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Technological parameters optimization by orthogonal array designs for steeping silk slices on small reels

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Abstract

A new steeping method is put forward in this paper, in which steeping assistant is added into the solution during the vacuum permeation for the treatment of silk slices on small reels. With this new method, the traditional process of steeping raw silk and drying can be omitted. Orthogonal array L_{16} (4^3) is used to study the new method. The result shows that the optimal technological parameters are 2 h, 1.25% and five times for balance drying time, assistant concentration and vacuum number, respectively. The test indicates that the properties of silk slices show no significant difference between steeping on small reels and conventional steeping, and the new process of steeping silk slices on small reels can mostly meet the technological requirements of steeping. It is also found that the properties of knitted fabric produced by the new steeping method and conventional steeping method are quite similar to each other. However, some special properties of the former, such as abrasion resistance, bursting strength and draping, are better than the latter. Compared with the conventional steeping of raw silk, the new steeping method has lower cost and higher efficiency.

Keywords

Raw silk, steeping, orthogonal array, knitted fabric, properties

Steeping is the process of steeping skein silk into aqueous solution with assistants, making sericin swell and soften, providing the raw silk with good performance properties, such as compliance, flexibility, lubricity and so on. It is also suitable for future processes and guarantees the quality of the product.[1,2]

At present, for the purpose of obtaining skein silk, the following processes are necessary: balance of silk slices on small reels → the vacuum permeation treatment of silk slices on small reels → balance of wet silk slices on small reels → re-reeling → balance of silk slices on large reels → lacing and checking → balance of silk slices → hank of silk → weighing of silk → color blending → packing → batch of products.[3] All of the mentioned processes are accomplished in a silk factory.

In a knitting factory, in order to meet the process requirements of winding, twisting, knitting and so on, it is necessary to treat skein silk by the processes of steeping raw silk, oven drying and airing. These processes need a large amount of manpower, material resources and lot of space for airing. Thence, the above-mentioned conventional process can be omitted if the skein silk meets the requirements of winding, twisting and knitting through adding a steeping assistant during the processes before re-reeling in the silk factory. This may improve production efficiency and reduce production cost. Therefore, it is necessary to find a suitable process. In this paper, a new steeping method is developed in which steeping assistant is added into the solution during the vacuum permeation treatment of silk slices on small reels, namely, steeping silk slices on small reels.

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Orthogonal array designs have been widely used with the advantage of reducing the number of the tests while giving reliable results to the optimize test.[4–7] This paper aims at evaluating the experimental conditions that provide the excellent performance of raw silk based on orthogonal array designs for steeping silk slices on small reels. Three parameters, namely, time of balance drying, concentration of assistant and vacuum number, are studied and optimized by a four-level orthogonal array design, with breaking strength, breaking elongation, compliance, loop elongation and loop strength as the performance index.

As a kind of natural protein fiber, the structure and performance of silk may be affected by different processing methods, which may further affect the quality of silk and fabric.[8] So it is necessary to compare silk slices on small reels after steeping and skein after conventional steeping, as well as raw silk without steeping in terms of quality and micro-structure of the silk. Furthermore, the comparison of the properties of the knitted fabric produced by new steeping method and conventional steeping method is made.

Experimental section

Materials

Raw silk was Grade 6 A silk slices on small reels (specification: 20/22 D, namely, 22.2/24.4 dtex), provided by Huzhou Zhejiang silk second factory. The conditioned weight fineness is 21.29 D (23.66 dtex).

Knitted fabric was steeping silk (specification: $20/22D \times 4$), used to weave the single jersey in the Z201 loop wheel machine's knitting motion. Then, the test samples were obtained after scouring.

Experimental methods

In this study, the balance drying time, assistant concentration and vacuum number, which effect properties of silk slices on small reels, are studied. The method of orthogonal array L_{16} (4^3) is used to determine the optimal technological parameters. Some other experimental parameters, such as degree of vacuum, temperature of water, bath ratio and the assistant applied to the steeping weft, are selected as 0.08 MP, $40 \pm 1°C$, 1:100 and EKL-101, respectively. The experimental parameters of conventional weft steeping are $40 \pm 1°C$, 1:100, EKL-101 and 3 h for water temperature, bath ratio, the assistant applied to steeping weft and steeping time, respectively.

A three-factor, four-level factorial design is used to evaluate the following factors effect on properties of raw silk: time of balance drying (A), concentration of assistant (B) and vacuum number (C). The factors and their respected levels are shown in Table 1. In order to estimate the best condition for steeping silk slices on small reels, 16 experiments are performed as shown in Table 2.

Table 1. Design of factor lever

	A	B	C
Lever	Time of balance drying (h)	Concentration of additive (%)	Vacuum number (time)
1	0	0.75	5
2	2	1.00	10
3	4	1.25	15
4	6	1.50	20

Table 2. L_{16} (4^3) orthogonal array

Trial numbers	A	B	C
1	1	1	1
2	1	2	2
3	1	3	3
4	1	4	4
5	2	1	2
6	2	2	1
7	2	3	4
8	2	4	3
9	3	1	3
10	3	2	4
11	3	3	1
12	3	4	2
13	4	1	4
14	4	2	3
15	4	3	2
16	4	4	1

Analysis of properties

Tensile properties. An XL-2 yarn tensile strength tester is used to test the breaking strength, breaking elongation, loop strength, loop elongation and elasticity modulus of raw silk. The test condition is as follows: the active length is 500 mm, the drawing speed is 500 mm/min and the original load is (0.05 ± 0.01) cN/dtex.

All mechanical tests should be carried out at the temperature of $(20 \pm 2.0)°C$ and relative humidity of $(65.0 \pm 4.0)\%$. These samples should be placed under the above condition for 12 h before tests.

The elasticity modulus reflects the compliance of raw silk, which is inversely proportional to the elasticity modulus.[9]

Sericin content test. The samples are degummed following the standard method for raw silk: 5-g samples are dried using a drying oven to balance weight, and marked as weight of before degumming. Then the samples are steeped in 0.5 g/L sodium carbonate solution at 98°C ± 2.0°C for 30 minutes under stirring. Afterwards, the samples are thoroughly washed with distilled water at 50–60°C.

The above degumming process is repeated three times to ensure that the sericin is removed completely. The weight after degumming is obtained by using the drying oven. After that, the sericin content is calculated as the follows:

$$X(\%) = \frac{W_1 - W_2}{W_1} \times 100 \tag{1}$$

where X is sericin content and W_1 and W_2 are the weight before degumming and after degumming, respectively.

Scanning electron microscopy observation. After gold coating, a JSM-5610LV (JEOL, Japan) scanning electron microscope is utilized to obtain the morphology of the raw silk with 5 kV acceleration voltage and 1000 × magnification.

X-ray diffraction analysis. X-ray diffraction (XRD) measurements are performed with an X'TRA X-ray diffractometer equipped with a Ni-filtered CuKα radiation source at 40-kV voltage and 40-mA tube current. The diffraction intensities are measured from $2\theta = 5°$ to $50°$ at a rate of $2\theta = 2°$/min.

Then the peaks are separated by using the PeakFit program to calculate the crystallinity of raw silk.[10,11] The equation is as follows:

$$X_c = \frac{A_c}{A_c + A_a} \times 100\% \tag{2}$$

where X_c is the crystallinity and A_c and A_a suggest the area of all crystal peaks and the amorphous peak, respectively.

Analysis of knitted fabric property

The following mechanical tests should be carried out at the temperature of (20 ± 2.0)°C and relative humidity of (65.0 ± 4.0)%. The knitted fabric samples should be placed under the above condition for 24 h before tests.

Tensile properties. A YG065 electronic fabrics strength tester is used to test the breaking strength and breaking elongation of fabric. The test conditions are as follows: the active length is 200 mm and drawing speed is 100 mm/min.

Bursting strength. A Y631 bursting strength tester is used to test the bursting strength of fabric. Five replicate specimens with a diameter of 60 mm were prepared.

Abrasion resistance. A YG(B)401E Martindale abrasion tester is used to test the abrasion resistance of knitted fabric. The test conditions are as follows: the pressure on the sample is about 583.1 cN and the relative speed of the sample and abrasive is (47.5 ± 2.5) r/min.

Draping. An XDP-1 fabrics draping tester is used to test the draping of fabric. The drape coefficient reflects the draping of knitted fabric. The fabric draping is improved with decreasing of the drape coefficient.

Results and discussion

Orthogonal array analysis

Without the blank column in the design of the orthogonal array, the repeated test is necessary to give reliable results.[12] Twenty samples are tested for every performance index. Initial data are omitted.

The orthogonal array, date analysis and analysis of variance (ANOVA) are obtained using SPSS software.[13,14] The ANOVA results are shown in Table 3.

The ANOVA indicates that concentration of assistant (B) and time of balance drying (A) play important roles in the properties of raw silk. By comparison, we find that the effect of vacuum number (C) is less important than the other two factors.

According to the assignment of the experiment, the mean of property for the corresponding factors at each level is calculated as shown in Table 4.

Effect of time of balance drying on properties of raw silk

Table 4 shows that the different balance drying times (drying for 0, 2, 4 and 6 h) affect breaking strength, breaking elongation, loop strength, loop elongation and elasticity modulus. When drying time is 2 h, breaking strength, breaking elongation, loop strength and loop elongation of raw silk reach a maximum, and the elasticity modulus of raw silk reaches a minimum at drying for 4 h. Based on an overall consideration, we select 2 h for drying as the optimum level.

Effect of concentration of assistant on properties of raw silk

Table 4 shows that the different assistant concentrations (0.75%, 1.00%, 1.25% and 1.50%) effect breaking strength, breaking elongation, loop strength, loop elongation and elasticity modulus. When the

Table 3. Analysis of variance of property of raw silk

Source of variance	Sum of square	df	Mean square	F-ratio	
Breaking strength					
A	0.793	3	0.264	4.331	***
B	2.700	3	0.900	14.748	***
C	2.083	3	0.694	11.377	***
Error	18.917	310	0.061		
Breaking elongation					
A	33.230	3	11.077	2.729	**
B	108.928	3	36.309	8.947	***
C	65.734	3	21.911	5.399	***
Error	1258.086	310	4.085		
Loop strength					
A	3.487	3	1.162	7.509	***
B	7.783	3	2.594	16.761	***
C	2.084	3	0.695	4.488	***
Error	47.985	310	0.155		
Loop elongation					
A	92.263	3	30.754	14.857	***
B	29.101	3	9.700	4.686	***
C	7.255	3	2.418	1.168	–
Error	641.726	310	2.070		
Elasticity modulus					
A	29.505	3	9.835	7.778	***
B	51.874	3	17.291	13.675	***
C	66.126	3	22.042	17.433	***
Error	391.963	310	1.264		

Note: $F_{0.1}$ (3,310) = 2.08, $F_{0.05}$ (3,310) = 2.6, $F_{0.01}$ (3,310) = 3.78; $F < 2.08$, the effect is negligible (–); $2.08 \leq F \leq 2.6$, the effect is mild (*); $2.6 \leq F \leq 3.78$, the effect is significant (**); $3.78 < F$, the effect is extreme (***).

Table 4. The mean of the four levels of each factor

Factors	Level 1	Level 2	Level 3	Level 4
Breaking strength				
A	3.790	3.919	3.844	3.896
B	3.796	3.935	3.971	3.774
C	3.831	3.915	3.956	3.746
Breaking elongation				
A	18.961	19.584	19.534	18.875
B	18.340	19.225	19.958	19.431
C	19.841	19.510	18.835	18.767
Loop strength				
A	5.430	5.509	5.254	5.285
B	5.304	5.296	5.636	5.241
C	5.262	5.429	5.464	5.322
Loop elongation				
A	8.566	9.236	8.309	7.741
B	8.525	8.393	8.889	8.046
C	8.588	8.602	8.229	8.434
Elasticity modulus				
A	21.599	21.939	21.530	22.293
B	21.947	22.219	22.030	21.164
C	21.329	22.042	22.482	21.506

concentration of assistant is 1.25%, breaking strength, breaking elongation, loop strength and loop elongation of raw silk reach a maximum, and the elasticity modulus of raw silk reaches a minimum at the assistant concentration of 1.50%. Based on an overall consideration, we select 1.25% for the concentration of assistant as the optimum level.

Effect of vacuum number on properties of raw silk

Table 4 shows that the different vacuum numbers (5 times, 10 times, 15 times and 20 times) affect breaking strength, breaking elongation, loop strength, loop elongation and elasticity modulus. As to breaking strength and loop strength, the values reach a maximum when the vacuum number is 15 times. As to breaking elongation and loop elongation, the value reaches a maximum when the vacuum number is 5 times and 10 times, respectively. At the level of 5 times for the vacuum number, the elasticity modulus of raw silk reaches a minimum. According to these results, it is difficult to determine the optimum level for vacuum number. Based on the above ANOVA calculations, it seems that the effect of vacuum number is less important than that of the other two factors. That is, vacuum number (C) is a secondary factor. Therefore, we can select the shortest vacuum number as 5 times taking other factors into account, such as production efficiency, convenience of operation and so on.

In general, to obtain good properties of raw silk, the technological parameters of steeping conditions for silk slices on small reels after optimization are determined as 2 h for balance drying, 1.25% for assistant concentration and 5 times for vacuum number.

Comparison of properties and micro-structure of skein after conventional steeping (a), silk slices on small reels after steeping (b) and raw silk without steeping (c)

Comparison of silk properties. The breaking strength, breaking elongation, loop strength, loop elongation,

Table 5. Comparison of silk properties

Sample	Breaking strength ($cN \cdot dtex^{-1}$)		Breaking elongation (%)		Loop strength ($cN \cdot dtex^{-1}$)		Loop elongation (%)		Elasticity modulus ($cN \cdot dtex^{-1}$)		Sericin content (%)
	Mean	CV (%)	Mean	CV (%)	Mean	CV (%)	Mean	CV (%)	Mean	CV (%)	
a	3.98	4.4	21.65	8.4	5.64	7.2	10.45	15.3	21.09	6.1	24.35
b	3.96	4.1	19.58	8.8	5.58	6.6	9.45	14.1	22.12	6.2	23.71
c	4.22	5.3	21.25	8.4	6.19	6.6	10.87	14.3	22.93	6.0	22.78

CV: coefficient of variation.
Note: The mean values in Table 5 are the average of 20 samples except sericin content.

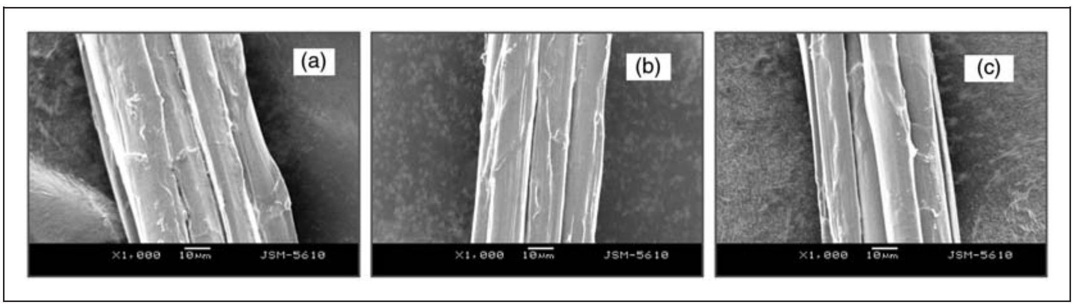

Figure 1. Scanning electron microscopy images of raw silk: (a) skein after conventional steeping; (b) silk slices on small reels after steeping; (c) raw silk without steeping.

elasticity modulus and sericin content of skein after conventional steeping (a), silk slices on small reels after steeping (b) and raw silk without steeping (c) are shown in Table 5. CV is coefficient of variation.

Comparison of sericin content. Sericin content is calculated using Equation (1) (see Table 5). There is no significant difference in the sericin content of the three samples. In addition, sericin content of samples a and b is slightly greater than sample c, namely, sericin content is slightly increased after steeping. This may be because strand silk absorbs a certain amount of oil after steeping.[15] There is no significant difference in CV.

From Table 5, we can see that the strength of samples a and b is slightly lower than sample c, namely, the strength of samples a and b is slightly decreased after steeping. This may be because the molecules are farther apart from each other, and intermolecular interactions decreases. There is no significant difference between samples a and b. Referring to GB/T 1797-2008 raw silk, we find that all breaking strength remain at the 6 A grade, which is the highest silk level. The CV of the three samples is similar.

As to elasticity moduli, from Table 5 we can see those of samples a and b are slightly lower than sample c, namely, the compliance of sample c is improved after steeping. This may be because water molecules enter the amorphous of fibroin, cracking intermolecular secondary bonds and making the molecular chain slipp.[16] In addition, compliance of sample b is slightly lower than that of sample a.

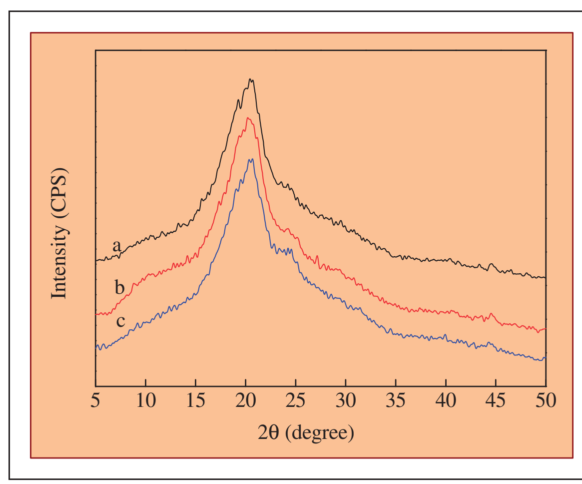

Figure 2. X-ray diffraction curves of raw silk: (a) skein after conventional steeping; (b) silk slices on small reels after steeping; (c) raw silk without steeping.

From Table 5, we can also see that both breaking elongation and loop elongation of sample b are the lowest among the three samples.

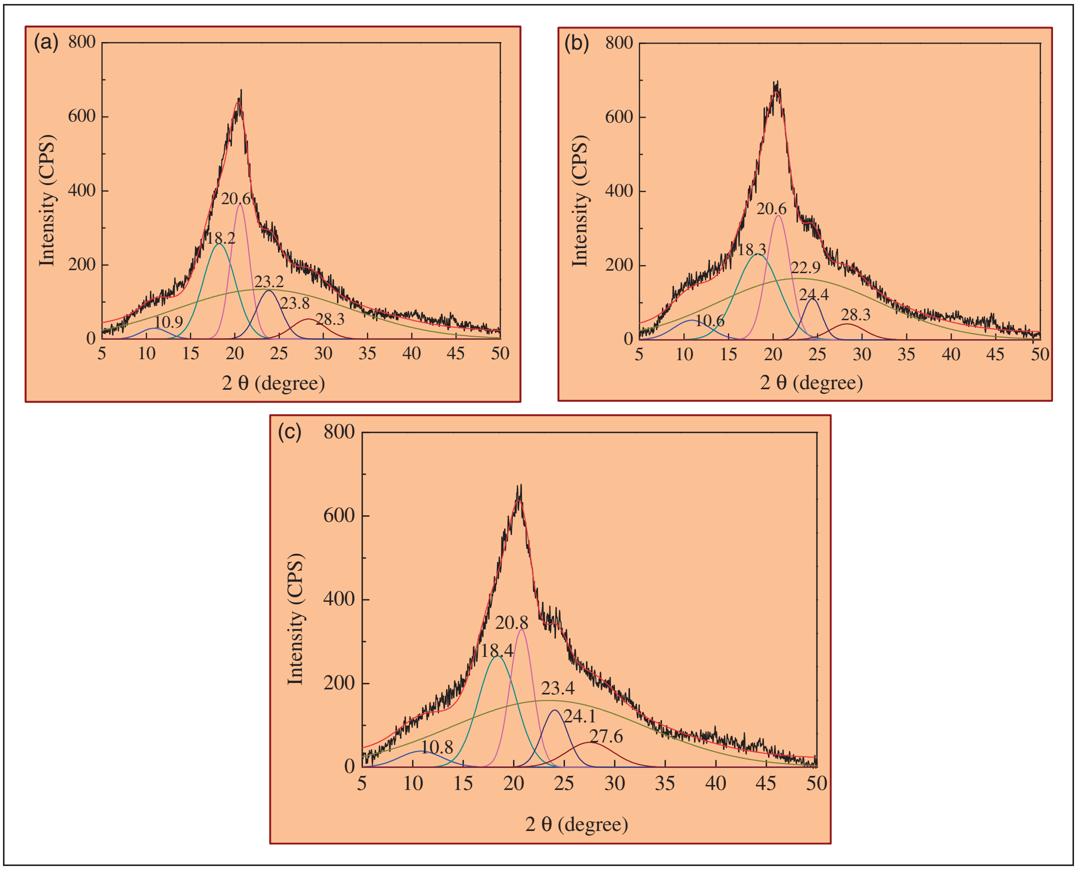

Figure 3. X-ray diffraction peak separation curves of raw silk: (a) skein after conventional steeping; (b) silk slices on small reels after steeping; (c) raw silk without steeping.

Comparison of raw silk structure

Surface morphology analysis. The surface morphologies of raw silk examined using scanning electron microscopy (SEM) are shown in Figure 1. Raw silk possesses several fibrils that are adhered to each other by sericin. The tiny particles on the surface of raw silk, which may be sericin tumors, lead to the rough surface of raw silk. It is difficult to distinguish obvious differences from each other, indicating that steeping silk slices on small reels has no effect on the surface morphology of raw silk.

XRD analysis. Figure 2 shows spectrographic evidence from XRD for the raw silk. There are no obvious differences among the three curves. While it is difficult to observe the diffraction peak from these curves, the PeakFit program is used to separate the overlapping bands, as shown in Figure 3. It is clear to see that the main diffraction peak appears at around $2\ \theta = 10.8°$, 18.3°, 20.6°, 24.4°, 28.3° (shown in Table 6). With the PeakFit program, the crystallinity of raw silk is calculated using Equation (2); the results are shown in Table 7. Table 7 shows that there are slight difference for the crystallinity of raw silk and no obvious difference in the structure of the three samples. The lower crystallinity of sericin on the raw silk surface leads to an effect on the crystallinity of silk.[17] In addition, the relative comparison of the crystallinity is made among the three samples.

Table 6. Diffraction peaks of raw silk

	Diffraction peaks (2θ/°)				
Sample	1	2	3	4	5
a	10.9	18.2	20.6	23.8	28.3
b	10.6	18.3	20.6	24.4	28.3
c	10.8	18.4	20.8	24.1	27.6

Comparison of knitted fabric properties

For further study, after scouring, the comparison of the properties of the knitted fabric Za and Zb produced from samples a and b is made. The result is shown in Table 8.

From Table 8, it can be found that the properties of the two knitted fabric are very close. In addition, the

Table 7. Crystallinity of raw silk

Sample	The area of crystal peaks					The area of amorphous peak	Crystallinity (%)
	1	2	3	4	5		
a	113.07	1172.25	965.67	462.79	254.17	3314.35	47.20
b	271.21	1383.27	1115.11	293.86	207.07	3773.73	46.40
c	204.16	1255.40	943.25	431.52	344.92	3843.97	45.30

Table 8. Comparison of knitted fabric properties

Sample	Breaking strength (N)		Breaking elongation (%)		Abrasion resistance (time)	Bursting strength (N)	Drape coefficient (%)
	Landscape orientation	Portrait orientation	Landscape orientation	Portrait orientation			
Za	87.6	132.0	189.5	67.4	1560	234.2	8.1
Zb	84.3	137.4	182.9	61.9	1600	251.4	7.9

value of abrasion resistance and bursting strength of knitted fabric Zb is greater than that of knitted fabric Za. The drape coefficient of knitted fabric Zb is lower than that of knitted fabric Za. Namely, abrasion resistance, bursting strength and draping of the knitted fabric produced from silk treated by the new steeping method is better than that treated by the conventional steeping method. The conclusion further indicates that the new steeping method is feasible.

Conclusions

The test indicates that the properties of silk slices show no significant difference between steeping on small reels and conventional steeping, and the new process of steeping silk slices on small reels can mostly meet the technological requirements of steeping. It is also found that the properties of knitted fabric produced by the new steeping method and conventional steeping method are similar to each other. However, some special properties of the former, such as abrasion resistance, bursting strength and draping, are better than those of the latter. Compared with the conventional steeping of raw silk, the new steeping method has lower cost and higher efficiency.

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