# Non-Formaldehyde Wrinkle Resistant Finishing on Silk Fabric with Polycarboxylic Acids

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***Abstract:*** *In the present study, an attempt is made to produce the wrinkle resistant silk fabric by using Citric acid an1, 2, 3, 4-Butanetetracarboxylic acid (BTCA) as non-formaldehyde crosslinking agents, Magnesium chloride as a catalyst and Polyethylene emulsion as a softener. Nine different concentrations of wrinkle resistant finishing solution are prepared and applied on silk sample fabrics using pad-dry-cure process. And then, the properties of the treated fabrics are analysed and compared with the untreated fabric. The results show that the non-formaldehyde crosslinking agents increase obviously the wrinkle properties of the silk fabric. The sample fabric treated with the mixture of 3% Citric acid and 3% BTCA gives the highest wrinkle recovery angle of 173˚ in warp direction and 168˚ in filling direction, respectively.*

***Key Words:*** *wrinkle resistant, silk fabric, citric acid, 1, 2, 3, 4-butanetetracarboxylic acid (BTCA), wrinkle recovery angle*

## 1. INTRODUCTION:

Silk is one of the most luxurious and beautiful natural fabric, possessing excellent lustre, wearing comfort, soft handle, good air permeability, excellent draping quality and aesthetic appearance in materials. However, silk fabric has low wet and dry resiliency. Hence the fabric can be wrinkle easily during home laundering or when wet. To improve these performance properties, silk fabric is required to be given chemical treatment known as wrinkle-resistant finishing [3].The recovery of wrinkles depend on crosslinks which hold closest molecular chains together and pull them back into position after the fiber is bent, so it preventing the formation of a wrinkles [3]. The wrinkle resistant finishing can be carried out by using formaldehyde based resins as a crosslinking agent. But the formaldehyde based resins are known as human carcinogen. For this reason, Polycarboxylic acids are used as substitutes for these resins. This research is concerned with the finishing of silk fabric by using Citric acid, 1, 2, 3, 4-Butanetetracarboxylic acid, and the mixture of two acids in order to obtain wrinkle resistant effect on silk fabric. Magnesium chloride and Polyethylene are used as catalyst and softener, respectively. Silk fabric is finished separately with different concentrations of Citric acid, 1, 2, 3, 4-Butanetetracarboxylic acid, as well as with the mixture of these two Polycarboxylic acids. After finishing, the treated silk fabric samples are analysed, compared and discussed.

## 2. MATERIALS AND METHODS:

### *2.1 Fabric Specification*

The silk fabric is purchased from Amarapura Township in Mandalay region. The construction properties of the silk fabric include the followings: warp count 45 Denier, weft count 45 Denier ends per cm 29, picks per cm 29 and the weight 43.07 $g/m^2$.

### *2.2 Conditioning of Fabric Samples*

All tests are carried out in the standard atmospheric condition and relative humidity at (20$\pm$ 2˚C, 65$\pm$ 2% RH) in the laboratory of Textile Testing and Quality Control of the Department of Textile Engineering, Yangon Technological University.

### *2.3 Preparation of Sample Fabrics for Wrinkle Resistant Finishing Treatment*

The sample fabrics [13 in×15 in(33.02 cm×38.10 cm)] are prepared for the wrinkle resistant treatments. The padder mangle and Auto mini tenter are used for padding, drying and curing the fabric samples.

### *2.4 Preparation of Wrinkle Resistant Finishing Treatment Solution*

Nine different concentrations of crosslinking solution, 2%, 4%, 6% and 8% of Citric acid (o.w.f), 2%, 4%, 6% and 8% of BTCA (o.w.f) and a mixture of 3% of Citric acid (o.w.f) and 3% of BTCA (o.w.f) are prepared respectively with 3% of magnesium chloride (o.w.f) and 5% of polyethylene emulsion (o.w.f). A material to liquor ratio of 1:20 is used in this study. Designation of samples and conditions for wrinkle resistant finish are shown in Table 1.

Table1. Designation of Samples and Recipes for Wrinkle Resistant Solution

| Sample Code | Crosslinking agent % (o.w.f) | Magnesium chloride % (o.w.f) | Polyethylene % (o.w.f) | M:L |
|---|---|---|---|---|
| $C_1$ | Citric acid 2% | 3% | 5% | 1:20 |
| $C_2$ | 4% | 3% | 5% | 1:20 |
| $C_3$ | 6% | 3% | 5% | 1:20 |
| $C_4$ | 8% | 3% | 5% | 1:20 |
| $B_1$ | BTCA 2% | 3% | 5% | 1:20 |
| $B_2$ | 4% | 3% | 5% | 1:20 |
| $B_3$ | 6% | 3% | 5% | 1:20 |
| $B_4$ | 8% | 3% | 5% | 1:20 |
| D | Citric acid 3% and BTCA 3% | 3% | 5% | 1:20 |

### *2.5 Wrinkle Resistant Finishes on Silk Fabric*

Firstly, the sample fabrics are immersed separately in the different concentrations of crosslinking solution which contained Citric acid, magnesium chloride and polyethylene. And then the impregnated fabrics are passed through the squeeze rolls to give wet pick-up of 80% on the weight of the fabric by using padder mangle. After that, the treated samples are dried at the temperature of 85˚C for 5 minutes and cured at the temperature of 130˚C for 3 minutes in auto mini tenter. Then, the treated samples are washed in 5g/l of sodium carbonate for 5 minutes, rinsed in cold water and dried at room temperature.

Secondly, the sample fabrics are padded in the different concentrations of crosslinking solution of BTCA, in place of Citric acid, with magnesium chloride and polyethylene. And then, the treatments are carried out according to the procedure described above.

Thirdly, the sample fabric is padded in the crosslinking solution of the mixture of Citric acid and BTCA, magnesium chloride and polyethylene under the same condition and procedure described above.

And then all of the samples are analysed and tested to investigate the effect of wrinkle resistant finish on physical properties of silk fabric.

## 3. RESULTS AND DISCUSSIONS:

### *3.1 Effect of Wrinkle Resistant Finish on some Physical Properties of Silk Fabric*

The properties of the treated fabrics such as fabric thickness, fabric weight, wrinkle recovery angle, air permeability, breaking strength and fabric stiffness are tested to evaluate the effect of wrinkle resistant treatment on silk fabric.

All tests are carried out according to the respective AATCC and ASTM standard test methods. The test results of untreated and treated fabric are shown in Table 2.

Table2. Test Results of the Untreated and Treated Samples

| Sample Code | Wrinkle Recovery Angle (˚) | | Fabric Thickness (mm) | Fabric Weight ($g/m^2$) | Air Permeability ($cm^3/cm^2/sec$) | Breaking Strength (kg) | | Overall Flexural Rigidity (mg-cm) |
|---|---|---|---|---|---|---|---|---|
| | Warp | Filling | | | | Warp | Filling | |
| Untreated | 86.95 | 92.30 | 0.13 | 43.07 | 226.20 | 45.30 | 42.20 | 4.30 |
| $C_1$ | 120.50 | 120.65 | 0.12 | 44.02 | 199.65 | 39.70 | 40.80 | 4.91 |
| $C_2$ | 131.75 | 134.50 | 0.12 | 46.73 | 198.90 | 35.25 | 36.00 | 9.71 |
| $C_3$ | 170.25 | 170.30 | 0.14 | 46.89 | 197.30 | 37.00 | 30.75 | 4.04 |
| $C_4$ | 144.75 | 145.25 | 0.13 | 46.98 | 197.00 | 25.05 | 28.55 | 6.95 |
| $B_1$ | 121.20 | 125.45 | 0.13 | 43.44 | 196.95 | 28.25 | 27.60 | 8.03 |
| $B_2$ | 139.50 | 131.15 | 0.13 | 43.65 | 195.80 | 29.25 | 24.30 | 9.82 |
| $B_3$ | 165.75 | 164.75 | 0.13 | 44.00 | 194.60 | 23.50 | 24.05 | 7.33 |
| $B_4$ | 123.75 | 134.60 | 0.13 | 45.97 | 193.80 | 20.60 | 20.25 | 7.80 |
| D | 172.85 | 168.25 | 0.14 | 49.96 | 191.95 | 29.15 | 30.60 | 9.33 |

*(1) Effect of Treatment on Wrinkle Recovery Angle*

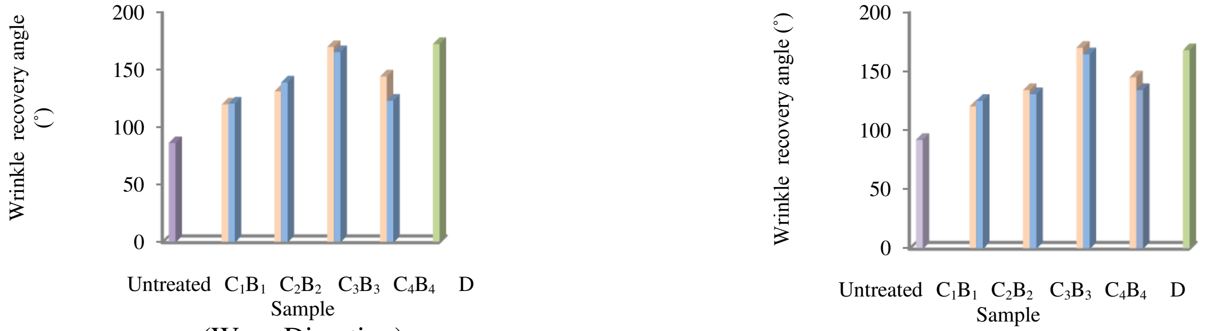
Wrinkle recovery is the property of a fabric that enables it to recover from folding deformations. In this study, wrinkle recovery angle is determined according to the standard method of AATCC (66-2008) by using Shirley Crease Recovery Tester. This test method is used to determine the wrinkle recovery of woven fabrics. It is applicable to fabrics made from any fibre, or combination of fibres.

The wrinkle recovery angle of the treated sample is obviously increased by wrinkle resistant finishing. By observing on Fig.1 (a) and (b), the wrinkle recovery angle is considerably increased at the 6% concentration of crosslinking solutions. It may be due to the fact that crosslinking agent produces crosslinks among fibroin molecules of the silk and masks the free hydroxyl side groups, which in turn reduces the swelling and subsequently improves the wrinkle resistant properties of treated fabrics.

In the case of both Citric acid and BTCA, with the increase of the concentration of crosslinking agents to 6%, the wrinkle recovery angle increases up to 170.25˚ and 165.75˚in warp direction and 170.30˚ and 164.75˚ in filling direction, while the use of 8% of crosslinking agents leads to the wrinkle recovery angle of 144.75˚ and 123.75˚ in warp direction and 145.25˚ and 134.60˚ in filling direction, respectively.

Beyond the 6% concentration of crosslinking agent, the decrease in wrinkle recovery angle can be observed at the sample $C_4$ and $B_4$ in both directions. It may be due to inadequate degree of crosslinking during the curing period. Another reason may be the fact that the fabric has an exceed crosslinking with polycarboxylic acid and the elasticity of fabric is decreased which in turn leads to the decrease of wrinkle recovery angle.

Use of BTCA is more costly than Citric acid. In order to reduce the expense of using BTCA, the required amount of BTCA in wrinkle resistant finish is reduced by mixing with Citric acid. Based on 6% of the total crosslinking agent concentration, wrinkle resistant solution is made by mixing 3% Citric acid with 3% BTCA since the maximum wrinkle recovery angle values is obtained at 6% of Citric acid and also at 6% of BTCA, respectively. The sample D gives the highest wrinkle recovery angle of 172.85˚ in warp direction.




(Warp Direction) (Filling Direction)

Fig.1 The Effect of Treatment on Wrinkle Recovery Angle

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*(2) Effect of Treatment on Fabric Thickness*

The thickness of a fabric is dependent on its mass per unit area, the type of yarns used, the weave structure and the finish. The effect of wrinkle resistant finish on the thickness of treated samples is shown in Fig.2. According to the test results shown in Table.2, the only BTCA crosslinking solution does not affect on the thickness of silk fabric. The thickness values of the treated fabric samples $C_3$ and D are slightly increased whereas the thickness values of sample $C_1$ and $C_2$ are slightly decreased as compared to that of the untreated sample. It is found that the sample $C_3$ and D are thicker than the untreated sample.

The increase in thickness may be due to formation of a layer on the surface of the fabric and stress relaxation of the fabric after treatment. The reduction in fabric thickness may be due to degradation of fibre during treatment and low concentration of Citric acid which is not enough to form a layer on the surface of silk fabric.

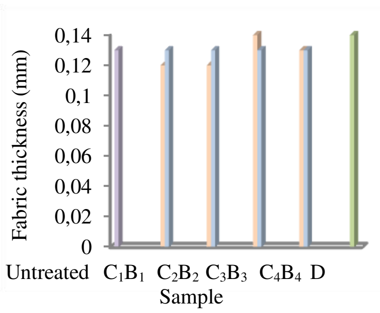



Fig.2 Effect of Treatment on Fabric Thickness

*(3) Effect of Treatment on Fabric Weight*

An important property of a fabric is its weight that is a criterion of the amount of material contained in it. Table.2 shows the mean values of the weight of the untreated and treated fabric samples. The results show that Citric acid and BTCA solution affect on the weight of the sample fabric. With the increase of the concentration of acids, the weight of the treated fabrics is greater than that of the untreated fabric as shown in Fig.3. It is obvious that the weight of the treated fabric samples is slightly increased as the concentration of acid in the padding liquor.

The weight of the treated sample D is the highest among the treated samples. It may be due to the fact that wrinkle resistant finish forms a layer on the surface of fabric and makes the fabric more compact, which leads to increase in weight of fabric.

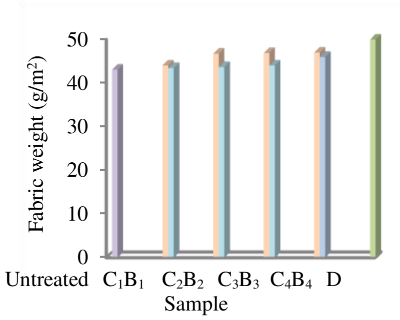



Fig.3 Effect of Treatment on Fabric Weight

*(4) Effect of Treatment on Air Permeability*

The air permeability of a fabric is the volume of air measured in cubic centimetres passed per second through $1cm^2$ of the fabric at a pressure of 1 cm of water. From the comfortable point of view, the greater the air permeability value of a fabric, the more the body comfort is.

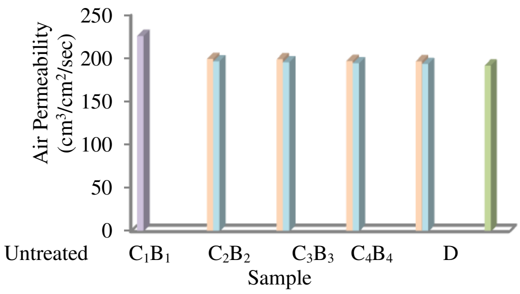


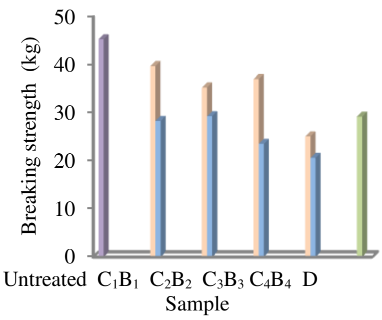

Fig.4 Effect of Treatment on Air Permeability

The results of air permeability of the treated samples are obviously reduced as compared to that of the untreated samples as shown in Table.2 and Fig.4. It may be due to the fact that the crosslinking agent fills partially the interstices between the yarns of the fabric that reduces the air permeability. Another reason may be that the surface coating is formed by wrinkle resistant finish.

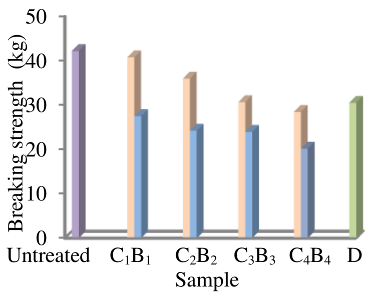
*(5) Effect of Treatment on Breaking Strength*

Breaking strength is a measure of the resistance of the fabric to a tensile load or stress in either warp or filling direction.

By observing on Fig. 5 (a) and 5 (b), the wrinkle resistant treated silk samples are significantly decreased in breaking strength when compared with the untreated silk sample. Apart from the sample $C_1$ in filling direction, the differences in breaking strength between the untreated sample and all other treated samples in both directions are greater than the least significant difference of 1.43 kg. It may be due to the acidity of the treatment solution and acid catalyzed depolymerisation during the curing process.




(Warp Direction)




(Filling Direction)

Fig.5 Effect of Treatment on Breaking Strength

It is learnt that silk fabric strength lower than 20 lb (9.09 kg) is not suitable to use for clothing [4]. From the results shown in Table.2, the breaking strengths in both warp and filling directions of the untreated silk fabric are 45.30 kg and 42.20 kg, respectively and after the finishing process the breaking strength decreases to 45% and 47.5% (20.6 kg and 20.25 kg) in warp and filling directions, respectively. In both directions, the breaking strength values of the BTCA treated samples are lower than that of the Citric acid treated samples at the same concentration. The higher the concentration of the crosslinking solution, the lesser the breaking strength of the treated samples. The breaking strength of the treated sample $B_4$ gives the lowest value among the other treated samples in both warp and filling directions. The breaking strength of the sample D is greater than that of the sample $C_4$, $B_3$ and $B_4$ in both directions.

*(6) Effect of Treatment on Fabric Stiffness*

The stiffness is also an important factor in obtaining high resistance to creasing. Fabric stiffness is defined as its resistance to bending. The results of the overall flexural rigidity of the untreated and treated samples are illustrated in Table.2. The results of overall flexural rigidity of the BTCA treated samples are greater than that of the Citric acid treated samples.

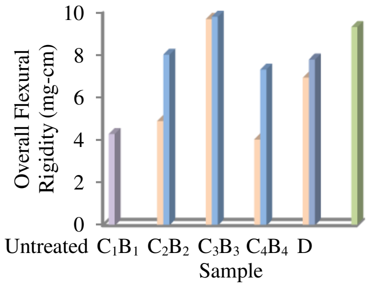



Fig.6 Effect of Treatment on Overall Flexural Rigidity

As seen in Fig.6, the results of overall flexural rigidity of the treated samples are influenced by the concentration of the crosslinking agent. The increase in overall flexural rigidity may be the fact that the crosslinks formed between the adjacent silk fibroin molecules control the degree of swelling and lower degree of swelling makes the fibre more compact and stiff. The greater the overall flexural rigidity, the stiffer the fabric is. The highest overall flexural rigidity is observed at 4% of the crosslinking solutions, $C_2$ and $B_2$.

## 4. CONCLUSIONS:

From this research, wrinkle resistant silk fabrics can be achieved by applying non-formaldehyde crosslinking agents with selective additives. It can be found that wrinkle resistant performance of both the Citric acid treated and BTCA treated fabrics reached the maximum when the concentration of the crosslinking agent solution is 6%. The treated samples $C_3$, $B_3$ and D give the higher wrinkle recovery angle values as compared to the other treated silk samples. The fabric weight and the overall flexural rigidity of the silk fabric are increased whereas the air permeability of the treated silk fabric is reduced due to non-formaldehyde wrinkle resistant finish. The breaking strength of the treated silk fabric is decreased in both warp and filling directions. The wrinkle resistant treated fabric results in moderate resistance to washing in this study.

## 5. RECOMMENDATIONS:

As for future study, an attempt should be done on the silk fabric by using other polycarboxylic crosslinking agents with different concentration at different curing temperature in order to produce the durable wrinkle resistant fabrics. In addition, other catalysts, softeners and additives should be used in wrinkle resistant treatment solution not only for improving the wrinkle recovery but also for retaining the strength and softness of the treated silk fabric.

## REFERENCES:

1. Bernard P. C. 1970. *Textiles Fibre to Fabric*. Sixth Edition. Gregg Division, McGraw-Hill Book Company
2. Ekstorm, D.E. 1928. *The Wearing Qualities of Certain Ready-made Garments*.B.S., Kansas State Teachers College, Emporia.

**Web references:**

- http://www.researchgate.net/publication/242148517 Effect of wrinkle resistance finish on cotton fabric properties
- http://www.inflibnet.ac.in/ojs/index.php/KJAS/article/view/956