

Effect of Washing and Subsequent Heat Treatment on Water Repellency and Mechanical Properties of Nylon 6, Triacetate and Silk Fabrics Treated with Hydrocarbon Resins

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Abstract— It is commonly known that water repellency of the fabric treated with fluorocarbon resin brings about a decrease by the washing and recovers by the subsequent heat treatment. In this article, effect of the water repellency was investigated on the nylon 6, triacetate and silk fabrics treated with hydrocarbon and silicon resins. Hydrocarbon and silicon resins have been widely used in the textile finishing as the softening and water proofing agents. The fabrics were treated with hydrocarbon resins, Paragium JQ and RC (Ohara Paragium Chemical Co.) and a silicon resin, Poron MR (Shinetsu Chemical Co.), and then washed and subsequently heat treated. Although the water repellency increased by the resin treatment, it decreased by the washing apparently and recovered a little by the heat treatment. The effect of the heat treatment was small comparing with that of the fluorocarbon resin. Furthermore, as a mechanical property of the treated fabric, KES shearing and bending hysteresis parameters, modulus and hysteresis width of the hydrocarbon resin-treated nylon 6, triacetate and silk fabrics decreased by the heat treatment after washing. Therefore, the treatment is effective at improving the softening of the fabric in water repellent finish.

Keywords: Repellency, hydrocarbon resin, textiles, washing and heat treatment, mechanical property

1. Introduction

It is well known that the surface of the fiber and fabric plays an important role in the textile finishing such as water repellency, wetting, adhesion, soil resistance, soil release, luster, electrostatic proofing and textile hand¹⁻⁷⁾. Fluorocarbon resins have widely used in textile finishing to improve water repellency and soil resistance. One of the problems is that the water repellency of the fluorocarbon resin-treated fabric is, as empirically well known, decreased by washing and recovered by heat treatment^{1,2)}. Although it has been explained based on the disturbance of the orientation of fluoroalkyl groups by washing¹⁾ and rotation of fluoroalkyl groups from surface to inside the polymer rejecting the hydrophilic environment³⁾. The details, however, are still unclear.

Previously, we investigated on the water repellency of the film and fabric by the treatment of the low

temperature plasma with carbon tetrafluoride (CF_4) gas²⁾ and of the fluorocarbon resin⁴⁾. The water and oil repellencies increased by the treatment considerably. The contact angle, critical surface tension and ESCA analysis were measured in relation to the water repellency during the washing and subsequent heat treatment.

The water repellency decreased by the washing and recovered considerably by the heat treatment. The behavior was considered on the basis of the critical surface tension by the Zisman plots and the extended Fowkes equation^{8,9)}, and by the ESCA analysis⁴⁾.

In this article, as an another water repelling agent, the nylon 6, triacetate and silk fabrics were treated with a commercial hydrocarbon and silicon resins by the pad/dry/cure procedure. The effect of the treatment on the water repellency after the washing and heat processings was evaluated by the JIS spray test method.

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Furthermore, to evaluate the mechanical property during the processing, KES shearing and bending parameters, the modulus and hysteresis width were obtained.

2. Experimental

As a fabric material, the nylon 6 Staner, triacetate Habutae, and silk Seikaplace (a kind of Chirimen) were treated with hydrocarbon resins, Paragium JQ (paraffin/zirconium acetate) and RC (octadecyl ethylene urea, Ohara Paragium Chemical Co., Ltd.) and silicon resin, Poron MR (Shinetsu Chemical Co.) by the following procedure. The fabrics were padded with 1 dip-1 nip in aqueous solutions containing 2% (JQ) or 3% (RC) of the resins, and then dried (105°C/ 45 s, JQ or 150°C/ 3 min, RC) and cured (160°C/1 min, JQ or 160°C/2 min.). Also the silk fabric was padded with an aqueous solution containing 3% of the resin, Poron MR and 1.5% of the catalyst, CAT-LZ-1, and then dried (120°C/45 s) and cured (160°C/1 min).

The fabrics were one time washed by the JIS L 0217-103 method, and subsequently heat treated under following conditions 80°C/3 min and 120°C/3 min with a pin tenter.

Water repellency of the fabrics was evaluated by the five steps of the JIS L 1092-98 6.2 spray method according to following method, 250 mL water sprayed onto fabric fitted on a 20 cm diameter circular frame rotating in a plane at 45° to the horizontal. The effect was evaluated with a pattern of the index between 1 and 5, where 1 represents complete wetting out and 5 total water repellency.

Furthermore, to obtain a parameter related to mechanical property of the fabric, KES (Kawabata Evaluation System F-7, Kato Tech) shearing and bending measurements were carried out, and the modulus G , B , and the hysteresis widths $2GH$, $2HG5$, and $2HB$ were obtained from the hysteresis curve.

3. Results and discussion

3.1 Water repellency

Table 1 shows the water repellency of the nylon 6 and triacetate fabrics treated with Paragium JQ and RC during the washing and subsequent heat processing. Although the repellency increased considerably by the JQ resin treatment, and decreased a little apparently after the washing, it recovered a little by the heat treatment. The effect of the JQ resin on the water repellencies of the fabrics are less smaller comparing with that of the fluorocarbon resin^{2,4)}.

Therefore, it is expected that change of the surface chemical composition of the fabric treated with hydrocarbon resin is comparably small during the washing and heat treatment.

Table 2. Water repellency of silk fabrics treated with various resins during washing and heat treatment

Treatment	Paragium JQ	Paragium RC	Poron MR
Untreated	1	1	1
Resin treated	5	3	5 ⁻
Washed and dried	1	1	4
Heat treated			
80°C/3 min	2	1	4
120°C/3 min	3 ⁻	1 ⁺	4 ⁺

Table 1. Water repellency of nylon 6 and triacetate fabrics treated with hydrocarbon resins during washing and heat treatment

Treatment	Nylon 6		Triacetate	
	Paragium JQ	Paragium RC	Paragium JQ	Paragium RC
Untreated	1	1	1	1
Resin treated	4	1	1	1
Washed and dried	1	1	1	1
Heat treated				
80°C/3 min	2	1	2	1
120°C/3 min	2 ⁺	1	3 ⁻	1

On the other hand, the RC resin treatment causes no change in the water repellency with both nylon 6 and triacetate fabrics, and by washing and heat treatment. It seems likely that Paragium RC having a octadecyl ethylene urea group is cellulose fiber-reactive resin and leads to show no effect at improving the water repellency of the fabrics.

From the result, it seems that the change of water repellency of the hydrocarbon resin-treated fabric during the washing and heat treatment is smaller than that of the fluorocarbon resin treatment. Detail of the mechanism is not clear at present.

Considering a change of the water repellency of the nylon 6, triacetate and silk fabrics treated with hydrocarbon, we conclude that the water molecules trapped on the resin surface by the washing and release of the water by subsequent heat treatment play an important role for the water repellency.

Table 2 shows the water repellency of the silk fabrics treated with Parajium JQ, RC and silicon resins during the washing and subsequent heat processing. Untreated silk fabric is considerably wettable because of the chemical composition of the peptide chain which contain many oxygen atom in the material. Although the water repellency was improved remarkably by the treatment with Paragium JQ and Porn MR, the effect decreased apparently by the washing and increased gradually by the subsequent heat treatment. We suppose that a little water molecules were bonded on the fabric surface by the washing and leads to a decrease of the water repellency, and the water was released from the surface by the heat treatment. As the result, water repellency was recovered.

3.2 KES shearing property

Hydrocarbon and silicone resin have already been used in the textile finishing as a softening agent. As one of the parameter of the mechanical property during the washing and subsequent heat treatment of the treated fabrics, shearing and bending behaviors play an important role for the fabric property.

It seems that shearing deformation corresponds well to the frictional coefficient of the interyarn. The shearing modulus G and the hysteresis width $2HG$ and

$2HG5$ of the nylon 6, triacetate and silk fabrics treated with Paragium JQ and RC were shown in Tables 3, 4 and 5, respectively. It is clear that G , $2HG$ and $2HG5$ were diminished generally by the resin treatment, and also subsequent heat treatment after washing, especially at 120°C . It seems that hydrocarbon and silicone resins on the fiber surface depress the surface tension of the fiber, and leads to a decrease of the adhesive force and frictional coefficient. It is clear that the effect contributes to a decrease of the shearing parameters, G , $2HG$ and $2HG5$.

Table 3. KES shearing parameters of nylon 6 fabrics treated with Paragium JQ and RC during washing and heat treatment

Treatment	Shear modulus (gf/cm degree)		Hysteresis width (gf/cm)
	G	$2HG$	$2HG5$
Untreated	0.97	2.40	5.43
Paragium JQ treated	0.80	1.27	3.08
Washed and dried	0.58	2.07	2.97
Heat treated			
$80^{\circ}\text{C}/3$ min	0.71	1.93	3.08
$120^{\circ}\text{C}/3$ min	0.55	1.20	2.17
Paragium RC treated	0.74	1.11	3.15
Washed and dried	0.75	2.52	4.23
Heat treated			
$80^{\circ}\text{C}/3$ min	0.65	2.18	3.85
$120^{\circ}\text{C}/3$ min	0.63	1.58	3.14

Table 4. KES shearing parameters of triacetate fabrics treated with Paragium JQ and RC during washing and heat treatment

Treatment	Shear modulus (gf/cm degree)		Hysteresis width (gf/cm)
	G	$2HG$	$2HG5$
Untreated	0.27	0.23	0.74
Paragium JQ treated	0.21	0.07	0.35
Washed and dried	0.22	0.08	0.37
Heat treated			
$80^{\circ}\text{C}/3$ min	0.22	0.07	0.37
$120^{\circ}\text{C}/3$ min	0.23	0.13	0.37
Paragium RC treated	0.23	1.11	0.44
Washed and dried	0.21	2.52	0.32
Heat treated			
$80^{\circ}\text{C}/3$ min	0.21	2.18	0.35
$120^{\circ}\text{C}/3$ min	0.21	1.58	0.34

**Table 5.** KES shearing parameters of silk fabrics treated with Paragium JQ and silicon resins washing and heat treatment

Treatment	Shear modulus (gf/cm degree)		Hysteresis width (gf/cm)
	G	2HG	2HG5
Untreated	0.24	0.14	0.56
Paragium JQ treated	0.21	0.06	0.27
Washed and dried	0.22	0.13	0.38
Heat treated			
80°C/3 min	0.22	0.11	0.31
120°C/3 min	0.20	0.10	0.36
Silicon resin treated	0.21	0.02	0.12
Washed and dried	0.24	0.16	0.35
Heat treated			
80°C/3 min	0.21	0.10	0.26
120°C/3 min	0.18	0.04	0.15

3.3 KES bending property

Tables 6 and 7 show the bending characteristics B and $2HB$ of the hydrocarbon resins treated nylon 6, and triacetate fabrics, respectively. On the other hand, although the fabrics were stiffened by the resin treatment, it decreased by the washing and heat treatment. It seems that excessive surface resin was removed and it became much more flexible for bending deformation.

4. Conclusions

In previous papers, we confirmed that the water repellency of the fabric treated with fluorocarbon resin decreased by the washing and recovered by the subsequent heat treatment remarkably. To investigate the effect of the washing and heat treatment on the water repellency of the fabric, nylon 6, triacetate and silk fabrics were treated with hydrocarbon resin (Paragium JQ) and silicon resin (Poron MR). After padding, drying and curing, water repellency was evaluated at each process of the washing and the heat treatment.

Considering a change of the water repellency of the nylon 6, triacetate and silk fabrics treated with hydrocarbon and silicon resins, we concluded that the water molecules trapped on the resin surface by the washing and release of the water by subsequent heat treatment play an important role for the water repellency.

Table 6. KES bending parameters of nylon 6 fabrics treated with Paragium JQ and RC during washing and heat treatment

Treatment	Bending modulus (gf cm ² /cm)	Bending hysteresis width (gf cm/cm)
	B	2HB
Untreated	0.0275	0.0273
Paragium JQ treated	0.0282	0.0260
Washed and dried	0.0227	0.0298
Heat treated		
80°C/3 min	0.0294	0.0270
120°C/3 min	0.0254	0.0270
Paragium RC treated	0.0294	0.0260
Washed and dried	0.0241	0.0298
Heat treated		
80°C/3 min	0.0230	0.0270
120°C/3 min	0.0268	0.0255

Table 7. KES bending parameters of triacetate fabrics treated with Paragium RC during washing and heat treatment

Treatment	Bending modulus (gf cm ² /cm)	Bending hysteresis width (gf cm/cm)
	B	2HB
Untreated	0.0148	0.0056
Resin treated	0.0148	0.0117
Washed and dried	0.0126	0.0066
Heat treated		
80°C/3 min	0.0145	0.0054
120°C/3 min	0.0103	0.0060

The water repellency decreased by the washing and recovered a little after the heat treatment. The effect on the water repellency was apparently smaller than the fluorocarbon resin treatment. Octadecyl ethylene water repellent agent as a cellulose fiber-reactive resin was not shown in effect at decreasing and recovering by the washing and heat treatment. KES shearing and bending parameters generally decreased by the heat treatment after washing. It is obvious that the treatment contributed to the softening of the fabric.

References

1. H. Ono, Hatusui Hatsuyuzaino Seinouni Tsuite, *Dyeing Industry*, **30**, 460-466(1982).
2. T. Wakida, T. Goto, H. Li, T. Sato, M. Lee, and J. Chen, Effect of Washing and Subsequent Heat Treatment on the Water Repellency of Poly(ethylene terephthalate) fabrics and Film Treated with Carbon Tetrachloride and Trifluoromethane Low temperature Olasma, *Sen'i Gakkaishi*, **50**, 533-537(1994).
3. F. Ide, "Surface Modification of Polymer", Kindai Henshusha, Tokyo, Jpn, p.162, 1987.
4. T. Wakida, H. Li, Y. Sato, H. Kawamura, M. Ueda, H. Mizushima, and S. Takekoshi, The effect of washing and heat treatment on the surface characteristics of fluorocarbon resin treated polyester, *J. Soc. Dyers Colour.*, **109**, 292-296(1993).
5. Y. Sato, T. Wakida, S. Tokino, S. Niu, and M. Ueda, Effect of Crosslinking Agents on Water Repellency of Cotton Fabrics Treated with Fluorocarbon Resin, *Textile Res. J.*, **64**, 316-320(1994).
6. Y. Ikada, "Fundamental and Application of Polymer Surface", Kagaku Donin, Tokyo, Jpn, A69, 1986.
7. T. Wakida, T. Mori, M. Saito, and C. Doi, A Change of Surface Characteristics of Commercial Water Repellent Fabrics by washing and Subsequent Ironing, *Jpn. Res. Assn. Text. End-Uses*, **42**, 113-119(2001).
8. T. Wakida, H. Kawamura, J. C. Song, T. Goto, and T. Takagishi, Surface Free Energy of Poly(ethylene terephthalate) and Nylon 6 Films Treated with Low temperature Plasma, *Sen'i Gakkaishi*, **43**, 384-389(1987).
9. Y. Kiatazaki and T. Hata, "Kobunshi Jikkengaku (Vol. 12)", Maruzen, Tokyo, Jpn, p.173, 1983.