JARQ 31, 287-294 (1997)

Characteristics of Wild Silk Fibers and Processing Technology for Their Use

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Abstract

The physical properties and processing technology of wild silk fibers, yarns and fabrics have been studied for the promotion of new uses of silk by developing novel fabrics made of fibers of wild silkworms different from those of the domesticated silkworm (*Bombyx mori*). Although wild silk fibers show advantages such as thickness, bulkiness, compressive elasticity, resistance to chemicals, they display many defects, including shrinkage with hot water, poor cohesion, development of bright specks in the yarn, low dyeing ability, etc. However, we succeeded in pro by combining mori silk with wild silk fibers, and by effectively utilizing the latter's characteristics for a wider demand of silk products. The mixed twisted yarns produced by the combination of wild silk fibers with mori silk with limited shrinkage enabled to improve the dimensional stability of wild silk fabrics. Also, the use of thick yarn enabled to prevent the occurrence of bright specks. The use of acid milling dyes or reactive dyes for wild silk yarns led to the induction of dark shades and rich, fast colors, especially since it had been hitherto difficult to use reactive dyes for mori silk products and wild silk fabrics.

Discipline: Sericulture/Agricultural environment
Additional key words: wild silk, physical property, silk fabrics, dyeing

Introduction

Cocoon filaments of the *Bombyx mori* silkworm consist of a pair of fibers spun out by the insect. The filament on an average is about 3 denier corresponding to a thickness of 20-30 microns, and shows a dense inner structure. There are various kinds of wild silk fibers in which, unlike in *mori* silk, the cocoon filament thickness varies considerably, and the filaments have a flat cross-section and a structure that includes voids with various sizes. They are different from *mori* silk in their silkiness, moisture absorption and thermal insulation properties and hand-feel, etc. Thus, wild silk fibers represent a fiber material with many potential applications in addition to their use for western style clothes.

We studied the physical properties of wild silk fibers as a starting material for making fabrics, and compared them with *mori* silk, to identify better applications and proper utilization[1,2,5].

Materials and methods

1) *Wild silk fibers*

The fabrics from wild silkworms that were used included Japanese tusser silk (*Antheraea yamamai*) which is indigenous to Japan, Chinese tusser silk (*Antheraea pernyi*) which is mainly produced in China, Indian tusser silk (*Antheraea mylitta*) which is produced mainly in India, Muga silk (*Antheraea assamensis*) and Eri silk (*Philosomai cynthia ricini*).

Japanese tusser, Chinese tusser, *mori* silk used throughout this investigation were supplied by Dr. H. Tanaka, Sanin Wild Silkmoth Research Center, (Kurayoshi, Tottori Prefecture, Japan) and also Indian tusser, Muga and Eri were given by Dr. S. K. Majhi, Central Tasar Research & Training Institute (Ranchi, India) (Plate 1)[4].

2) *Degumming method*

Since sericin of wild silk fibers is mostly fixed with tannin, coloring matters, wax, etc., it is difficult to boil off sericin using soap, or soap and sodium.

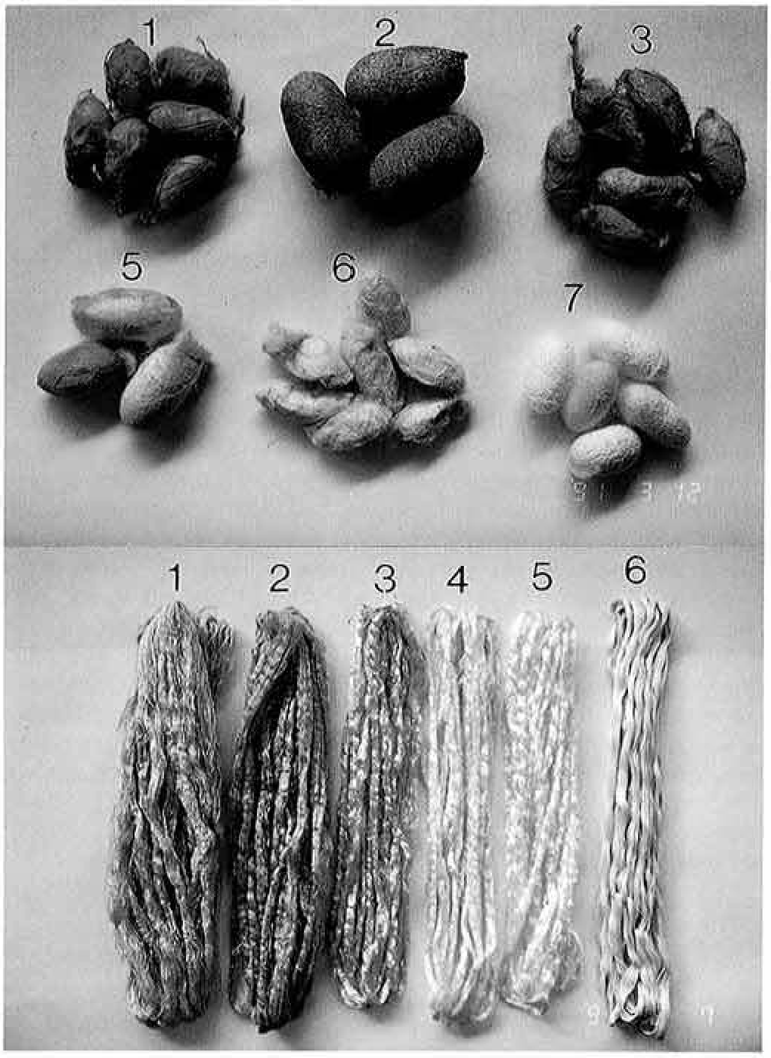

Plate 1. Wild silk cocoons (upper) and reeled raw silk yarns used in the experiment (below)
1: Indian tusser (*A. proylei*). 2: Indian tusser (*A. mylitta*). 3: Muga silk (*A. assamensis*). 4: Chinese tusser (*A. pernyi*). 5: Japanese tusser (*A. yamamai*). 6: Eri silk (*Philosamai cynthia ricini*). 7: *Mori* silk (*Bombyx mori*).

Also, the use of a higher concentration of alkali agents is likely to decrease the strength of wild silk yarns or raise the naps. However, it was found that reeled yarn of wild silk cocoon filaments could be well degummed with proteolytic enzymes like papain, alkali protease by the application of the decomposition method of sericin. Composition of degummed bath is as follows:

Pre-treatment:

Sodium carbonate (Na_2CO_3)	1 g/l
Sodium hydrosulfite ($Na_2S_2O_4 \cdot 2H_2O$)	1 g/l
Liquor ratio	1:40
Temperature-Time	98°C-30 min

Enzyme treatment:

Alkali protease (Alkaraze 2.5L)	1 g/l
Sodium hydrogencarbonate ($NaHCO_3$)	1 g/l
Nonionic surface active agent (Noigen HC)	1 g/l
Liquor ratio	1:40
Temperature-Time	60°C-60 min

Bleaching treatment:

Hydrogen peroxide (H_2O_2)	10 g/l
Sodium silicate (Na_2SiO_3)	2 g/l
Nonionic surface active agent (Noigen HC)	1 g/l
↓ (continued)	
Sodium hydrosulfite ($Na_2S_2O_2 \cdot 2H_2O$)	1 g/l
Sodium hydrogencarbonate ($NaHCO_3$)	1 g/l
Temperature-Time	90°C-60 min

3) Fabrics containing wild silk fibers

Twenty m/m yarn-scoured fabrics were prepared in plane weave, twill weave and satin weave, according to the design presented below, using *mori* silk yarn as warp and Japanese tusser, Chinese tusser, or *mori* silk yarn as weft yarn, and the characteristics of the fabrics containing Japanese tusser, i.e. structure, physical properties, gloss, etc. were studied and compared with those of the samples of *mori* silk and Chinese tusser fabrics.

Design of fabrics containing wild silk fibers:

[Warp] Raw silk 21 d × 2, plied yarn, reed 25/cm, 4 yarn beating, heald 8 pieces, turn passing, heald width 47.5 cm.

[Weft] Japanese tusser (24.2 d), Chinese tusser (25.9 d), *mori* silk (27.8 d), yarn 4/Z 200, single twist, beating spaces, plain 38/cm, twill 46/cm, satin 45/cm.

Characteristics of wild silk fibers

1) Morphological characteristics

The wild silk fibers are 2-3 times thicker than the *mori* silk fibers (Table 1). They display a greater variation in size and diameter and invariably show a flat cross-section. The high gloss and unique feel of wild silk fabric is mainly derived from these morphological characteristics of the fiber. The specific gravity of Japanese tusser is lower than that of Chinese tusser, which is lower than that of *mori* silk. Generally, wild silk fibers are lighter.

2) Physical properties

The load-elongation curves of wild silk fibers are characterized by clear-cut yield points. These materials show a plastic flow beyond the yield points (Fig. 1)[2]. As a result, if a load exceeding the yield point is applied, they cannot recover properly from the strain even when the load is removed. Their strength and Young's modulus are lower than those of *mori* silk, and the wild silk fibers show a greater elongation. They also exhibit a greater variability in these parameters. As a result, wild silk fabrics develop bright specks and it is difficult to weave them into thin fabrics.

All the wild silk fibers shrink by 5-8% of the

Table 1. Physical properties of wild silk fibers used throughout this investigation

Type of silk (denier of yarn)	Species	Mean no. of cocoon filaments in yarn (pieces)	Size of cocoon filament (denier)	Strength (g/d)	Elongation (%)	Young's modulus (g/d)	Degumming loss (%)	Shrinkage[a] (%)	Regain (%)	Specific gravity (g/cm^3)
Mori silk (26.3 d)	*Bombyx mori*	9	2.9	4.14	18.9	83.8	23.5	0.8	7.8	1.338
Japanese tusser (45.7 d)	*Antheraea yamamai*	8	5.7	3.24	32.6	60.5	12.4	7.7	8.7	1.274
Chinese tusser (37.9 d)	*A. pernyi*	6	6.3	3.01	31.1	45.6	7.83	6.5	11.9	1.281
Indian tusser (Tropical) (53.4 d)	*A. mylitta*	6	8.9	2.50	25.5	54.1	3.69	7.3	10.5	1.274
Indian tusser (Temperate) (91.6 d)	*A. proylei*	13	7.0	2.26	22.4	42.4	6.72	5.5	9.8	1.271
Muga silk (38.6 d)	*A. assamensis*	7	5.5	2.84	28.8	34.7	7.64	7.7	10.2	1.271
Eri silk (77.5/2 S250)	*Philosamai cynthia ricini*	-	-	2.73	29.2	46.0	4.5	7.3	9.4	1.283

Eri silk used consisted of spun yarn, and all the others of reeled raw silk yarn.

a): The value of shrinkage observed in the degumming process.

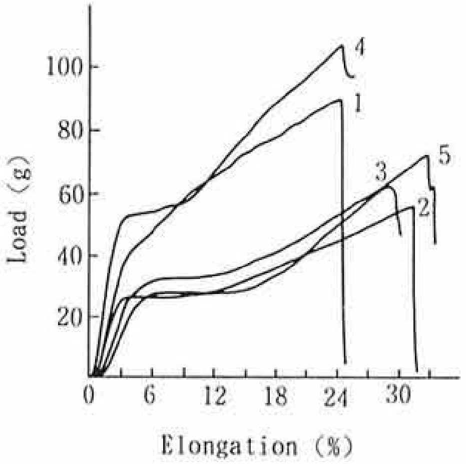

Fig. 1. Load-elongation curves of reeled raw silk yarns
1: *Mori* silk (30.0 d). 2: Japanese tusser (25.0 d). 3: Chinese tusser (28.0 d). 4: Indian tusser (48.1 d). 5: Muga silk (27.6 d).
( ): Size of reeled raw silk yarn.

original length when they come into contact with moisture or steam, which leads to a remarkable increase of elongation of the degummed yarn. In general, wetting and drying steps are necessary in various types of processing, like degumming, dyeing and finishing, before the material can be made into a product. Since the large shrinkage is one of the shortcomings of wild silk fibers, combining them with raw silk with reduced shrinkage, such as in mixed *mori* silk-Chinese tusser twisted yarns, would be essential for the improvement of the use of wild silk fibers.

The extent of shrinkage of wild silk fibers should be taken into account in designing mixed fabrics containing wild silk fibers. For example, Chinese tusser silk, once subjected to steam heat treatment does not shrink appreciably further even if it is boiled in water in a subsequent stage. Therefore, if the starting material yarn is preshrunk by steam heat treatment, the fabric would show a better dimensional stability. Fabrics made of large amounts of Chinese tusser have a rough feel. However, since sliding of the yarns is suppressed, the slip resistance of the stitches improves. Since Japanese tusser silk and Chinese tusser silk have similar physical properties, similar effects can be expected with Japanese tusser too.

Twisting and weaving of wild silk fibers

1) Twistability

Wild silk fibers show a poor cohesion compared to *mori* silk. This is why they are more susceptible to opening up, which requires that the filaments be twisted before weaving. The conventional soaking treatment used for twisting raw *mori* silk sometimes further reduces the cohesion of the fibers and causes shrinkage, making twisting even more difficult. Therefore, care should be taken if this procedure is used for the twisting of wild silk fibers. The degree of shrinkage depends on the amount of moisture. Thus, processing methods that use less moisture should be developed. Presently, specially developed oils are used for this purpose. Techniques like spraying oil on skeins are also employed to prevent misalignment of yarns.

With poorly bound fibers, it is preferable to use the so-called "rolling method" for taking the yarn out from the bobbins. Up-twisters like the Italian style twisters are more suitable than ring-twisters

Table 2. Properties of yarn in bright specks of wild silk fabrics

	Yarn with bright specks		Normal weft	
	Mean	S. D.	Mean	S. D.
Size (d)	99.3–117.4		109.4–122.3	
Crimp percentage (%)	1.39	0.608	2.13	0.347
Strength (g/d)	3.96	0.286	3.64	0.177
Elongation (%)	34.8	13.67	47.2	2.57
Young's modulus (g/d)	45.5	19.28	23.7	2.59

Wild silks fabric consisted of yarn-scoured plane weave with Japanese tusser.

which use a traveller. Steam heat treatment at 90°C for 15–20 min, or about 40 min for high twist yarn, is sufficient for twist-fixing of the wild silk fibers.

2) Causes of bright specks on wild silk fabrics and their prevention

Wild silk fabrics develop bright specks. Japanese tusser and Chinese tusser silk are more prone to the development of bright specks, regardless of whether they are prepared in plain weave, twill weave, or satin weave. This is a characteristic defect of wild silk fabrics, which can be considered as a unique feature also. Yarn in the bright specks in Japanese tusser fabric shows a lower crimp percentage, larger Young's modulus, lower elongation, and higher scattering of these values, compared to the weft yarns in the normal part of the fabric (Table 2).

Bright specks develop mainly because of the dynamic characteristics of wild silk fibers. The flatness of the fiber, knots, and unevenness of the yarn are the main causes, and the weft yarns are subjected to an excess of tension. For preventing the occurrence of bright specks, the yarn must be carefully arranged to minimize the unevenness in the yarn and in cohesion so that it slides well and abnormal tension is not experienced by the weft yarns.

A conventional technique is the use of thick yarn. Another useful technique that has been suggested is to draw the yarn under a tension exceeding the yield point to increase the Young's modulus of the weft yarns before weaving.

Physical properties of wild silk fabrics

1) Optical characteristics

A unique characteristic of Japanese tusser fabric is its gloss. Generally, the gloss of a fabric varies depending on the fiber direction from which it is observed, in particular in the case of fabrics containing Japanese tusser. When we determined the anisotropy level (maximum/minimum gloss ratio) after measuring the intensity of specular gloss from different directions, in the fabrics containing *mori* silk as warp yarns and wild silk or *mori* silk as weft yarns, the anisotropy was found to be maximum in the fabrics containing Japanese tusser, followed by the fabrics with *mori* silk and those with Chinese tusser. This trend was particularly conspicuous in the case of twill weave. The absolute value of the gloss was almost the same in the fabric made of Japanese tusser and that made of *mori* silk, while the fabric with Chinese tusser showed a lower value (Table 3).

2) Structural and physical properties of wild silk fabrics

Fabric samples showed almost the same specific gravity and void volume in the 3 types of weaves irrespective of whether the weft was made of Japanese tusser or *mori* silk. In the twill weave and satin weave fabrics, which have fewer crossing points, both the compressibility and compressive elasticity were large and the resistance against drawing and the bending resistance were low (Table 4). Wild silk fibers

Table 3. Gloss of wild silk composite fabric

Fabric weave	Weft	Maximum		Maximum		Anisotropy	
		Direction (°)	Gloss	Direction (°)	Gloss	Max–Min	Max/Min
Plain	Japanese tusser	0	8.63	120	7.21	1.42	1.20
	Chinese tusser	0	6.10	60	5.55	0.55	1.10
	Mori silk	180	6.55	60	5.91	0.64	1.11
Twill	Japanese tusser	180	8.21	60	7.05	1.16	1.16
	Chinese tusser	0	6.96	45	6.36	0.60	1.09
	Mori silk	90	9.04	15	8.20	0.84	1.10
Satin	Japanese tusser	0	13.39	90	10.01	3.38	1.34
	Chinese tusser	0	11.50	105	9.34	2.16	1.23
	Mori silk	0	13.71	105	11.31	2.40	1.21

Warp yarn: *Mori* silk.
Method of measurement: JIS Z 8741 Specular Gloss. Method 3, angle of projection 45°.
Direction of fabric: The angle formed by the projection of the beam entering the fabric and the weft direction.

Table 4. Properties of wild silk composite fabric

Fabric weave	Weft	Mezuke ($g \cdot m^2$)	Thickness (mm)	Apparent sp. gr (g/cm^3)	Void volume[a)] (%)	Compression characteristics (%)		Slope of load-elongation curve (g/%·cm)		Bending resistance (g·cm)		Crease resistance (%)	
						Compressibility	Comp-elasticity	Warp dir.	Weft dir.	Warp dir.	Weft dir.	Warp dir.	Weft dir.
Plain	Japanese tusser	88.78	0.164	0.541	58.8	15.2	80.8	6.2	7.3	0.437	0.500	59.5	52.7
	Chinese tusser	93.27	0.179	0.521	61.4	25.1	51.1	5.2	7.3	0.346	0.776	57.9	52.4
	Mori silk	86.70	0.159	0.556	59.3	19.6	74.2	5.3	74.2	0.332	0.839	54.2	60.8
Twill	Japanese tusser	96.27	0.245	0.393	70.0	25.8	71.4	12.0	12.5	0.206	0.262	62.6	66.8
	Chinese tusser	90.91	0.243	0.374	72.3	23.0	59.7	14.0	9.9	0.198	0.231	66.6	66.0
	Mori silk	91.66	0.235	0.390	71.5	27.9	62.4	13.2	23.5	0.206	0.336	57.7	73.6
Satin	Japanese tusser	94.66	0.255	0.371	71.6	25.2	75.8	17.4	8.6	0.170	0.211	63.4	74.7
	Chinese tusser	103.75	0.274	0.379	71.9	22.7	56.5	15.9	9.3	0.182	0.284	64.3	67.9
	Mori silk	88.00	0.224	0.394	71.2	24.8	66.7	17.2	20.1	0.156	0.226	59.1	74.3

a): Void volume (%) = (S − S'/S) × 100(%), where S is the specific gravity of the fiber and S' is the apparent specific gravity.

become so aligned in the fabric that their flat sides are adjacent. Therefore, the friction between fibers is large and the fabric shows a low crease recovery in the direction that will cause the wild silk yarns to bend, i.e. across the weft direction. However, the fabrics from wild silk yarns as weft display a higher crease resistance across the warp direction because of the bulkiness of the wild silk yarns. Therefore, the decrease of the crease resistance of mixed fabrics containing wild silk can be minimized if the wild silk yarns are alternated with *mori* silk yarns.

Gloss and hand-feel of the fabric composed of Japanese tusser yarn are closer to those of *mori* silk fabric, and give a denser feel compared to the fabric containing Chinese tusser. However, in terms of formation of bright specks, fabric stretch ability, and crease resistance, the fabric is closer to the Chinese tusser fabric.

Dyeing behavior of wild silk fibers

1) Dye adsorption isotherms

The type of dyeing mechanism that operates at a certain temperature can be predicted from the dye adsorption isotherms which express the relationship between the dye uptake by the fiber and the dye concentration in the bath, at equilibrium. Fig. 2 shows such isotherms of wild silk fibers and *mori* silk for the 0.5 – 5.0% o.w.f. range of acid dye concentrations in the bath. This type of adsorption curve clearly indicates that dye uptake occurs through ionic bonding.

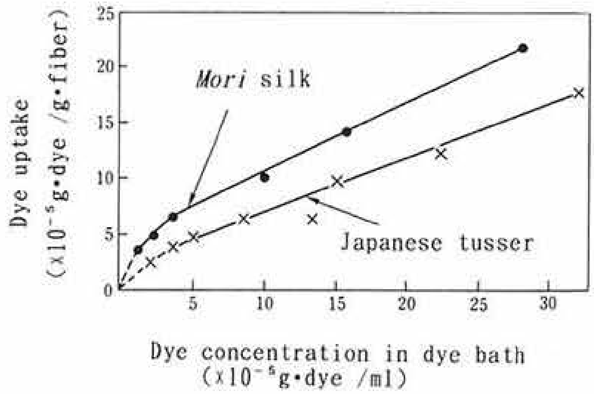

Fig. 2. Characteristics of the uptake of acid dye by Japanese tusser and *mori* silk
Dye: Acid Violet 5B.

With further increase in the dye concentration in the bath, the dye uptake increased almost linearly, although the slope was more gentle. In such a type of dyeing system, dye adsorption occurs due to physical bonding, including hydrogen bonding or "van der Waals" force bonding, rather than through ionic bonding. Dye adsorption isotherms of the type shown in Fig. 2 are typically obtained when wild silk fibers or *mori* silk are dyed with non-level acid dyes. The adsorption isotherms depicted in Fig. 2 can be defined using the following combined equation under the assumption that Langmuir adsorption through ionic bonding and partitioning adsorption through non-ionic bonding had occurred simultaneously[3].

$$(D)_\phi = \frac{k \times (S)_\phi \times (D)_\sigma}{1 + k \times (D)_\sigma} + K \times (D)_\sigma$$

where $(D)_\phi$ is the dye uptake, $(D)_\sigma$ is the dye bath concentration, $(S)_\phi$ is the saturation Langmuir adsorption, and k and K are constants of Langmuir and partitioning adsorption, respectively.

The value of K, the parameter related to the intensity of the partitioning adsorption, calculated by the least square method was 0.586 for Japanese tusser, a value slightly higher than in *mori* silk, 0.534. This fact may reflect the physical differences in the fine structure between the 2 kinds of fibers and their differences in the types and amounts of functional groups that take part in the bonding with the dye.

2) Effect of acid concentration and dye bath pH

Fig. 3 shows the relationship between the hydrochloric acid concentration in the dye bath as a function of the pH of the dye bath after dyeing. It appears that there is a large difference in the amount of hydrochloric acid between the *mori* silk and the wild silk in the hydrochloric acid concentration range of 1-3% o.w.f. When 2% o.w.f. hydrochloric acid was present in the dye bath, the residual bath pH was about 3.0 in the case of *mori* silk and about 6.0 for wild silk fibers. This difference of 3.0 pH units is very large from the viewpoint of dye adsorption. Fig. 3 shows the results obtained with Chinese tusser, while Japanese tusser, Muga and Eri also gave similar results.

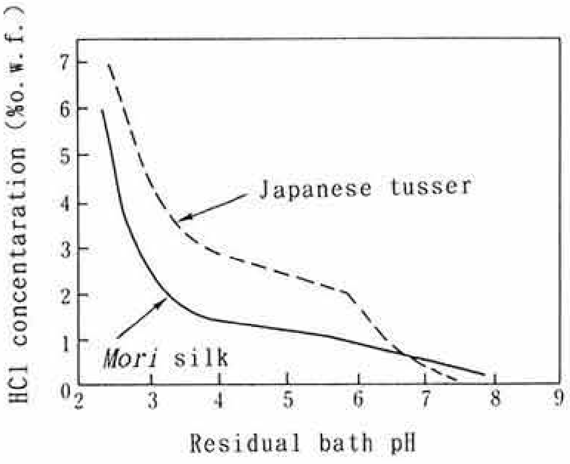

Fig. 3. Concentration of hydrochloric acid in the dye bath and pH of the residual bath
Dyeing conditions:
Dye concentration: 1.5% o.w.f.
Temperature: 80°C.
Duration: 60 min.
Liquor ratio: 1:100.

Thus, wild silk fibers show some buffering action towards acids. As a result, the pH of the dye bath does not decrease in the course of the dyeing process of *mori* silk, for the same amount of acid added. If the amount of acid added to the bath increased to the level at which the pH of the residual bath becomes the same as in *mori* silk dyeing, the percentage of dye uptake would be about the same in the 2 types of silk.

The dyes with a low molecular weight, like Orange II show a higher affinity for wild silk fibers compared to *mori* silk[6]. When semi-level or non-level dyes are used, there is hardly any difference in the affinity between wild silk fibers and *mori* silk. It is well known that the relationship between the dye uptake and the residual bath pH shifts toward a higher pH value when the affinity is higher[7]. With acid milling dyes, this relationship can be represented by a single curve as shown in Fig. 4.

Dyeing methods for wild silk fibers

1) Acid dyes

Neutral dyeing, which is responsible for the decrease of the number of dyeing specks, is commonly used for *mori* silk dyeing. However, acid dyeing at high temperature, i.e. dyeing in an acidic bath at a high temperature of 90-95°C, is recommended

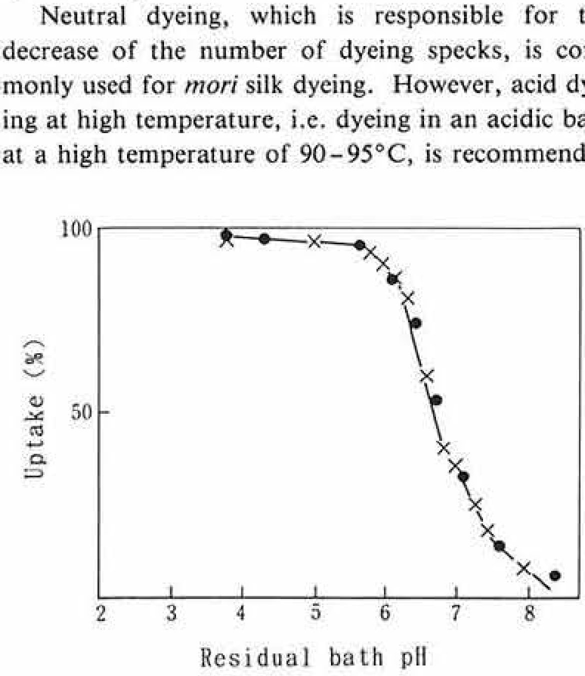

Fig. 4. Dye uptake and pH of the residual bath for degummed silk yarns
×: Japanese tusser, ●: *Mori* silk.
Dyeing conditions:
Dye: Acid Red 85.
Dye concentration: 1.5% o.w.f.
Acid added: Hydrochloric acid.
Temperature: 80°C.
Duration: 60 min.
Liquor ratio: 1:100.

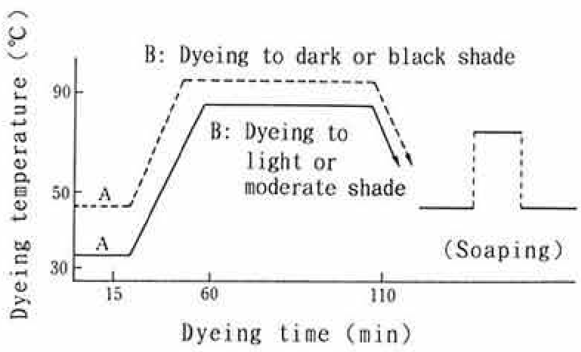

Fig. 5. Schematic dyeing conditions for wild silk yarns with acid dyes

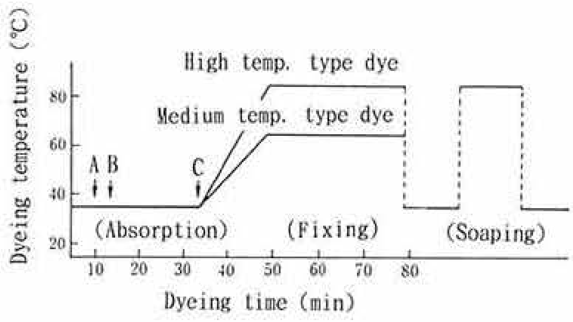

Fig. 6. Schematic dyeing conditions for wild silk yarns with reactive dyes
A: X% o.w.f. of moderate temperature or high temperature-reactive dyes.
B: 100 g/l of andydrous Glauber's salt.
C: 1-2 g/l of sodium carbonate.

for wild silk fibers. The dyeing temperature of the bath should be maintained at 95°C or at around the boiling temperature when dyeing to dark colors like black or navy blue is performed. After dyeing, it is preferable to remove the dyed material from the bath until the bath temperature decreases to about 70-80°C after turning off the heat source. Thorough soaping is needed after dyeing. If possible, finishing or bleaching treatment should be applied with weak acetic acid or tartaric acid to give a good scrooping effect on the silk fabric, and to improve the color development and hand-feel. Schematic dyeing conditions for wild silk fibers with acid dyes are presented in Fig. 5.

Metal complex salt dyes, instead of ordinary acid dyes, are recommended for improving color fastness and 2:1 metallized dyes show a high affinity to silk around the neutral zone.

2) Reactive dyes

There are many types of reactive dyes with different reactive groups. Cold-dyeing reactive dyes show a very low affinity to wild silk fibers. Most of the cold-dyeing reactive dyes become adsorbed. On the contrary, hot-dyeing or moderately hot-dyeing reactive dyes, that show a relatively low reactivity but can be used at a high temperature become adsorbed onto wild silk fibers. For using such dyes, the concentration of anhydrous Glauber's salt needs to be as high as about 50-100 g/l. The dyeing temperature should be 60-90°C and 1-2 g/l of sodium carbonate should be used as a dye-fixing agent. Soaping process after dyeing is essential for producing a good color fastness. Soaping should be performed using 0.2-0.3 g/l weak-alkali agent and 1-2 g/l non-ionic surfactant, at 70-90°C for 10-15 min.

Fig. 6 illustrates the conventional method of dyeing using moderate temperature-reactive dyes and high temperature-reactive dyes.

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(Received for publication, January 24, 1997)