

## Low-Stress Physical/Mechanical Properties of Cochineal-dyed Cotton, Silk, Nylon, and Polyester Fabrics subjected to Chitosan-Pretreatment

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### Abstract

Chitosan has been widely applied to the products in various industries such as textile fabrics, apparels, foods, medical area, etc. Cochineal has long been employed as one of natural dyestuffs in the textile industry. The effect of chitosan pre-treatment on the low-stress physical and mechanical properties of cochineal-dyed fabrics including cotton, silk, nylon and polyester fabrics was investigated in this study. The chitosan treatment and mordanting of the fabrics changed the bending, shear, compression, and surface properties of the fabrics. In cotton fabric specimens, while the increase of B(bending rigidity) of cotton is relatively high, the increase of G(shear rigidity) of cotton is relatively low. In nylon and PET fabric specimens, while the increase tendency of B is relatively low, that of G is high compared to the corresponding cotton fabrics.

**Key words:** chitosan, cochineal, low-stress, mordant

### I. Introduction

Recent years, environment-friendliness or “green” oriented product development/marketing has become an important issue in the textile industry. Cochineal, a natural dyestuff, is a red dye manufactured from the dried bodies of the female *Dactylopius coccus*. The animal is a scale insect that manufactures a deep maroon pigment and stores this pigment in body fluids and tissues. This precious dyestuff was available to the Aztecs and continues to be applied in foods, drinks, cosmetics, and pharmaceuticals. On top

of this, the pigment is known to be a natural ant repellent<sup>1)</sup>. The dyestuff develops different colors depending on various mordanting processes as well as the pH value of the dye bath.

Peirce<sup>2)</sup> was the pacesetter in the investigation of the relationship between fabric hand and mechanical properties. His works led to the development of various measuring equipment.

Kawabata<sup>3)</sup> developed a series of instruments called the KES-FB instruments to accurately measure bending, compression, tensile and shear, and surface properties at low levels of strain similar to the deformations involved when a

person handles a fabric. These low-stress mechanical/physical property measurements are important to product development and needed for effective communication among textile mills, finisher, and apparel manufacturers.

Chitosan is widely used in the fields of textiles, food processing, medical use, and cosmetics. It is also known as an antimicrobial polysaccharide due to the amino group.

The chitosan treatment has been reported to impart fabric samples crisp and stiff hand<sup>4)</sup>. Moreover the chitosan, in the textile finishing processes, has been widely employed as an antibacterial agent on the condition that the chitosan meets specific molecular weight requirement for the antibacterial action. The treatment effects are especially needed for suitings worn in hot and humid weather, or for hospital uses.

The purpose of this study is to investigate the effect of chitosan pre-treatment and cochineal dyeing on the low-stress physical and mechanical properties of fabrics.

## II. Samples and Experiments

### 1. Fabric samples and chemicals

#### 1) Fabric Samples

Fabric specimens were white standard cotton, polyester, nylon, and silk fabrics conforming to the KS K 0905 specifications for dyeing fastness test<sup>6)</sup>. These were purchased from the KATRI (Korea Apparel Testing and Research Institute).

#### 2) Dyestuffs

Cochineal dyestuffs were purchased from Mikwang International Co. in powder form.

#### 3) Mordants

Copper sulfate ( $\text{CuSO}_4 \cdot 5\text{H}_2\text{O}$ , Duksan Pure Chemicals, Korea) was employed as a copper mordant.

#### 4) Chitosan

Chitosan was prepared in the laboratory. It has the characteristics of Mn (number average molecular weight) 95,600, Mw (weight average molecular weight) 120,000, and polydispersity of 1.26. The readings were obtained using a GPC (Gel permeation chromatography). The degree of de-acetylation was determined as 100%.

## 2. Experimental procedures

### 1) Preparation of acetic acid solution of chitosan

Chitosan was dissolved in 1%(w/w) acetic acid solution using a mechanical stirrer for 6 hours resulting in 0.7% chitosan solution. Chitosan solution was applied to the fabric immediately after the dissolution in order to minimize the possible chitosan chain breakage.

### 2) Chitosan treatment of fabric specimens

Specimen size of 30×30cm was prepared for chitosan treatment. The cut specimens were dipped in the prepared chitosan solution for 6 hours. The fabric specimens were treated using a mangle roller (Werner Mathis AG, Switzerland) with its pressure automatically adjusted. The wet pick up ratios were 95%, 25%, 45%, and 130% for the cotton, polyester, nylon, and silk fabrics, respectively. A lab tenter was employed for heat-treatment of the treated fabrics.

### 3) Mordanting treatment

1%(o.w.f.) mordant was added to a bath of de-

ionized water of liquor ratio 1:75. Fabric specimen was dipped in the bath at 40°C bath temperature. The bath temperature was steadily raised from 40 to 60°C for 20 minutes. The fabric specimen was further mordanted for 40 minutes at 60°C, and the bath was cooled down to room temperature.

#### 4) Dyeing of fabric specimen

Cochineal was used to prepare for dyeing solutions of liquor ratio 1:75. After water bath was heated to reach 40°C, specified amount of dyestuff was introduced to the warm bath and solubilized. Three pieces of 30 × 30cm fabric specimen were dipped in the bath and the dyeing temperature of 60°C was maintained for 60minutes. When the dyeing is finished the dye liquor was cooled down to room temperature. The dyed specimens were rinsed with enough de-ionized water and air-dried.

#### 5) KES measurements

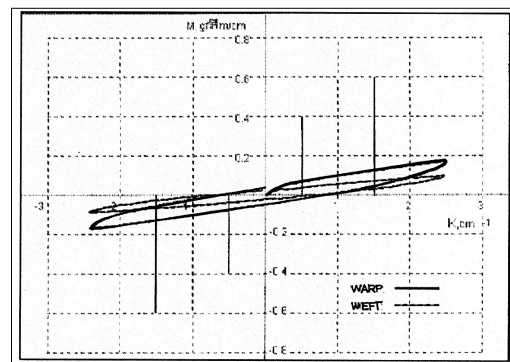
The fabric specimens were cut in the size of 20 × 20cm. Low stress physical/mechanical properties were measured using the KES-FB(Kawabata Evaluation System for Fabrics) series equipment. The properties including tensile, shear, bending, compression, surface friction and surface contour (roughness) were measured with the equipment. Stress-strain curves, similar to those from a universal tensile tester, are generated except that the peak load readings are much lower. The readings are comparable to those encountered in the actual wearing. Specimens are subjected to stress until a specified load is reached. The stress is then gradually reduced. In the shear test, effective size of the test specimen is 20 × 5 cm. Starting from the initial position, the specimen is shear to 8

degrees in one direction and then the shearing motion is reversed in order to get the information on recovery behavior.

In bending test, the fabric specimen is bent in the warp and filling directions. In the compression test, a specified area of the specimen is subjected to a peak pressure and the pressure is gradually reduced. The pressure is applied using a movable plunger that slowly moves up and down. In the surface test, two independent probes are used to record the geometric roughness of the fabric specimen and the friction coefficient (MIU) during the specimen's movement underneath the two probes.

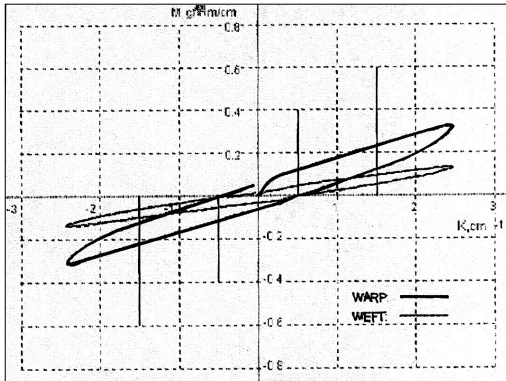
### III. Results and Discussion

When the fibers comprising a yarn, either warp or weft, are bonded together, the yarn tends to make the structure stiff. As in the cases of a sized yarn or a highly twisted yarn, these exhibit higher bending stiffness than their counterparts, an unsized or a low twisted yarn. The chitosan treatment imparts the fabric higher bending stiffness as shown in <Figs. 1 and 2>. Chitosan treated and unmordanted fabric sample shows



<Fig. 1> KES bending chart, Cotton, control.

higher slope than the control cotton fabric. In <Tables 1 and 2>, bending rigidity (B) of the control cotton fabric increased 80.6% after chitosan treatment (0.042 to 0.076). B of the control silk fabric increased 133.3% after



<Fig. 2> KES bending chart, cotton, chitosan treated and unmordanted fabric

<Table 1> KES Bending properties of fabric samples

Chitosan	Untreated	Treated	Untreated	Treated	
Cu Mordant	Untreated		Treated		
B	Cotton	0.0423	0.0764	0.0384	0.0429
	Silk	0.0102	0.0238	0.0094	0.0099
	Nylon	0.0339	0.0377	0.0340	0.0353
	PET	0.0621	0.0688	0.0619	0.0581
2HB	Cotton	0.0600	0.0750	0.0553	0.0618
	Silk	0.0027	0.0090	0.0025	0.0029
	Nylon	0.0290	0.0283	0.0256	0.0249
	PET	0.0331	0.0330	0.0306	0.0164

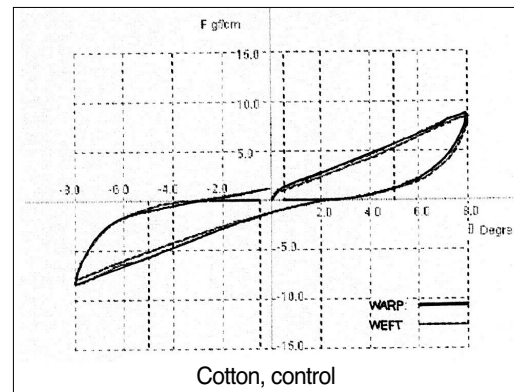
<Table 2> KES Bending rigidity change and the chitosan wet pick-up ratio

Sample	Wet pick-up ratio (%)	B Increase (%)
cotton	95	80.6
silk	130	133.3
nylon	45	11.2
PET	25	10.8

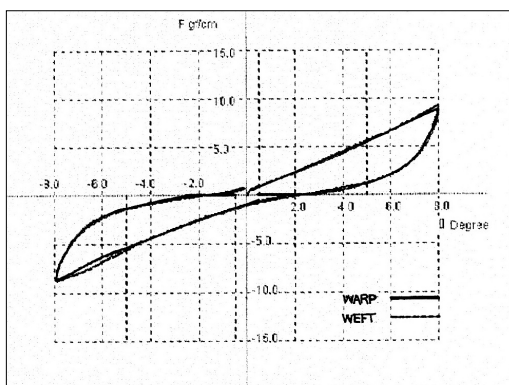
chitosan treatment. However, B of nylon control fabric increased 11.2%, B of PET control fabric increased 10.8% after the chitosan treatment. This may be explained from the fact that there were wet pick-up ratio differences among the fabrics. The higher the wet pick-up ratio, the higher the B increase rate.<Table2> Due to the differences in fiber composition, yarn and fabric structural differences, the pick-up ratio became different with the selected mangle setting.

Mordanting process, in this study, seemed to have lowered the B values and 2HB values. During the wet treatment, some structural relaxation in the fabric weave or yarn tightness might have occurred in the specimens.

<Figs. 3 and 4> show the shear deformation curves of cotton control fabric specimen and chitosan treated unmordanted cotton fabric specimen. The two charts are, overall, relatively similar in shape. The increase of the shear rigidity(G) is low (4.7%). When we compare the shear and bending charts of corresponding cotton fabrics, however, the increase of the slope is readily noticeable (80.6% increase of B) in bending deformation curves of the cotton control and chitosan treated unmordanted cotton fabrics.



<Fig. 3> KES shear chart, cotton, control



<Fig. 4> KES shear chart, cotton, chitosan treated and unmordanted fabric

<Figs. 1 and 2> It is postulated that the chitosan is impregnated within the yarns comprising fabric sample in the case of cotton fabrics, and that the region of the intersection of the warp and weft is not adhered together by the chitosan treatment, ensuring relatively free movement of the warp and weft yarns during the shear deformation movement of the fabric within 8 degrees. Even if the chitosan wet pick-up ratios are low in cases of nylon and PET fabrics, the G increase rates are relatively high compared to the corresponding cotton fabrics. (20.3% and 14.9%, respectively)

<Table 3> In the case of synthetic filament yarns (nylon and PET), the chitosan might not have evenly permeated due to their low surface affinity resulting in higher chitosan coating on the yarn surface than inside the yarn. This could have resulted in the adhesion of warp and weft yarns at the intersection region, thereby higher shear rigidity than the corresponding cotton samples. It may be interesting if the effect is investigated in terms of fiber-chitosan-cochineal system in a continuing study.

The G value of control nylon fabric is 0.59. After the Cu mordanting, the G value increased to 0.70 (18.6%), which is high compared to other three

<Table 3> KES shear properties of fabric specimens

Chitosan		Untreated	Treated	Untreated	Treated
Cu Mordant		Untreated		Treated	
G	Cotton	0.86	0.90	0.88	0.88
	Silk	0.21	0.23	0.21	0.21
	Nylon	0.59	0.71	0.70	0.71
	PET	1.41	1.62	1.36	0.81
2HG	Cotton	2.49	1.86	2.63	2.50
	Silk	0.09	0.11	0.09	0.11
	Nylon	2.16	2.30	2.21	2.01
2HG5	PET	2.45	1.56	2.21	1.29
	Cotton	4.56	4.36	4.68	4.65
	Silk	0.22	0.34	0.21	0.24
	Nylon	3.63	4.51	4.19	4.31
	PET	7.16	6.97	6.61	3.64

fabric specimens. The G value of cotton after Cu mordanting barely increased. That of silk is identical even after Cu mordanting, and that of PET decreased down to 1.36 from 1.41(3.6%). The Cu mordanting does not seem to affect the G value of fabrics in general.

<Table 4> shows the tensile properties of fabrics.

WT (tensile energy) can be interpreted as another measure of softness. The higher the WT value, the easier to elongated the fabric sample under specified amount of load. Therefore, cotton, silk, and nylon fabrics after the chitosan treatment without mordanting may be regarded as stiffer based on the WT value change. The WT values of mordanted cotton fabric samples, either chitosan untreated or chitosan treated, increased up to 0.66. The EMT values of cotton after the chitosan treatment without mordanting decreased appreciably from 2.24 to 1.56.

<Table 5> shows the compression properties of chitosan treated, and mordanted fabrics dyed

with cochineal. WC (compression energy), generally, decreased after the chitosan treatment without mordanting. This could be interpreted that the fabric structure became stiffer. To (initial thickness) and Tm (thickness at Pm) values of silk and PET fabrics after the chitosan treatment without mordanting increased appreciably.

<Table 6> shows the surface friction coefficient (MIU), mean deviation of MIU (MMD), and surface roughness (SMD). MIU values of chitosan treated and unmordanted fabrics decreased compared to the control fabrics. This means that the chitosan coating lowered the surface friction and made the fabric feels slippery. The same tendency appeared in the Cu mordanted fabrics series. However, the Cu mordant treatment increased the MIU of the cotton and nylon fabrics.

<Table 4> KES tensile properties of fabric specimens

Chitosan		Untreated	Treated	Untreated	Treated
Cu Mordant		Untreated		Treated	
LT	Cotton	0.964	0.920	0.978	0.848
	Silk	0.907	0.711	0.874	0.864
	Nylon	0.856	0.900	0.904	0.711
	PET	0.824	0.608	0.825	0.837
WT	Cotton	0.55	0.38	0.66	0.66
	Silk	0.65	0.44	0.53	0.52
	Nylon	0.18	0.13	0.15	0.13
RT	PET	0.07	0.08	0.08	0.09
	Cotton	42.7	53.7	42.7	44.2
	Silk	68.7	187.3	78.8	72.9
EMT	Nylon	51.9	45.2	45.8	48.6
	PET	41.7	44.3	60.0	61.4
	Cotton	2.24	1.56	2.66	2.97
EMT	Silk	3.43	2.33	2.60	2.61
	Nylon	0.88	0.64	0.63	0.65
	PET	0.34	0.46	0.37	0.42

<Table 5> KES compression properties of fabric specimens

Chitosan		Untreated	Treated	Untreated	Treated
Cu Mordant		Untreated		Treated	
LC	Cotton	0.562	0.658	0.600	0.374
	Silk	0.667	0.563	0.667	0.137
	Nylon	0.737	0.480	0.558	0.115
	PET	0.857	0.667	0.800	0.100
WC	Cotton	0.061	0.049	0.057	0.057
	Silk	0.010	0.009	0.009	0.010
	Nylon	0.007	0.006	0.005	85.0
RC	PET	0.003	0.004	0.004	83.3
	Cotton	40.8	53.4	48.2	50.0
	Silk	53.3	92.6	66.7	63.3
	Nylon	81.0	82.4	92.9	85.0
To	PET	88.9	75.0	90.9	83.3
	Cotton	0.583	0.481	0.554	0.554
	Silk	0.164	0.181	0.166	0.137
	Nylon	0.139	0.137	0.132	0.115
Tm	PET	0.105	0.117	0.110	0.110
	Cotton	0.366	0.332	0.364	0.374
	Silk	0.134	0.149	0.139	0.137
	Nylon	0.120	0.112	0.115	0.115
Tm	PET	0.098	0.105	0.100	0.100

<Table 6> KES Surface properties of fabric specimens

Chitosan		Untreated	Treated	Untreated	Treated
Cu Mordant		Untreated		Treated	
MIU	Cotton	0.131	0.124	0.145	0.133
	Silk	0.156	0.116	0.154	0.148
	Nylon	0.109	0.106	0.117	0.101
	PET	0.099	0.091	0.097	0.092
MMD	Cotton	0.0168	0.0157	0.0175	0.0190
	Silk	0.0148	0.0195	0.0131	0.0141
	Nylon	0.0138	0.0162	0.0149	0.0148
	PET	0.0132	0.0136	0.0123	0.0130
SMD	Cotton	5.896	5.420	5.095	5.005
	Silk	2.722	2.710	2.095	2.517
	Nylon	3.240	3.391	3.298	3.486
	PET	3.508	3.792	3.735	3.948

#### IV. Conclusions

The effect of chitosan pre-treatment on the low-stress physical and mechanical properties of cochineal-dyed fabrics including cotton, silk, nylon and polyester fabrics was investigated in this study. The chitosan treatment and mordanting of the fabrics changed the bending, shear, compression, and surface properties of the fabrics. In cotton fabric specimens, while the increase of B of cotton is relatively high, the increase of G of cotton is relatively low. In nylon and PET fabric specimens, while the increase tendency of B is relatively low, that of G is high compared to the corresponding cotton fabrics. It might be interesting if this effect is investigated in terms of fiber-chitosan-cochineal system in a subsequent study.

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