higher for satin fabrics than plain woven fabrics.

Statistical analysis of the outcomes of this investigation revealed significant information related to the effect of acid cellulase (Bactosol GETU) on pilling, tensile strength, tear strength and berger whiteness of the cellulosic fabrics. This study found that acid cellulose could also improve pilling resistance of the woven fabrics. Pilling resistance of the fabrics changed by increasing the concentration of acid cellulase (Bactosol GETU), however it did not significantly affect strength and berger whiteness of the fabrics.

Table 6

Analysis of variance (ANOVA) Berger Whiteness

	_					
Source	Sum of	df	Mean	F-value	p-value	
Model	0.913	2	0.456	56.84	< 0.0001	significant
A-Weave	0.714	1	0.714	88.9	< 0.0001	
C-Pretreatment	0.199	1	0.199	24.79	0.0001	
Residual	0.136	17	0.008			
Cor Total	1.049	19				
Std. Dev.	0.09		R	R²	0.86991484	
Mean	8.603		Adjusted RÂ ²		0.8546107	
C.V. %	1.042		Predic	eted RÂ ²	0.81995134	
			Adeq Precis	ion	16.6359489	



Fig. 4. Effect of Fabric Weave on Berger Whiteness at 5% bio-polishing agent of 100% Regenerated Bamboo (Non-Singed) (A), Singed (B), 50:50 Regenerated Bamboo: Cotton (Non-Singed) (C), Singed (D)

4. Conclusion

Pilling gives unaesthetic appearance to the fabrics which results in the rejection of the material means it can't be employed for manufacturing of products. There is a need to employ those fabrics having higher resistant to the pilling no matter constructed of different types of materials especially blends.

Bio-polishing by Bactosol GET U proved one of the ways to improve pilling resistance of the Regenerated Bamboo and Regenerated Bamboo-Cotton blended, woven fabrics without compromising on its physical characteristics. Statistical analysis of the significant factors discovered their effects on the response variables of bio polishing of pure and blended bamboo woven fabrics. Maximum improvement in the pilling of woven fabrics of 100% Regenerated Bamboo and Regenerated Bamboo-Cotton blend was observed at 5% Bactosol GET U and this enhancement is better in plain fabrics. However, different concentrations of Bactosol GET U did not affect weft tensile strength, tear strength and Berger whiteness of the woven fabrics. Moreover, the models have been verified for adequacy and it is established that the hypothesis of normality and independency are not violated. The high R2 values recommend that models accounted most of the variability.

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