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Changes in Physical Properties of Hot Mercerized Ring and Open-end Spun Cotton Yarns

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ABSTRACT

The effects of hot mercerization on open-end and ring spun yarns in slack and under tension conditions were compared. It was found that hot mercerizing at optimum temperature had different effects on cotton yarns of different structures. The results indicated more pronounced effect of treatment temperature on physical properties of ring yarn, while the mercerized open-end spun yarns showed higher dye uptake and tensile strength compared to the ring spun yarns. The degree of mercerization of ring and open-end spun yarns was obtained by measuring the barium activity number. X-ray and infrared spectroscopic analyses (the ratio of $\alpha_{1372\ cm^{-1}}/\alpha_{2900\ cm^{-1}}$) were used to measure the crystallinity of cotton yarns mercerized at various temperatures. Results showed that mercerization treatments lead to a decrease in cellulose crystallinity as assessed by determining the increase of the reactivity of cellulose towards dye sorption. The decrease in crystallinity was varied with experimental conditions. It was also revealed that the higher the mercerizing temperature, the more shrinkage occurred on both yarns, where ring spun yarns have shown higher shrinkage.

Key Words:

hot mercerizing;
yarn structure;
ring spun yarn;
open-end spun yarn;
dye uptake;
infrared spectroscopy.

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INTRODUCTION

Mercerization is one of the most common wet processing of cotton materials to improve their dyeing and physical properties. Changes in mirostructure, morphology, and conformation of the cellulose chains occur during mercerization. These changes increase sorption, tensile strength, extensibility, luster, roundness of the fibres, and also affect the handling of the resulting fabrics. The extent of the changes that occurs depends on the processing time, caustic concentration, temperature, degree of polymerization, and source of cellulose, slack or tension treatment, the degree of applied tension during the treatment and the physical state of cellulose [1-8].

Processes in which the cellulosic materials are immersed in hot caustic solution afford better penetration of the alkali into the fibres than mercerization at ambient temperature. In hot mercerization the fibres and textile structure become more pliable and less elastic than when they are treated with cold concentrated caustic solution. Hot mercerization produces high tensile strength than cold mercerization and improves dimensional stability of the fibres for two main reasons. Firstly, owing to the total penetration of the hot caustic soda into the fibre structure, a far greater proportion of the cellulose is modified [9]. Secondly in the presence of concentrated caustic soda solution at an elevated temperature, the fabric becomes highly plastic and less elastic and therefore it is capable of being readily stretched, leading to improvement in the properties of the fabric [9,10].

Another advantage of hot mercerization is that wetting agent can be avoided as it is necessary even when mercerizing grey fabrics or yarns. Furthermore, the desizing stage can be eliminated where the sizing material is starch-based, modified starch, carboxy methyl cellulose, or a synthetic size such as polyvinyl alcohol [9,10]. Wakida et al. found that crystallite of the cotton fibre was transformed from cellulose I to cellulose II by decreasing the mercerizing temperature, especially by treatment at temperature below 60ºC [4]. Ahmad et al. concluded that improvement in dye uptake during mercerization between 5ºC and 45ºC is almost the same and there was further increase in dye sorption on mercerizing at 55ºC and when using cold water instead of warm water at 70ºC for the first wash [8].

It was found that the extent of change in the physical properties of the mercerized yarn depends on its structure [11]. Huh et al. showed that structural differences in staple yarns related to the processing conditions and spinning technologies lead to different yarn properties [12]. Open-end spun yarns are known to have good uniformity, less hairiness, and better strength, but their mean breaking strength is lower than the corresponding ring spun yarns. This has been attributed to the different structure of the open-end spun yarn [11]. The mean fibre position or fibre migration, fibre packing density of the yarn and orientation angle of the yarn structure is the main parameters that determine the yarn physical properties.

Open-end spun yarn has a relatively high packing density around the yarn axis while the ring spun yarn has an intermediate packing density. The mean fibre position represents the overall tendency of fibres to be located near the surface or near the centre of the yarn. The higher value of the mean fibre position means that more fibres are located near the peripheral region of the yarn, thus the possibility of finding fibre ends increases on the outer layer of the yarn. The mean fibre position of ring spun yarn is higher than that of the open-end spun yarn [12]. Pillay et al. observed an increase of 2.5% to 25.9% in the tenacity of the open-end spun yarn, depending on the type of cotton and the degree of stretch, but detected a decrease of 10% to 20% in tenacity for slack mercerized yarn [13]. Pillay et al. concluded that mercerization is more beneficial for open-end spun yarns than ring spun yarns.

With rapid recent increase in energy costs, it would be useful to study the possibility of eliminating the scouring stage of the cotton yarn finishing process using hot mercerizing process, and to optimize the mercerizing temperature for ring and open-end spun cotton yarns. Thus, in the present study, the scouring stage was eliminated as the greige cotton yarns were mercerized and the effect of hot mercerization on ring and open-end spun cotton yarns was examined. Barium activity number, infrared crystallinity index, X-ray diffraction, moisture regaining, dye sorption, tensile strength of hot mercerized ring and open-end spun yarns were evaluated in the present study.

EXPERIMENTAL

Materials

Grade-one type cotton fibre (Gorgan, Iran) with average length of 28 mm was used in open-end and ring spinning lines to produce 100% cotton yarns. The yarns' physical properties are summarized in Table 1. Analytical grade caustic soda (99/5% purity) was purchased locally. Remazol Red RB (C.I. Reactive Red 198) a vinyl sulphone reactive dye was used in dye uptake measuring experiments.

Mercerization

Ring and open-end yarn hanks in greige state were

Table 1. Characteristics of the yarns.

Sample	Yarn count (Tex)	Twist (T/M)	Tenacity (cN/Tex)	Elongation-at-break (%)
Ring spun yarn	29.5	690	16.1	6.1
Open-end spun yarn	29.5	790	11.9	6.2

treated with caustic soda (300 g/L) solution in slack and stretched conditions at temperatures ranging from 20ºC to 90ºC with 10ºC intervals for 5 min in order to find the optimum mercerization temperature. A square shape frame with adjustable dimensions was used for applying tension on the yarn hanks. Five hanks of yarn, each 24 cm long, were fixed to their original length using a screw type stretching device installed on the frame before immersion. To remove the excess caustic soda after the end of mercerization procedure, the immersed yarn hanks were washed with hot and cold water. Any remaining alkali was finally neutralized with dilute acetic acid solution (10 g/L), followed by cold rinsing step.

Bleaching and Dyeing

Mercerized yarns were bleached in alkali hydrogen peroxide solution in an exhaustion procedure and dyed in a dyeing machine at a liquor ratio of 40:1. Dyeing solution containing 3% (o.w.f) Remazol Red RB (Reactive dye), 10 g/L Na_2CO_3 and 30 g/L NaCl was adjusted at 25ºC and then raised to 60ºC at a rate of 2ºC/min and maintained at this temperature for 90 min. Dyed samples were then washed in a boiling soap solution for removal of hydrolyzed dyes and were dried at 50ºC in an oven.

Measurements

Infrared ratio, $\alpha_{1372\ cm^{-1}}/\alpha_{2900\ cm^{-1}}$ is proposed for measuring crystallinity indexes in mercerized cellulosic yarns [14]. This ratio was obtained using an infrared (IR) spectrophotometer (IR-470, Shimadzu Co.). Measurement was performed by means of a diffuse reflection method on a tablet made by compressing cut fibre segments and potassium bromide [15]. The infrared ratio was estimated according to the literature [14]. The methods of drawing the baselines are indicated in Figure 1. For the band at 2900 cm^{-1} the intensity at the adjacent shoulder near 3000 cm^{-1} was chosen as the base. For the band at 1372 cm^{-1} a line was drawn between the maxima at approximately 1290 and 1410 cm^{-1} giving a common baseline for the group of three bands which occur close together in this region and which are changed simultaneously.

The crystallinity was measured using an X-ray diffractometer (Model: 5000, Siemens Co.). Crystallinity was defined as the ratio of the integrated crystalline scattering intensity to the total scattering intensity ranging from 10º to 45º given in eqn(1).

$$\text{Crystallinity} = \{I_c / (I_a + I_c)\} \times 100 \quad (1)$$

I_c = Integrated diffraction intensity of the crystalline region

I_a = Integrated diffraction intensity of the amorphous region

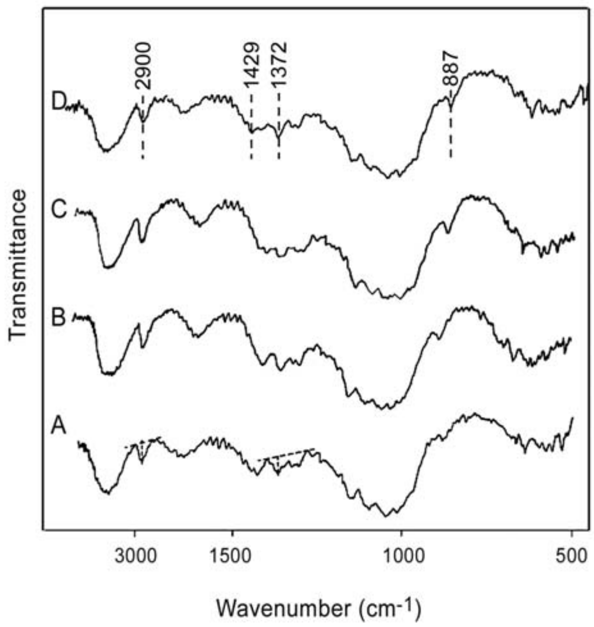

Figure 1. IR spectra of yarns with different treatment temperatures: (A) untreated yarn, (B) slack mercerized open-end spun yarn (20ºC), (C) slack mercerized ring spun yarn (20ºC), and (D) tension mercerized open-end spun yarn (20ºC).

Barium activity number was measured by AATCC test method 89-1998. One gram of mercerized and unmercerized cotton yarns were each cut into small pieces and consequently treated with 30 mL of 0.25 N barium hydroxide solution in 100 mL flasks. After 2 h, 10 mL of the solution was titrated with hydrochloric acid (0.1 N). A blank was also run without any yarn sample. For A, B , and C being the titration readings of the blank, mercerized and unmercerized samples, respectively, the barium activity number may be given as in eqn(2).

$$\text{Barium activity number} = \{(A-B)/(A-C)\} \times 100 \quad (2)$$

Dye uptake was measured using K/S values of dyed samples which were originally determined using Kubelka-Munk equation (eqn 3), where K and S are the absorption and scattering coefficients, respectively and R is the reflectance. A Tex-flash reflectance spectrophotometer set for illuminant D65 and CIE 1964 standard observer was used in colour measurements.

$$K/S = (1-R)^2 / 2R \quad (3)$$

Moisture regain was determined according to the following procedure: The hanks yarn was dried for 1 h at 105ºC (W_1), and then kept for 48 h at 65% RH (W_2). Entering the weight of dry and humid samples in eqn (4) the yarn's moisture regaining may be calculated:

$$\text{Moisture regaining (\%)} = \{(W_2-W_1)/W_1\} \times 100 \quad (4)$$

The tensile strength and elongation were measured by Zwick tensometer (Model 1446) based on the CRE method. For strength and elongation-at-break, 30 specimens were tested for each sample on the basis of standard ASTM D 2256.

Eqn (5) was used to measure the shrinkage percentage happened in slack mercerization process.

$$\text{Shrinkage (\%)} = \{(L-L') / L\} \times 100 \quad (5)$$

Where L and L' are the average length of the hanks, before and after the slack mercerization, respectively.

RESULTS AND DISCUSSION

X-ray Diffraction

The crystallinity of cotton yarns treated with caustic soda solution is shown in Table 2. As shown in Table 2, all caustic mercerization treatments caused a decrease in the crystallinity of cellulose. The decrease of crystallinity varied with experimental conditions. The crystallinity of the yarns tension mercerized is higher than that of the yarns slack mercerized as this may related to the greater degree of orientation in tension mercerization. The crystallinity of the mercerized ring spun yarn is higher than the mercerized open-end spun yarn, at all conditions. The open-end spun yarn has a relatively high packing density around the yarn axis, while the ring spun yarn has an intermediate packing density distribution [12], thus, the primary penetration of caustic soda into the open-end spun yarn is easier than that ring spun yarn.

Changes of Crystallinity Index

The infrared absorption spectra for the untreated yarn and treated yarns with sodium hydroxide are shown in Figure 1. Infrared spectra reflect the differences in the OH vibrating region at 700 cm^{-1}, and 3300 cm^{-1}. Differences were also noted at 2900 cm^{-1}, 1372 cm^{-1},

Table 2. The effect of mercerizing temperature on crystallinity of mercerized cotton yarns.

Treatment temperature (ºC)	Crystallinity of tension mercerized ring spun yarn (%)	Crystallinity of slack mercerized ring spun yarn (%)	Crystallinityof tension mercerized open-end spun yarn (%)	Crystallinity of slack mercerized open-end spun yarn (%)
20	55.3	53.7	54.2	52.3
60	55.8	54.3	54.9	53.5
80	49.3	48.8	48.8	48.2
Untreated cotton	64.0	-	-	-

Table 3. The effect of mercerizing temperature on crystallinity index of infrared ratio.

Treatment temperature (°C)	Crystallinity index of tension mercerized ring spun yarn	Crystallinity index of slack mercerized ring spun yarn	Crystallinity index of tension mercerized open-end spun yarn	Crystallinity index of slack mercerized open-end spun yarn
20	0.83	0.76	0.75	0.69
40	0.78	0.73	0.73	0.67
60	0.73	0.71	0.73	0.69
70	0.76	0.73	0.75	0.70
80	0.75	0.72	0.74	0.69
Untreated cotton	0.98	-	-	-

1429 cm^{-1}, and 893 cm^{-1}. The strong peak at 1590 cm^{-1} is an evidence for formation of hydrogen bonds in amorphous regions of the cellulose. The infrared ratio $\alpha_{1372\ cm^{-1}}/\alpha_{2900\ cm^{-1}}$ for estimating the crystallinity in cellulose samples with mixed lattices was calculated for all mercerized samples. It was suggested that this ratio should have considerable advantage in studies of natural cellulosic fibres which are subjected to caustic treatment during textile finishing [14].

The effect of mercerizing temperature on crystallinity index is shown in Table 3. According to the data, the crystallinity index of cellulose fibres is decreased in all mercerization treatments. Reduction in crystallinity index was varied with mercerization conditions. Crystallinity index was higher for yarns mercerized under tension compared to the slack mercerized yarns which may be related to the greater degree of orientation in tension mercerized yarns. It was also observed that the crystallinity index of sodium hydroxide treated yarns first decreased; reached a minimum and then increased with raising the mercerizing temperature. Initial decrease in crystallinity index can be related to the formation of cellulose II while increase in the crystallinity index can be attributed to the fact that increasing treatment temperature leads to the formation of a product similar to cellulose I. The initial decrease in crystallinity index and its further increase usually happens faster in the mercerized open-end spun yarn compared to the mercerized ring spun yarn.

It was reported that the crystallinity index of the cotton fibres first decreased and then increased with an increasing mercerization temperature [16]. The crystallinity index is higher for mercerized ring spun yarn compared to the mercerized open-end yarn in all conditions which is in agreement with the X-ray results. The results indicated the more pronounced effect of treatment temperature on crystallinity index of the ring spun yarns.

Barium Activity Number

The barium activity number is widely used to express the degree of mercerization [2,11]. Table 4 shows the barium activity number of mercerized yarns in various mercerizing temperatures. Based on results shown in Table 4, the barium activity number of mercerized yarns has increased by increasing the treat-

Table 4. The effect of mercerizing temperature on barium activity number of mercerized yarns.

Treatment temperature (°C)	Barium activity number of tension mercerized ring spun yarn	Barium activity number of slack mercerized ring spun yarn	Barium activity number of tension mercerized open-end spun yarn	Barium activity number of slack mercerized open-end spun yarn
20	139	146	142	152
40	144	152	147	152
60	148	153	145	151
70	142	149	140	146
80	141	148	143	150

ment temperature. Boost in mercerization degree was higher in ring spun yarn compared to the open-end spun yarn during the hot mercerization procedure. Slack mercerized yarns usually showed higher barium activity number compared to the yarns mercerized under tension. Easier diffusion of chemicals in the slack mercerization process can be the main reason behind this phenomenon which is in agreement with the reduction in crystallinity level. It is reported that the barium activity number increases as the immersing time increases and the barium activity number of cotton mercerized with the degassed solution is higher than that of cotton treated with the saturated solution, Thus, the penetration ability of alkali into the yarn structure is a very important factor [2].

The barium activity number of mercerized yarns in most cases first increased, reached a maximum, and then diminished with further increase in treatment temperature. There is an inverse correlation between crystallinity index and barium activity number of mercerized yarns at various temperatures, as confirmed by the data shown in Tables 2, 3, and 4.

Moisture Regain

Generally, the crystalline region in cotton fibres is decreased by mercerization thus, the mercerized cotton fibres have higher moisture regain compared to unmercerized fibres. Table 5 shows the changes in moisture regain of slack mercerized cotton yarns at different temperatures. The moisture regain of untreated cotton was 7.5%. Mercerized open-end spun yarns at 40ºC and mercerized ring spun yarns at 60ºC show the highest level of moisture regain with 10.19% and 10.4%, respectively. The moisture regain of sodium hydroxide treated yarns first increased, reached a maximum, and then decreased by increasing the treatment temperature. This can be related to the changes in the crystallinity of fibres due to temperature change. Saapan et al. reported that cellulose II is formed in cotton fibres following their treatment in sodium hydroxide [16]. The extent of conversion was believed to be dependent on the experimental conditions, i.e., caustic mercerization at 20ºC caused higher conversion from cellulose I to cellulose II than what is obtained by swelling in NaOH at 90ºC.

Table 5. The effect of mercerizing temperature on moisture regain of slack mercerized yarns.

Treatment temperature (ºC)	Moisture regain of slack mercerized Ring spun yarn (%)	Moisture regain of slack mercerized open-end spun yarn (%)
20	9.10	9.39
40	10.02	10.40
60	10.19	10.05
70	9.06	9.15
80	8.80	8.90
Untreated cotton	7.50	-

Dye Uptake

The mercerized yarns were bleached in hydrogen peroxide and caustic soda and then dyed with a reactive dye. Increase in dye uptake of mercerized samples is shown in Figures 2 and 3. The results indicate a higher dye uptake of mercerized samples compared to the unmercerized yarns. This effect could be related to the destruction of crystalline regions during the swelling and structural change of mercerized cotton. The dye uptake of slack mercerized yarns was higher than that of yarns mercerized under tension. Mercerized open-end yarns have gained higher increase in their dye uptake compared to the ring spun

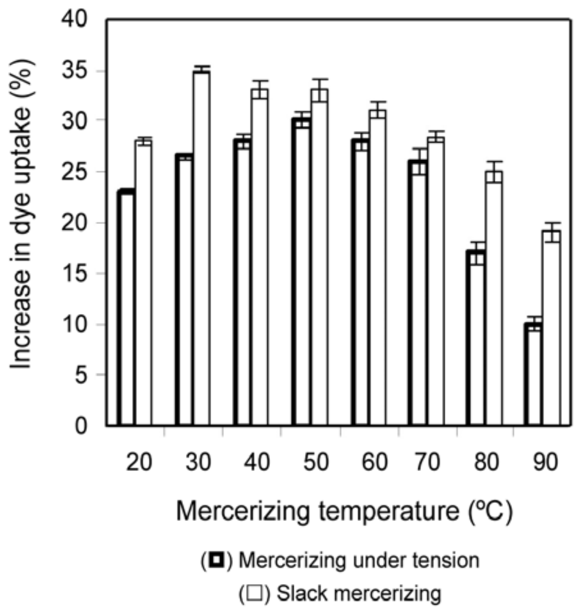

Figure 2. Effect of mercerizing temperature on dye uptake of open-end spun yarn.

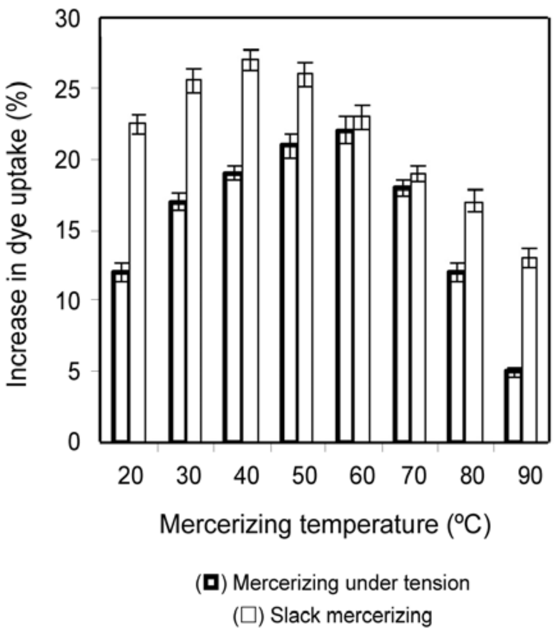

Figure 3. Effect of mercerizing temperature on dye uptake of ring spun yarn.

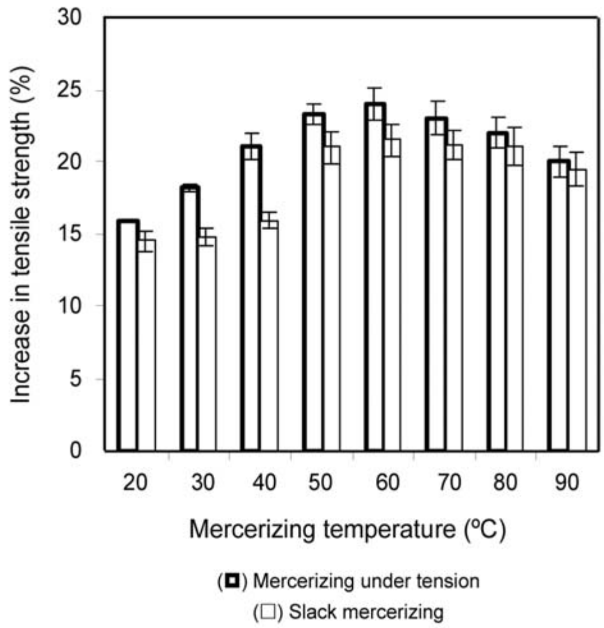

Figure 4. Effect of mercerizing temperature on tensile strength of ring spun yarn.

yarns which could be related to their porous yarn structure. The results have also shown that slack mercerization at 30ºC for open-end spun yarns and at 40ºC for ring spun yarns lead to the highest level of 35% dye uptake for the open-end spun yarn and 27% for the ring spun yarn. Maximum dye uptake increase was observed at 50ºC for the tension mercerized open-end spun yarn and at 60ºC for ring spun yarns which were about 30% and 22%, compared to unmercerized yarns, respectively. It has been reported that the mercerized fabrics show maximum dye uptake at 55ºC [8].

Tensile Strength

Figures 4 to 5 show the tensile strength of the mercerized ring and open-end spun yarns. The tensile strength of the slack and under tension mercerized yarns clearly show increases in all conditions. The major reasons for the increased tensile strength may be related to the alleviation of internal stresses and the deconvoluting of the cotton fibres in the yarn during swelling process.

Based on observations, the higher tensile strength of the mercerized open-end spun yarn compared to ring sample can be due to the fibre packing density difference or the mean fibre position. The mean fibre position of ring spun yarn is higher than that of the open-end spun yarn [12]. Mercerization increases the yarn tensile strength by improvement of fibre packing, fibre orientation, and fibre tenacity. Thus, improvement in tensile strength of mercerized open-end spun yarns was greater than the mercerized ring spun yarns. Hari et al. concluded that tension

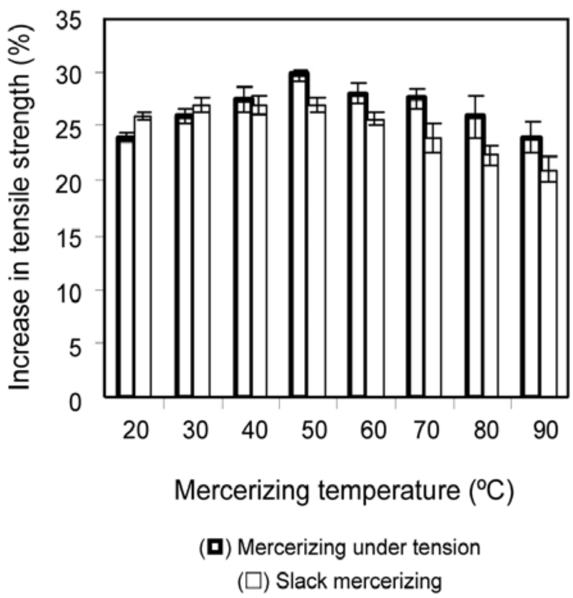

Figure 5. Effect of mercerizing temperature on tensile strength of open-end spun yarn.

mercerization increases yarn tenacity primarily by improvement of fibre packing [11]. Tension mercerization at 50ºC for open-end spun yarns and at 60ºC for ring spun yarns resulted in the highest level of increase in tensile strength with 30% for the open-end spun yarn and 24% for the ring spun yarn. In addition, the results have revealed that slack mercerization at 40ºC for open-end spun yarns and at 50ºC for ring spun yarns leads to the highest level of tensile strength with 27% for the open-end spun yarn and 22.5% for the ring spun yarn, compared to un-mercerized yarns, respectively.

Elongation-at-break

One of the important mechanical properties of various kinds of yarn is breaking elongation. Huh et al. reported that yarn rupture depends on the mean fibre position in the yarn where fibres placed parallel to the yarn axis could be crucial to rupture elongation [12]. The breaking elongation of ring spun yarn is lower than that of the open-end spun yarn. The low percentage of breaking elongation of ring spun yarn can be explained by the axis-parallel alignment of fibres in the yarn centre [12]. The elongation-at-break for the mercerized ring and open-end spun yarns are shown in Tables 6 and 7. As shown in these Tables, an increase in treating temperature could enhance the elongation of slack mercerized ring and open-end spun yarns, while the elongation of the tension mercerized samples decreased which could be related to the improvement of fibre packing in the yarn. The elongation of mercerized open-end spun yarns in slack state is higher than comparable ring samples which may be due to the easier penetration of caustic soda into the open-end spun yarn structure.

Table 6. The effect of mercerizing temperature on elongation-at-break (%) of ring spun yarns.

Treatment temperature (ºC)	Elongation-at-break of slack mercerized ring spun yarn (%)	Elongation-at-break of tension mercerized ring spun yarn (%)
20	4.5	4.8
40	4.8	4.9
60	6.0	4.7
70	6.4	3.4
80	8.2	3.7
90	7.8	3.9

Table 7. The effect of mercerizing temperature on elongation-at-break (%) of open-end spun yarns.

Treatment temperature (ºC)	Elongation-at-break of slack mercerized open-end spun yarn (%)	Elongation-at-break of tension mercerized open-end spun yarn (%)
20	4.6	4.2
40	5.0	4.6
60	6.3	3.4
70	7.8	3.5
80	9.0	3.3
90	10.4	3.4

Shrinkage

Shrinkage of slack mercerized ring and open-end spun yarns at various temperatures is shown in Table 8. The experimental results show that increase in treatment temperature enhances the shrinkage in yarn length. The maximum shrinkage was observed at 80ºC for both ring and open-end spun yarns. Shrinkage in the ring spun yarns was higher than that of the open-end spun yarns. This may be due to the difference in fibre packing between both yarns. The open-end spun yarn has a relatively high packing density around the yarn axis while the ring spun yarn has an intermediate packing density distribution [12]. Mercerization at temperatures lower than 30ºC and higher than 70ºC have resulted in lower shrinkage

Table 8. The effect of slack mercerizing temperature on shrinkage (%) of ring and open-end spun yarns.

Treatment temperature (ºC)	Shrinkage of slack mercerized ring spun yarn (%)	Shrinkage of slack mercerized open-end spun yarn (%)
20	5.7	5.1
40	8.5	7.0
60	15.5	13.9
70	16.9	15.3
80	23.5	22.6
90	22.9	21.8

differences between both yarn types. This effect at higher temperature could be related to the easier penetration of caustic soda into the yarn structures.

CONCLUSION

Ring and open-end spun yarns made up of 100% cotton fibres were treated with caustic soda solutions at different temperatures, under tension and relaxation conditions. The extent of changes occurred during the mercerization was observed to be dependent on yarn structure, treatment temperature, and slack or under tension style of treatment. Treatment temperature has shown different effects at optimum temperature on ring and open-end spun yarns. The effect of treatment temperature on physical properties of the ring spun yarn was higher than that of open-end spun yarn, while the mercerized open-end yarn shows higher degree of mercerization compared to ring spun yarn. The dye uptake, moisture regaining, and barium activity number of the mercerized yarns first increased and reached to a maximum and then decreased by increasing the treatment temperature.

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