Mehdi Akhbari[1], Abdulreza Zahiri[2], Seyed Jalal Eddin Bassam[3]

Optimization of Parameters Influencing Mercerization Using the RSM Method in Order to Increase the Tensile Strength of Mercerized Yarn

[1]Deptartment of Textile,
Islamic Azad University,
Kashan, Iran
E-mail: m_a4949@yahoo.com

[2]Islamic Republic of Iran,
Ministry of Commerce,
Tehran, Iran
E-mail: ariyana_1354@yahoo.com

[3]Institute of Technical and Vocational Higher Education,
Ministry of Jahad Agriculture,
Tehran, Iran
E-mail: bassamsje@hotmail.com

Abstract

In this study, five parameters of a mercerising machine, four parameters of untreated yarn and three parameters of the caustic soda bath affecting the tensile strength of mercerised yarn were investigated. Experiments were designed with the aid of the Response Surface Method. Accordingly different types of yarns were mercerised on different machines and with production settings, then relevant trends were studied. Results show that the strength of the mercerised yarn is highly affected by the cylinder normal pressure, warm rinsing temperature and caustic soda bath concentration, while the effect of the cold rinsing temperature is observed only for higher qualities of cotton fibers and yarns. Increasing the cylinder rotation duration results in a reduction in the strength of carded yarns, but has little effect on combed yarns. Results also show that optimum settings are highly affected by the type of cotton fiber, yarn structure and number of plies, but the linear density of single yarn and the mercerising machine type have no significant effect.

Key words: *mercerisation, mercerising machine, caustic soda bath, Iranian cotton, Egyptian cotton, Uzbek cotton.*

Introduction

Mercerisation is one of the most common chemical treatments of cotton materials to improve their properties. Most of researches in this field have been carried out in laboratory scale with special emphasis on fabric mercerisation, focusing on the improvement of appearance properties. However, the effects of this process on the mechanical properties of mercerised yarn have almost been neglected. Yarn mercerisation with the aim of increasing dye uptake and light reflection properties while not considering some other technical aspects sometimes may bring about a reduction in yarn strength and an increase/decrease in yarn elongation [1 - 7].

McDonald's *et al.* (1957) investigations on six varieties of cotton fibres showed that mercerisation in a slack and stretched condition results in an increase in the evenness of all yarn properties and a reduction in their standard deviation. However, moisture absorption, fibre and yarn extension and swelling in slack mercerisation are higher than when the yarns are treated under tension. Yarn and fibre strength in tension mercerisation are, respectively, 60% and 53% higher than those in slack treatment [8].

Balasubramanian studies on 29.5 tex open end yarns showed that the strength of these yarns with the minimum twist (twist factor 4.25) may be improved by 52.8% in tension mercerisation, while this figure is 46% in slack mercerisation. He also found that although the degree of mercerisation and improvement in the strength of low twist yarns are higher than those of high twist yarns, yarns with an optimum twist always possess better strength. In other words, the effect of mercerisation on improving yarn strength is not comparable to that of the twist. He also observed that two folded (and multi-ply) yarns may be mercerised better than single ones, provided that the twist direction of plying is opposite to the single yarn spin. This is because the movement of fibres within the folded yarns takes place more easily; hence they may be better arranged in the yarn structure [9].

In 1989, some researches were carried out on the effect of temperature on mercerisation treatment. In these studies, Egyptian cotton was mercerised over 24 hours using 24% caustic soda. An x-ray examination showed that the amorphous regions of cotton fibres increased from 27% to 34% during mercerisation. The results demonstrated that raising the temperature has no effect on the extent of amorphous regions. Since the mercerising process changes cotton properties through the conversion of crystalline areas to amorphous areas, it is expected that changing the temperature of the caustic soda bath would have no effect on mercerisation. The results confirmed that the mercerisation degree in the same fibres changes very little when the temperature of the process rises from 5 degrees to 55 degrees centigrade. The research explained that controlling the mercerisation time facilitates the absorption caustic soda and converting cellulose I to cellulose II, and at the same time increases the efficiency of the treatment [10].

In this study, the parameters influencing mercerised yarn strength are identified and optimised. To this end, 19.7 tex, 11.8 tex, R 39.4 tex/2, R 23.6 tex/2, R 59.1 tex/3 and R 35.4 tex/2 carded and combed yarns made of cotton fibres from Iran, Uzbekistan and Egypt were prepared. Each one of them was mercerized using a Jacob Jaeggli MM-6 die machine in five different settings of the machine parameters (cylinder normal pressure, warm rinsing temperature, cold rinsing temperature, duration of hank rotation in caustic soda and duration of tensioning) and also in five levels of the caustic soda parameters (temperature, concentration and percentage of wetting agents). According to RSM experiment design, 51 different samples were produced to identify and optimise the effective parameters influencing the tensile strength of mercerised yarn.

Then the samples were processed using two other mercerising machines (Jakob

Jaeggli Extensa and Rite Aurora) with similar settings to investigate the effect of machine type on the mercerised yarn strength.

Materials and methods

Yarns

Yarns employed in this study were all 100% cotton. Technical specifications of fibres are given in ***Table 1. Table 2*** gives some details of untreated and mercerised yarns. The twist factor (α_{tex}) for Egyptian, Uzbekistan and Iranian yarns are 3930, 4250 and 4792, respectively.

Chemical materials

Chemical materials in all tests were the same, as follows:
- Avolan IW detergent with alkyl polyglycol ether from Bayer
- 99 - 100% pure flake caustic soda from Saliva
- Wetting agent Invadin MET
- Acetic acid 98% Merck
- Sodium carbonate Merck.

Design of experiments

Due to the numerous independent variables, the optimum points in the survey space were determined using RSM (Response Surface Method) as a powerful method to optimise processes mathematically. Eight independent variables were considered as input data in the ranges specified, as follows:
- Cylinder normal pressure (Cp): 0.375 - 0.575 MPa with 0.05 - MPa intervals
- Duration of cylinder rotation in caustic soda bath (Rt): 60 - 100 seconds with 10 - second intervals
- Tensioning time in caustic soda bath (Dt): 40 - 120 seconds with 20 - second intervals
- Temperature of warm rinsing water (Tw): 60 - 80 degrees centigrade with 5 - degrees centigrade intervals
- Temperature of cold rinsing water (Tc): 5 - 25 degrees centigrade with 5 - degrees centigrade intervals
- Caustic soda bath concentration (Sc): 15% - 35% with 5 - percent intervals
- Temperature of caustic soda bath: 10 - 30 degrees centigrade with 5 - degree centigrade intervals
- Wetting agent concentration: 0.5% - 2.5% with 0.5 - percent intervals

A plan of the essential settings of the machine and caustic soda bath was prepared by RSM to perform the experiments. The average of three strength tests in each setting was recorded as the response and strength of the relevant sample.

Table 1. *Technical specifications of fibres; *Short fibres are defined as fibres with 12 mm length and less.*

Cotton	Effective length, mm	Short fibers*, %	Bundle strength, cN/tex	Linear density, tex×10^{-3}	Maturity degree, %
Egypt (Giza 70)	35.9	5.2	33.0	122	92
Uzbekistan	33.0	8.4	29.3	130	85
Iran	30.5	10.5	26.2	142	82

Table 2. *Twist and tensile strength of untreated and mercerized yarns; ** Twist of yarns have been measured according to ASTM D1423 (Standard test method for twist in yarns by direct counting).*

Yarn type	Twist**, t.p.m.			Tenacity of untreated yarn, cN/tex			Tenacity of mercerized yarn in optimum case, cN/tex		
	single	two folded	three folded	single	two folded	three folded	single	two folded	three folded
19.7 tex yarn, Egyptian cotton	884.6	627.2	511.1	19.5	19.0	21.3	29.6	26.5	24.8
19.7 tex yarn. Uzbekistan cotton	958.9	677.0	549.3	18.0	18.3	20.4	27.0	26.7	22.3
19.7 tex yarn, Iranian cotton	1080.4	765.4	624.4	13.6	14.2	18.6	18.7	18.8	19.4
11.8 tex yarn. Uzbekistan cotton	1238.1	878.4	714.6	19.6	19.9	18.9	29	24.6	24.5
11.8 tex yarn, Egyptian cotton	1141.8	807.1	668.0	21.0	19.5	20.8	31.7	25.5	23.2

Treatments

Curing process

A bath of caustic soda 5% with Lizapon-N 10% detergent with a liquid ratio of 1:10 and maximum temperature of 80 °C was prepared to cure the yarns. The temperature was raised to 100 °C while feeding the hanks. The curing process took 30 minutes.

Mercerization process

According to the RSM output plan, the samples were mercerised in 51 different settings of the machine and caustic soda bath.

Neutralising process

Neutralising was performed with a solution as follows:
- acid: acetic acid 98%
- bath concentration: 1%
- pH = 4
- liquid ratio = 1:15
- temperature: 25 °C.

Strength test

Strength tests were performed using an Instron Tensile Strength Tester under the following standard conditions:
- Lab temperature: 20 ± 2 °C
- Lab relative humidity: 65 ± 2 percent
- Sample gauge length: 500 mm
- Pre-tension force: 0.5 cN/tex
- Extension rate: 4% per second
- Instrument error: 0.2%

Results & discussion

A model for changes in the mercerised yarn strength based on eight parameters was set up using RSM, CCD (Central Composite Design) and DX7 (Design Expert 7, software prepared specifically for design experiments, which is free from the inaccuracies and shortcomings of other software for designing experiments).

In this model, the main , interactive and quadratic effects of the parameters are examined. Significant effects remain in the model and insignificant ones are removed.

In all cases, hypothesis H_0 is that the parameter considered has no effect on the strength. If the significance is less than 0.05, which is in the area of the hypothesis rejection, the effect of the parameter considered will be significant and remain in the model. On the other hand, when the significance is greater than 0.05, the effect of the parameter considered is not significant; hence it is excluded from the model.

Figure 1.

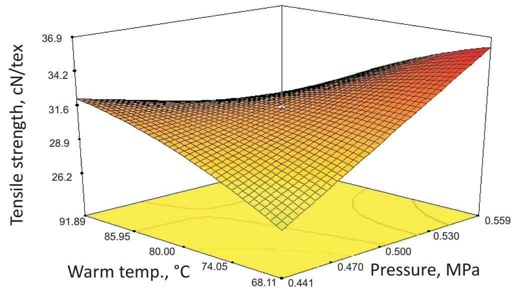

Figure 2.

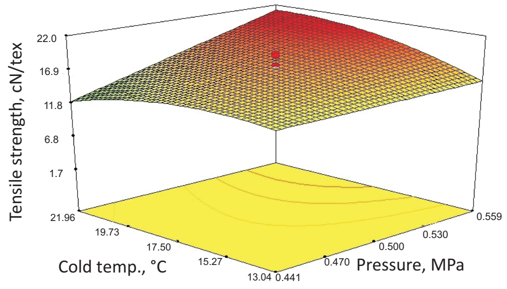

Figure 3.

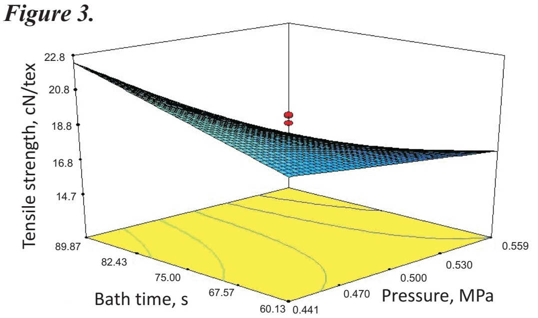

Figure 1. *Effect of cylinder normal pressure and warm rinsing temperature on the strength of Egyptian R 59.1 tex/3 mercerized combed yarn.*

Figure 2. *Effect of cylinder normal pressure and cold rinsing temperature on the strength of Iranian R 59.1 tex/3 mercerized combed yarn.*

Figure 3. *Effect of cylinder normal pressure and immersion time in the caustic soda bath on the strength of 19.7 tex mercerized combed yarn spun from Iranian cotton.*

In the regression equations, the parameter with the highest absolute value of the coefficient has the greatest effect on the strength. The plus or minus sign of coefficients of the parameters shows the increasing or decreasing effect on the strength, respectively.

Effect of machine parameters on the tensile strength of mercerised yarn

Cylinder normal pressure with a large coefficient in the regression equations has an enormous, basic and positive effect on the strength of the mercerised yarn. Increasing this parameter to an optimum level increases the strength of all mercerised carded/combed yarns with various linear densities and numbers of plies. Results show that in almost all cases, the maximum strength of mercerised yarn is obtained at 0.559 MPa of cylinder normal pressure and is independent of the technical specifications of the untreated yarn. As can be seen in the following figures, the cylinder normal pressure is optimum at 0.559 MPa. Thus for all other parameters, the optimum case is investigated at this pressure.

Effect of cylinder rotating duration

Cylinder rotation duration is not present in the regression equation of the strength of 19.7 tex mercerized combed yarn, but it has a small coefficient in the regression equation of the strength of 11.8 tex mercerised combed yarn. Therefore it can be assumed that this parameter has a small effect on the strength of mercerised combed yarns. However, increasing the cylinder rotation duration reduces the strength of all carded yarns, especially those spun from Iranian cotton fibres.

Effect of the warm rinsing temperature

The presence of a warm rinsing temperature in all regression equations shows its influence on the strength of mercerised yarn. Negative and large absolute values of its coefficients in thicker yarns and a higher number of plies demonstrate the high significance of this parameter on the strength of samples. ***Figure 1*** shows the effect of the cylinder normal pressure and warm rinsing temperature on the strength of Egyptian R 59.1 tex/3 mercerised combed yarn. As is seen, at lower cylinder normal pressures, the mercerised yarn strength increases as the temperature rises, while at the optimum cylinder normal pressure, the strength-temperature curve has a negative slope. On the other hand, at lower temperatures, which are more desirable, the strength of the yarns increases as the normal pressure increases to a maximum of 0.559 MPa. At higher temperatures the strength decreases as the normal pressure increases, which suggests that in order to reduce production costs and achieve high strength yarns, it is better to increase the normal pressure instead of increasing the temperature.

Effect of the cold rinsing temperature

Figure 2 shows the effect of the cylinder normal pressure and cold rinsing temperature on the strength of R 59.1 tex/3 mercerised combed yarn. As is seen, at a normal cylinder pressure the strength of the yarn increases as the temperature rises, with the highest strength being achieved at around 20 °C. Comparison of the regression equations shows that the effect of the cold rinsing temperature is remarkably significant in higher quality fibres and yarns. In other words, for better quality fibres and yarns (combed yarn spun from Egyptian and Uzbek cotton fibres), increasing the cold rinsing water temperature from 13 to 22 °C increases the yarn strength to an optimum level. By decreasing the quality of fibres and yarns (carded yarn spun from Iranian cotton), the effect of the cold rinsing temperature diminishes and even lies outside the significance range; hence it is excluded from the model.

Effect of the immersion time of the hank in the saustic soda bath

The immersion time in the caustic soda bath appears in the regression equations of the carded and combed yarns with

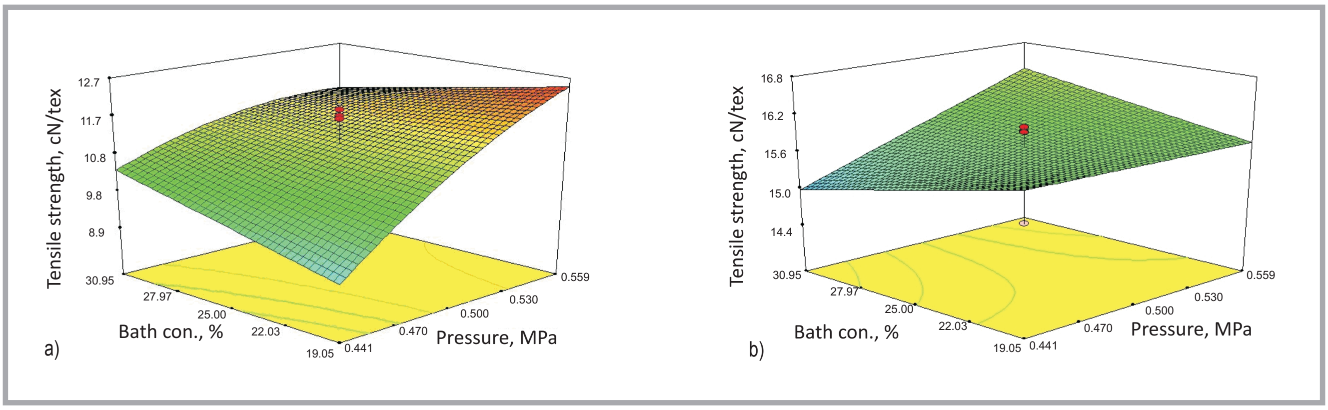

Figure 4. *Effect of cylinder normal pressure and Caustic Soda concentration on mercerized yarn strength; a) R 39.4 Tex/2 mercerized combed yarn spun from Iranian cotton, b) R 39.4 Tex/2 mercerized combed yarn spun from Uzbek cotton.*

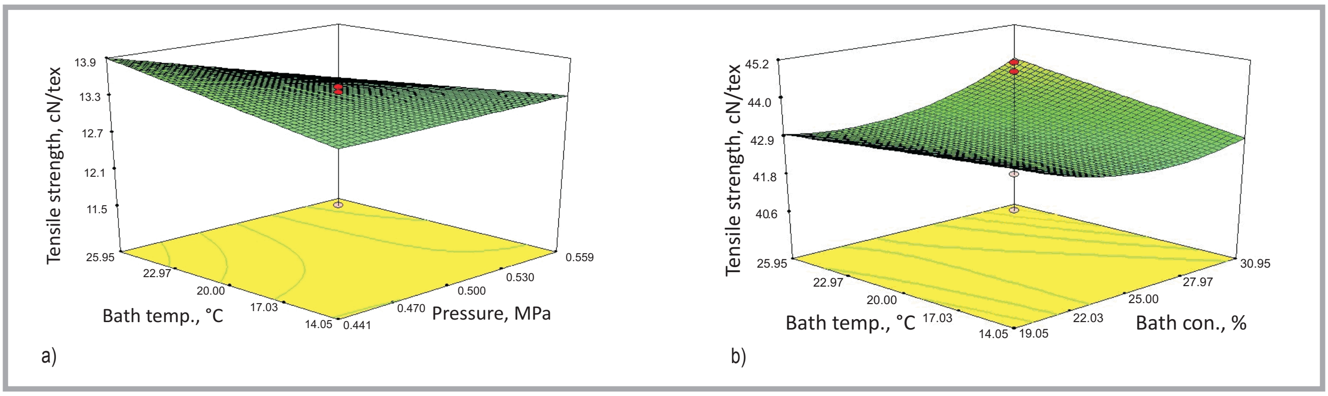

Figure 5. *Effect of cylinder normal pressure and Caustic Soda bath temperature on mercerized yarn strength; a) R 39.4 Tex /2 mercerized carded yarn spun from Uzbek cotton, b) R 39.4 Tex /2 mercerized combed yarn spun from Uzbek cotton.*

small negative and positive coefficients, respectively. ***Figure 3*** shows the effect of the cylinder normal pressure and immersion time in the caustic soda bath on mercerised yarn strength. At a cylinder normal pressure of 0.559 MPa, the strength of the yarn decreases slightly as the immersion time increases.

Effects of caustic soda bath parameters on the tensile strength of mercerised yarn

Effect of caustic soda bath concentration

The concentration of the caustic soda bath is seen in all regression equations except single and two folded carded yarns spun from Iranian cotton. The coefficients of this parameter in the regression equations of optimum settings of two folded and three folded yarns show the significant effect of this parameter on the strength. ***Figure 4.a*** shows that by increasing the concentration of the caustic soda bath, the strength of R 39.4 tex/2 mercerised combed yarn increases with a steady slope as the normal pressure rises. However, observing the change in yarn strength at each normal pressure shows that at lower pressures the strength increases by increasing the bath concentration, whereas at the maximum pressure a slight decrease is present. In ***Figure 4.b***, on the other hand, it is seen that the strength has a negative slope at lower pressures, but a positive slope at a cylinder normal pressure of 0.559 MPa.

For fine single yarns, increasing the caustic soda concentration to a level higher than the optimum leads to excessive absorption of caustic soda by the yarn, which in turn results in brittleness and a decrease in the strength of mercerised yarns. For all combed yarns, particularly multi-ply 19.7 tex, the coefficients of caustic soda bath concentration are positive, but their absolute values are less than other coefficients, meaning that for thicker and multi-ply yarns, increasing caustic soda bath concentration results in an increase in the strength.

Effect of the caustic soda temperature

The caustic soda temperature appears in the regression equations of carded and combed samples with relatively small and large coefficients, respectively. As is seen from ***Figure 5.a***, the yarn strength increases with a steady slope by increasing the bath temperature at lower pressures, whereas it decreases slightly at higher normal pressures. In ***Figure 5.b***, however, the strength increases at higher normal pressures. This slope is steeper for yarns spun from Egyptian and Uzbek cotton. The coefficients of caustic soda bath temperature are much higher for the regression equations of two folded and three folded combed yarns.

Effect of wetting agent concentration

Wetting agent concentration appears in none of the regression equations of single and two folded carded yarns. However, in the case of three folded carded yarns, it is present with small coefficients. Its influence is incremental in lower linear densities of single combed yarns (including Egyptian, Uzbek or Iranian cotton single or multi-ply combed yarns). In the case of three folded combed yarns, this parameter has a larger coefficient.

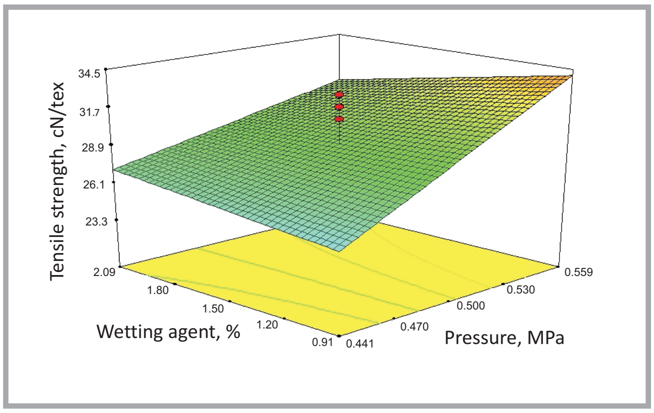

Figure 6. Effect of cylinder normal pressure and wetting agent concentration on strength of 19.7 tex mercerised combed yarn

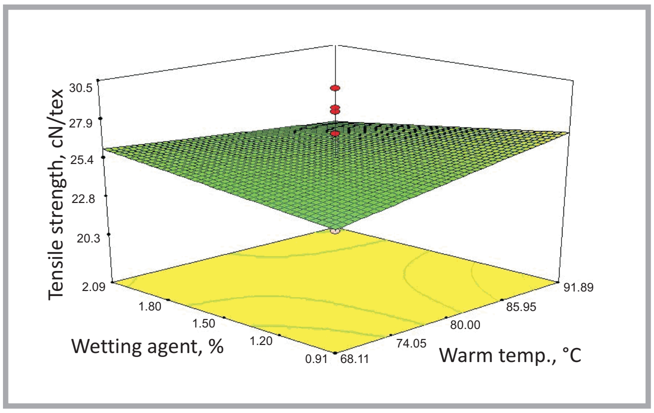

Figure 7. Effect of warm rinsing temperature and wetting agent concentration on 19.7 tex mercerised combed yarn.

Figure 6 shows the effect of the cylinder normal pressure and wetting agent concentration on the strength of 19.7 tex mercerised combed yarn, and ***Figure 7*** shows the effect of the warm rinsing temperature and wetting agent concentration on 19.7 tex mercerised combed yarn.

Effect of yarn technical specifications and machine type on optimum settings of the mercerising machine

Results of variance analysis reject the hypothesis that the linear density of yarns affects the optimum settings of the mercerising machine. However, the hypothesis that yarn structure and fibre type affect the strength of mercerised yarn is confirmed at a confidence level of 99%. This suggests that the optimum mercerising machine settings to obtain the maximum strength of mercerised yarn strongly depend on the fibre type and yarn structure.

The Duncan test also shows that at a significance level of 0.01, there is a significant difference between the optimum machine settings in achieving the maximum strength for carded and combed yarn. Variance analysis, even at a confidence level of 99%, does not reject that the number of plies affects the optimum machine settings in achieving the maximum strength. ***Table 3*** summarises some results.

According to ***Table 4***, the Duncan test also shows that a significant difference exists in the optimum settings of single, two folded and three folded yarns in achieving the maximum strength.

As is seen in ***Table 5***, the Duncan test results and comparison of the averages of fibre types show significant differences among Iranian, Uzbek and Egyptian cotton fibres in the optimum settings to achieve the maximum strength of mercerised yarn. On the other hand, the variance analysis and Duncan test demonstrate no significant difference between optimum settings of the various machines.

Table 3. *Effect of number of plies, yarn structure, fineness, fibre type and machine type on optimum machine setting; R squared = 0.415 (Adjusted R squared = 0.414).*

Tests of between - subjects effects Dependent parameter: response					
Source	**Type III sum of squares**	**df**	**Mean square**	**F**	**Sig.**
Corrected model	6.416E8	8	8.020E7	325.1	0.000
Intercept	1.493E9	1	1.493E9	6051.3	0.000
No. of layers	5.701E8	2	2.850E8	1155.4	0.000
Structure	2686700	1	2686700	10.89	0.001
Fineness	21090	1	21090	0.085	0.770
Type	6.534E7	2	3.267E7	132.4	0.000
Machine	275800	2	137900	0.559	0.572
Error	9.037E8	3663	246700		
Total	4.413E9	3672			
Corrected total	1.545E9	3671			

Table 4. *Effect of number of plies on optimum machine settings to achieve maximum strength; Based on means observed. Error term is the mean square (Error) = 246700. a) Uses harmonic mean sample size = 1224. b) alpha = 0.01.*

Duncan[a,b]				
No. of layers	**N**	**Subset**		
		1	**2**	**3**
2 No. of layers	1224	577.0		
1 No. of layers	1224		634.2	
3 No. of layers	1224			1440
Sig.		1.000	1.000	1.000

Conclusion

- Results show that some mercerizing machine settings and some technical characteristics of untreated yarn have a considerable effect on the strength of mercerised yarns, while the others have little effect.
- Cylinder normal pressure has a considerable effect on the strength of all kinds of mercerised yarns in the range studied. It is optimised at 0.595 MPa and is independent of yarn technical characteristics.
- In the next stage, the warm rinsing temperature and caustic soda bath concentration, especially for thicker yarns with a higher number of plies, have a noticeable effect on the mercerised yarn strength.
- The duration of immersion in the caustic soda bath has a diverse effect on carded and combed yarns. By increasing the immersion time, the strength

Table 5. *Effect of machine type on its optimum settings to achieve maximum strength of mercerized yarn; Error term is the mean square (Error) = 246700. a) Uses harmonic mean sample size = 1224. b) alpha = 0.01.*

Duncan[a,b]		
Machine	**N**	**Subset**
		1
Machine 2	1224	877.2
Machine 3	1224	878.1
Machine 1	1224	896.0
Sig.		0.381

of combed/carded yarns increases or decreases, respectively.

- The effect of the wetting agent concentration is elevated by decreasing the yarn linear density and/or increasing the number of plies.
- The effect of the cold rinsing temperature is more obvious in higher quality yarns and fibres. The effect of the cold rinse temperature on Egyptian and Uzbek cotton combed yarns is more obvious than for carded yarns spun from Iranian cotton fibres.
- Statistical analysis shows that fibre type, yarn structure (carded/combed) and the number of plies have a significant effect on the optimum settings of the mercerising machine in achieving the maximum strength of mercerised yarn, while the linear density of single yarns and the mercerising machine type have no significant effect.

Acknowledgments

The cooperation of Mr. Naziri, Mr. Abbaszadeh and other staff of Sabalan Silk Co., who helped us in all production processes and tests, is acknowledged.

References

1. Rollin SO, Albert WB, Frederick RA, James NG.. Physical properties of mercerized and decrystallized cottons. *Textile Res. J.* 1959; 29: 349-355.
2. Haga T, Takagishi T. Structural change in mercerized cotton fibres on cellulose treatment. *J Appl Polym Sci*. 2001; 80: 1675-1680.
3. Abrahams DH. Improving on mercerizing processing. *Am Dyestuff Reptr.* 1994; 83: 78.
4. Kim SI, Lee ES, Yoon HS. Mercerization in degassed sodium hydroxide solution. *Fibre Polym.* 2006; 7: 186-190.
5. Wakida T, Lee M, Park SJ, Hayashi A. Hot mercerization of cottons. *Fibre* 2002; 58: 304-307.
6. Yuichi Y, Kunihiro H, Yoshio S. The liquid ammonia treatment of cotton fibres. *Fibre* 2006; 62: 100-105.
7. Freytag R, Donze´J-J, Alkali treatment of cellulose fibres. In: *Handbook of Fibre Science and Technology*. Lewin M, Sello SB(Eds), Marcel & Dekker, New York, Vol. 1, Part A, 1983, 94-165.
8. McDonald AW, Rollin S. Orr, Geraldine Couturier Humphreys, James N. Grant, Physical Properties of Chemically Modified Cottons, Part III: Effects of Mercerization. *Textile Research Journal* 1957; 27; (8): 641.
9. Hari PK, Balasubramanian P, Sengupta AK, Chavan RB, conditions. Effect of mercerization on the tensile mercerization was observed to be dependent on yarn properties of rotor spun yarn. *Textile Res J* 1985; 55: 122-124.
10. Saafan AA. Effects of structural changes on thermal properties and pyrolysis of modified cotton fibres. *Journal of Thermal Analysis and Calorimetry* 1989; 35, 7: 2367-2377.

Received 25.10.2010   Reviewed 26.03.2012