Effect of Chitosan on Dyeability of Cotton Fabric Dyed with Natural Dye Extract

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Abstract: *This research is concerned with investigation on the influence of chitosan treatment on dyeability of cotton fabric. In this work, natural dye was extracted from leaves of Indian almond in water medium. Before dyeing operation, cotton fabric was treated with three different concentrations of chitosan and pad-dry-cure method was used for chitosan treatment. Then, chitosan treated and untreated fabrics were dyed with the extracted natural dye and cold dyeing method was used for all dyeing operations. The colour yield, colour difference and fastness properties of chitosan treated and untreated dyed samples were analysed and evaluated. The effect of chitosan concentration on dye absorption of samples was also investigated. All the chitosan treated dyed samples had higher colour saturation compared with untreated dyed samples and fabric treated with 1.5% chitosan had higher K/S value. The impact of chitosan treatment and natural dye extract on strength and stiffness of chitosan treated and untreated dyed samples were also examined. There was no strength loss due to the chitosan treatment and chitosan treated dyed samples were stiffer than the untreated one.*

Key Words: *cotton, chitosan, natural dye, dyeability*

1. INTRODUCTION:

Today, a number of synthetic dyes have been employed to acquire desirable colours and to enhance dye affinity of textile materials. Although the synthetic dyes grant to develop esthetic colours and good dye affinity, many of these dyes are toxic to humans and do not simply degrade in the environment. For these occasions, the use of natural materials in colouration and dye fixation of textile materials is one of the solutions. There is a tendency to suppose that biodegradable natural materials are more harmless and better than artificial materials. Surface modification of textile fibres by using some natural polymers such as chitin or chitosan in textile finishing processes is considered to be the best route to overcome these problems. Chitosan is a potentially useful biopolymer obtained by alkaline deacetylation of chitin [1-2]. Chitin and chitosan are naturally occurring β-1, 4-linked linear polysaccharides similar to cellulose. Chitin is essentially a homopolymer of 2-acetamide-2-deoxy-β-D- glucopyranose. Chitosan is the N-deacetylated derivative of chitin and the majority of its glucopyranose residues are 2-amino-2-deoxy-β-D-glucopyranose [3]. The application of chitosan in textiles can be categorized into two main topics: the production of man-made fibres and textile wet processing, which include dyeing (improving the dyeability), finishing (antimicrobial properties), and printing (as a print-paste thickener) [4-5]. The application of chitosan for improving dyeability of cotton fabric has been widely studied. Chitosan can easily absorb anionic dyes, such as direct, acid and reactive dyes, by electrostatic attraction due to its cationic nature in an acidic condition. Moreover, chitosan has number of unique properties such as biocompatibility, biodegradability, non-toxicity and antimicrobial activity [5-6]. There are many plant materials that can be used for dyeing textile materials: roots, bark, leaves, berries, seeds, twigs, branches, tubers, and nut hulls, each capable of producing a range of colours with various mordant. In recent years, there has been an interest manifested towards natural dyes because of the ecological movement, biodegradability and higher compatibility of natural dyes with environment. Other advantages associated with natural dyes include lower toxicity and allergic reaction in relation to synthetic dyes. In addition, natural dyes are also eco-friendly dyes and so the uses of natural dyes in textile process are very welcoming. This research focused on influence of chitosan treatment on dyeing properties (dye absorption, colour strength, and colour fastness) of cotton fabric dyed with natural dye extracted from Indian almond leaves. Additionally, other physical properties of chitosan treated and untreated dyed fabrics were also investigated.

2. EXPERIMENTAL PROCEDURE:

2.1. MATERIALS:

Bleached cotton fabric with the constructional design of plain weave was selected for this research. Chitosan (85% Deacetylation) and acetic acid (99.5%) were collected in Analytical Reagent (AR) grade.

2.2. METHODS:

Pad-dry-cure method was used for all chitosan treatments and Laboratory Padding Mangle and Chamber Dryer were employed for this treatment. Cold extraction method was applied for natural dye extraction from Indian almond leaves. Similarly, cold dyeing method was used for dyeing cotton fabric with natural dye extract. The fixation process was also carried out by means of post mordanting method using alum as mordant.

2.3. ANALYSIS OF SAMPLE FABRIC:

The sample was conditioned for six hours at the standard atmospheric condition having relative humidity of 65 ± 2% R.H and temperature of 20 ± 2°C. Fabric analysis tests of sample fabric were carried out according to the respective ASTM standard test methods in the standard atmospheric condition. The summary of test results was described in Table I.

TABLE I
SUMMARY OF TEST RESULTS ON ANALYSIS OF ORIGINAL FABRIC (C_{00})

Sr. No.	Test Method		Test Results (Mean Value)
1	Fabric Width (cm)		102.21
2	Fabric Thickness (mm)		0.24
3	Fabric Weight (g/m^2)		102.30
4	Fabric Count	Ends/cm	46.00
		Picks/cm	27.00
5	Yarn Number (Tex)	Warp	13.11
		Filling	13.22
6	Crimp (%)	Warp	2.80
		Filling	15.39
7	Air Permeability (cm^3/cm^2/sec)		38.00
8	Breaking Strength (kg)	Warp	52.50
		Filling	27.80
9	Bending Length (cm)	Warp	1.30
		Filling	0.92

2.4. FINISHING TREATMENT WITH CHITOSAN

The sample fabric 13" × 15" (33.02 cm × 38.10 cm) that suited with the available machine facility was prepared first. Prior to finishing treatment, the prepared sample was conditioned in the standard atmospheric condition for about six hours. The required amount of chitosan, acetic acid and distilled water were calculated based on the weight of prepared sample. In this research, the material to liquor ratio, 1:15, was used for chitosan treatment. In the preparation of chitosan solution, the calculated amount of chitosan was dissolved in 1% acetic acid solvent and the mixture was stirred frequently in order to acquire the complete solution. Pad-dry-cure method was used for this treatment. The sample was dipped in the prepared liquor for about 3 minutes and then the fabric was padded through Padding Mangle. And then, the padded sample was dried at 80°C for 5 minutes in the Chamber Dryer and the dried sample fabric was cured at 150°C for 3 minutes in the same dryer again. After that, the pad-dry-cure sample was rinsedwith tap water in order to eliminate the trace of acid residue and finally, dried at room temperature. The treating procedure is the same for all chitosan treatments at each concentration of chitosan. The detailed description concerned with chitosan treatment was shown in Table II and the process flow diagram of finishing treatment was shown in Figure 1.

TABLE I
DETAILED DESCRIPTION CONCERNED WITH CHITOSAN TREATMENT

Sample Code	Chitosan Concentration (%)	M:L	Drying		Curing	
			Temp. (°C)	Time (min)	Temp. (°C)	Time (min)
C_{00}	-	-	-	-	-	-
C_{01}	0	-	-	-	-	-
C_{11}	0.5	1:15	80	5	150	3
C_{21}	1.0	1:15	80	5	150	3
C_{31}	1.5	1:15	80	5	150	3

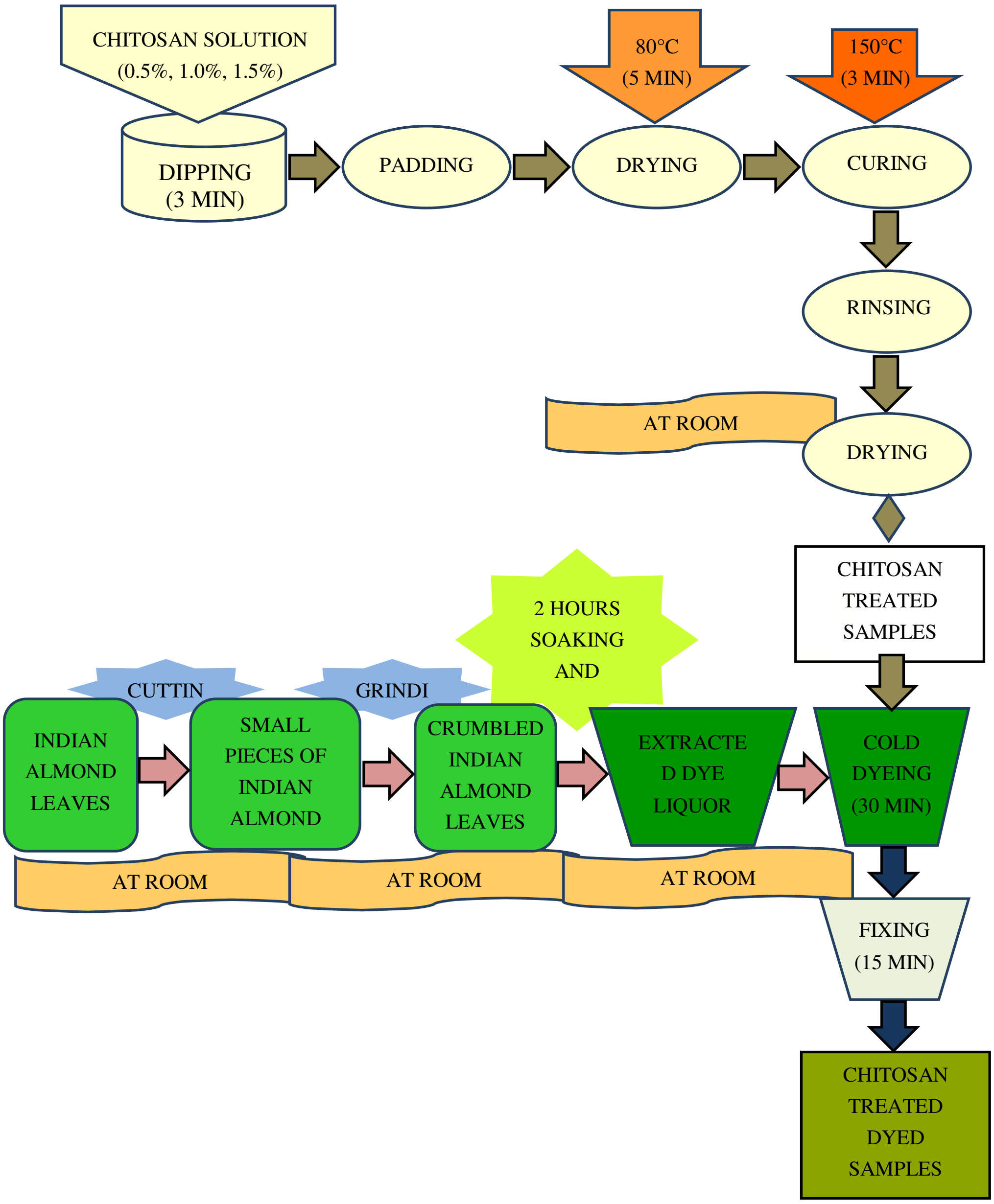

Figure 1. Process flow diagram of chitosan treatment, dye extraction and dyeing process

2.5. Dye Extraction

Natural dyes can be obtained from plant materials: roots, bark, leaves, berries, seeds, twigs, branches, tubers, and nut hulls. In this research, natural dye liquor was extracted from the Indian almond leaves which could be available plentifully in YTU campus. Moreover, Indian almond leaves could be plucked easily in anytime, anywhere and any season in Myanmar. Indian almond leaves were collected from YTU campus and then rinsed with tap water to remove dust, dirt and other undesirable impurities. After that, the cleaned Indian almond leaves were cut into small pieces in order to facilitate in the subsequent step. And then, the small pieces of Indian almond leaves were ground to crumble using the electronic blender. In this case, it is necessary to add small amount of water to facilitate the grinding operation and the material to liquor ratio used was 1:3. And then, the crumbled leaves were poured into the steel pot and soaked in water medium for about two hours at room temperature. During soaking, it is necessary to agitate and crush in every 10 minutes. After soaking period, the crumbled leaves were squeezed out and removed and the extracted dye liquor was filtered in order to remove the remaining trace of crumbled leaves. Consequently, the natural dye liquor is ready to apply in dyeing (Figure 2). The pH value of extracted dye liquor was assessed using the HI2211 pH/ORP meter and the resultant pH value was 4.5 ~ 5.

Figure 2. Indian almond leaves and extracted dye liquor

2.6. DYEING:

Before starting the dyeing process, the chitosan treated and untreated samples were conditioned in the standard atmospheric condition for about six hours. The required amount of dye liquor was calculated based on the weight of the sample to be dyed and the material to liquor ratio used was 1:25. Chitosan treated and untreated samples were dyed with the respective amount of prepared natural dye liquor by means of cold dyeing method. Dyeing process was carried out for 30 minutes at room temperature. During dyeing process, it is needed to stir up in every 5 minutes in order to achieve the level dyeing result. After dyeing, the dyed sample was lifted out, squeezed by hand and allowed to set in the open air in at room temperature for about 5 minutes for the better penetration of dye liquor. In this research, post-mordanting method was used in order to achieve dye fixation using alum as mordant. The pH value of fixing liquor was kept between 4 ~ 5 and the amount of fixing liquor required were determined based on the weight of dyed sample. In this case, the material to liquor ratio used was also 1:25. The dyed sample was soaked in the fixing liquor for 15 minutes at room temperature. During soaking, it is needed to stir up in every 3 minutes in order to acquire even result. After fixing process, the sample fabric was rinsed in cold water and dried at room temperature that free from direct sunlight. At the end of dyeing process, weight add-on % was determined after treating the sample with chitosan and dyed with natural dye liquor and compared the initial weight (before treatment with chitosan) and final weight (after treatment with chitosan and natural dye extract).

$$Weight\ add-on\ (\%) = \frac{(W_2 - W_1)}{W_1} \times 100 \quad (1)$$

Where, W_1 *= Weight of sample before treatment with chitosan*
W_2 *= Weight of sample after treatment with chitosan and natural dye extract*

2.7. ANALYSIS OF COLOUR PRODUCED AND COLOUR STRENGTH:

Benchtop Spectrometer (X-rite Ci7600) was used to analyze the colour obtained on chitosan treated and untreated dyed fabrics by CIELAB colour attribute (L*, a*, b*, C*, h°) and dye absorption concentration on fabrics by K/S values examined under D65-10 light. K/S values were computed at 600 nm and the colour strength was calculated in terms of K/S values.

$$K/S = \frac{(1-R)^2}{2R} \quad (2)$$

Where K is coefficient of absorption, S is coefficient of scattering, and R is reflectance value of the sample.

The colour difference (ΔE) and relative colour strength between chitosan treated dyed and untreated dyed samples were also investigated.

$$\Delta E^* = \sqrt{(\Delta l^*)^2 + (\Delta a^*)^2 + (\Delta b^*)^2} \quad (3)$$

$Where\ \Delta E^* = total\ colour\ differences$
$\Delta l^* = l^*_{treated\ dyed} - l^*_{untreated\ dyed}$
$\Delta a^* = a^*_{treated\ dyed} - a^*_{untreated\ dyed}$
$\Delta b^* = b^*_{treated\ dyed} - b^*_{untreated\ dyed}$

l^* refers to lightness-darkness values from 100 to 0 representing white to black, a^* describes redness if positive and greenness if negative and b^* represents yellowness if positive and blueness if negative.

$$\text{Colour strength (\%)} = \frac{\frac{K}{S}\ of\ chitosan\ treated\ dyed\ sample}{\frac{K}{S}\ of\ untreated\ dyed\ sample} \times 100 \quad (4)$$

2.8. ANALYSIS OF FASTNESS PROPERTIES:

The fastness properties of chitosan treated and untreated dyed sample were assessed by means of such methods as fastness to washing and fastness to light. The samples were washed by ISO Test No.3, CO3 as stated in the washing test (ISO R-105) to determine the change in colour and staining of adjacent fabrics after washing. The changes of colour due to washing were compared with the colour of the untreated dyed sample and the colour transfer on cotton fabrics were determined by using Grey Scales. The rating scale of washing fastness for colour change was from 1 (very poor), 2 (poor), 3 (fair), 4 (good), to 5 (excellent). Light fastness test was carried out according to AATCC test method. The rating scale of light fastness was from 1 (very poor), 2 (poor), 3 (fair), 4 (fairly good), 5 (good), 6 (very good), 7 (excellent), to 8 (outstanding).

2.9. ANALYSIS OF PHYSICAL PROPERTIES:

All the samples were conditioned in the standard atmospheric condition for six hours. All tests were carried out according to the respective ASTM standards mentioned. Fabric weight (ASTM D 1910-59T), Fabric stiffness (ASTM D 1388-55T) and Fabric strength (ASTM D 1682-59T) were tested.

3. RESULTS AND DISCUSSIONS

3.1. COLOUR PRODUCED AND COLOUR STRENGTH:

The colour developed on the chitosan treated and untreated dyed samples are ascertained by CIELAB system coordinates. The change in colour of cotton fabric modified with different concentration of chitosan prior to dyeing is shown in Table III. According to the resultant values, the colours developed on chitosan treated and untreated dyed samples are light greenish yellow with a little trace of green and red shade (with L*a*b* value of 70.18 ~ 81.97 lightness, -0.32 ~ -2.10 green and only 2.37 red, 24.05 ~ 37.64 yellow). From the value of b*, it can be observed that the yellowness value of chitosan treated dyed samples slightly increase as compared to the untreated one. According to L* results, there is slight decrease in lightness values by increasing the concentration of chitosan.

TABLE III
COLOUR DEVELOPED AND COLOUR STRENGTH OF CHITOSAN TREATED AND UNTREATED DYED SAMPLES

Sample Code	CIEL*a*b* System					*ΔE**	K/S	Colour Strength (%)
	L*	a*	b*	C*	h°			
C_{00}	-	-	-	-	-	-	-	-
C_{01}	81.97	-2.10	24.05	24.14	94.98	-	0.08	100
C_{11}	77.08	-0.32	29.21	29.21	90.64	7.33	0.14	175
C_{21}	75.48	-0.48	35.16	35.16	90.78	12.97	0.16	200
C_{31}	70.18	2.37	37.64	37.72	86.40	18.54	0.25	313

t can be found that K/S values of chitosan treated dyed samples are greater than that of the untreated dyed sample. As the concentration of chitosan increases, the dye uptake also increases. It is learnt that cotton fibres form cross linking with chitosan resulting positive dyesites on the fibre surface. The reason is that the enhancement of dye absorption occurred due to the higher concentration of chitosan and also due to more dyesites on fabric surface. Making reference to these results, it can be anticipated that the chitosan has a positive effect in textile dyeing processes. Based on chroma values and K/S values, maximum colour saturation and maximum absorption are achieved on 1.5% chitosan treated dyed samples. Also, the colour strength of chitosan treated dyed samples is higher than that of untreated dyed one and the maximum colour strength is achieved on 1.5% chitosan concentration. It can be found that the chitosan present in the fabric enhances the dyesite causing deeper shade.

3.2. FASTNESS PROPERTIES:

Colour fastness results of chitosan treated and untreated dyed samples are revealed in Table IV.

TABLE IV
SUMMARY OF TEST RESULTS ON FASTNESS PROPERTIES OF CHITOSAN TREATED AND UNTREATED DYED SAMPLES

Sr. No.	Sample Code	Washing Fastness		Light Fastness
		Change of Shade	Staining on Cotton	
1	C_{00}	-	-	-
2	C_{01}	1	5	4
3	C_{11}	2	5	4-5
4	C_{21}	2-3	5	4-5
5	C_{31}	2	5	4-5

Based on the resultant data, it can be seen that staining on cotton fabric reveals good fastness grade for both chitosan treated and untreated dyed samples. Although the change of shade rating of chitosan treated dyed samples is higher than that of the untreated dyed sample, the ratings show poor in washing fastness except the sample treated with 1% chitosan. This may be due to the fact that the temperature used in washing fastness test is beyond the temperature used in dyeing process. Because cold dyeing method was used in this research, it is shown that the chitosan treated dyed fabrics should not be washed in warm water. As for light fastness property, acceptable results can also be achieved in all samples.

3.3. PHYSICAL PROPERTIES:

Since chitosan is a high molecular weight polymer, its application on cotton fabric can affect the physical properties. Therefore, some related physical properties of chitosan treated and untreated dyed samples are analyzed and the summary of results is described in Table V.

TABLE V
SUMMARY OF TEST RESULTS ON PHYSICAL PROPERTIES OF CHITOSAN TREATED AND UNTREATED DYED SAMPLES

Sr. No.	Sample Code	Fabric Weight (g/m^2)	Weight Add-on (%)	Breaking Strength (kg)		Bending Length (cm)	
				Warp	Filling	Warp	Filling
1	C_{00}	102.30	-	52.50	27.80	1.30	0.92
2	C_{01}	105.40	3.03	47.70	27.40	1.35	0.97
3	C_{11}	106.18	3.79	53.40	32.70	1.59	1.10
4	C_{21}	106.95	4.35	57.60	32.90	2.14	1.45
5	C_{31}	107.73	5.31	58.10	34.80	2.32	1.54

The weight of the untreated dyed sample is greater than that of the original one and also the weight of chitosan treated dyed samples increases as the concentration of chitosan increases. This is due to the increase in weight add-on percent as the increase in the concentration of chitosan. According to the resultant data, it can be observed that the breaking strength in the warp direction of the untreated dyed sample is less than that of the original fabric. But there is not much difference in breaking strength in filling direction. On the other hand, the breaking strength of chitosan treated dyed samples is greater than that of the original fabric and also the untreated dyed sample in both warp and filling directions. In such case, the highest breaking strength is achieved with 1.5% chitosan concentration. There is no strength loss and even increase in strength because of chitosan treatment. Therefore, depletion in strength due to natural dyeing with alum as mordant could be tolerated.

As for fabric stiffness, the bending length in both warp and filling direction of chitosan treated dyed samples is considerably greater than that of the original and the untreated dyed samples. It can be seen that the bending length of chitosan treated dyed sample increases with the increase in percent concentration of chitosan.The results are shown in Figure 3, Figure 4 and Figure 5 respectively.

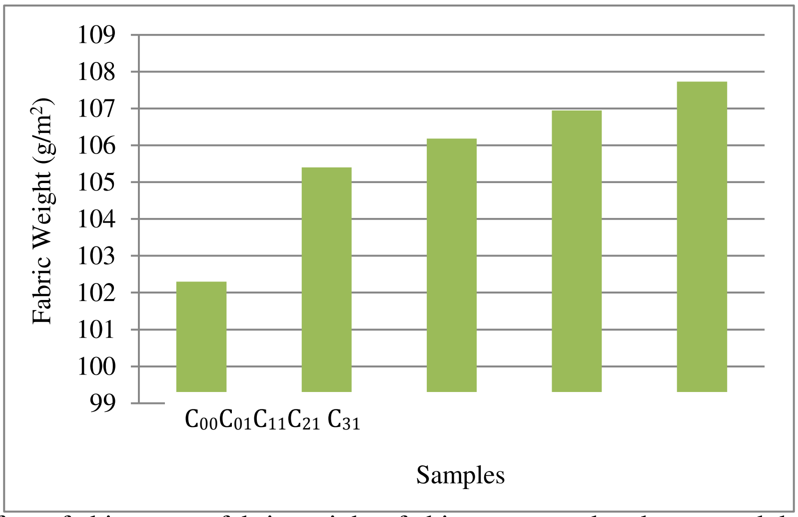

Figure 3. Effect of chitosan on fabric weight of chitosan treated and untreated dyed samples

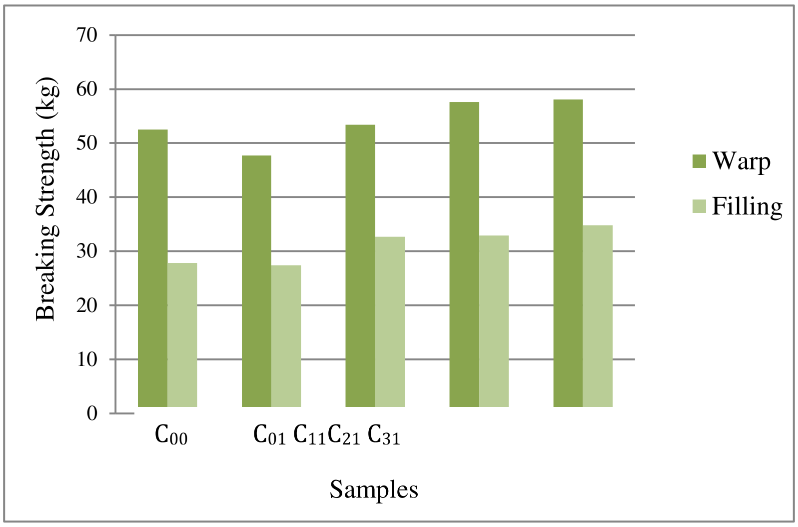

Figure 4. Effect of chitosan on breaking strength of chitosan treated and untreated dyed samples (warp and filling directions)

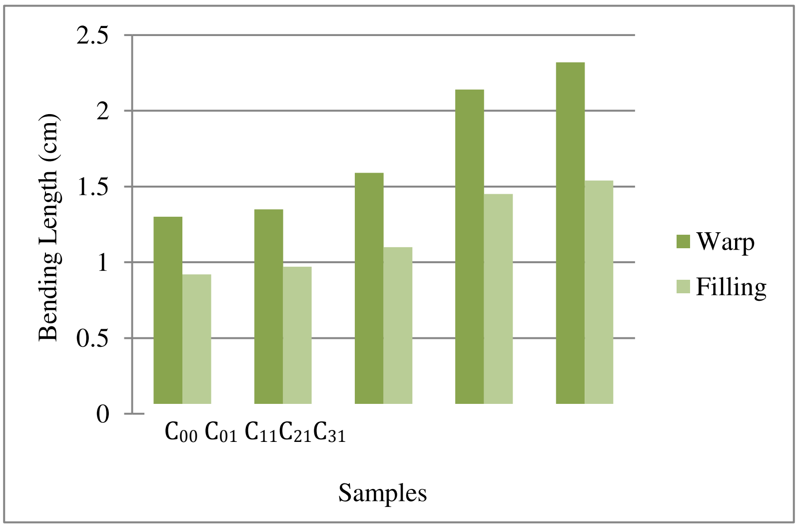

Figure 5. Effect of chitosan on fabric stiffness of chitosan treated and untreated dyed samples (warp and filling directions)

4. CONCLUSIONS:

In this research, the use of chitosan on dyeability of cotton fabric has been examined. For this intention, the cotton fabric was treated with varying concentrations of chitosan and dyed with natural dye extract from Indian almond leaves. It was found that the pretreatment of cotton fabric with chitosan enhances the dye uptake. Also the colour strength of chitosan treated dyed samples improved significantly compared with that of the untreated dyed samples. Based on the K/S values, it was observed that the higher depth of shade of chitosan treated dyed samples developed as the concentration of chitosan increases. As for change of shade in washing fastness test, the better fastness result is achieved with 1% chitosan concentration and there is no stain on adjacent cotton fabric by applying natural dye extract. Colour fastness properties of chitosan treated dyed samples are good in light fastness. Additionally, the breaking strength of chitosan treated dyed samples was also improved in both warp and filling directions. On the other hand, there is somehow affect on fabric stiffness due to chitosan treatment.

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