Examining the Effect of Mercerization Process Applied Under Different Conditions via the Degree of Whiteness and Color Efficiency

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ABSTRACT: In this study, 100% cotton woven fabrics having three different patterns are mercerized by chained mercerizing machine. The temperature is kept at 60°C for all applications. Application types and process period are the variables of this experiment. The effects of mercerization process applied on different conditions are examined in terms of color efficiency and degree of whiteness. Fabrics with different patterns are also compared with each other to understand whether fabric patterns are effective in whiteness and color efficiency. © 2011 Wiley Periodicals, Inc. J Appl Polym Sci 121: 202–209, 2011

Key words: mercerization; finishing; cotton fabric; the degree of whiteness; color efficiency

INTRODUCTION

Finishing processes applied on woven fabrics include all applications that are performed to gain desired properties to the fabric according to the customer's demands after weaving. It is possible to gain structures, which are similar to superior properties of synthetic fibers, to cotton fiber with modern finishing processes applied nowadays. One of the prefinishing processes changing the characteristical and physical properties of fiber and the most important one is mercerization.

The mercerization process is based on the principle of passing fabrics or yarns through 15–20% cold caustic soda (sodium hydroxide) solution. After being exposed to sodium hydroxide, yarn or fabric is rinsed again and again. Fabric is subjected to tension by a stenter on which most quantity of caustic soda is removed by hot water and basic residuals are neutralized by cold acidic liquor. With liquor application, removal of the residual acid is provided. This is a continuous process. For conventional mercerization, the fabrics or yarns are exposed to tension during finishing process. Good results will be taken with an appropriate saturation, adequate tension, and completely fine washing. Moreover, the same results can be held for round-knitted products by the aid of special equipments.[1,2]

Mercerization processes applied in different working conditions have different effects. In other words, structural and physical properties of mercerized material are completely different from those of the raw material. These changes are observed in terms of luster, degree of whiteness, hydrophilicity, and strength.[3]

Some heavy metals carried by textile material, water, or machine parts cause some problems in textile industries. Therefore, acid usage is common to remove these metals in prefinishing processes. Cotton material is bleached with peroxide after it has been treated with sulfuric, citric, hydrochloric, *o*-phosphoric, and gluconic acid. Degree of whiteness, hydrophilicity, mineral material content, and desizing degree are investigated. These are compared with conventional prefinishing applications.[4]

Enzymatic processes are investigated to be an alternative for conventional applications. Materials are desized with one-step enzymatic process and treated with two-step enzymatic process and bases. These specimens are compared with raw material. It is reported that enzyme type and concentration have an important effect on hydrophilicity, degree of desizing, weight loss, degree of whiteness, and dyeability.[5]

Mercerization process

Because of the use of concentrated sodium hydroxide liquor, reactions occurring with cellulosic fibers are intramicellar reactions. In other words, sodium hydroxide liquor with this concentration penetrates into

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micellars, and a composition called hydrate cellulose is constructed.[6]

Mercerization changes cellulose at either molecular or macromolecular level. For instance, absorbency of mercerized fibers increases because of the changes seen on fiber structure.[7]

Reduction in crystallinity and increase in amorphous region lead to increase in reactivity of hydroxyl groups of cellulosic fibers.[8] Some structural changes are observed during mercerization process, such as

- Increase in solubility
- Increase in absorption of fiber
- Increase in reaction with oxygen
- Increase in luster of fiber[6]

Effects of mercerization

Fiber structure

Cotton fiber has a kidney-type cross-sectional view and a ribbon-like twist longitudinal view. As a result of swelling during mercerization process, the fiber cross-sectionally changes from kidney shape to round and longitudinally enlarges to cylinderic form.[6]

Luster

Luster is related with light permeability and reflection. Because of uneven decrease in fiber surface, light reflection increases. As a result of this, yarns or fabrics have lustrous appearance.[6,9]

Hydrophilicity

Mercerization is a process decreasing hydrophilicity. Being the last process among prefinishing applications is the disadvantage for mercerization process. Therefore, the best hydrophilic values are observed in raw material mercerizations. Because low hydrophilicity resulted from mercerization is developed by further applications.[3]

Decrease in hydrophilicity is a result of changes on crystalline areas of cotton fiber. It is considered that hydrophilicity changes on the fabric side and in the middle of fabric. In other words, hydrophilicity is not same along the fabric length and width. This causes color differences along fabric surface.[10]

Parameters of mercerization process

- Temperature: For optimum swelling, the temperature must be kept between 10°C and 15°C because the viscosity of chemical (sodium hydroxide) decreases at low temperature.[11]
- Time: It was found that the required time for swelling is 60 s when a scoured cotton fabric is immersed into mercerization flotte. However, this time is decreased by the help of mechanical effect of mercerizing machines. In a chainless mercerizing machine, required time for dry product is 12 s to supply full impregnation with sodium hydroxide flotte, and it is 20 s for wet product.[9]
- Chemical concentration: Specimens having same structural and physical properties are subjected to mercerizatoin with different concentrations. It is seen that mercerization results are not same in different concentrations.[9]
- Mechanical effect: Mercerization process makes fiber structure more regular, and also it causes crystalline areas to replace more parallelly to fiber axis. Thereby, the number of bonds between molecules increases. Unfortunately flexibility decreases.[12]

MATERIAL AND METHOD

Material

In this study, fabrics that are constructed with 20/1 Ne open-end 100% cotton yarn and chosen from three different patterns will be used. Fabric weights of all specimens are the same. Properties of these fabrics are given in Table I.

Machines and devices

Experiments made on fabrics are performed by using machines and devices in Kipaş Dyeing and Finishing Departments. These machines and devices are as follows:

- Osthoff Scouring and Desizing Machine in which desizing and scouring are combined.
- KÜSTERS Combined Bleaching Machine in which hot bleaching can be done. It has six

TABLE I
The Properties of raw Material Used in This Study

	Plain weave	Twill	Dobby weave
Warp yarn Ne	20/1 OE	20/1 OE	20/1 OE
Weft yarn Ne	20/1 OE	20/1 OE	20/1 OE
Warp sett	38.3	39.7	41.4
Weft sett	16.5	21	21
Raw material width (cm)	159.5	160	159.5
Raw material weigth g/m^2	181	202	207
Warp strength of raw material (kgf)	86.25	96.65	94.25
Waft strength of raw material (kgf)	28.75	37.25	37
% Warp shrinkage of raw material	−14.5	−14.5	−14
% Weft shrinkage of raw material	−4	−4.5	−4.5

washing cabins, with two at the front and four at the rear.
- BENİNGER Mercerizing Machine has five washing cabins at the rear and has stabilization unit and chained-tenter unit after caustic cabin.
- WUMAG Drying Machine with contact-drying system provides exit-moisture control with pleva device and has 20 drying drums running with steam.
- KÜSTERS Pad-batch Dyeing Machine in which padding of dye and chemical is performed according to the method of cold-padding has unique dyeing bath.
- Beaker, graduated cylinder, pipette, titration assemblies, etc., are used for chemical tests at the laboratory of chemical.

SDL strength device, full-automatic washing machine, and drum-drying machine are used for physical tests in laboratory of product test.

Chemicals and their properties

Desizing.

- Enzyme: It is used for desizing. Torazym NT is sensitive to high temperature and pH value is 6.5. It is a kind of alpha amylase enzyme.
- Antifoaming agent: It is used to prevent foaming. Foaming occurs because of material and flotte motion. Antifoaming agent must be added because it causes decrease in surface tension. Schnellnetzer NT is used as antifoaming agent. It is produced by Textile Color.
- Wetting agent: It is used to decrease surface tension between material and flotte. In this study, Schnellnetzer NT is preferred. It is a nonionic and colorless wetting agent produced from phosphoric acid esters and oil–alcohol polyglycol ethers. Its pH value is 7. It is produced by Textile Color.
- Emulgator: It is used to mix two or more liquids homogenously without phase formation. Emulgator BEO is a nonionic, colorless derivative of ethylene oxide condensation. Its pH value is 7. It is produced by Textile Color.
- Washing agent: It is used to remove remained chemicals and impurities from material surface. Nevertheless soap is insufficient to remove impurities and poor to resist. Colorless and nonionic washing agent is preferred in this study. It is produced by Textile Color.
- Air removing: It is used to prevent air formation between material and flotte. Rotta Entlüfter BK is a nonionic and colorless chemical and also an organic hydroxyl compound. Its pH value is between 4.5 and 6.5. It is produced by Rotta.[14–15]

Bleaching.

- Sodium hydroxide: It is a strong base having 13–14 pH value. It is the main chemical used in bleaching. It is a colorless chemical.[13]
- Hydrogen peroxide: It is an oxidizing agent and used for increasing the pH value. It is generally available as 50% concentrated form. It is a colorless and odorless chemical.[13]
- Gemsol WA: It is a kind of wetting agent with synergic and stable composition of surface-active materials. It is a nonionic and colorless chemical with a pH value of 7.
- Sevalin D: It is used for increasing hydrophilicity. It consists of oil aminopoly-glycolether. It is a nonionic, yellow-colored chemical with a pH value of 9–10.
- Rohstoff ST/OS: It is a kind of bleaching stabilizer used for preventing phase formation in an emulsion solution. It is a kind of organic acid derivative. It has a brown color, and a pH value of 8.5–9.5.

Other auxiliary chemicals.

- Floranit 4028: It is a kind of wetting agent used in mercerization process. It is an anionic and yellow-colored chemical with pH value of 10–11. It is a derivative of alcohol.
- Erkantol AS: It is a wetting agent produced from inorganic acid ester. Its color is yellow, and it is an anionic chemical with a pH value between 6 and 9.
- Silikat: It consists of sodium hydroxide and silisium dioxide. It works as a kind of buffer solution with a pH value of 12. It is generally available with a concentration of 38–40°Be.[14–15]

Method

Prefinishing processes are applied to fabrics by considering recipes and conditions listed below before mercerization process. First of all, scouring and desizing process is applied to fabrics.

Scouring and desizing process is performed by OSTHOFF-patented Scouring and Desizing Machine in which a multifunctional scouring machine and a bath used for impregnation of desizing agent to fabric are combined.

Recipe: 2.5 g/L Torozym NT, 2 g/L Schnellnetzer KE, 1 g/L Emülgatör BE-O, 1 g/L Entlüfter BK; speed: 70 m/min; burner position: vertical/double face of

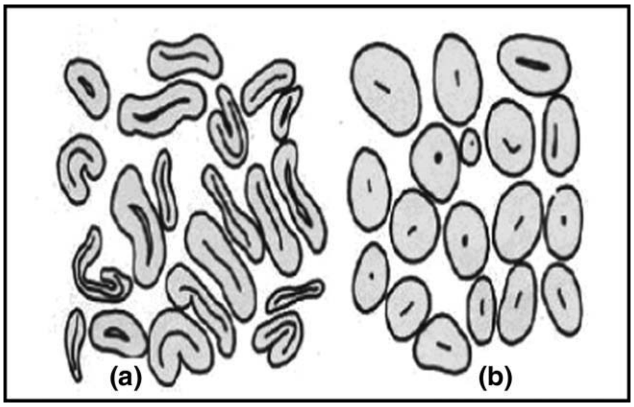

Figure 1 Cross-sectional views of cotton fiber (a) without mercerization and (b) after mercerization.

material; burner distance: 8 mm; pH: 6–7, temperature: 60–65°C, and waiting: 6 h by rotating on beam.

Recipe shown below is applied to fabric to make CMC sizing agent, with which the fabric is sized, soluble in water. Rotation machine is used to balance and to prevent dripping on any side. After rotating sized fabric during 6 h, it is exposed to bleaching process.

Bleaching process is performed with Mega-Bleaching Machine, which is marketed by KÜSTERS and has two prewashing cabins, Flexnip for impregnating solution, steam cabin suitable for stuffing, and four washing machines for output washing. Technical data and recipe are as follows:

Recipe: 35 mL/L %50 H_2O_2, 15 mL/L NaOH 48°Be, 2 mL/L Gemsol W, 2 mL/L Rohstoff ST-OS, 1 mL/L Sevalin D; speed: 60 m/min; steam cabin: stuffing; and waiting: 20 min, 98°C.

The temperatures of prewashing and washing cabins are adjusted to 90–95°C. Fabrics taken from rotation are bleached by considering the recipe above and by being passed through working conditions in bleaching machine. Then drying or mercerizing is applied according to the stage of process.

Drying is performed by WUMAG-patented drying machine, which operates with contact drying method and has 20 drums heated by steam. Moisture of the output material is controlled by Pleva hygrometer. Technical data are as follows: speed, 40 m/min; temperature, 140°C at drum drying machine; and moisture, 8% ± 2%.

Mercerization process is applied to the fabrics after drying process. Mercerization process is performed according to the plan by using caustic cabin at 60°C and 30°Be NaOH in chained-mercerizing machine produced by BENİNGER. This process is done by stretching fabric length at a ratio of 3% along warp direction and by stretching fabric width till +1 cm at pinned-tenter after stabilization part along weft direction.

TABLE II
Mercerization Experimental Plan of Fabrics

Experiment number	Mercerization conditions: Process level	Time of exposuring to caustic bath (s)
1	Drying after bleaching (dry)	15
2	Drying after bleaching (dry)	10
3	Drying after bleaching (dry)	7.5
4	Drying after bleaching (wet)	15
5	Drying after bleaching (wet)	10
6	Drying after bleaching (wet)	7.5
7	Mercerization with wetting agent after bleaching (wet+wetting agent)	15
8	Mercerization with wetting agent after bleaching (wet+wetting agent)	10
9	Mercerization with wetting agent after bleaching (wet+wetting agent)	7.5

Fabrics are dyed with reactive dyestuffs after mercerization process. Dyestuffs and chemicals are impregnated into fabric by KÜSTERS-patented pad-batch dyeing machines working with unique padding and squeezing method, and dyed fabrics are rotated for preventing dripping during fixation. Technical data and recipes are as follows:

Recipe: 14.8 g/L Cibacron blue CR, 5.6 g/L Cibacron deep red CD, 12.1 g/L Cibacron yellow CRG, 1 g/L Erkantol AS, 4.84 g/L caustic and 48 °Be, 50 g/L silicate; fixation: waited by rotating 14 h after dyeing. After fixation, fabrics were washed by hot-boiling washing and dried at 120°C.

Experimental plan of fabrics being applied on mercerizing machine

Fabrics having the properties shown in Table I were evaluated by being passed through mercerizing machine according to plan below. First, second, and third samples were dried after bleaching and before

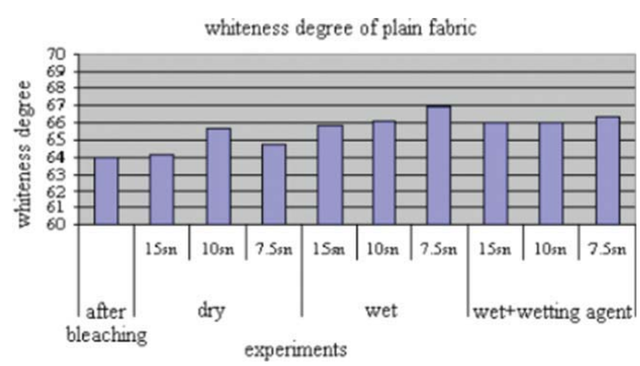

Figure 2 Changes in whiteness degree of plain fabric depending on process and time. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

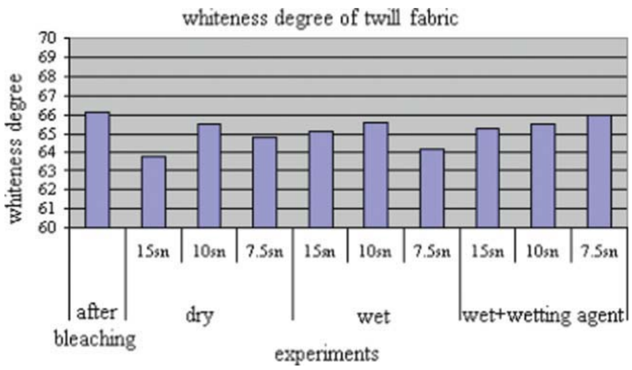

Figure 3 Changes in whiteness degree of twill fabric depending on process and time. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

mercerization process and were fed into caustic cabin as dry material. Fourth, fifth, and sixth samples were fed into caustic cabin as wet material after bleaching process. Seventh, eighth, and ninth samples were fed into caustic cabin without drying as wet material, and 5 g/L wetting agent was added into caustic cabin.

After applying finishing processes to the fabric samples, whiteness degree of these samples were measured. Measurement was done by considering D65/10 daylight in terms of Berger at Macbath Color Eye 7000 Spectrophotometer device. Samples whose values were till 100 refer to whiteness levels of ready-to-dyeing white fabrics. Samples whose values are 100 or 100 above refer to optical values of fabrics.

Samples that were taken from fabrics dyed by dyestuffs in reactive dyestuffs recipes under the same conditions were read by Macbath Color Eye 7000 Spectrophotometer device. By taking up references of fabric not mercerized after bleaching, color efficiency and deepness of samples were compared. Comparison is done under the light of D65/10 daylight by considering dL values.

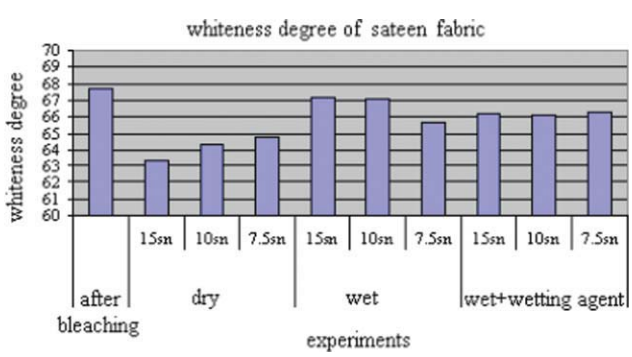

Figure 4 Changes in whiteness degree of dobby woven fabric depending on process and time. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

TABLE III
Color Efficiency of Plain Fabric

Mercerization conditions		Experiment number	Difference between readings	Efficiency (%)
Bleached fabric			Reference	0
Dry	15 sn	1	4.73	98.3
	10 sn	2	4.66	96.9
	7.5 sn	3	4.14	86.1
Wet	15 sn	4	4.81	100
	10 sn	5	3.99	82.9
	7.5 sn	6	3.85	80
Wet + wetting agent	15 sn	7	4.76	98.9
	10 sn	8	4.39	91.3
	7.5 sn	9	4.23	87.9

RESULTS AND DISCUSSION

Results of whiteness degree determination

Fabric type is important on whiteness degree. It was shown that mercerization is a factor on whiteness degradation at twill and dobby woven structures, but results are two or three times higher at plain woven structures corresponding to bleached fabric. As it is seen in Figures below, fabric types having maximum whiteness degradation are dobby woven fabrics because maximum whiteness degrees are obtained on dobby woven fabrics between bleached fabrics. Samples on which mercerization is applied as dry process generally have the least whiteness degrees. Drying temperature is important because yellowing seen after drying can affect this process. The results of whiteness degrees determined by all samples are approximately between 65 and 66 values. Maximum whiteness degree is obtained at plain woven structures as wet processing with the average result of 66.2. The best results for twill fabric is 65.6 with wet processing by using wetting agent and for dobby woven fabric is 66.6 with only wet processing. The results obtained after wet processing are better than those obtained after dry process. However, adding wetting agent does not cause a

TABLE IV
Color Efficiency of Twill Fabric

Mercerization conditions		Experiment number	Difference between readings	Efficiency (%)
Bleached fabric			Reference	0
Dry	15 sn	1	8.69	100
	10 sn	2	7.74	89.1
	7.5 sn	3	7.7	88.6
Wet	15 sn	4	8.17	94
	10 sn	5	6.82	78.5
	7.5 sn	6	5.96	68.6
Wet + wetting agent	15 sn	7	7.16	82.4
	10 sn	8	6.86	78.9
	7.5 sn	9	6.79	78.1

TABLE V
Color Efficiency of Dobby Woven Fabric

Mercerization conditions		Experiment number	Difference between readings	Efficiency (%)
Bleached fabric			Reference	0
Dry	15 sn	1	7.18	100
	10 sn	2	7.14	99.4
	7.5 sn	3	5.71	79.9
Wet	15 sn	4	5.6	78.4
	10 sn	5	5.5	77
	7.5 sn	6	5.2	72.8
Wet + wetting agent	15 sn	7	5.31	74.4
	10 sn	8	5.34	74.7
	7.5 sn	9	4.9	68.6

difference. Each result differs approximately one or two times higher, but not clear in observation.

Results of color efficiency determination

Comparison of color efficiency is performed by using Macbath Color Eye 7000 Spectrophotometer device. Mercerized samples are read one-by-one by taking "fabric dyed after bleaching" as reference. Then, prints about the differences of dL read on screen and results are graphically printed. These graphics are seen on Figures 5, 6, 7, 8, 9 and 10. The highest difference shows 100% efficiency, and the other differences are calculated according to this value. At the process of dyeing after mercerization, dyestuff is used approximately 30–40% less than other dyeing processes. When mercerized fabrics are dyed by using dyestuff 30% less than conventional dyeing at laboratories, the obtained color is the same as seen on fabric dyed after bleaching according to normal recipe, but more exhilarated and lustrous.

Drying process applied before mercerization increases color efficiency. If wet and dry processed fabrics are compared with each other, samples exposed to dry processing are more efficient than wet processed fabrics.

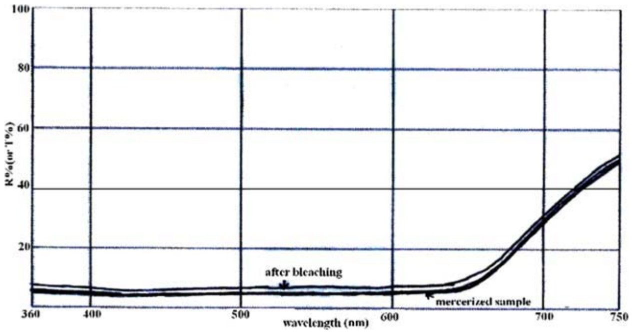

Figure 5 Visible spectrum graphic of plain fabric. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

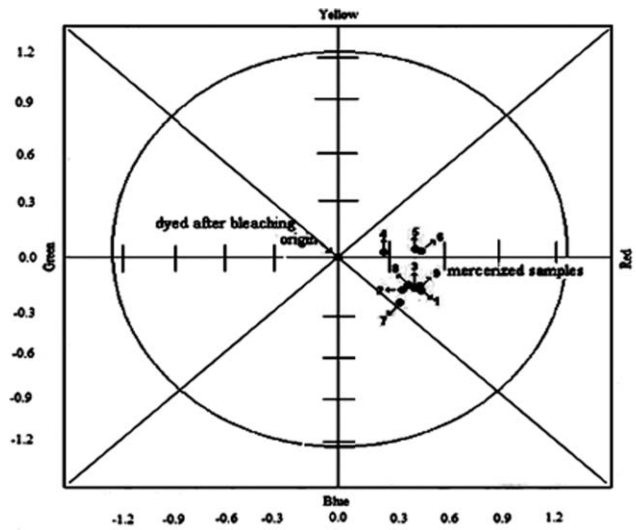

Figure 6 Color space graphic of plain fabric.

On comparing differences of color readings, it is noted that the highest differences are determined on samples treated during 15 s. Because samples of wet processing give highly different results, sample treated during 15 s of wet processing is called as 100% efficient. Others are determined according to this.

If the time of caustic treatment is decreased, swelling of fibers decreases and color efficiency decreases too. As seen in Table III, decrease in treatment time causes a decrease in difference of color readings; therefore, color efficiency decreases. This difference is extremely observed between samples exposed to wet processing in plain fabrics.

As it is seen from Figure 5, R% in visible spectrum of fabric dyed after bleaching is higher because this fabric reflects less light (because of dispersion of light indiscriminately). Mercerized samples reflect more light; therefore, the color of fabric is dark, and R% is lower (because of reflection of light without dispersion).

Color space graphic is obtained by comparing mercerized samples with accepted bleached fabric origin, and it is shown in Figure 6. When comparing

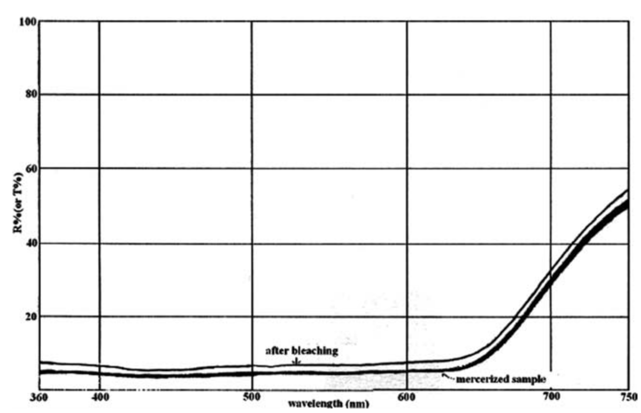

Figure 7 Visible spectrum graphic of twill fabric.

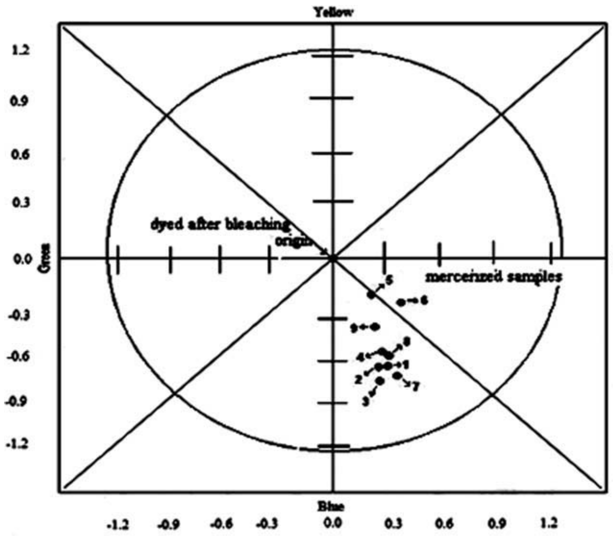

Figure 8 Color space graphic of twill fabric.

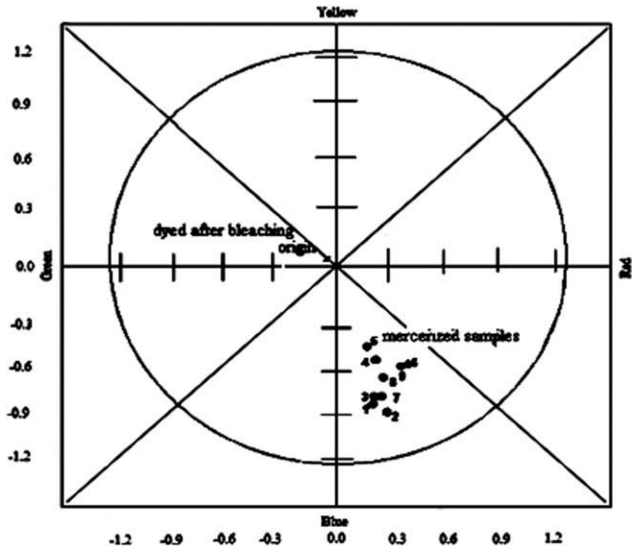

Figure 10 Color space graphic of dobby woven fabric.

the differences at color readings of twill fabric, the highest difference was seen for sample treated during 15 s. Because the highest reading difference is in dry processing and at the sample treated during 15 s of twill fabric, this sample has 100% efficiency.

As seen in plain fabric, if the treatment time of caustic soda decreases (swelling of fiber decreases), decrease in color efficiency is also seen in twill fabrics. As it is seen in Figure 7, color efficiency decrease on wet-processed fabric is higher than that of others.

Figure 7 shows the visible spectrum of twill fabric. As well as it is noticed in plain fabric, *R*% of bleached fabric is higher than that of other samples. In other words, color of sample is seen lighter than the color it is dyed. Figure 8 shows the color space graphic of twill fabric.

When comparing the differences between color readings of dobby woven fabric, all values obtained from dry-processed samples have higher results than both wet processed and wet processed + wetting agent samples. A 100% efficiency was obtained at dry-processed sample treated during 15 s because higher difference is seen on reading of this sample.

It was noticed that decrease depending on time seen on plain and twill fabrics causes higher differences on dobby woven fabrics, and results of other processes are close to each other. Visible spectrum graphic of dobby woven fabric is demonstrated in Figure 9. Same result is determined as the other fabric types have, and *R*% of samples are lower because of darker samples. Color space graphic of dobby woven fabric is given in Figure 10.

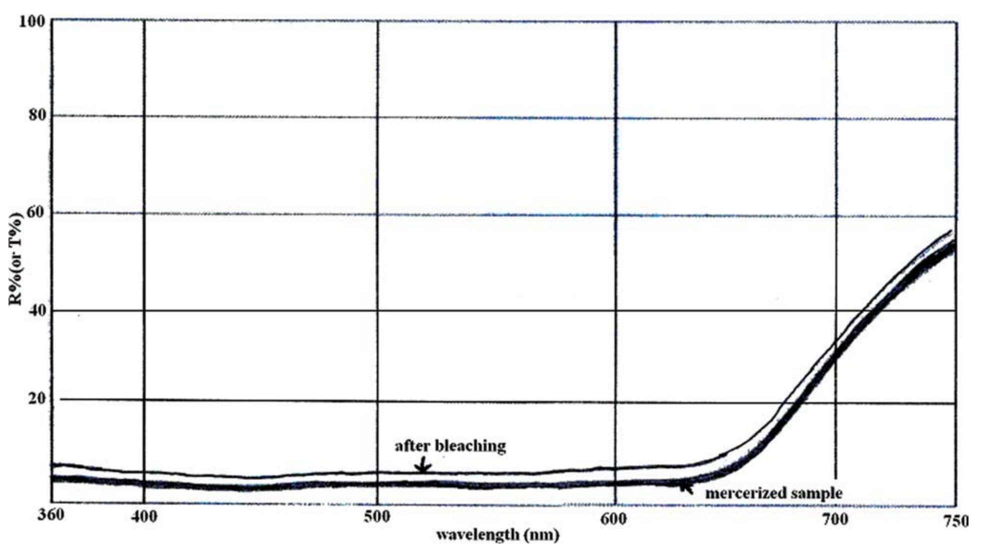

Figure 9 Visible spectrum graphic of dobby woven fabric. [Color figure can be viewed in the online issue, which is available at wileyonlinelibrary.com.]

If it is desired to evaluate generally, fabrics mercerized slowly seem darker and percentage of color efficiency is determined higher. Clear difference of faster process seen on plain and twill fabrics is not obtained either on wet-processed or wet-processed + wetting agent fabrics.

Dry-processed fabrics always give higher results, followed by wet-processed fabrics and wet processed + wetting agent fabrics. Wetting agent usage has no increasing effect on color efficiency.

CONCLUSIONS

It is possible to perform mercerization by treating material with caustic during different time intervals and different processes. These different applications and changeable periods have some positive and negative effects on material.

To perform an efficient mercerization and provide desired properties, following processes are suggested.

burning— desizing— scouring— bleaching
— drying— mercerizing

Drying can be performed depending on preference. There is no clear and definite difference among mercerizing periods. Longer periods have improving effects in a few special treatments but not in all. It is preferred to mercerize fabric during shorter periods. The advantages of shorter mercerization processes can be expressed as follows:

- Prevention of fabric from mercerizing faults. Because longer mercerizing processes refers to longer application on fabric.
- Lower cost and energy consumption.
- Elimination of space requirements.

As all results are generally evaluated, results and average values of wet processes are better for all types in terms of shorter periods. The shorter application period is sufficient for both economic and desired mercerization.

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