MICROWAVE DYEING OF REGULAR AND CARRIERLESS DYEABLE POLYESTERS WITH DISPERSE DYES

by

JAW HUA CHIAO-CHENG

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Approved by:

Barbara M. Reagan
Major Professor

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INTRODUCTION

Approximately 60% of the energy required to produce a textile is consumed in the wet processing area (i.e., preparing, dyeing, and finishing textiles). As a result, considerable research has been directed towards reducing energy consumption in wet processing. Microwave dyeing and finishing technique are being explored because they can provide a more uniform and more rapid method of heating and drying textiles than conventional heating techniques with considerable energy savings. In addition, microwave heating results in a more even distribution of dyes and finishes and faster rates of dye and finish penetration.

In 1966, Ciba-Geigy obtained one of the earliest patents for using microwave heating in dyeing and printing fibrous textile material with reactive dyes [1]. Adams [1], Belton [5], Dawson [15], Delaney [17], Evans [20], and Pendergrass [26] explored the possibilities of using microwave heating techniques for dyeing and finishing textiles. Recently, Needles [25] and Berns [6] investigated the feasibility of using microwaves for a variety dyeing and finishing processes. The application potential of microwave energy is endless.

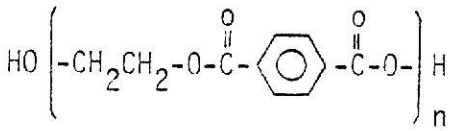
The purpose of this research project was to develop methods suitable for dyeing regular and carrierless dyeable polyester fabrics with selected disperse dyestuffs and to compare the results with conventional dyeing techniques in terms of uniformity, dye penetration or diffusion, color yield, and changes in physical properties.

REVIEW OF LITERATURE

Polyester Dyeing

Polyester first appeared on the world market in the early 1950's and gained rapid acceptance both as a filament yarn and as a staple fiber. Today, polyester fibers are sold under approximately 17 different trade names (i.e., Encron, Dacron, Fortrel, Kodel, Lanese, Strailine, and Trevira polyester) and according to type or variation(i.e., Dacron Type 54).

Regular polyester fibers are long chain, synthetic polymers composed of an ester of a dihydric alcohol such as ethylene glycol and terephthalic acid.



Polyethylene terephthalate(PET)

When first produced, PET polyester had little affinity for dyestuffs traditionally used on cellulosic and protein fibers, thus, disperse dyes, initially developed for dyeing acetate fibers, were perfected as a means for coloring polyester. In addition, special dyeing methods (i.e., carrier dyeing and thermosoling) and machinery (i.e., jet dyer) have been developed for improving the color yield, quality, and efficiency in polyester dyeing.

The PET polyester is moderately crystalline and markedly hydrophobic, therefore, it is difficult to dye and often presents dyeing problems.

Considerable advances were made in polyester dyeing when the carrier method was introduced. It was discovered that a numbers of organic compounds such as phenols, amines, and aromatic hydrocarbons, when either dissolved or suspended in the dyebath, accelerated the adsorption of dispersed dye by the fiber [32].

Many theories have been postulated to explain the mechanism of carrier action. These include: (1) increased swelling of the fiber, (2) increased water absorption, (3) transport theory, (4) increased solubility of dyes, (5) film theory, (6) liquid fiber theory, (7) molecular lubrication theory, (8) loosening of fiber structure theory and (9) increased dye site theory.

In Colour Index [13], the major methods given for dyeing polyester fiber include: N (normal temperature), Nc (normal temperature + carrier), HT (high temperature, i.e., in pressurised systems), 3Az (3 bath azoic), 2Az (2 bath azoic), T (pad/Thermofix), and Pad/steam which is used to fix prints.

The Thermosol Process, which was developed by E.I. du Pont de Numerous and Co., is a rapid and continuous method which was designed primarily for polyester/cotton blends [33]. It involves padapplying the dye liquor onto the fabric, predrying the fabric to minimize dye migration, and then exposing the goods to a suitable heat treatment (180-220^{o}C,30-60 sec) which diffuses the dye into the fibers. Thermosol fixation of disperse dyes depends on the molecular size, shape, volatility of the dyestuff. The heat treatment in the Thermosol Process opens up the polymer structures and makes the fibers more permeable to the dye molecules.

In the thermosol dyeing of polyester, the migration of dis-

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perse dye particles during predrying and thermal fixation will significantly affect the fixation, color yield, and appearance of the dyed products. Thus, the control of dye migration is very important. The factors which influence migration include fabric structure, moisture regain, thermal conductivity as a function of regain, meth- of heating, ease of evaporation and removal of the water vapor, substantivity of the solute for the fabric, and surface tension and viscosity of the solution. Technical Migration, M_T, is a common expression for the effect of both particulate and molecular migration of disperse dyes in the Thermosol Process. It has been shown that M_T is a function of fabric structure, auxiliary concentration, and concentration of dye [19].

The Thermosol Process has the following advantages over other methods of dyeing polyesters: (1) it is a continuous process, (2) no carrier is required when applying disperse dyes to polyesters, (3) utilization of dye is excellent, (4) the fabric can be processed full width thus eliminating rope marks, and (5) dyeability is not affected by heat setting [33]. Sommers [31] discussed important factors to be considered when selecting disperse dyes for thermosol dyeing. These include dye characteristics, effects of various finishes, colorfastness, pH sensitivity, and build-up properties.

Regular polyester fibers usually are dyed with disperse dyes. The dye affinity of polyester fibers is modified by incorporating other molecules into the polymer. The methods of modification generally can be divided into (1) the addition of ionic groups on the acid structures which impart affinity for cationic or anionic

dyestuffs (i.e., ionic dyeable polyester) and (2) the addition of small molecules or plasticizers into fiber structure which open up the fiber structure, enhance the dye uptake, and eliminate or decrease the use of carriers during dyeing (i.e., carrierless dyeable polyester).

Microwave Heating Principle

Microwave are high frequency radio waves which are capable of penetrating many materials and causing heat to be generated in the process. The wavelengths of microwaves are in the range of 100-1 cm. This range also can be expressed in terms of frequency (i.e., megahertz), and corresponds to 300-30,000 MHz.

Various applications of microwaves have been used in communications, radar, physical research, medicine and industrial measurements, and heating and drying of agricultural, food, paper, and textile products. In order to avoid interference with the communication and radar systems, the only frequencies that can be used for microwave processing are 915 ± 25 and 2450 ± 50 MHz. The major difference between using these frequencies is the power output of the magnetron tubes. The 915 MHz tubes produce an output of 25 KW, whereas, 2450 MHz tubes are restricted to a much lower output of 2.5 KW. Therefore, 915 MHz is used in high-power industrial heating equipment and 2450 MHz is used for domestic microwave ovens [29].

The ability of materials to absorb microwave energy is determined by the polarity and the presence of dissolved salts. Microwave radiation is absorbed by molecules having resonant frequencies similar to the frequencies of microwave energy. When an electric field is applied at microwave frequencies, the polar molecules rotate

in an attempt to rearrange their dipole moment with the changing electric field. Energy is absorbed and heat is generated by the internal friction between the rotating molecules [14, 29].

The materials which only absorb microwaves to a limited extent are termed 'dielectric' and are used as construction materials in microwave processing equipment. 'Loosy' materials, on the other hand, can be heated by microwaves. The amount of microwave absorption or heat genetration depends on the lossiness of a material and the amount of lossiness varies with the radiation frequency, temperature, and the type of material being heated [5].

Due to the penetration properties of the microwaves, the energy is transferred directly to the moisture throughout the volume of a wet body rather than through the surface as in the conventional heating methods. When wet textile materials are heated by microwaves, the average efficiency of power transfer from the main supply is more than 50% [29]. Dry textile materials usually are not heated to any great extent when irradiated by microwaves, and must be surrounded by a medium capable of generating heat when exposed to microwave energy.

In general, fibrous materials contain interconnected cavities or capillaries of irregular shapes and sizes. When the material is wet, cavities are filled in varying degrees with water or solvent. In the drying process, water in the material is vaporized and transferred to the surrounding air. Drying time depends on the rate of thermal energy transfer to the site of evaporation.

There are numerous ways to apply this thermal energy. These can be divided into two categaries: surface heating and internal

heating. In surface heating, heat is transferred from an external heat source to the surface of the material by convection, conduction, radiation or a combination of these. Microwave heating, on the other hand, is a type of internal heating in which the energy is absorbed from the electromagnetic field which provides heating throughout the whole exposed material. The drying rate with microwave energy is much more rapid than with surface heating and the danger of overheating the surface of material is lessened.

Typical drying curves for conventional heating are shown in Figures 1 and 2 [23].

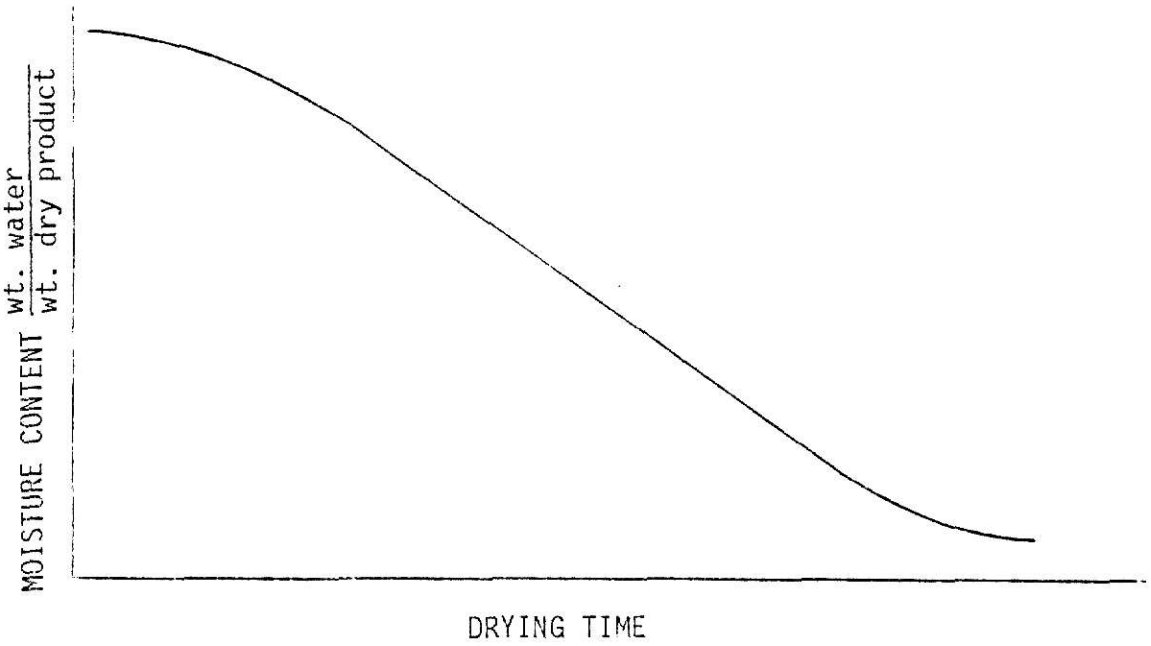

Figure 1: Typical Drying Curve Using Conventional Heating (Taken from D. W. Lyons's Ph.D. Thesis, Atlanta, Georgia, Georgia Inst. Technol., 1966).

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The portion AB of the curve in Figure 2 is the initial warm-up period which may or may not be significant in the total processes. Curve BC is the constant rate period. Point C is termed the point of critical moisture content, and curve CD represents the falling rate period. Within this period, the rate of the moisture flow to the surface of the body is no longer sufficient enough to maintain the saturation at the surface, therefore, the surface becomes less wet and the plane of vaporization moves into the body. Consequently, a temperature gradient is established between the surface of material and vapori-

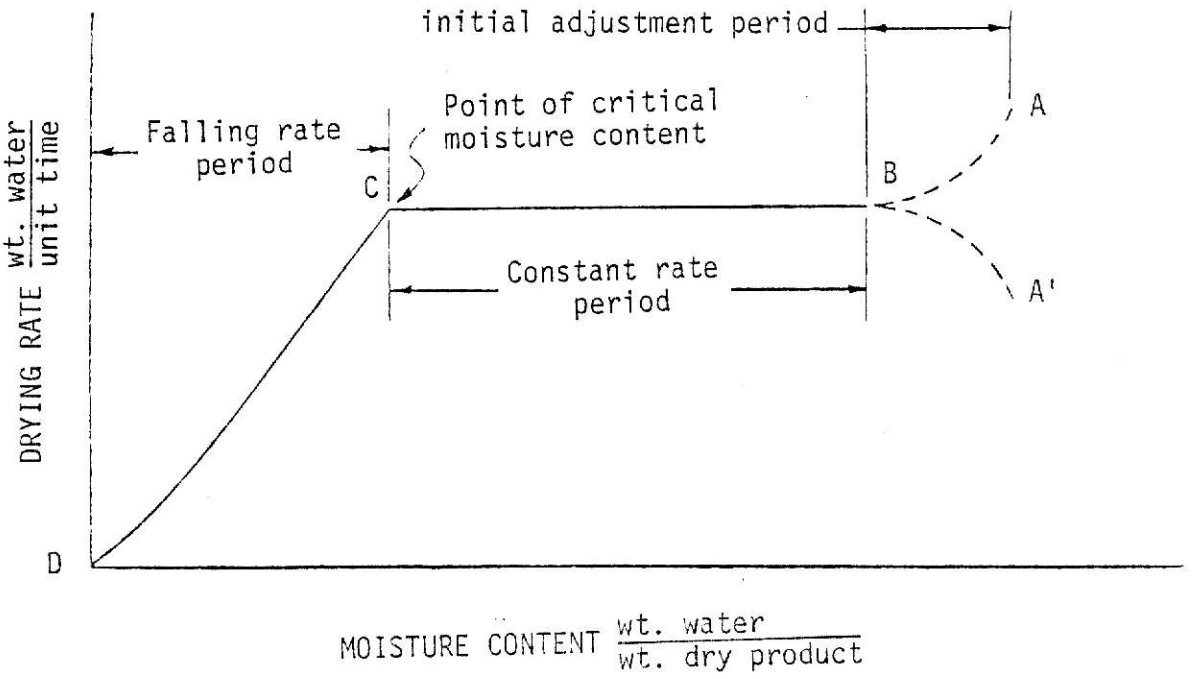

Figure 2: Typical Drying Rate Curve for Drying Using Conventional Heating (Taken from D. W. Lyons's Ph.D. Thesis, Atlanta, Georgia, Georgia Inst. Technol., 1966).

zation plane, and heat is added to the moisture by this gradient in order to achieve vaporization of the moisture within the material. Because the drying rate decreases as the dry insulating layer thickens, the drying curve for the falling rate period has a progressively decreasing slope.

During the microwave drying process, both temperature and moisture content are uniform within the material. The temperature of material will increase gradually to the boiling point of liquid and is retained at this constant condition throughout as long as liquid is present. Thus, in a wet material it is possible to apply energy rapidly to the water inside without causing damage to the surface of the material by over heating [23].

Microwave Dyeing and Finishing

The textile industry has investigated many uses for microwave energy from heating, drying, dye fixing, curing of resin-finished fabrics [18, 21, 27, 28] to disinfesting wool fabrics [30, 31].

In 1966, a patent was granted to Ciba for a dyeing process that utilized microwaves to heat batches of textile materials which had been previously pad-applied with reactive dyes. Results showed that the reaction rate between the dye and the fiber was increased as the temperature increased during microwave heating [12]. Adams [1] varied the direction of the microwave frequency with respect to the warp and filling directions of the woolen substrate which permitted selective and mixed color effects to be obtained.

Reports dealing with the use of microwaves to accelerate the dyeing processes were published by Belton [5] and Evens [20]. The rate of dye fixation on wool and nylon was much more rapid with

microwave heating than with conventional heating techniques. Dawson [15] conducted a series of dye fixation experiments by varying the type of fiber, microwave intensities, and steam power. He concluded that microwave fixation units should be designed in such a way so that the humidity remains as high as possible. In the treatment chamber, the radiated energy must be designed uniformly across the material to reduce irregularities in dye fixation. In general, those dyes which fix most rapidly in steam fixation methods also are the ones suitable for rapid microwave fixation.

A combinational heating system was designed by Metaxas [24] for dye fixation in which microwave energy was used to preheat the fibers prior to steaming. Compared to the conventional steaming system, this method was more economical, improved dye fixation, and eliminated the frosting problem.

Pendergrass [26] compared the dye migration in textiles dried at different rates with microwave and conventional heating. Dry times for microwave heated samples ranged from 9 to 36 minutes, whereas the average drying time for convective surface heating was much longer and ranged from 5 to 7 hours. A uniform dye concentration was observed throughout the microwave dried sample. As a result, dye migration was minimized during microwave drying.

Uniform deposition of dyes in wool fabrics also has been explored by Delaney [16, 17]. He introduced the microwave heating technique to decrease the batching time required for the IWS (International Wool Secretarial) Pad-Batch (cold) dyeing processes from 24 hours to one hour. Pepperman [27, 28] used microwave drying in the application of flame retardants to cotton fabric. There were

no significant improvements in the physical and chemical properties of the finished fabrics over hot-air drying. The flame retardant finished fabrics that were dried with microwaves exhibited poorer tearing strength and an increase in stiffness. However, the microwave drying process did not affect the chemical properties and durability of the finish to laundering. The speed of drying and the feasibility of using alternate energy sources were accepted as the inherent advantages of microwave drying.

Needles and Berns [25, 6] investigated the feasibility of using microwaves for a variety dyeing and finishing processes. They indicated that a polar molecule such as water, DMF, DMSO, or ethylene glycol is needed to absorb microwaves and induce heating in the textile material. When compared with the external heating and curing processes for grafting of polymers, microwave heating of the wet textiles was more efficient and less degrading to the fibers. Microwave heating can impart uniform deposition of dyes on textile, accelerated dye adsorption and penetration, increase the rate of grafting, and reduce dye and finishing migration. Furthermore, Berns [6] pointed out that microwave-induced molecular oscillations within dyebaths caused an increase in the dyeing rate, faster dye diffusion, and increased solvent-induced plasticization within the fibers.

The potential of using microwave radiation as a heating source for dye fixation directly after pad-applying the dyebath to the fabric has not been investigated widely. Research in this area has been confined to limited fabric substrates (i.e., acrylic, cotton, nylon, polyester, rayon, and wool), dyestuffs (i.e., acid, basic,

direct, disperse, and reactive), and solvent systems (i.e., DMF, DMSO, DMF-H_2O, EG, and H_2O). Few studies have investigated the feasibility of using microwaves to dye polyester fibers with disperse dyes, and only one study attemped to elucidate the effects of microwave irradiation on fiber structures [6].

Because of the energy saving potential, additional research is needed in the area of microwave dyeing. This study examined two types of polyester fibers (i.e., regular and carrierless dyeable polyester) and three solvent systems (i.e., 100% water, 10% aqueous urea and 45% aqueous urea). In total, seven dyeing methods that utilized microwave radiation were developed, evaluated, and compared to conventional thermosol dyeing in terms of color yield, dye uniformity, and fiber structural changes.

Objectives

The specific objectives of this study were: (1) to develop a suitable microwave dyeing method for 100% regular polyester and carrierless dyeable polyester, (2) to evaluate the color yield by computing K/S values from the UV/visible reflectance spectra of the dyed fabrics, (3) to evaluate dye uniformity by using CIE L*, a*, b* and total color difference ΔE, (4) to evaluate dye penetration in fibers by examining photomicrographs of fiber cross sections, and (5) to evaluate the structural changes in the polyester caused by microwave exposure. Test methods used to evaluate sturctural changes were density gradient analysis, breaking strength and elongation, and small-angle light scattering.

PROCEDURES

In order to develop a suitable microwave dyeing method for regular polyester and carrierless dyeable polyester, numerous dyeing procedures were explored with different solvent systems, water temperatures, and methods of exposure. The results obtain with microwave dyeing were compared to that obtained with the conventional thermosol method. Parameters examined included depth of shade, evenness of color, dye penetration, and changes in the physical properties of the polyester fabrics after microwave irradiation.

Fabrics

The fabrics selected for this study were a regular dyeable, Dacron 54 polyester (Testfabrics, Inc.) and carrierless dyeable, Treveria 403 polyester (Celanese Fibers Marketing Company) both of which were specifically woven for this study. These fabrics were medium weight, filling satins containing 2-ply, spun filling yarns and filament warp yarns. The yarn and fabric construction characteristics of these fabrics, as presented in Table 1, were determined according to the procedures in ANSI/ASTM D 1244, Designation of yarn construction; and ANSI/ASTM D 1910, Construction Characteristics of woven fabrics [4, 2].

Twenty-five yards each of the regular dyeable polyester and the carrierless dyeable polyester were cut into specimens weighing 25 g and randomly assigned to the various dyeing procedures explored in this study. Prior to dyeing, all specimens were scoured to remove surface dirt or soil that may have resulted in an uneven dyeing.

Table 1: Construction Characteristics of Polyester Fabrics.

Fabric	Yarn Construction	Fabric Count (endsxpicks/cm)	Fabric Width (cm)	Fabric Weight (g/m^2)
Dacron 54 (regular dyeable)	Warp: 150 den. f 26 Z 13 tpi Filling: R 14.68 cc S 12 tpi/2 Z 16 tpi	38.6x35.8	117.60	209.35
Treveria 403 (carrierless dyeable)	Warp: 75 den. f 37 t 0 Filling: R 10.00 cc S 14 tpi/2 Z 20 tpi	39.4x29.9	116.84	207.66

Scouring

All specimens of the regular dyeable and carrierless dyeable polyester fabrics were scoured prior to dyeing in baths containing 1 g/liter of Gafterge 317 (anionic surfactant) and 1 g/liter of soda ash using a 30:1 liquor-to-goods ratio. Thus, 750 ml of the scouring solution was used for each 25 g specimen.

The specimens and scouring solution at 25°C were placed in individual, high-temperature Launder-Ometer canisters. The canisters were closed and placed in the Atlas Launder-Ometer, Model LHT. After slowly raising the temperature to 60°C, the specimens scoured for 30 minutes, then removed, thoroughly rinsed in distilled water, and air dried in a standard atmosphere for testing (20 ± 1°C and 65 ± 2% RH).

Dyeing

Numerous microwave-dyeing procedures were investigated in this study. They varied in solvent type, dyeing time, and method of exposing the specimens in the microwave oven. The microwave dyeing experiments that did not produce satisfactory results are discussed in the results section of this thesis.

Selected dyeing procedures which utilized microwave energy were more thoroughly investigated and are described herein. Experimental results were compared to those obtained in conventional thermosol dyeing in terms of depth of shade, dye penetration into the interior of the fiber, and evenness of the color. The dyeing procedures discussed below include:

Code	Description
I.	Thermosol dyeing with conventional predry
II.	Thermosol dyeing with microwave predry
III.	100% H_2O pad-bath with microwave heating and heated water bath
IV.	100% H_2O pad-bath with microwave heating and boiling water bath
V.	10% urea pad-bath with microwave heating and heated water bath
VI.	10% urea pad-bath with microwave heating and boiling water bath
VII.	45% urea pad-bath with microwave heating and heated water bath
VIII.	45% urea pad-bath with microwave heating and boiling water bath

All experimental procedures utilizing microwaves were conducted in a 2450 MHz, Sharp Carousel microwave oven, Model R-6770, operating at full power (0.65 KW). Three disperse dyes were used in this study. They were selected on the bases of hue, chemical class, and recommended dyeing procedure (see Table 2). According to Colour Index and BASF, these are medium to high energy disperse dyes which are suitable for Thermosol dyeing [13, 11].

The regular dyeable and carrierless dyeable polyester fabrics were dyed under identical procedures using the same dyestuffs and conventional and microwave dyeing methods. Subsequent to dyeing, comparisons of depth of shade, degree of penetration, and evenness were made among dyeing methods and between fabric types.

Table 2: Disperse Dyestuffs.

Generic Name	Commercial Name	Chemical Class
C. I. Disperse Blue 200	Dispersol Blue D-4GL	Monoazo
C. I. Disperse Violet 33	Dispersol Rubine C-BL	Monoazo
C. I. Disperse Yellow 126	Dispersol Yellow D-7GL	Monoazo

Dyeing Method I

Thermosol Dyeing with Conventional Predry

Conventional Thermosol dyeing is a widely used, industrial method of dyeing polyester and polyester/cotton blends. The steps include pad-applying the dye liquor, predrying to reduce the moisture content, and then exposing the fabric to heat (i.e., 180-220^{o}C, 30-60 sec) which causes the fiber structure to open up, thereby enabling the vaporized dye to be absorbed into the interior of the fiber.

Dyeing. In this study, the dried and conditioned specimens of regular dyeable and carrierless dyeable polyester fabrics to be Thermosol-dyed were passed twice through the specified disperse dye pad-bath which contained the following quantities of dyestuff and auxiliries:

Disperse dye pad-bath

Disperse dye	5% dpu (regular polyester-96 g/liter, and carrierless dyeable polyester-77 g/liter)
Superclear N-100 (antimigrant)	20 g/liter
Irgasol DA (dispersing agent)	2 g/liter
Monosodium phosphate (to adjust pH to 5.5)	

The concentration of the pad-bath was adjusted according to the wet pick-up of the fabrics which was 52% for the regular dyeable polyester and 65% for the carrierless dyeable polyester. The dye solutions were pad applied in a Butterworth 3-roll laboratory padder at 40 psi and 25 $\pm$ 1^{o}C.

After being padded with the dye solution, the polyester fabric specimens were predried in a Custom Scientific Laboratory oven for 7 minutes at 130 ± 5°C. The specimens were held taut during predrying by placing them over wooden frames and inserting the warp ends into the grooves of the frames with cotton cord. To prevent the specimens from touching the wire racks in the oven, the edges of the frames were placed on top of four small beakers positioned inside the oven. After predrying, the polyester specimens were immediately removed from the oven and conditioned for 15 hours in a standard atmosphere for testing.

The specimens were remounted in wooden frames coverd with aluminum foil to prevent undesirable shrinkage during thermofixation. The procedure was similar to that previously described, except all four edges were securely held in place with the cotton cord placed over the edges and pressed into the grooves of the wooden frames.

The disperse dyes were then thermofixed on the polyester fabrics by heat-treating the specimens in a modified Custom Scientific, Model 17, laboratory oven for 60 seconds at 205 ± 5°C. The laboratory oven was modified for this study to prevent heat loss when opening the oven door to insert the specimens. Because the exposure time is so short during thermosoling, it is important that there is no heat loss when the specimens are being placed in the oven, otherwise the temperature would be too low for proper thermofixation.

The oven was modified to minimize the heat loss by placing a tightly-fitting, asbestos-covered, metal plate inside the oven

cavity. A horizontal slot (5 x 28.5 cm) was cut out of the metal plate so that the wooden frames used for thermofixation could be inserted into the oven. Fiberglass string leaders attached to the wooden frames and these were used in removing the frames from the oven. The modified Custom Scientific Latoratory oven used in this study is shown in Figure 3.

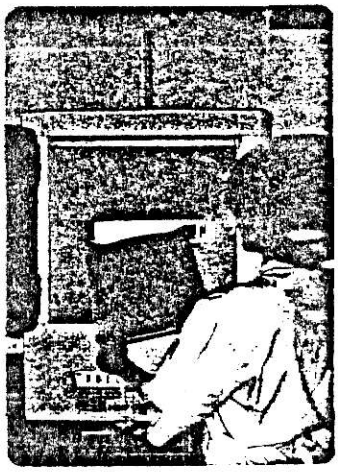

Figure 3: The Modified Custom Scientific Laboratory Oven.

Reduction Clear. A reduction clear process usually is given to a disperse-dyed polyester in order to remove the unfixed dyes and auxiliary residues on the surface of the fiber. This treatment usually will improve the crock fastness and wet fastness of the dyed fabrics.

After thermofixation, the fabric specimens were placed immediately in the following reduction clear bath at 25°C:

Reduction clear bath

Gafterge S-111 (nonionic surfactant)	1 g/liter
Sodium hydrosulfite	2 g/liter
Sodium hydroxide	6 g/liter
Liquor-to-goods ratio	30:1

The temperature of the bath was raised to 75°C and the specimens were treated for 30 minutes. After the reduction clear treatment, the specimens were rinsed thoroughly in distilled water and placed in a standard atmosphere for testing.

Dyeing Method II

Thermosol Dyeing with Microwave Predry

Since research has shown that microwave drying is a more efficient method of removing moisture from textiles than are conventional drying methods, the feasibility and advantages of using microwave energy for predrying fabrics during Thermosol dyeing was investigated. It was hoped that the 7-minute predrying step could be substantially reduced while minimizing dye migration which frequently is a problem when drying textiles during dyeing.

Specimens of the regular dyeable and carrierless dyeable polyester fabrics were subjected to the same scouring, dyeing, and reduction clear procedures, except for being exposed to microwaves for 2 mimutes to predry the textiles instead of the conventional 7 minutes predry described above. During microwave predrying, the specimens were placed on a glass tray without any stress in the center of the glass carousel plate inside the microwave oven. The glass tray and carousel plate were cooled to room temperature before each exposure.

Thermosoling with Microwaves

The six additional microwave dyeing methods evaluated in this study differed in solvent type (i.e., 100% water, 10% urea, and 45% urea) and the temperature of the water bath below the specimens during microwave irradiation (i.e., heated to 75°C or 100°C).

As previously described, these methods are identified as dyeing methods III-VIII. The composition of the disperse dye pad-bath and the reduction clear bath used in these methods were identical to those which were used in conventional Thermosol dyeing, except for the diluent or dissolving medium which contained 10% urea in Dyeing Methods V and VI and 45% urea in Dyeing Methods VII and VIII.

After the regular dyeable and carrierless dyeable fabric specimens were padded with the disperse dyebath formulations, they were attached to glass frames, constructed from 1/4 in glass rods, by sewing together the edges as shown in Figure 4.

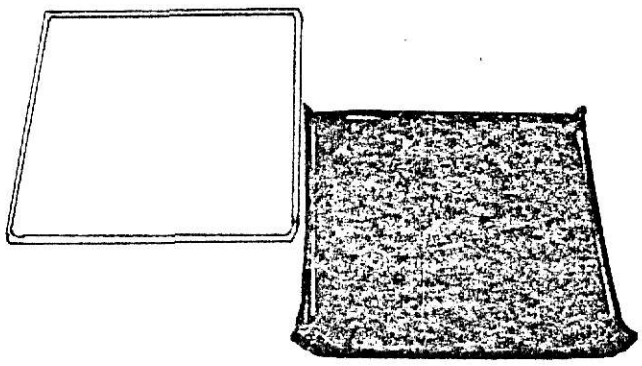

Figure 4: The Fabric Specimen with Glass Frame.

The glass frames measured 23.8 x 23.8 cm and were specifically made for this study by Mr. Mitsugi Ohno, Department of Physics, Kansas State University.

In Dyeing methods III, V, and VII (100% H_2O, 10% urea, and 45% urea) a Pyrex glass dish (25.7 x 21.9 x 5.7 cm) containing 200 ml of distilled water was placed on the carousel plate inside the

oven, covered with a similar Pyrex glass dish, then exposed to microwave radiation for 3 minutes to preheat the water which served as a steam bath. After the water bath had been preheated, the glass frames containing the polyester fabric specimens were placed singly between the Pyrex glass dishes and exposed to microwaves for 10 minutes.

In dyeing methods IV, VI, and VIII (100% water, 10% urea, and 45% urea) the regular dyeable and carrierless dyeable polyester specimens were thermosoled-dyed with microwaves by placing them over a water bath which was heated to a boil for 7 minutes in a microwave oven.

Methods of Analysis

Dye depositional characteristics of the regular polyester and carrierless dyeable polyester after microwave heating and conventional thermofixation were evaluated and compared by colorimetric measurements and microscopic examination of fiber cross sections. Colorimetric measurements used in this study included (1) K/S values for assessing depth of shade or concentration, and (2) total color difference, ΔE, for assessing dye uniformity. Small-angle light scattering, density gradient analysis, and breaking strength and elongation tests were used to evaluate the fiber structural changes induced by the dyeing methods.

K/S Values

Based on the Kubelka-Munk equation, the K/S values of a colored textiles at a given wavelength are proportional to the concentration of dye thereon. Hence, a straight line relationship often exists between K/S values and concentration.

In this study K/S values were used to determine the depth of shade or concentration of the dyestuff associated with the eight dyeing methods on both types of polyester. Three reflectance spectra for each of the dyeing methods were recorded on a Cary 219 UV/visible spectraphotometer. Specimens were folded to obtain three thicknesses placed on the reflectance sphere with a 500 g weight, and scanned from 380 to 720 nm.

The reflectance (R) of the dyed specimens was measured at the wavelength of maximum absorption for each dye:

Dye	λmax
C. I. Disperse Blue 200 (Dispersol Blue D-4G)	640nm
C. I. Disperse Violet 33 (Dispersol Rubine C-B)	540nm
C. I. Disperse Yellow 126 (Dispersol Yellow D-7G)	448nm

From the average of three reflectance values for each specimen (see Figure 5), the K/S values were calculated from the Kubelka-Munk equation as follows:

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

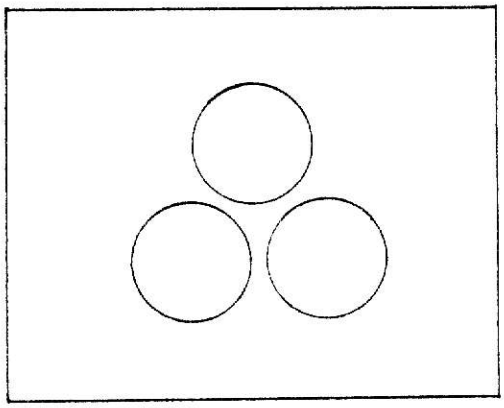

Figure 5: Areas on Specimen for K/S Value Determination.

Dye Uniformity

End-to-end and side-to-side variations in the uniformity of the dye on regular dyeable and carrierless dyeable polyester specimens dyed by the conventional Thermosol and microwave methods were evaluated by comparing the variability in CIE L*a*b* values. Readings were taken at nine specific locations on each specimen as follows: one reading from the center which served as the control and eight additional readings from the top, bottom, and side areas as shown in Figure 6.

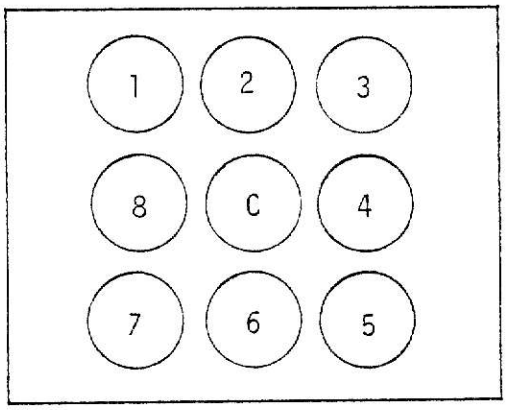

Figure 6: Areas on Specimen for Dye Uniformity Evaluation.

CIE L*a*b* values were taken on a calibrated Hunterlab D25M-4 color difference meter and a Tektronix programmable calculator by using a 5 cm sample port and block "0" on Hunterlab software Tape 3.

Dye Penetration

During the initial stages of a dyeing operation, the dye frequently is deposited on the exterior of the fiber. Prolonged dyeing times, temperature increases or suitable fixation methods resulted in the absorption of the dye into the interior of the fiber. The

presence of ring-dyed fibers (i.e., dye remains on outer surface) frequently is used to assess the lack of dye diffusion or incompleted dye penetration.

In this study, fiber cross sections of the dyed polyester specimens were prepared for determining the extent of dye diffusion into the fiber interior for each of the eight dyeing methods. Fiber cross sections, prepared on a Schwartz microtome using collodion as the embedding medium, were examined with a American Optical compound microscope, Model 150.

Photomicrographs were taken of fibers representing the degree of dye penetration obtained with the eight dyeing methods and two fabric types.

Evaluation of Effects of Dyeing Methods on Fiber Properties

The morphological characteristics of the crystalline and amorphous regions inside the fiber can be altered by various textile processing treatments (i.e., stress, solvents, heat, etc.,). Because the mechanism of microwave heating differs from conventional heating methods, the types of structural changes which are induced by these methods may vary. Limited research has been done to characterize microwave-induced morphological changes within polyester fiber.

In this study, density gradient analysis, small-angle light scattering, and breaking strength and elongation tests were used to assess the structural changes that occurred in the regular dyeable and carrierless dyeable polyester fibers during conventional Thermosol dyeing and with the seven dyeing methods which used micro-

wave heating. The specimens evaluated by these test methods were subjected to the same dyeing procedures as previously described, except that the dyestuff was eliminated from the pad-bath. The untreated control specimens were scoured prior to evaluation as were the dyed specimens.

Density Gradient Analysis

The density gradient method is a sensitive technique for distinguishing among commerical polyester fibers that differ in chemical composition, origin, manufacturing treatments, and consumer use [6]. It also has been used in determining the percentage of fiber crystallinity, in quantitatively assessing fiber additives, and in monitoring polymer degradation.

In this study, a density gradient column was prepared according to the procedures specified by Bresee [7]. Ten fractions of carbon tetrachloride/n-heptane varying in density were prepared and added dropwise successively to a 100 ml graduated cylinder positioned in a constant temperature bath at 35±0.8^{o}C by using a stepwise addition method. The resulting density gradient column had a continuous change from 1.3320 to 1.4040g/cm^{3}.

Four standard floats with known densities were deaerated in n-heptane with a light vacuum for 2 minutes in order to remove air bubbles in the voids on the surface of the floats. After deareation, the floats were placed in the density gradient column and allowed to reach an equilibrium over 24 hours. The location of the calibration floats were then recorded and used to construct a calibration curve by calculating the least squares line of best fit.

Fiber specimens representative of each fiber type and dyeing method were cut into 5-8 mm lengths, marked, and then dried

in a desiccator for 24 hours; after which they were deareated by the same procedure described above. The fiber specimens were carefully placed, one-by-one, in the density gradient column and allowed to reach equilibrium for 24 hours. The density of each fiber specimen was then determined by noting the density on the calibration curve corresponding to the fiber's location in the density gradient column.

Breaking Strength and Elongation

Breaking strength and elongation tests were performed on single yarns removed from the regular dyeable and carrierless dyeable polyester specimens after dyeing in order to evaluate the effects of the various dyeing methods on structural properties. Changes in polymer chain length, interfiber bonding or degree of crystallization are often detected by changes in tensil strength and elongation. The procedures followed for determining tensile strength and elongation were in accordance with the ANSI/ASTM D 2256-76, Breaking Load (strength) and Elongation of Yarn by the Single-Strand Method [3].

Five filling yarns from the control and each of the dyed specemins were randomly selected and conditioned for 12 hours in a standard atmosphere for testing. The tests were conducted on the Scott Tester Model CRE with a 3-inch distance between the yarn clamps and 20±3 second time to break.

Small-Angle Light Scattering (SALS)

The SALS technique is a very sensitive tool that can be used to discriminate both among and within generic fiber types, to identify the same fibers which have been subjected to different

manufacturing treatments and consumer uses, and to characterize fiber cross-sectional shapes, surface topograph, opacity, and various forms of bicomponent fiber [8 - 10]. Bresee [9] at Kansas State University has pioneered the use of the SALS technique in forensic fiber analysis. His apparatus, which was used in this study, is shown in Figure 7.

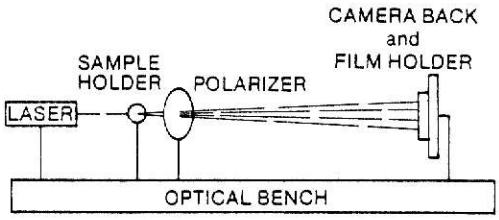

Figure 7: The SALS Apparatus

The SALS apparatus contains a continuous-wave, helium-neon laser (Huges 5 mW) which emits high intensity, monochromatic light of 632.8 nm. The photographic detection system in the SALS apparatus consists of a 10 x 13 cm Polaroid film holder and camera back.

The specimens to be evaluated were taped at both ends and hung singly on the specimen holder with a small metal chip weighing 0.1 g. Another metal chip was attached to the fiber's lower end which aided in the removal of fiber crimp and twist. The analyzer was adjusted to achieve minimum light intensity with no fiber scattering in order to get an Hv pattern of the fiber. The Hv pattern was recorded on Type 55 P/N Polaroid film with 3-second shutter speed and a 20 to 25 second development period.

Statistical Analysis

The data for K/S values, density gradient analysis, and breaking strength and elongation tests were analyzed for significance using an

analysisi of variance procedure and the Duncan's Multiple Range Test for variance. The statistical testing was performed at the Kansas State University Computing Center using statistical package SAS 79.3A.

Hypotheses of Study

H_o: Type of polyester will have no significant effect on dye fixation, density or breaking strength and elongation.

H_o: Dyeing method will have no significant effect on dye fixation, density or breaking strength and elongation.

H_o: The temperature of the water bath will have no significant effect on dye fixation, density or breaking strength and elongation.

H_o: The concentration of urea will have no significant effect on dye fixation, density or breaking strength and elongation.

RESULTS AND DISCUSSION

Numerous preliminary tests with different temperatures, exposure times, solvent systems, and methods of microwave irradiation were explored to determine the dyeing conditions which had the greatest potential or warranted further study. In total, seven dyeing methods which utilized microwave heating were throughly investigated in terms of depth of shade, degree of dye penetration, evenness, and subsequent effects on the morphological structure and physical properties of regular dyeable and carrierless dyeable polyesters.

Because specific thermosoling conditions vary in the literature, it first was necessary to evaluate and select the most appropriate thermosoling procedures which would be compatible with existing laboratory equipment and produce the best color yield. To accomplish this goal, selected predrying temperatures (i.e., 105±5, 115±5, 125±5, and 135±5^{o}C) and times (i.e., 2, 5 and 7 minutes) were evaluated with thermofixation conditions of 60 seconds at 205±5^{o}C. The best result was obtained with a conventional predrying of 7 minutes at 130±5^{o}C.

The feasibility of substituting microwave heating for conventional heating to predry the dye-padded fabric prior to Thermosoling was investigated since drying times can be substantially reduced with microwave irradiation. Microwave drying times of 1, 2, 3 and 5 minutes were evaluated in this study. A 2-minute microwave heating time was sufficient to reduce the moisture content in the dye-padded regular dyeable and carrierless dyeable polyester fabrics. Thus, microwave

predrying with conventional Thermosoling was one of the dyeing procedures that was more throughly investigated in subsequent tests.

To develop a suitable microwave-fixation method for dyeing polyester, the researcher investigated various solvent systems as given in Table 3 and microwave fixation times (i.e., 2, 2.5, 5 and 10 minutes).

The 50% urea dyebath with 10 minutes of microwave exposure produced results which were comparable to those obtained with dyeing method II (i. e., conventional thermofixation with microwave predrying). However, 50% urea presented problems because it would recrystallize at room temperature; thus, the concentration was changed to 45%.

In order to maintain the moisture content of fabric during microwave thermofixation, the specimens were enclosed in polyethylene bags prior to exposure. This method did not work well because the plastic bags melted during microwave irradiation. Thus, alternative methods for maintaining the moisture content during microwave irradiation were explored. The most satisfactory results were obtained by placing a glass Pyrex dish containing heated or boiling water underneath the specimens during microwave exposure.

After comparing the results from successive microwave fixation trials, it was noticed that the edges of fabric specimens that came in contact with the wooden frame had a much deeper color than did the central area. This indicated that the wooden frames or moisture therein was preferentially absorbing the microwave energy. Therefore, glass frames were substituted for the wooden frames.

The preliminary tests described above were conducted with both the regular dyeable and carrierless dyeable polyesters using the three disperse dyestuffs as given in Table 2.

Table 3: Solvent Systems Evaluated for Microwave Dyeing.

Chemical Name	Concentration (%)
N,N-dimethyl foramide	10%, 20%
Dioxane	10%
Ethylene glycol	10%, 20%, 100%
Glycerol	10%, 50%
Water	100%
Urea	10%, 20%, 50%

K/S Values

The K/S values were calculated using the Kubelka-Munk equation. The percent reflectance of the dyed fabric specimens at the wavelength of maximum absorbance for each dye was recorded. According to the principles of reflectance spectroscopy, K/S values are proportional to the dye concentration in the fabric.

Three readings were taken from each specimen and statistically analyzed. The independent variables in the analysis were type of polyester and dyeing method. A second statistical analysis was performed on only the six dyeing methods which utilized microwave radiation for thermofixation. In this analysis. the independent varibles were type of polyester, temperature of the water bath, and urea concentration. A 0.05 level of confidence was used in all statistical analysis.

Analysis of the Eight Dyeing Methods

The mean K/S values for the regular dyeable and carrierless dyeable polyester specimens associated with the eight dyeing methods and three dyestuffs are presented in Figures 8-10. The data used in constructing the bar graphs are given in Appendix A.

In general, the differences in dye concentration on the regular dyeable and carrierless dyeable polyesters associated with eight dyeing methods were similar for the three disperse dyes investigated in this study. However, the differences between the conventionally thermosoled specimens and those dyed using microwave energy for fixation were slightly less for polyester specimen dyed with C. I. Disperse Yellow 126 (Dispersol Yellow D-7G). Similarities in dyeing behavior among the three dye types were expected since they are all classified as monoazo dyes and are recommended for Thermosol dyeing in Colour Index [13]. Dyes suitable for Thermosol dyeing usually are larger molecules which require higher

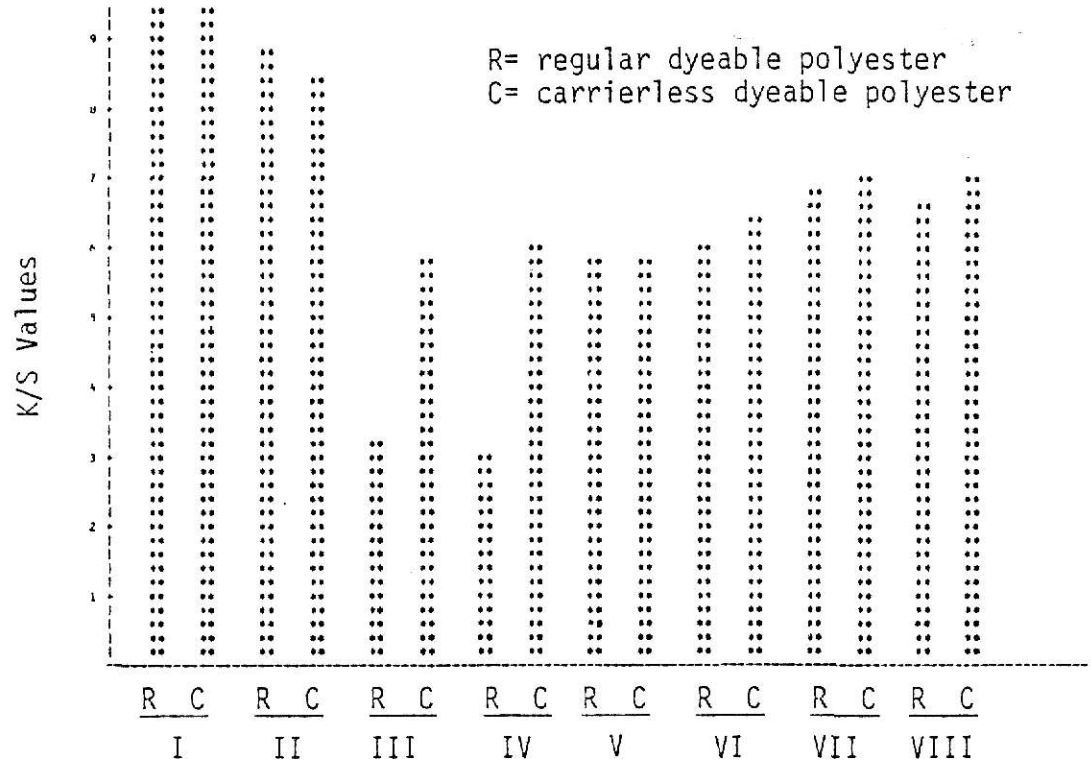

Figure 8: K/S Values for Polyester Dyed with C. I. Disperse Blue 200 (Dispersol Blue D-4G).

Dyeing Methods: I (Thermosol), II (microwave predry/ conventional thermofixation), III (100% water, heated water, microwave-fixation), IV (100% water, boiling water, microwave-fixation), V (10% urea, heated water, microwave-fixation), VI (10% urea, boiling water, microwave-fixation), VII (45% urea, heated water, microwave-fixation), VIII (45% urea, boiling water, microwave-fixation).

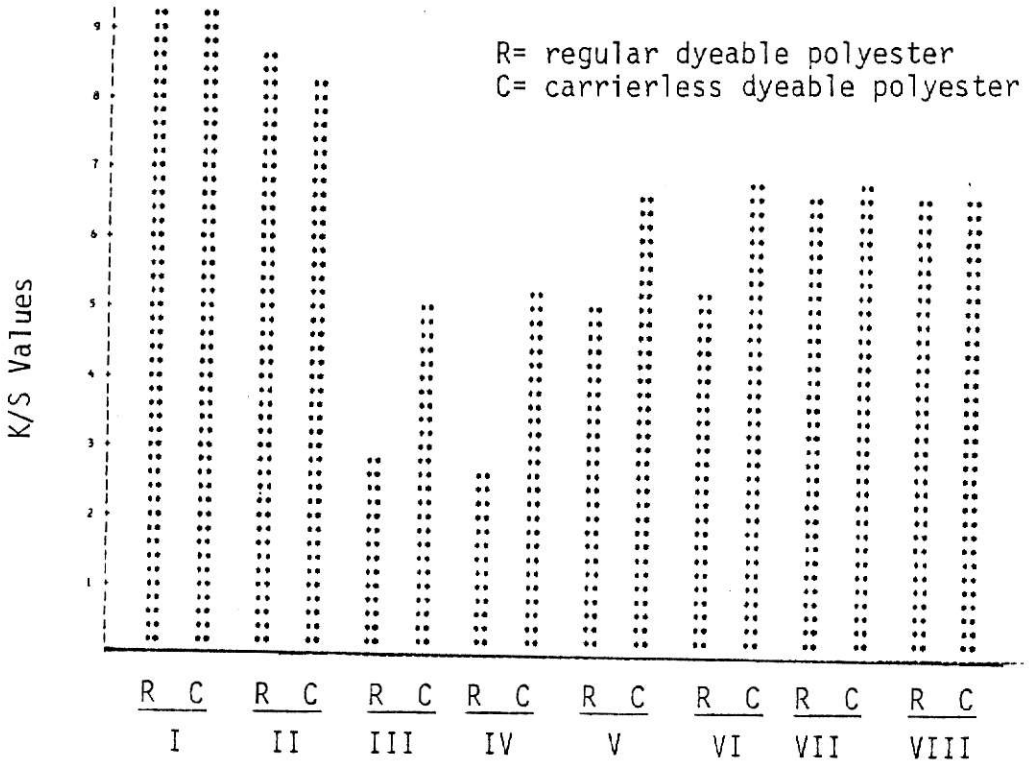

Figure 9: K/S Values for Polyester Dyed with C. I. Disperse Violet 33 (Dispersol Rubine C-B).

Dyeing Methods: I (Thermosol), II (microwave predry/ conventional thermofixation), III (100% water, heated water, microwave-fixation), IV (100% water, boiling water, microwave-fixation), V (10% urea, heated water, microwave-fixation), VI (10% urea, boiling water, microwave-fixation), VII (45% urea, heated water, microwave-fixation), VIII (45% urea, boiling water, microwave-fixation).

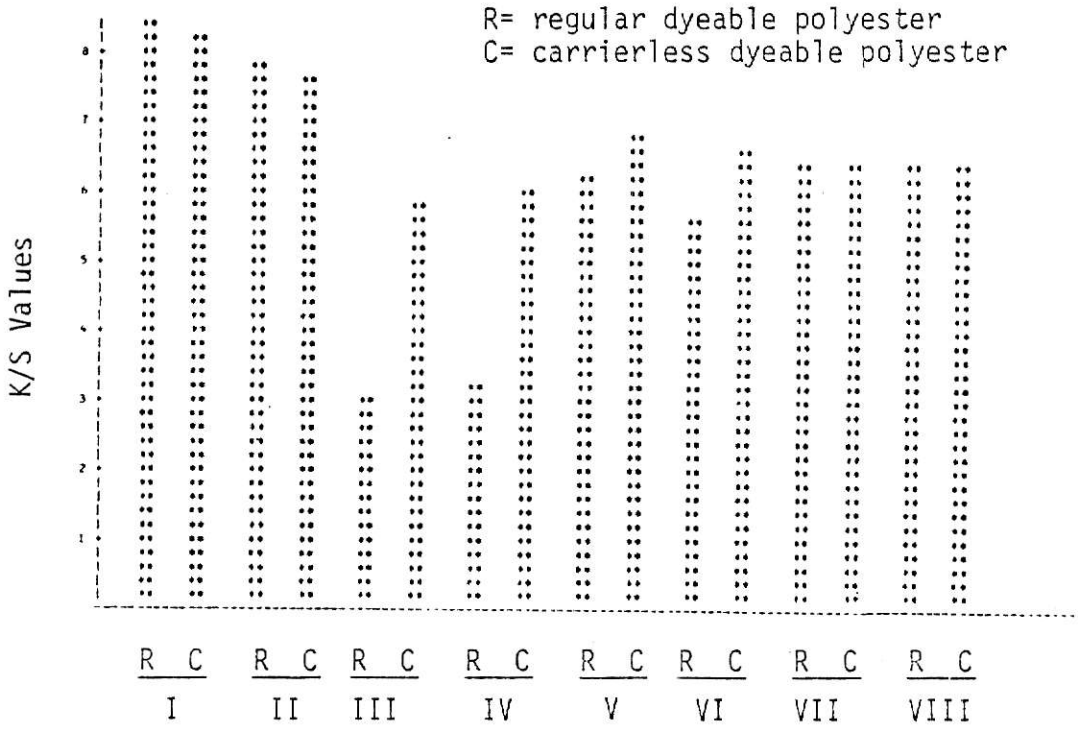

Figure 10: K/S Values for Polyester Dyed with C. I. Disperse Yellow 126 (Dispersol Yellow D-7G).

Dyeing Methods: I (Thermosol), II (microwave predry/ conventional thermofixation), III (100% water, heated water, microwave-fixation), IV (100% water, boiling water, microwave-fixation), V (10% urea, heated water, microwave-fixation), VI (10% urea, boiling water, microwave-fixation), VII (45% urea, heated water, microwave-fixation), VIII (45% urea, boiling water, microwave-fixation).

temperature for dyeing than are required for other dyeing methods.

The analysis of variance test results for the three dyestuffs and eight dyeing methods are presented in Tables 4 through 6. The independent variables in these analyses were type of polyester, method of dyeing, and fiber type x dyeing method interaction. All variables had a significant influence on the K/S values of the dyed specimens; however, they were confounded by an interaction between the main effects.

Generally, the carrierless dyeable polyester exhibited significantly higher K/S values than did the regular dyeable polyester. These results were not unexpected since the carrierless dyeable polyester has a more open structure, thus increasing dye up-take. The differences between the regular dyeable polyester and the carrierless dyeable polyesters were more apparent with dyeing methods III, IV, V, and VI which used microwave heating and either 100% water or 10% urea dyebath. Since lower temperatures were attained during fixation with these methods, it appears that the carrierless dyeable polyester is able to open-up more readily at lower temperature. At higher temperatures, such as those attained in methods I (conventional thermosoling), II (micorwave predry and conventional thermofixation), VII (45% urea pad-bath with microwave fixation over heated water), and VIII (45% urea pad-bath with microwave fixation over boiling water), few difference were observed in the depth of shade on the regular dyeable and carrierless dyeable polyester fabrics.

Presented in Table 7 are the results of the Duncan's Multiple Range Test computed on mean K/S values for the regular dyeable and carrierless dyeable polyesters. The rank order and nonsignificant grouping of the means for the eight dyeing methods are represented

Table 4: Analysis of Variance for the Eight Dyeing Methods with C. I. Disperse Blue 200 (Dispersol Blue D-4G).

Source of Variations	Degrees of Freedom	Sum. of Squares	F	PR>F
Type of Polyester (P)	1	7.58	131.51	0.0001*
Dyeing Methods (D)	7	127.56	315.99	0.0001*
P X D	7	16.98	42.07	0.0001*

* Significant variables and interaction at the 0.05 level of confidence.

Table 5: Analysis of Variance for the Eight Dyeing Methods with C. I. Disperse Violet 33 (Dispersol Rubine C-B).

Source of Variation	Degrees of Freedom	Sum. of Squares	F	PR>F
Type of Polyester (P)	1	11.36	217.29	0.0001*
Dyeing Methods (D)	7	152.53	416.83	0.0001*
P X D	7	14.99	40.96	0.0001*

* Signicant variables and interaction at the 0.05 level of confidence.

Table 6: Analysis of Variance for the Eight Dyeing Methods with C. I. Disperse Yellow 126 (Dispersol Yellow D-7G).

Source of Variations	Degrees of Freedom	Sum. of Squares	F	PR>F
Type of Polyester (P)	1	8.76	172.49	0.0001*
Dyeing Methods (D)	7	74.64	210.08	0.0001*
P X D	7	16.36	46.04	0.0001*

* Significant variables and interaction at the 0.05 level of confidence.

Table 7: Duncan's Multiple Range Test on K/S Values for the Eight Dyeing Methods.

C. I. Disperse Blue 200 (Dispersol Blue D-4G)			C. I. Disperse Violet 33 (Dispersol Rubine C-B)			C. I. Disperse Yellow 126 (Dispersol Yellow D-7G)			
Method	Mean	Grouping*	Method	Mean	Grouping*	Method	Mean	Grouping*	
I	9.41	A	I	9.23	A	I	8.24	A	
II	8.56	B	II	8.35	B	II	7.65	B	
VIII	6.87	C	VII	6.69	C	V	6.49	C	
VII	6.84	C	VIII	6.65	C	VII	6.43	C	
VI	6.24	D	VI	5.94	D	VIII	6.35	C	D
V	5.84	E	V	5.85	D	VI	6.10		D
IV	4.53	F	IV	3.86	E	IV	4.57	E	
III	4.49	F	III	3.84	E	III	4.36	E	

* Means with the same letter are not significantly different.

in this table.

According to the Duncan's Multiple Range Tests, the greatest depth of shade was obtained with dyeing methods I and II which used convective heating for thermofixation. The K/S values for the specimens dyed by method I (Thermosol) were only slightly higher than those obtained by method II which utilized microwave predrying. Thus, microwave predrying can shorten predrying times and increase production speeds with inducing comparable dyeing results. It also was observed that after conventional thermosoling, the portion of the specimens which was against the wooden frames were slightly stained, however, in the specimens that were predried with microwaves, this area was unstained. Since thermosoling conditions were identical, this difference was due to predrying conditions. In microwave predrying, the temperature did not exceed the glass transition temperature of the polyester fiber, therefore, no dye penetration occurred. Dye penetration did occur in the specimens that were dried by conventional heating because the temperature of the oven was above the fiber's glass transition temperature. Thus, microwave predrying eliminated premature dye penetration.

In the microwave-fixation procedures, methods VII and VIII produced significantly greater depths of shade than the other methods investigated, but the K/S values for these specimens were significantly lower than those obtained with conventional thermofixation. The lowest K/S values were recorded for the polyester fabrics that were pad-applied with dyebaths containing 100% water and subjected to microwave-fixation. In general, higher dye concentrations could be achieved with the microwave-fixation methods as the concentration of the urea in the pad-bath increased. Since

urea has a higher dipole moment than water, the increased concentration of urea in the dyebath caused more absorption of microwave radiation. Thus, there appeared to be a correlation between the concentration of urea in the pad-bath and the temperature of microwave-fixation. The results of analysis of variance and Duncan's Multiple Range Test for the three dyestuffs and various urea concentrations are presented in Tables 8 through 11.

Based on the statistical analyses, the temperature of the water bath (i.e., heated or boiling) below the specimens duing microwave-fixation did not have a significant effect on depth of shades. The statistical analysis did not agree, however, with visual assessment of the dyed specimens. It was visually apparent that deeper shades were obtained with the boiling water baths for C. I. Disperse Violet 33 and C. I. Disperse Blue 200. The rank order of the means for dyeing methods III and IV and V and VI depict the visual differences observed, however, statistically they were not considered different.

Dye Uniformity

The dye uniformity of the microwave and conventionally dyed samples were evaluated by comparing the variability in CIE L*a*b* values of the specimens. The mean ΔE values for the different types of polyester, dyeing methods, and dyestuffs are given in Table 12.

A comparison of the ΔE values between polyester types and among dyeing methods and dyestuffs revealed that there were no apparent trends in dye uniformity. Uneven dyeings were obtained with many of the microwave-fixation methods, but they did not appear

Table 8: Analysis of Variance for the Various Urea Concentrations with C. I. Disperse Blue 200 (Dispersol Blue D-4G).

Source of Variations	Degrees of Freedom	Sum. of Squares	F	PR>F
Type of Polyester (P)	1	11.33	157.60	0.0001*
Urea Concentration (U)	2	34.04	236.67	0.0001*
P X U	2	12.92	89.83	0.0001*

* Significant variables and interaction at the 0.05 level of confidence.

Table 9: Analysis of Variance for the Various Urea Concentrations with C. I. Disperse Violet 33 (Dispersol Rubine C-B).

Source of Variations	Degrees of Freedom	Sum. of Squares	F	PR>F
Type of Polyester (P)	1	17.06	339.85	0.0001*
Urea Concentration (U)	2	50.84	506.53	0.0001*
P X U	2	8.99	89.59	0.0001*

* Significant variables and interaction at the 0.05 level of confidence.

Table 10: Analysis of Variance for the various Urea Concentrations with C. I. Disperse Yellow 126 (Dispersol Yellow D-7G).

Source of Variations	Degrees of Freedom	Sum. of Squares	F	PR>F
Type of Polyester (P)	1	13.16	183.73	0.0001*
Urea Concentration (U)	2	28.27	197.30	0.0001*
P X U	2	11.61	81.05	0.0001*

* Significant variables and interaction at the 0.05 level of confidence.

Table 11: Duncan's Multiple Range Test on K/S Values for the Various Urea Concentrations.

C. I. Disperse Blue 200 (Dispersol Blue D-4G)			C. I. Disperse Violet 33 (Dispersol Rubine C-B)			C. I. Disperse Yellow 126 (Dispersol Yellow D-7G)		
Urea Conc.	Mean	Grouping*	Urea Conc.	Mean	Grouping*	Urea Conc.	Mean	Grouping*
45%	6.86	A	45%	6.67	A	45%	6.39	A
10%	6.04	B	10%	5.90	B	10%	6.29	A
0%	4.51	C	0%	3.85	C	0%	4.46	B

* Means with the same letter are not significantly different.

Table 12: Mean ΔE Values of the Dyed Specimens.

Dyeing Methods	C. I. Disperse Blue 200 (Dispersol Blue D-4G)		C. I. Disperse Violet 33 (Dispersol Rubine C-B)		C. I. Disperse Yellow 126 (Dispersol Yellow D-7G)	
	Regular PET	Carrierless PET	Regular PET	Carrierless PET	Regular PET	Carrierless PET
I	0.84	0.72	0.76	0.53	0.59	0.51
II	1.16	0.77	2.00	1.67	0.56	0.73
III	0.83	1.03	0.86	0.98	0.99	1.19
IV	0.60	0.54	1.38	1.16	0.84	1.53
V	1.06	2.60	1.97	2.39	0.97	1.51
VI	0.62	1.31	1.00	1.55	0.83	1.73
VII	0.48	0.51	0.78	0,47	0.62	0.48
VIII	0.61	0.43	1.24	0.69	1.08	0.60

to be associated with a specific method. Less variation in dye uniformity was observed in the specimens that were Thermosol dyed with conventional predrying. Based on previous research [5, 20, 25, 26], less dye migration and higher uniformity was expected to be achieved with microwave heating because the mechanism of microwave heating differs from conventional heating in that heating and water vaporization takes place internally. These inconsistent results may be attributed to the design of the microwave oven used in this study. After microwave exposure, the following patterns were observed in many of the polyester specimens:  and  . Other factors which may have caused the uneven dyeing could be nonuniformity in the application of the dye when it was pad-applied to the fabric and uneven steaming from water bath during microwave exposure. The data obtained from this test method are given in Appendix A.

Dye Penetration

The degrees of dye penetration was evaluated by examining fiber cross sections of the microwave and conventionally dyed specimens. Because the fibers dyed with C. I. Disperse Yellow 126 were too bright to distinguish under the microscope, fiber cross sections were made only for the specimens dyed with C. I. Disperse Blue 200 and C. I. Disperse Violet 33.

Results showed that the dye penetration characteristics were quite similar for these dyestuffs. In general, the carrierless dyeable polyester had better dye penetration than did the regular dyeable polyester. These findings were not unexpected because carrierless dyeable polyester has a looser molecular structure which allows for greater dye penetration. The most uniform dye penet-

ration resulted from dyeing method II, VII, and VIII which used microwave radiation for predrying or fixation. The dye penetration from dyeing method I (conventional thermosoling) was much deeper than that observed from dyeing methods III, IV, V, and VI, but somewhat less than which was obtained with dyeing methods II, VII, and VIII. Some of the fibers taken from the specimens dyed by methods III, IV, V, and VI were ring-dyed or partially ring-dyed. The variations in dye penetration were less between the center and edges of fiber specimens from dyeing methods V and VI than from III and IV. Therefore, it appeared that when the temperature was high enough, as in methods VII and VIII, microwave irradiation increased the ability of the fiber to open up so that the dye molecules could penetrate into the interior more readily. Also interesting was the observation that dye penetration was increased when microwave heating was used in predrying the polyester specimens before conventional thermofixation. Dyeing methods III, IV, V, and VI resulted in partial ring-dyed fibers because the temperature was not high enough during microwave-fixation. Consequently, an increase urea concentration of dyebath enhanced the absorption of microwave radiation and achieve better dye penetration.

Density Gradient Analysis

Density values are commonly used for monitoring structural changes within polyester fibers. For example, changes in the degree of crystallization or the structural arrangement in the crystalline and non-crystalline regions in the fiber have been determined by density [22]. In addition, fiber properties often are influenced by structural changes within the fiber.

In this study, density gradient analysis was used to monitor structural changes in the fiber for the eight dyeing methods. Density values were obtained from single fibers taken from the central portion of the undyed (control) specimens of the regular dyeable or carrierless dyeable polyester and those polyester specimens that were subjected to the eight dyeing methods.

Density data was statistically analyzed by the analysis of variance procedure and the Duncan's Multiple Range Test to determine which dyeing methods had a significant effect on fiber density. Because the computer could not accept any undefinite values, the density value for the regular polyester specimen from dyeing method II was changed from >1.3868 to 1.4000 g/cm^3 in order to facilitate statistical analysis. This adjustment was necessary because the highest value in the density gradient column was 1.4040 g/cm^3.

The density values for the regular deyable and carrierless-dyeable polyesters associated with the untreated control (C) and the eight dyeing methods (I through VIII) are given in Figure 11. The results of the analysis of variance test are given in Table 13.

There was no significant difference in the mean density values for regular dyeable and carrierless dyeable polyester fabrics. The density values of the untreated controls were quite similar and in general, both types of polyester exhibited similar increase or decrease in density, depending on the dyeing conditions.

Based on the analysis of variance test, dyeing method had a significant effect on the density of the polyester fibers, thus

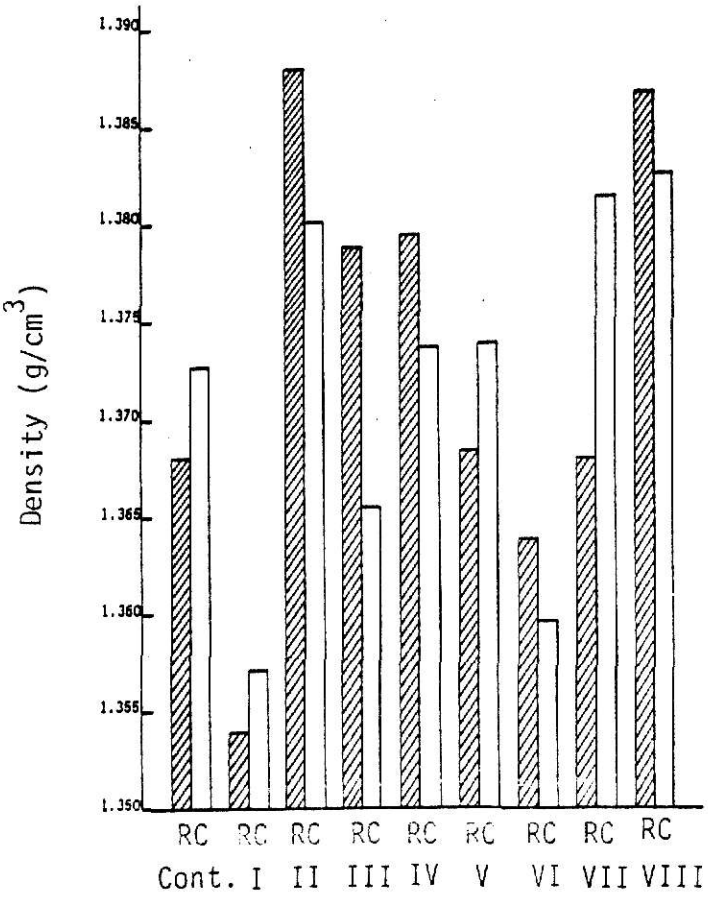

Figure 11: Density Values for Regular Dyeable (R) and Carrierless Dyeable (C) Polyesters.

Table 13: Analysis of Variance for Density Values.

Source of Variations	Degrees of Freedom	Sum. of Squares	F	PR>F
Type of Polyester (P)	1	0.000023	0.45	0.5226
Dyeing Methods (D)	8	0.001773	4.24	0.0283*

* Significant at the 0.05 level.

indicating the structural changes did occur in the arrangement of the amorphous regions or the degree of crystallization. The Duncan's Multiple Range Test was performed on the mean density values for the various dyeing methods to determine which dyeing conditions produced major structural changes within the polyester. The results from the Duncan's Multiple Range Test for dyeing methods are given in Table 14. According to the groupings from this test, the only density value which was significantly different from the untreated polyester was that obtained from dyeing method II (microwave predrying with conventional thermofixation), which increased markedly.

For the regular dyeable polyester, decreases in density occurred with dyeing procedures I (conventional thermosoling) and VI (10% urea pad-bath with microwave fixation). All other dyeing methods caused an increase in fiber density. The greatest increases in fiber density occurred with dyeing methods II (microwave predrying with conventional thermofixation) and VIII (45% urea with microwave-fixation).

The carrierless dyeable polyester exhibited similar changes in density with dyeing methods I, III, and VI resulting in a decrease in density and all other methods causing an increases.

The increases and decreases in fiber density resulted in significant differences between values obtained for the various dyeing methods. There was a significant difference between the density of the polyester specimens dyed by method II and those dyed by using methods I, V, and VI. Similarly, fibers dyed by method VIII had a significantly higher density than did those dyed by method I.

These changes in fiber density may be attributed to the

Table 14: Duncan's Multiple Range Test for Dyeing Methods..

Density (g/cm^3)	Dyeing method	Grouping*
1.3901	II	A
1.3847	VIII	A B
1.3766	IV	A B C
1.3748	VII	A B C
1.3722	III	A B C D
1.3712	V	B C D
1.3704	Control	B C D
1.3617	VI	C D
1.3556	I	D

* Means with the same letter are not significantly different.

temperatures attained during the dyeing process or the degree of internal heating which occurred therein, and the presence of the urea in the pad-bath. Research has shown, for example, that low molecular weight substances can influence crystallinity by increases the mobility of the polymer chains. The decrease in crystallinity after thermosoling probably was due to the temperature used in the thermofixation. The interaction between the low molecular weight urea and water molecules and the heat genetrated internally during microwave-fixation resulted in an appreciable increase in density for specimens pad-applied with 45% urea and exposed to microwaves over a boiling water bath.

Breaking Strength and Elongation

The single strand yarn method was used to measure the breaking strength and elongation of the regular dyeable and carrierless dyeable polyester specimens before and after dyeing. Structural changes within the fiber frequently are reflected in tensile strength properties. An increase in crystallinity may result in a significant increase in tensile strength such as that exhibited by fibers after drawing.

Breaking strength and elongation data for the polyester specimens are presented in Figures 12 and 13, respectively. Because the initial tensile strength values of the regular dyeable and carrierless dyeable polyesters differed markedly, the statistical analyses were performed on percent change rather than the actual values in kilograms of force at the time of failure. The raw data

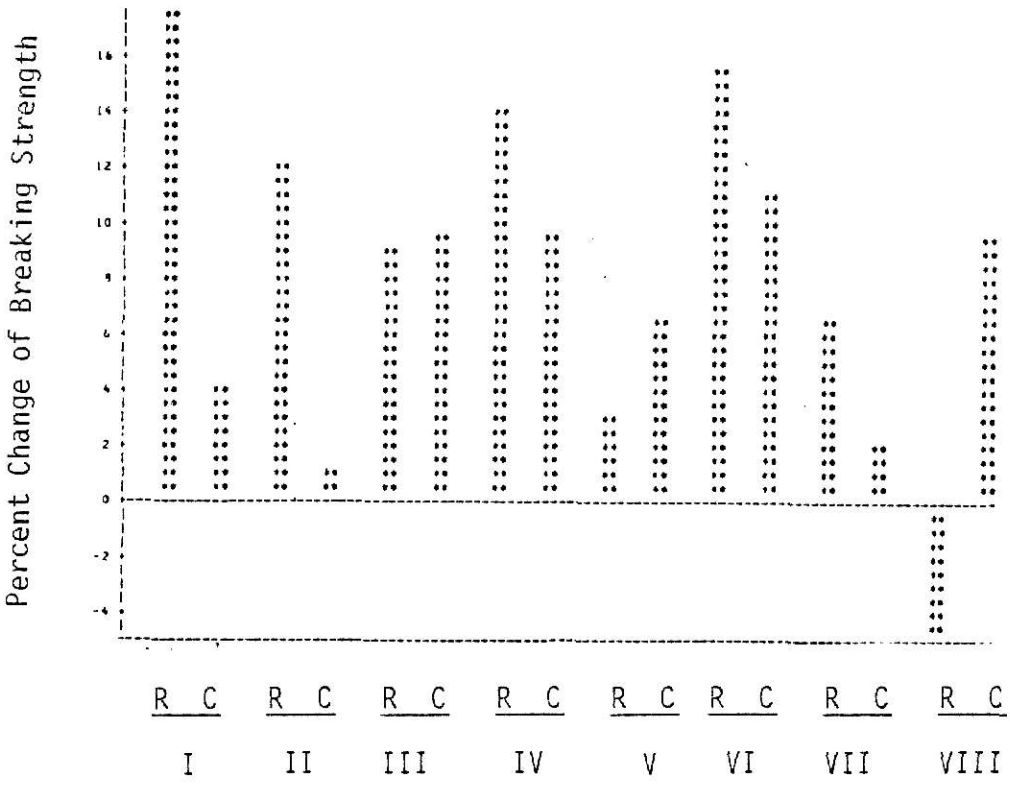

Figure 12: Percent Change of Breaking Strength for Regular Dyeable (R) and Carrierless Dyeable (C) Polyesters.

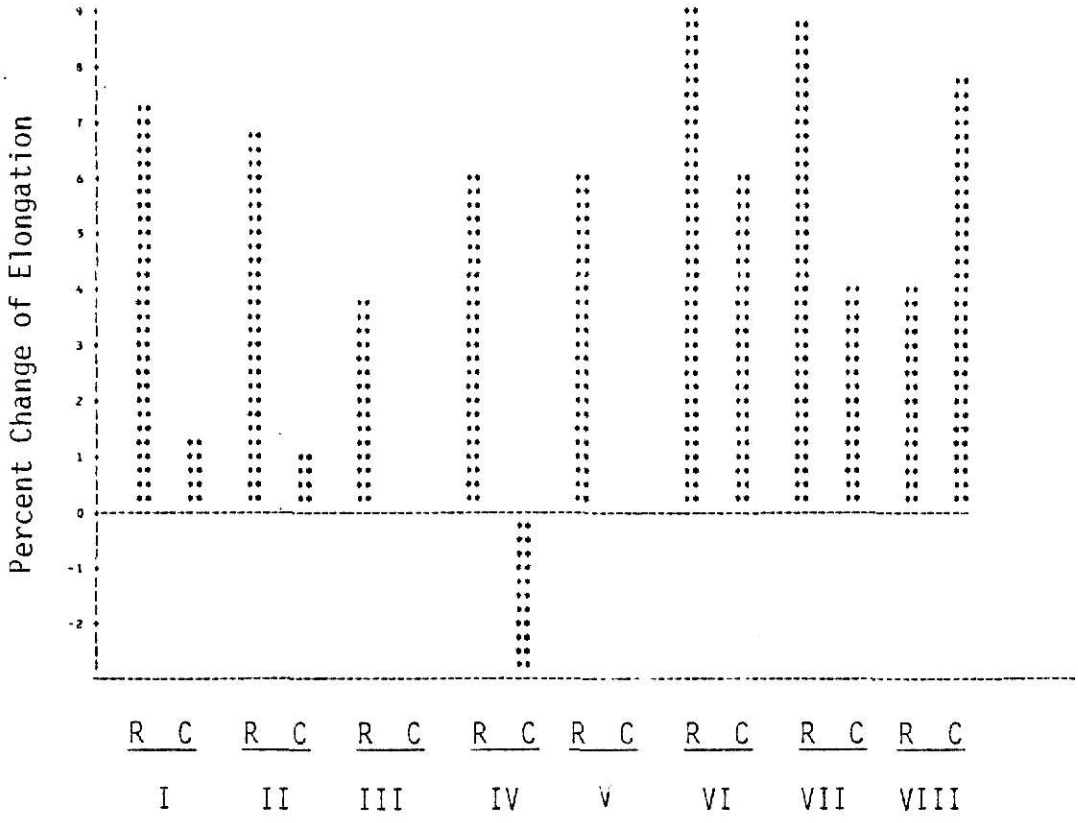

Figure 13: Percent change of Elongation for Regular Dyeable (R) and Carrierless Dyeable (C) Polyesters.

for breaking strength and elongation are given in Appendix B.

Based on the analysis of variance tests, as shown in Tables 15 and 16, type of polyester and dyeing method had no significant effect on the tensile strength values of the test specimens; however the percent change in elongation was significantly greater in the regular dyeable polyester. Minimal changes occurred in the elongation properties of the carrierless dyeable polyester as a result of the eight dyeing methods.

Small-Angle Light Scattering (SALS)

The fiber surface characteristics of the control specimens and those dyed by the eight methods previously described were evaluated by using the SALS technique and are illustrated in Figures 14 through 31.

Generally, carrierless dyeable polyester exhibited more light scattering than did the regular dyeable polyester specimens, both initially and after each of the eight dyeing treatments. This may have been attributed to the non-circular cross section, rougher fiber surface, higher fiber cohesion, and greater migration of oligomers on the fiber surface of the carrierless dyeable polyester during dyeing.

When compared to the untreated controls, the eight dyeing treatments reduced the amount of light scattering in the regular dyeable polyester; whereas for the carrierless dyeable polyester, the initial amount of light scattering was increased after dyeing, except after dyeing method I in which there was no change and dyeing method IV which reduced the amount of light scattering. For the carrierless dyeable polyester, the amount of light scattering was proportional to the urea concentration (except dyeing method V) and water bath temperature. However, with regular dyeable polyester, there was an inverse

Table 15: Analysis of Variance for Breaking Strength Values.

Source of Variations	Degrees of Freedom	Sum. of Squares	F	PR>F
Type of Polyester (P)	1	21.44	0.57	0.4745
Dyeing Methods (D)	7	218.24	0.83	0.5936

Table 16: Analysis of Variance for Elongation Values.

Source of Variations	Degrees of Freedom	Sum. of Squares	F	PR>F
Type of Polyester (P)	1	72.25	10.95	0.0130*
Dyeing Methods (D)	7	63.28	1.37	0.3443

* Significant at the 0.05 level.

Figures 14-19: SALS Photographs.

Figure 14 : CR, Control Specimen of Regular Polyester.

Figure 15 : 1R, Regular Polyester : Dyeing Method I (Thermosol).

Figure 16 : 2R, Regular Polyester : Dyeing Method II (microwave predrying with conventional thermofixation).

Figure 17 : 3R, Regular Polyester : Dyeing Method III (100% water dyebath, heated water, microwave fixation).

Figure 18 : 4R, Regular Polyester : Dyeing Method IV (100% water dyebath, boiling water, microwave fixation).

Figure 19 : 5R, Regular Polyester : Dyeing Method V (10% urea dyebath, heated water, microwave fixation).

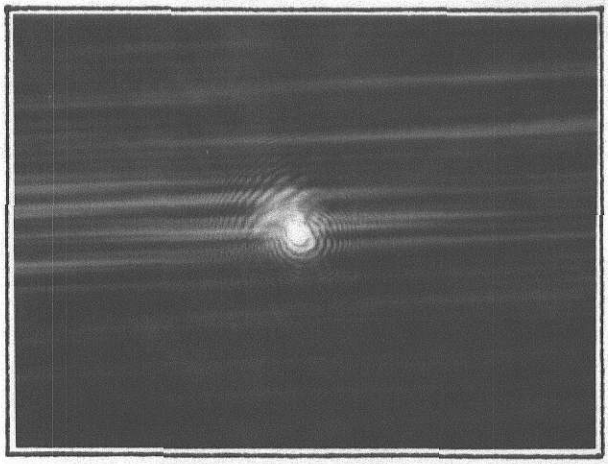

Figure 14

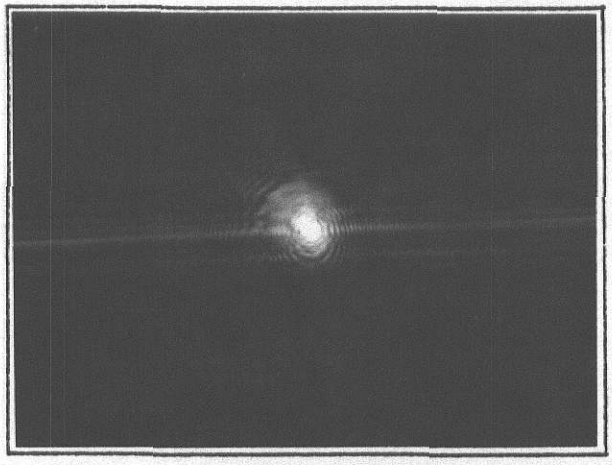

Figure 15

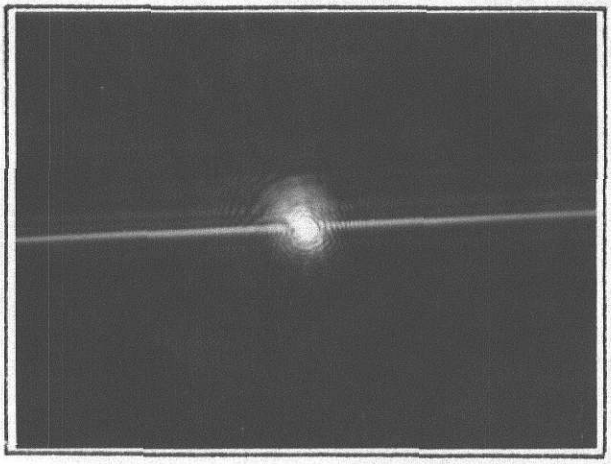

Figure 16

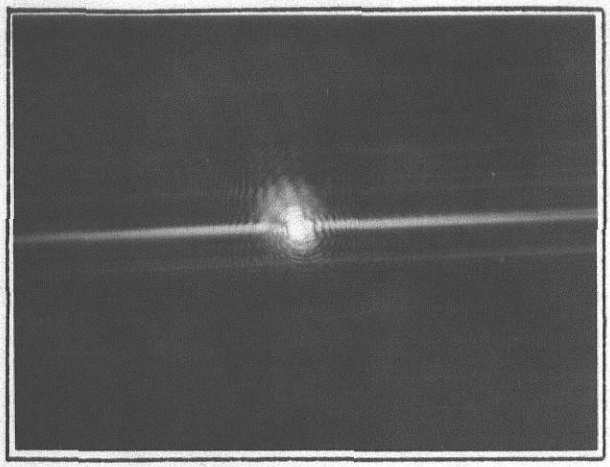

Figure 17

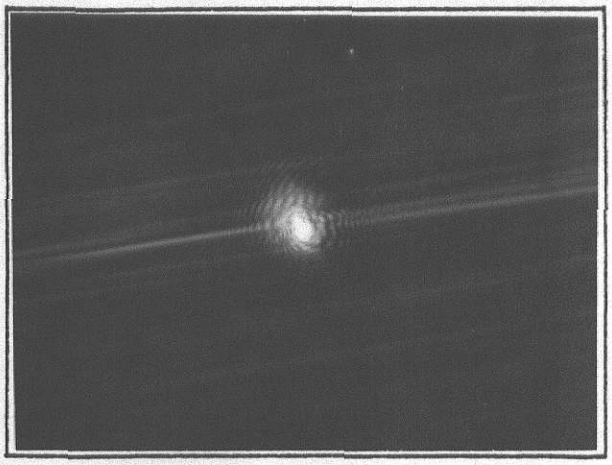

Figure 18

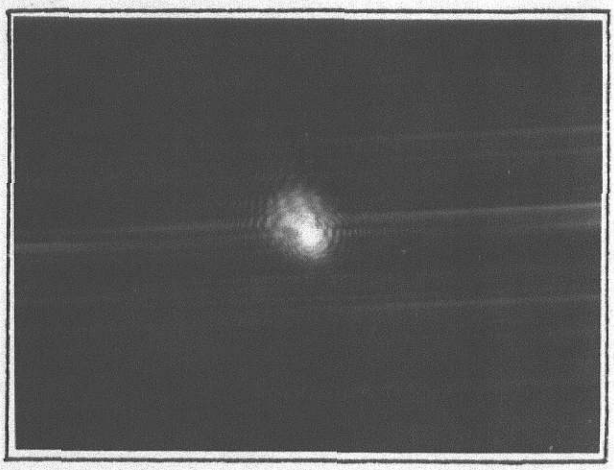

Figure 19

Figures 20-25: SALS Photographs.

Figure 20 : 6R, Regular Polyester : Dyeing Method VI (10% urea dyebath, boiling water, microwave fixation).

Figure 21 : 7R, Regular Polyester : Dyeing Method VII (45% urea dyebath, heated water, microwave fixation).

Figure 22 : 8R, Regular Polyester : Dyeing Method VIII (45% urea dyebath, boiling water, microwave fixation).

Figure 23 : CC, Control Specimen of Carrierless Dyeable Polyester.

Figure 24 : 1C, Carrierless Dyeable Polyester : Dyeing Method I (Thermosol).

Figure 25 : 2C, Carrierless Dyeable Polyester : Dyeing Method II (microwave predrying with conventional thermofixation).

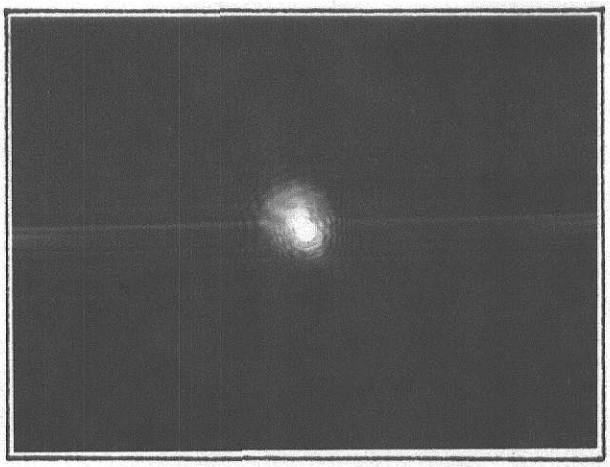

Figure 20

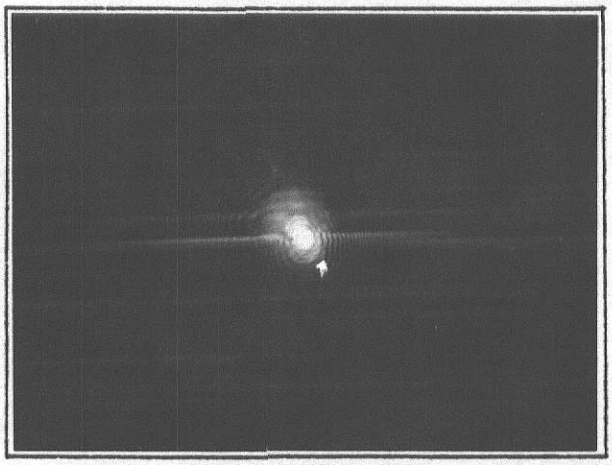

Figure 21

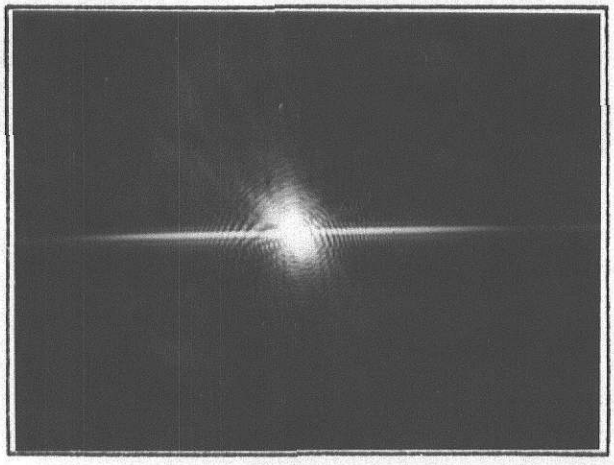

Figure 22

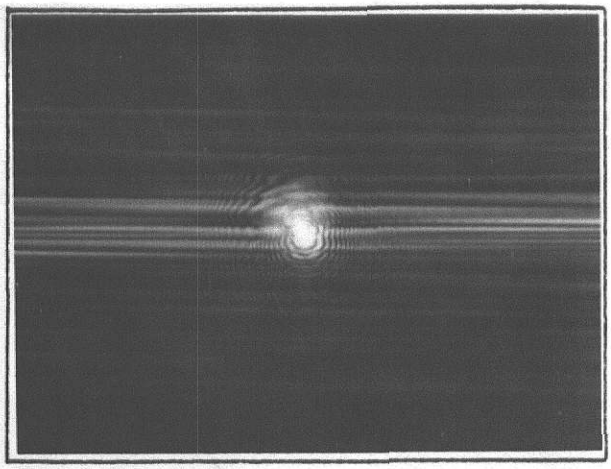

Figure 23

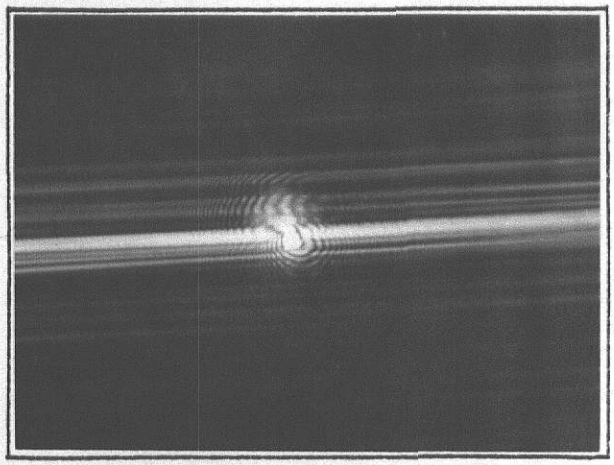

Figure 24

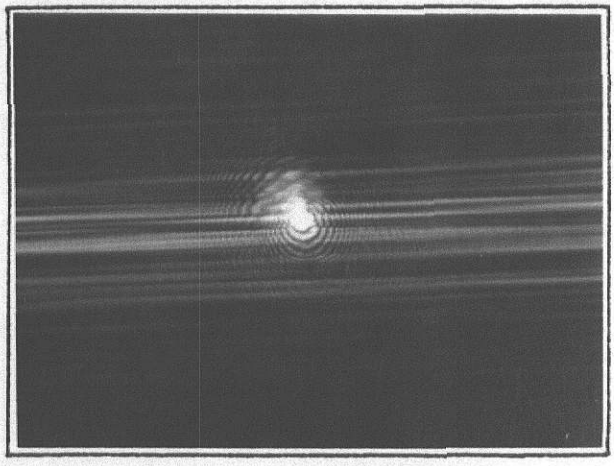

Figure 25

Figures 26-31: SALS Photographs.

Figure 26 : 3C, Carrierless Dyeable Polyester : Dyeing Method III
(100% water dyebath, heated water, microwave fixation).

Figure 27 : 4C, Carrierless Dyeable Polyester : Dyeing Method IV
(100% water dyebath, boiling water, microwave fixation).

Figure 28 ; 5C, Carrierless Dyeable Polyester : Dyeing Method V
(10% urea dyebath, heated water, microwave fixation).

Figure 29 : 6C, Carrierless Dyeable Polyester : Dyeing Method VI
(10% urea dyebath, boiling water, microwave fixation).

Figure 30 : 7C, Carrierless Dyeable Polyester : Dyeing Method VII
(45% urea dyebath, heated water, microwave fixation).

Figure 31 : 8C, Carrierless Dyeable Polyester : Dyeing Method VIII
(45% urea dyebath, boiling water, microwave fixation).

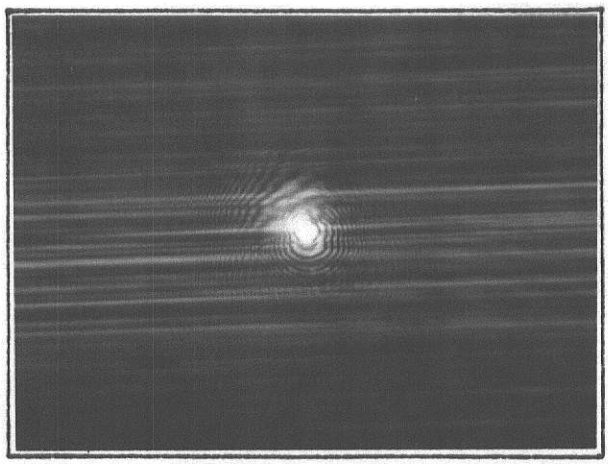

Figure 26

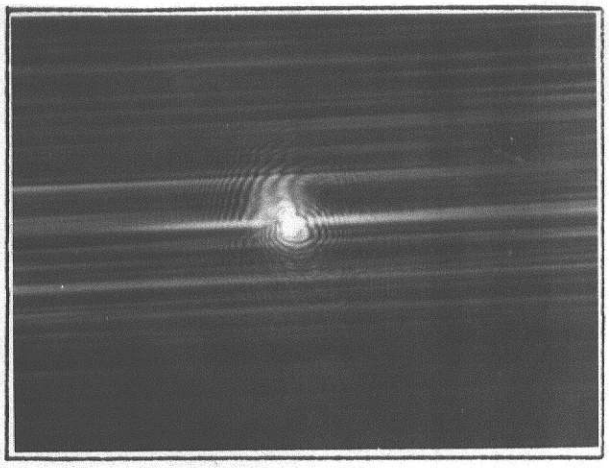

Figure 29

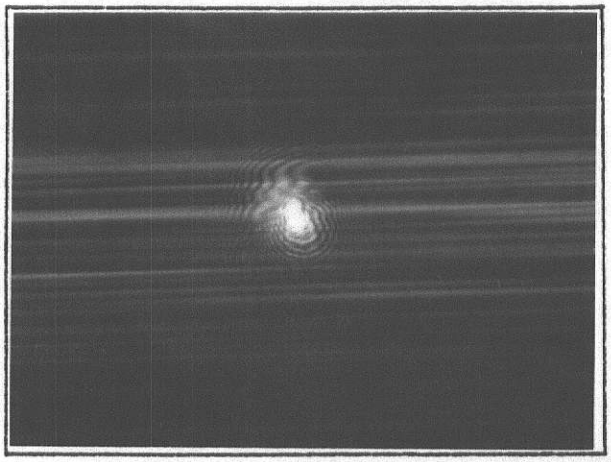

Figure 27

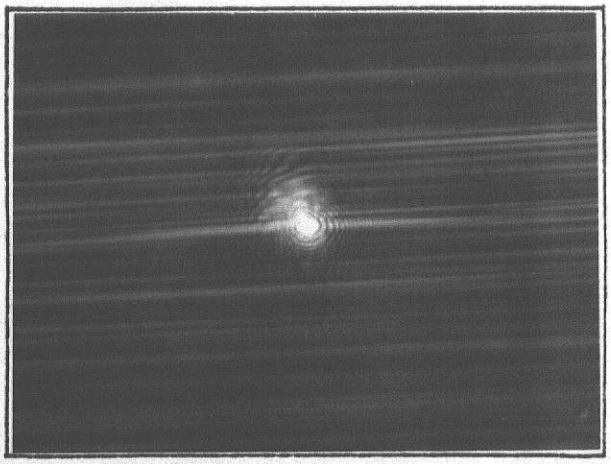

Figure 30

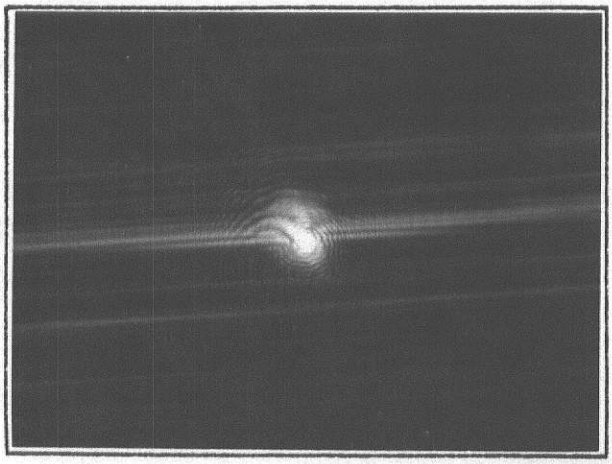

Figure 28

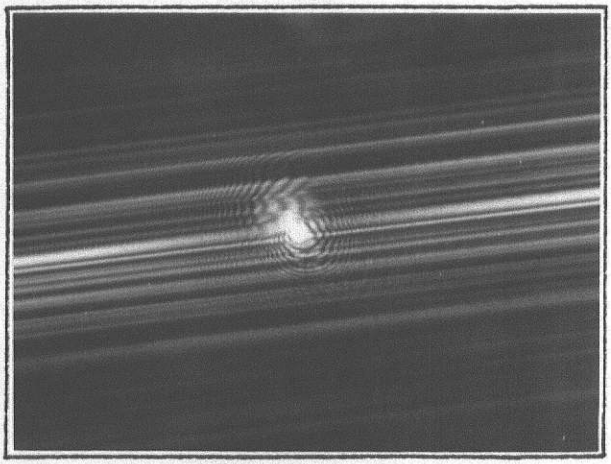

Figure 31

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relationship between urea concentration and temperature and the amount of light scattering.

In general, for both of the polyester, the fibers from various microwave-fixation methods had more scattering than the fibers from dyeing method I and II. Furthermore, the microwave predried specimens exhibited more light scattering than did the specimens that were predried by conventional heating prior to thermofixation.

Interrelationship among Variables

In order to further investigate the interrelationship among the various dependent and independent variables, Pearson's correlation coefficient was computed on the K/S values and density, breaking strength and elongation data for the regular dyeable and carrierless dyeable polyester specimens that were dyed by the eight methods previously described. The results from these statistical tests are given in Tables 17 and 18.

K/S Values among Different Dyestuffs

Variations in depth of shade among dyeing methods for both polyester types were similar for each dye type. Thus, there was significantly high, positive correlation coefficient (r) between each color ((i.e., C. I. Disperse Violet 33 vs. C. I. Disperse Blue 200 ($r = 0.993$), C. I. Disperse Blue 200 vs. C. I. Disperse Yellow 126 ($r = 0.988$), and C. I. Disperse Violet 33 vs. C. I. Disperse Yellow 126 ($r = 0.976$))) for regular dyeable polyester. Similar results were obtained for the carrierless dyeable polyester, except the correlation coefficient was slightly lower for the C. I. Disperse Blie 200 vs. C. I. Disperse Yellow 126 ($r = 0.887$). The similarities in dyeing behavior exhibited by the three disperse dyestuffs were

Table 17: Pearson's Correlation Coefficient for Carrierless Dyeable Polyester.

	Elongation	Density	K/S Values C. I. Disperse Blue 200 (Dispersol Blue D-4G) [K/S,B]	C. I. Disperse Violet 33 (Dispersol Rubine C-B) [K/S,R]	C. I. Disperse Yellow 126 (Dispersol Yellow D-7G)
Breaking Strength	0.120	-0.332	-0.625	-0.639	-0.609
Elongation		0.137	0.142	0.218	0.046
Density			-0.155	-0.173	-0.264
[K/S,B]				0.918*	0.887*
[K/S,R]					0.979*

* Significant at the 0.05 level of confidence.

Table 18: Pearson's Correlation Coefficient for Regular Dyeable Polyester.

	Elongation	Density	C. I. Disperse Blue 200 (Dispersol Blue D-4G) [K/S,B]	C. I. Disperse Violet 33 (Dispersol Rubine C-B) [K/S,R]	C. I. Disperse Yellow 126 (Dispersol Yellow D-7G)
Breaking Strength	0.529	-0.348	0.106	0.067	0.011
Elongation		-0.487	0.421	0.367	0.414
Density			-0.105	-0.060	-0.124
[K/S,B]				0.993*	0.988*
[K/S,R]					0.976*

* Significant at the 0.05 level of confidence.

attributed to the commonalities in chemical structure.

K/S Values vs. Density Values

The correlation coefficients ("r" values) for K/S values and density were similar for the regular dyeable or carrierless dyeable polyester. There was a negative correlation between K/S values and density for both fiber types, but they were non-significant. Thus, as K/S values increased, there was a slight decrease in density. This general trend was observed with dyeing methods III and IV (microwave-fixation with 100% water) and V and VI (microwave-fixation with 10% urea). Specimens dyed by methods V and VI had higher K/S values but lower density values than did those dyed by methods III and IV. Perhaps the increase in molecular oscillation and heat generation due to the urea in dyeing methods V and VI resulted in greater molecular chain movement and increased dye diffusion.

Specimens dyed by dyeing method I had the lowest density values and higher K/S values compared to the specimens from dyeing methods II through VIII. The specimens from dyeing method II had the highest density values. However, their K/S values were only slightly lower than the specimens from the conventional predry and thermofixation. The density values of specimens dyed by dyeing methods VII and VIII were lower than specimens from dyeing method II and much higher than those from dyeing method I, but their K/S values were lower than the specimens from both dyeing methods I and II. There was no obvious correlation between the density and K/S values for dyeing methods I, II, VII, and VIII. However, different dyeing methods did cause changes in density.

K/S Values vs. Breaking Strength

For regular dyeable polyester, there was a non-significant

positive correlation between K/S values and breaking strength; however, there was a non-significant negative correlation between these two variables for the carrierless dyeable polyester. This difference might be due to the differences in fiber structures of these two types of polyester. The looser structures of the carrierless dyeable polyester may have increased its sensitivity to the heat treatments. The negative correlation between K/S values and breaking strength for the carrierless dyeable polyester applied to those specimens dyed by methods III, V, and VII. The lack of correlation between K/S values and breaking strength was attributed to the non-significant changes in breaking strength after dyeing. Thus, changes in fiber structures (i.e., degree of crystallinity, size of crystals and orientation) did not result in significant changes in breaking strength measurement. In other words, changes breaking strength were not as sensitive as density gradient analysis and small-angle light scattering in detecting fiber structural changes.

K/S Values vs. Elongation

The correlation coefficients for K/S values and elongation were positive but non-significant for both polyesters. The "r" values were higher for regular dyeable polyester than for the carrierless dyeable polyester. The differences between the polyester types may have been attributed to differences in chemical or morphological properties of the fibers. In the analysis of variance test, dyeing method was not a significant variable that caused changes of elongation. Furthermore, there was no obvious correlation between K/S values and elongation for dyeing methods I, II, IV, VI and VIII.

Breaking Strength vs. Elongation

There was a non-significant positive correlation between breaking

strength and elongation for both types of polyester. This correlation was higher for regular dyeable polyester than for the carrierless dyeable polyester. Although the type of dyeing method did not cause statistically significant differences in breaking strength and elongation, some interesting changes did occur. For the regular dyeable polyester, all dyeing methods caused an increase in breaking strength and elongation, except dyeing method VIII which caused a decrease in breaking strength. Similar results were observed for carrierless dyeable polyester, however, dyeing methods III and VI did not affect the percent change of elongation and dyeing method IV caused a decreases in elongation. In total, regular dyeable polyester had exhibited greater changes in breaking strength and elongation than did the carrierless dyeable polyester.

Breaking Strength and Elongation vs. Density

There were similar non-significant negative correlation between density values and breaking strength for both types of polyester. However, there was non-significant positive correlation between density values and elongation for carrierless dyeable polyester but a non-significant negative correlation for regular dyeable polyester. Thus, there was no obvious trend between these variables for the different dyeing methods. The lack of correlation was due to the lack of significant changes in breaking strength and elongation after the various dyeing methods, whereas the type of dyeing method caused significantly changes in fiber density values. Changes in density could be attributed to many factors (i.e., crystal perfection, degree of crystallinity, chemical constitution and molecular orientation) which were not re- ıgth flected in the breaking strength and elongation values of the tested yarns.

Photographs from the SALS test showed that the amount of light scattering of various treated fibers were different from the control. These phenomena illustrated that method of dyeing did cause changes in the fiber structures. In order to more thoroughly explore internal structural changes in the fiber, the SALS test should be repeated with fibers mounted in a similar refractive index liquid.

After comparing the results from various color measurements, physical property tests, analysis of variance and Duncan's Multiple Range Test; dyeing methods II (i.e., 100% water dyebath with microwave predrying and conventional thermofixation) and VII (i.e., 45% urea dyebath with heated water bath and microwave thermofixation) have potential for further development as industrial dyeing methods. Higher K/S values would be expected with microwave ovens having higher energy output and penetration properties. Again, for industrial scale operations, microwave ovens could be designed so that steaming, irradiation, and dye fixation were more uniform.

In this study, dyeing method II and VII had high K/S values, and uniform dye penetration without undesirable changes of physical properties. Microwave predrying could eliminated the premature dye penetration with considerable energy saving and shorter predrying time. The advantages of dyeing method VII are (1) applicable to a continuous dyeing method, (2) elimination of predrying and elevated temperature thermofixation with considerable energy saving, and (3) recycling of urea from the dyebath residues.

SUMMARY AND CONCLUSIONS

Numerous dyeing procedures were explored with different solvent systems, water temperatures, and methods of exposure, in order to develop a suitable microwave dyeing method for regular polyester and carrierless dyeable polyester. The results obtain with microwave dyeing were compared to that obtained with the conventional thermosol method in terms of depth of shade, evenness of color, dye penetration, and changes in the physical properties of the polyester fabrics after microwave irradiation.

The K/S values were varied significantly by the type of polyester, dyeing method, and urea concentration. Generally, carrierless dyeable polyester had higher k/S values than the regular dyeable polyester. Results from dyeing methods I and II had the highest K/S values. The K/S values were proportional to the increase in urea concentration for dyeing methods III through VIII in which microwave radiation was used for fixation. Since the energy output of the microwave oven used in this study was relatively low, higher K/S values (i.e., greater depth of shade) may be obtained by using a microwave oven with the same frequency (2450 MHz) but with a greater energy output or an industrial microwave oven (915 MHz). From the examination of fiber cross-sections it was evident that microwave heating resulted in better dye penetration than conventional thermosol dyeing. However, there was no significant correlation between K/S values and physical property data (i.e., density, breaking strength and elongation). Therefore, the dye molecules may have diffused into the fibers before any changes of fiber structures occurred during microwave-fixation.

There was no significant difference in breaking strength caused by the various types of polyester, dyeing method, water bath temperature or urea concentration. Elongation was only varied by the type of polyester. The method of dyeing had a significant effect on fiber density. Theoretically, changes in breaking strength should be proportional to increase in fiber density. The lack of correlation between these variables may have been attributed to the lack of sensitivity in the breaking strength test. The changes in fiber density also may not have been great enough to cause significant changes in yarn strength.

The SALS technique proved to be a sensitive method for examining both internal and external changes in the fibers. Because the fibers were mounted in air, most of the results from SALS only reflected the surface changes on the fiber. The fibers from dyeing methods II-VIII had more light scattering than the fiber specimens which were dyed by using method I (conventional predrying with thermofixation). In order to more fully understand the internal structural changes caused by the different heating methods, the SALS technique should be repeated with fibers mounted in a similar refractive index liquid. This procedure would eliminate light scattering caused by external irregularities. Thus, the resulting patterns would depict internal changes.

Under suitable conditions, microwave irradiation could cause direct heating and vaporization of the moisture inside the fabric. Comparing dyeing methods II to I, microwave predrying eliminated the undesirable premature dye penetration with shorter heating time period and comparable dye fixation. In microwave dyeing methods III through

VIII, the degree of dye fixation was increased with the increase of urea concentration, since the microwave-induced molecular oscillations (i.e., urea and water molecules) in fabrics achieved faster dye diffusion and increased plasticization within the fibers. Consequently, the microwave heating method could be introduced to the textile dyeing processes as predry or thermofixation method to obtain high color yield, uniform dye penetration and without significant changes in the physical properties of fibers.

RECOMMENDATIONS

Various microwave dyeing methods were explored in this study for regular dyeable and carrierless dyeable polyester fabrics. Some suggestions for further research include:

1) The study should be repeated using 2450 MHz microwave oven with a higher energy output to detect the relationship between the amount of microwave energy and dye fixation.
2) Further research should compare the degree of dye penetration obtained with microwave ovens differing in penetrating power (i.e., 2450 and 915 MHz).
3) Research should be conducted to determine the feasibility of using microwave energy to dye ionic-dyeable polyesters.
4) This study should be repeated using the same dyeing methods with a microwave oven which is capable of monitoring changes of temperature and moisture in the fabric during exposure.
5) The fiber specimens dyed in this study should be mounted in a similar refractive index liquid and evaluated by the SALS technique in order to detect the internal structural changes caused by microwave exposure.
6) Further research is needed to explore the potential of using microwaves in textile printing processes to save time and energy and to eliminate premature dye penetration.

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APPENDICES

APPENDIX A

COLOR DATA

Table A1: K/S Values for Regular Dyeable Polyester.

Dyeing Method	C. I. Disperse Blue 200 (Dispersol Blue D-4G)	C. I. Disperse Violet 33 (Dispersol Rubine C-B)	C. I. Disperse Yellow 126 (Dispersol Yellow D-7G)
I	9.44	9.23	8.46
	9.44	9.44	8.46
	9.44	9.23	8.12
II	9.03	8.64	7.96
	8.64	8.64	7.65
	8.46	8.29	7.65
III	3.30	2.72	3.06
	3.23	2.77	3.09
	2.85	2.62	2.88
IV	3.16	2.54	3.16
	3.23	2.57	3.30
	2.85	2.57	3.09
V	5.70	5.07	6.50
	6.08	4.79	6.08
	5.62	5.14	5.89
VI	6.61	5.53	5.98
	5.70	5.14	5.45
	5.70	4.73	5.14
VII	6.84	6.84	6.50
	6.72	6.61	6.39
	6.72	6.50	6.39
VIII	6.84	6.72	6.39
	6.72	6.61	6.28
	6.50	6.61	6.39

Table A2: K/S Values for Carrierless Dyeable Polyester.

Dyeing Method	C. I. Disperse Blue 200 (Dispersol Blue D-4G)	C. I. Disperse Violet 33 (Dispersol Rubine C-B)	C. I. Disperse Yellow 126 (Dispersol Yellow D-7G)
I	9.23	9.03	8.12
	9.44	9.44	8.29
	9.44	9.03	7.96
II	8.64	8.29	7.65
	8.46	8.29	7.65
	8.12	7.96	7.36
III	5.89	5.07	5.79
	5.89	4.92	5.70
	5.79	4.92	5.62
IV	5.98	5.29	5.98
	6.18	5.07	5.89
	5.79	5.14	5.98
V	5.98	6.84	6.72
	5.98	6.28	6.84
	5.70	6.97	6.84
VI	6.97	7.36	7.36
	6.08	6.39	6.39
	6.39	6.50	6.28
VII	6.84	6.84	6.50
	6.97	6.72	6.39
	6.97	6.61	6.39
VIII	7.10	6.72	6.39
	7.10	6.61	6.28
	6.97	6.61	6.39

Table A3: Dye Uniformity Measurements for Regular Dyeable Polyester with C. I. Disperse Blue 200 (Dispersol Blue D-4G).

Dyeing Method	Areas for ΔE Readings								
	1	2	3	4	5	6	7	3	Average
I	0.72	0.70	0.71	0.47	1.07	1.05	0.73	0.31	0.72
II	0.25	0.30	0.72	1.18	0.93	0.76	1.06	0.95	0.77
III	0.73	1.16	0.50	1.54	0.28	1.17	0.57	2.26	1.03
IV	0.11	0.38	0.35	0.62	0.44	1.23	0.26	0.89	0.54
V	4.27	2.25	2.74	0.35	3.64	1.81	2.98	2.76	2.60
VI	1.50	0.46	1.51	0.74	3.06	0.61	1.65	0.93	1.31
VII	0.46	0.35	0.18	0.55	0.77	0.43	0.62	0.71	0.51
VIII	0.21	0.30	1.04	0.50	0.87	0.18	0.17	0.16	0.43

Table A4: Dye Uniformity Measurements for Regular Dyeable Polyester with C. I. Disperse Violet 33 (Dispersol Rubine C-B).

Dyeing Method	Areas for ΔE Readings								
	1	2	3	4	5	6	7	8	Average
I	0.53	0.47	0.22	0.27	1.00	0.50	0.61	0.66	0.53
II	0.99	1.55	1.49	2.08	1.76	2.38	1.49	1.59	1.67
III	1.57	0.91	0.36	0.82	1.11	1.26	1.16	0.64	0.98
IV	2.26	0.73	0.65	1.17	0.79	1.21	0.90	1.54	1.16
V	4.21	1.72	0.97	2.30	4.93	0.83	3.47	0.68	2.39
VI	2.54	0.25	0.67	0.94	3.85	1.32	1.23	1.56	1.55
VII	0.72	0.36	0.37	0.87	0.46	0.07	0.19	0.71	0.47
VIII	0.55	0.20	0.89	0.99	0.99	0.46	1.06	0.40	0.69

Table A5: Dye Uniformity Measurements for Regular Dyeable Polyester with C. I. Disperse Yellow 126 (Dispersol Yellow D-7G).

Dyeing Method	Areas for ΔE readings 1	2	3	4	5	6	7	8	Average
I	0.60	0.43	0.69	0.13	0.84	0.53	0.57	0.30	0.51
II	0.62	1.63	0.47	0.89	0.27	0.76	0.53	0.64	0.73
III	1.27	1.36	0.50	1.60	0.24	1.80	1.49	1.26	1.19
IV	2.19	0.16	1.37	0.56	3.33	0.61	2.98	1.03	1.53
V	0.98	1.46	2.96	0.56	0.87	2.07	2.60	0.58	1.51
VI	2.41	2.03	1.90	0.28	2.03	1.12	3.17	0.88	1.73
VII	0.60	0.40	0.65	0.59	0.24	0.43	0.73	0.23	0.48
VIII	0.28	0.44	0.46	0.61	1.14	0.58	0.76	0.50	0.60

Table A6: Dye Uniformity Measurements for Carrierless Dyeable Polyester with C. I. Disperse Blue 200 (Dispersol Blue D-4G).

Dyeing Method	Areas for ΔE readings								
	1	2	3	4	5	6	7	8	Average
I	0.70	0.96	0.68	0.12	1.28	1.43	1.27	0.28	0.84
II	1.08	1.07	0.52	0.92	1.51	1.61	1.69	0.89	1.16
III	0.62	1.09	0.53	1.03	0.75	1.49	0.32	0.84	0.83
IV	0.81	0.70	0.70	1.52	0.23	0.21	0.30	0.31	0.61
V	0.80	1.24	0.94	0.78	1.65	0.24	1.83	0.98	1.06
VI	o.50	0.71	0.20	0.30	0.19	0.57	0.92	1.58	0.62
VII	0.22	0.38	0.51	0.33	0.41	0.64	0.39	0.92	0.48
VIII	0.74	0.22	0.78	0.65	0.88	0.44	0.62	0.52	0.61

Table A7: Dye Uniformity Measurements for Carrierless Dyeable Polyester with C. I. Disperse Violet 33 (Dispersol Rubine C-B).

Dyeing Method	Areas for ΔE readings								
	1	2	3	4	5	6	7	8	Average
I	1.07	0.71	0.07	1.03	1.11	0.53	1.08	0.48	0.76
II	2.10	1.62	2.54	2.09	1.84	2.10	1.63	2.08	2.00
III	0.60	0.62	0.69	1.19	1.23	0.54	0.50	1.50	0.86
IV	1.54	0.54	2.51	1.67	1.53	0.62	1.12	1.52	1.38
V	2.56	2.07	2.31	1.49	3.51	1.40	1.91	0.51	1.97
VI	0.66	1.09	0.88	2.07	1.07	0.53	0.79	0.92	1.00
VII	0.74	1.09	0.56	1.03	0.84	0.82	0.59	0.56	0.78
VIII	1.02	0.52	1.35	1.40	1.79	1.13	2.15	0.56	1.24

Table A8: Dye Uniformity Measurements for Carrierless Dyeable Polyester with C. I. Disperse Yellow 126 (Dispersol Yellow D-7G).

Dyeing Method	Areas for ΔE readings 1	2	3	4	5	6	7	8	Average
I	0.79	0.23	0.55	0.24	0.71	0.77	1.00	0.46	0.59
II	0.69	0.72	0.05	0.88	0.52	0.41	0.56	0.63	0.56
III	0.49	1.27	1.12	0.97	0.71	0.85	1.23	1.30	0.99
IV	0.64	0.34	0.30	1.10	0.76	2.17	1.17	0.27	0.84
V	1.04	0.58	1.45	0.44	0.97	0.68	1.85	0.74	0.97
VI	1.26	1.01	0.20	0.79	0.49	0.76	0.85	1.30	0.83
VII	1.27	0.84	0.48	0.19	0.23	0.43	0.82	0.72	0.62
VIII	1.01	1.37	0.65	0.83	0.92	1.47	1.03	1.34	1.08

APPENDIX B

PHYSICAL PROPERTY DATA

Table B1: Density Values for Regular Dyeable and Carrierless Dyeable Polyesters.

Dyeing Method	Density Values (g/cm^3) Regular PET	Carrierless Dyeable PET
Control	1.3681	1.3727
I	1.3539	1.3572
II	>1.3868	1.3801
III	1.3788	1.3655
IV	1.3794	1.3737
V	1.3684	1.3739
VI	1.3638	1.3596
VII	1.3681	1.3815
VIII	1.3868	1.3826

Table B2: Breaking Strength and Elongation Values for Regular Dyeable Polyester.

Dyeing Method	Breaking Strength (Kg)	Elongation (in)
Control	1.57	1.65
	1.68	1.86
	1.64	1.80
	1.57	1.73
	1.78	1.85
I	1.69	1.92
	1.88	2.04
	1.74	1.75
	1.54	1.65
	1.73	1.76
II	1.59	1.76
	1.63	1.66
	1.78	1.73
	1.77	2.03
	1.57	1.88
III	1.85	1.63
	1.90	2.00
	1.74	1.67
	1.73	1.76
	1.84	1.86
IV	1.84	1.87
	1.54	1.44
	1.74	2.00
	2.12	1.87
	1.80	1.34
V	1.73	1.93
	1.88	1.70
	1.79	1.84
	1.78	1.66
	1.63	1.76
VI	1.88	2.00
	1.68	1.95
	1.90	1.93
	1.68	1.86
	2.00	2.04
VII	1.80	1.82
	1.73	1.98
	1.72	1.92
	1.53	1.83
	1.64	1.95

Table B2: Breaking Strength and Elongation Values for Regular Dyeable Polyester. (continued)

Dyeing Method	Breaking Strength (Kg)	Elongation (in)
VIII	1.16	1.77
	1.00	1.55
	1.00	1.67
	1.08	1.64
	0.96	1.52

Table B3: Breaking Strength and Elongation Values for Carrierless Dyeable Polyester.

Dyeing Method	Breaking Strength (Kg)	Elongation (in)
Control	0.95	1.44
	1.28	1.64
	1.10	1.57
	1.15	1.57
	0.96	1.34
I	1.07	1.58
	1.22	1.62
	1.58	1.68
	1.26	1.84
	1.25	1.94
II	1.28	1.54
	1.22	1.57
	1.20	1.64
	1.21	2.02
	1.20	1.77
III	1.16	1.78
	1.22	1.67
	1.23	1.63
	1.20	1.54
	1.14	1.50
IV	1.15	1.65
	1.32	1.77
	1.17	1.62
	1.38	1.74
	1.20	1.67
V	1.07	1.60
	1.14	1.66
	1.10	1.78
	1.00	1.60
	1.29	1.80
VI	1.26	1.90
	1.27	1.86
	1.30	1.77
	1.48	1.87
	1.00	1.50
VII	1.05	1.57
	1.20	1.78
	1.27	1.97
	1.17	1.67
	1.10	1.85

Table B3: Breaking Strength and Elongation Values for Carrierless Dyeable Polyester. (continued)

Dyeing Method	Breaking Strength (Kg)	Elongation (in)
VIII	1.70	1.73
	1.88	2.07
	1.70	2.20
	1.84	1.90
	1.91	2.14

APPENDIX C

UV/VIS SPECTRA

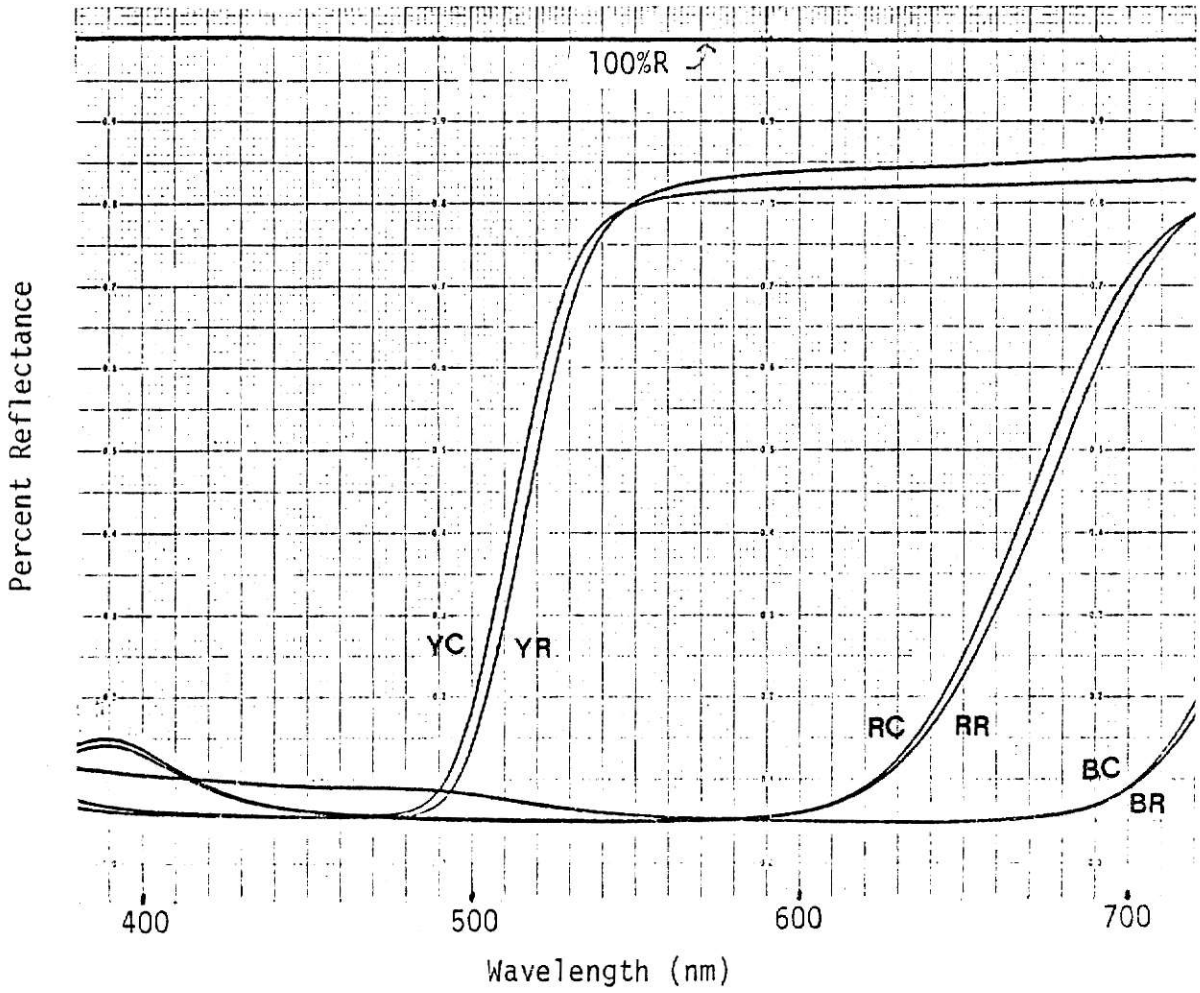

Figure C1: UV/VIS Spectra for Regular Dyeable (R) and Carrierless Dyeable (C) Polyester fabrics with Dyeing Method I (Thermosol) and Three Disperse Dyestuffs.

Disperse Dyestuffs:
B- C. I. Disperse Blue 200 (Dispersol Blue D-4G)
R- C. I. Disperse Violet 33 (Dispersol Rubine C-B)
Y- C. I. Disperse Yellow 126 (Dispersol Yellow D-7G)

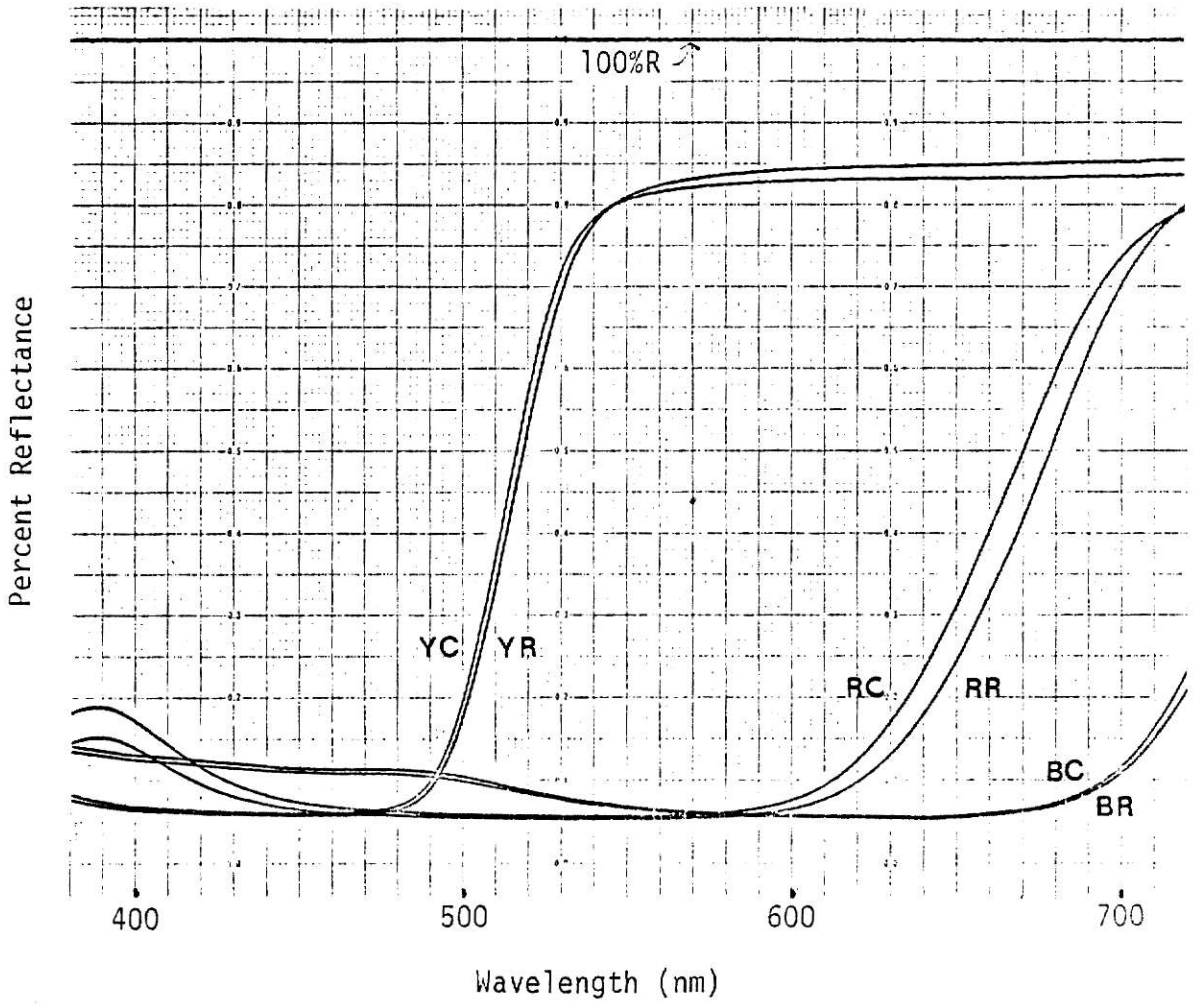

Figure C2: UV/VIS Spectra for Regular Dyeable (R) and Carrierless Dyeable (C) Polyester fabrics with Dyeing Method II (microwave-predry/conventional thermofixation) and Three Disperse Dyestuffs.

Disperse Dyestuffs:
B- C. I. Disperse Blue 200 (Dispersol Blue D-4G)
R- C. I. Disperse Violet 33 (Dispersol Rubine C-B)
Y- C. I. Disperse Yellow 126 (Dispersol Yellow D-7G)

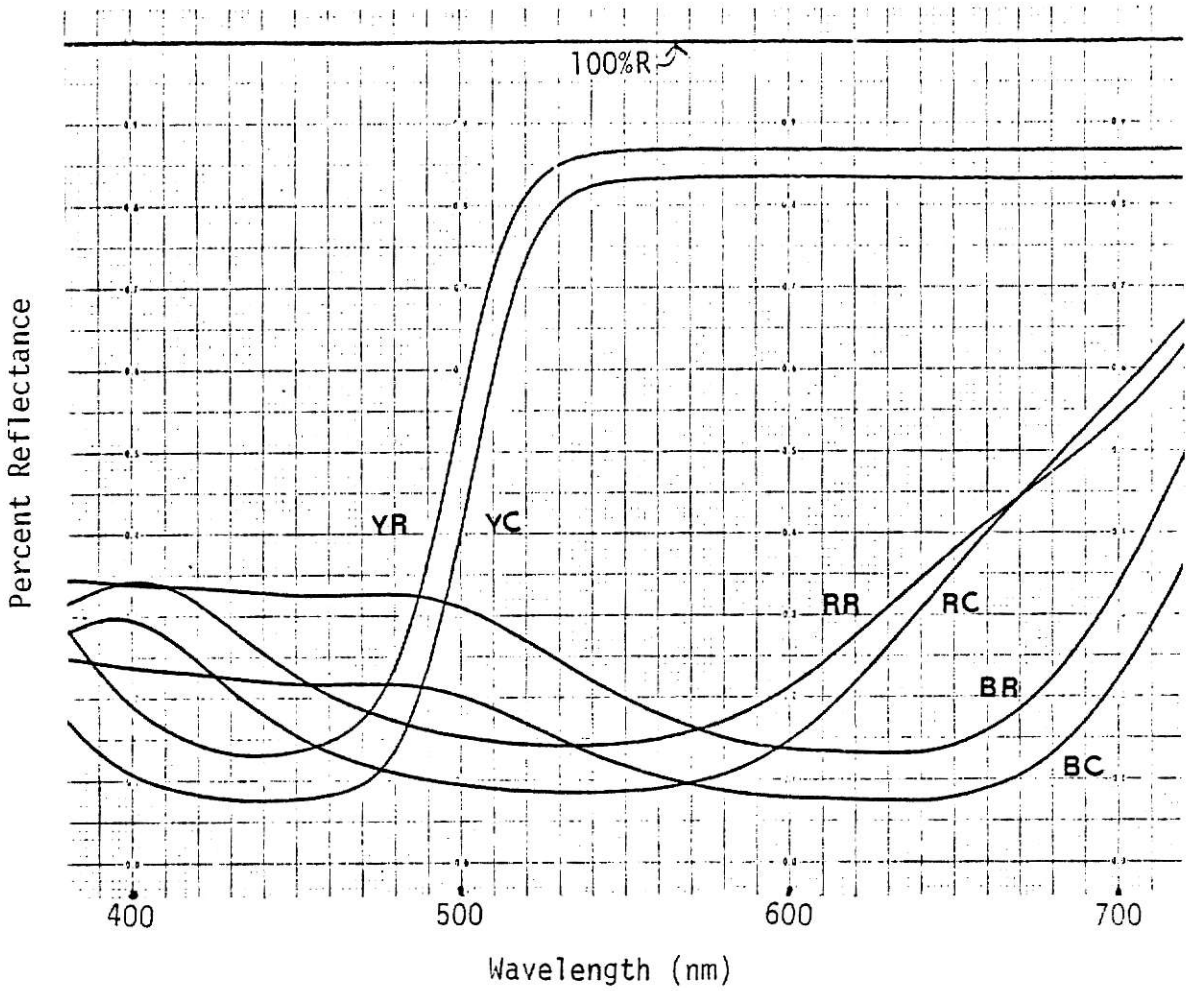

Figure C3: UV/VIS Spectra for Regular Dyeable (R) and Carrierless Dyeable (C) Polyester fabrics with Dyeing Method III (100% water, heated water, microwave-fixation) and Three Disperse Dyestuffs.

Disperse Dyestuffs:
B- C. I. Disperse Blue 200 (Dispersol Blue D-4G)
R- C. I. Disperse Violet 33 (Dispersol Rubine C-B)
Y- C. I. Disperse Yellow 126 (Dispersol Yellow D-7G)

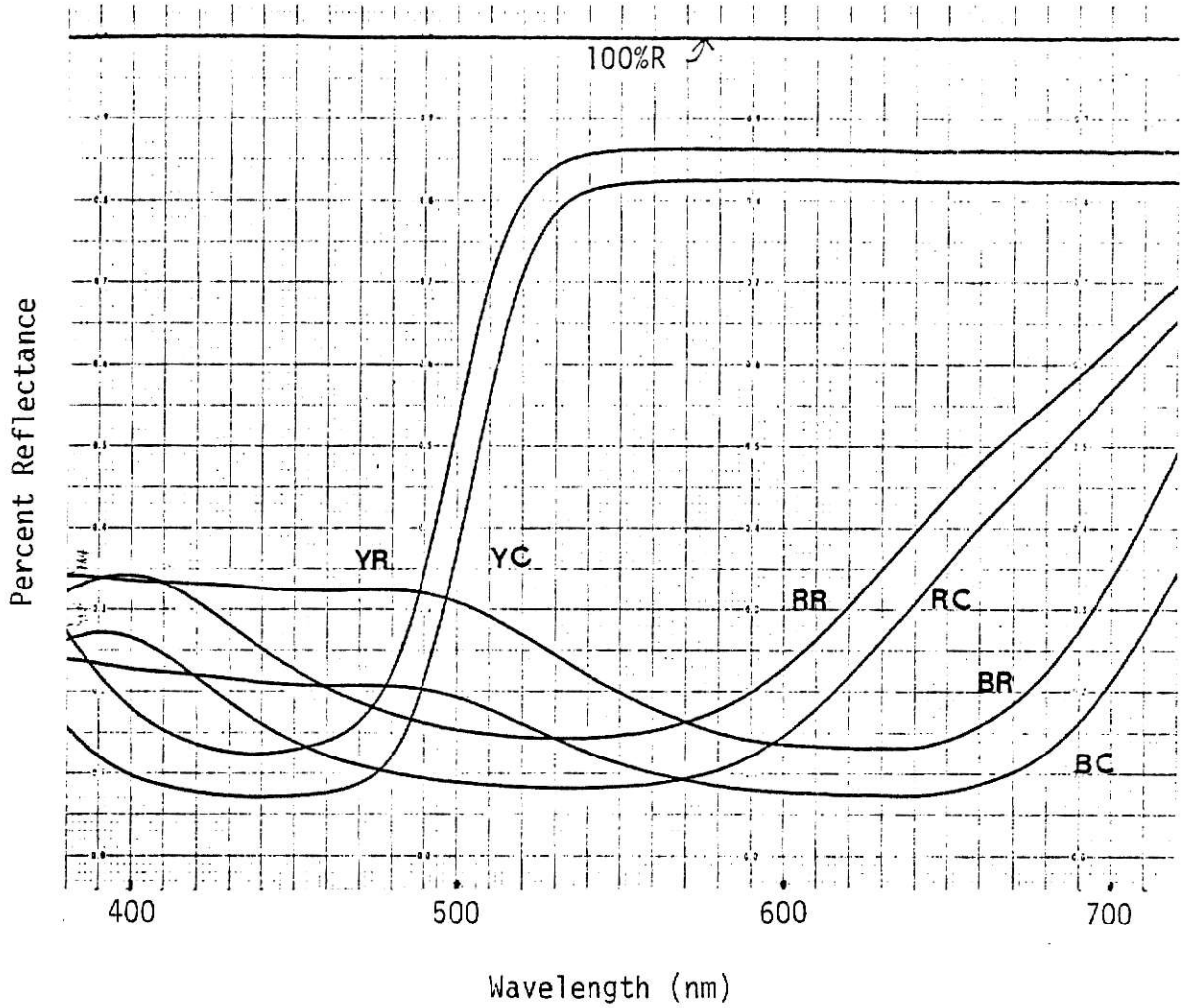

Figure C4: UV/VIS Spectra for Regular Dyeable (R) and Carrierless Dyeable (C) Polyester fabrics with Dyeing Method IV (100% water, boiling water, microwave-fixation) and Three Disperse Dyestuffs.

Disperse Dyestuffs:
B- C. I. Disperse Blue 200 (Dispersol Blue D-4G)
R- C. I. Disperse Violet 33 (Dispersol Rubine C-B)
Y- C. I. Disperse Yellow 126 (Dispersol Yellow D-7G)

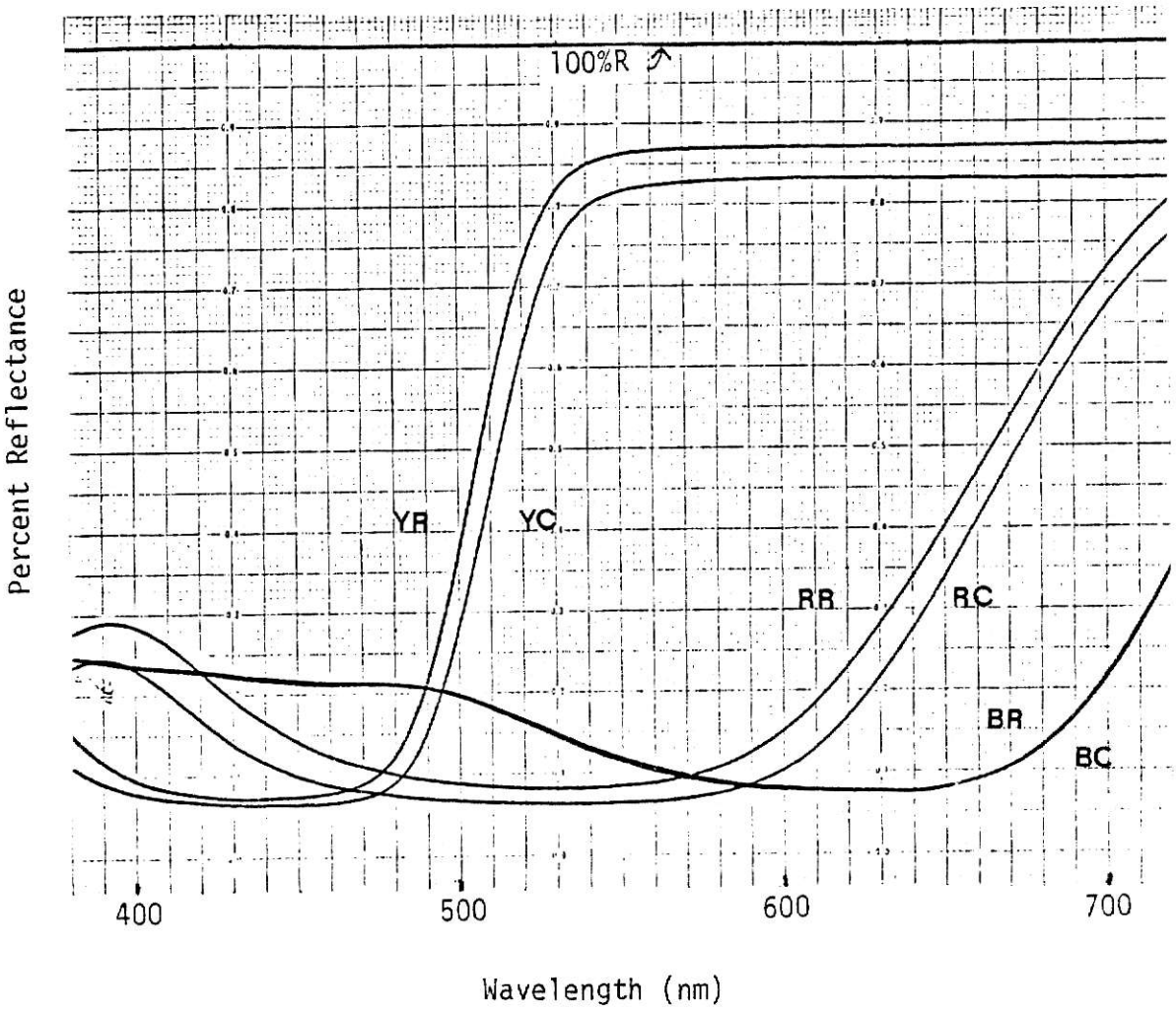

Figure C5: UV/VIS Spectra for Regular Dyeable (R) and Carrierless Dyeable (C) polyester fabrics with Dyeing Method V (10% urea, heated water, microwave-fixation) and Three Disperse Dyestuffs.

Disperse Dyestuffs:
B- C. I. Disperse Blue 200 (Dispersol Blue D-4G)
R- C. I. Disperse Violet 33 (Dispersol Rubine C-B)
Y- C. I. Disperse Yellow 126 (Dispersol Yellow D-7G)

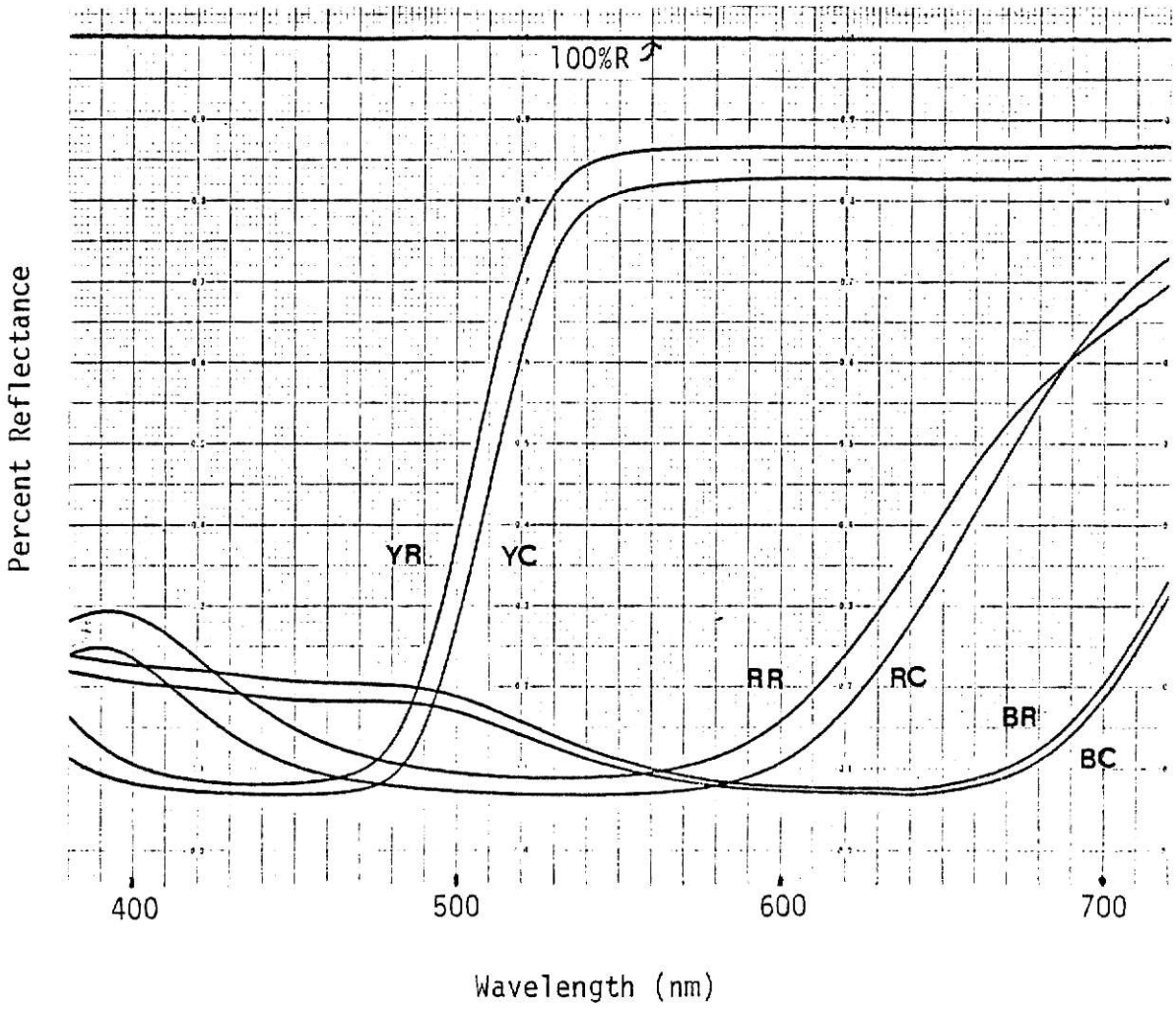

Figure C6: UV/VIS Spectra for Regular Dyeable (R) and Carrierless Dyeable (C) Polyester Fabrics with Dyeing Method VI (10% urea, boiling water, microwave-fixation) and Three Disperse Dyestuffs.

Disperse Dyestuffs:
B- C. I. Disperse Blue 200 (Dispersol Blue D-4G)
R- C. I. Disperse Violet 33 (Dispersol Rubine C-B)
Y- C. I. Disperse Yellow 126 (Dispersol Yellow D-7G)

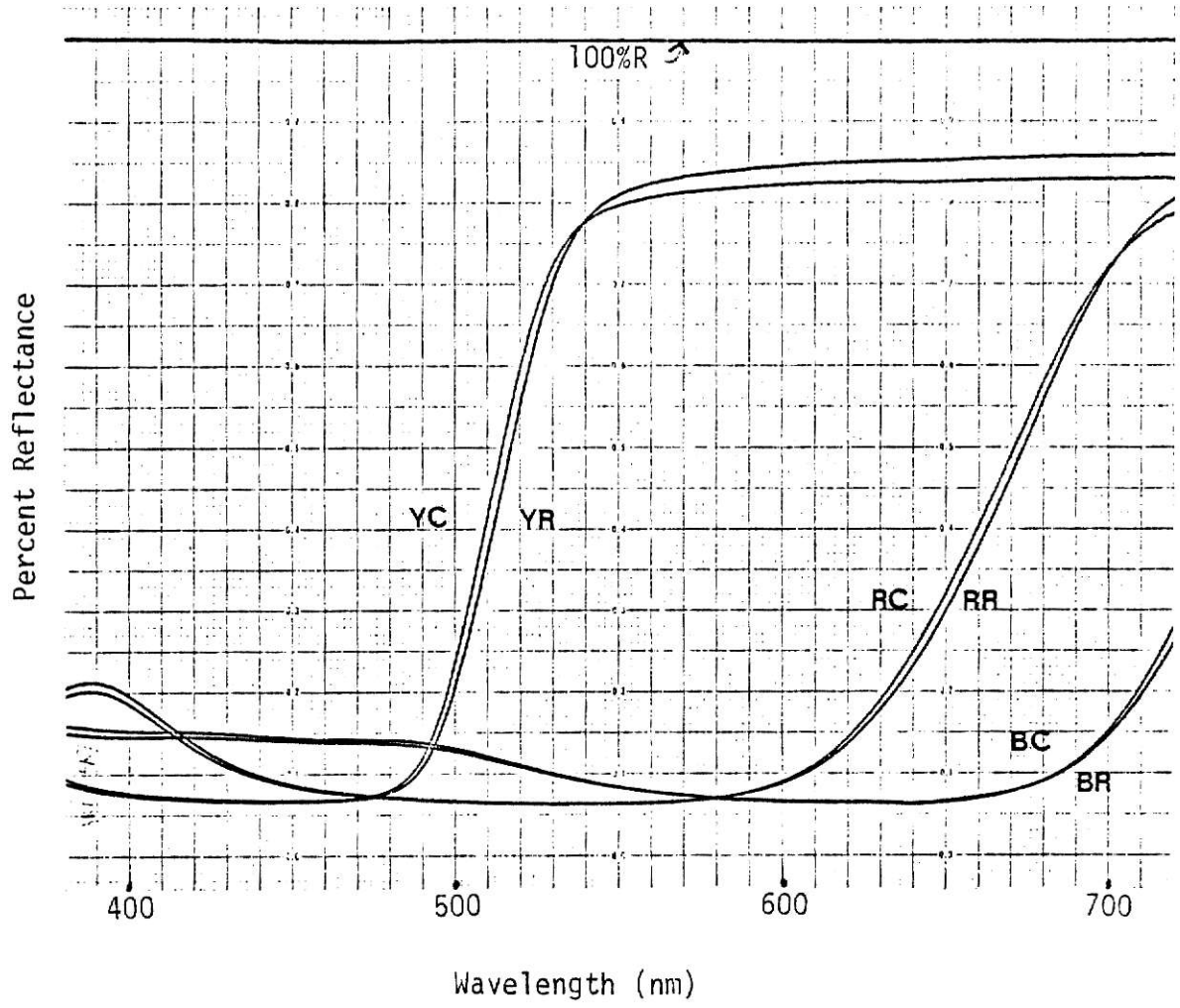

Figure C7: UV/VIS Spectra for Regular Dyeable (R) and Carrierless Dyeable (C) Polyester fabrics with Dyeing Method VII (45% urea, heated water, microwave-fixation) and Three Disperse Dyestuffs.

Disperse Dyestuffs:
B- C. I. Disperse Blue 200 (Dispersol Blue D-4G)
R- C. I. Disperse Violet 33 (Dispersol Rubine C-B)
Y- C. I. Disperse Yellow 126 (Dispersol Yellow D-7G)

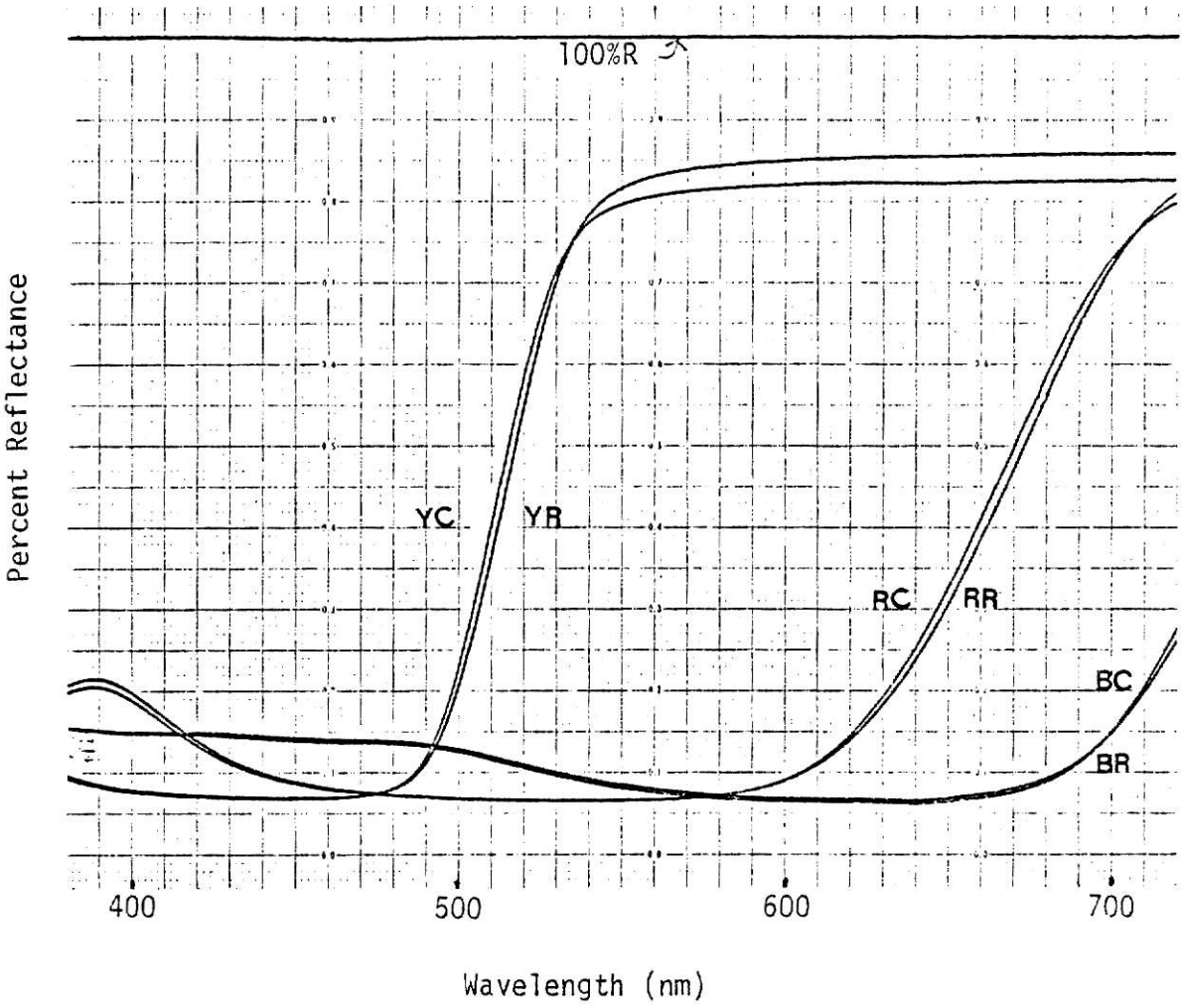

Figure C8: UV/VIS Spectra for Regular Dyeable (R) and Carrierless Dyeable (C) Polyester fabrics with Dyeing Method VIII (45% urea, boiling water, microwave-fixation) and Three Disperse Dyestuffs.

Disperse Dyestuffs:
B- C. I. Disperse Blue 200 (Dispersol Blue D-4G)
R- C. I. Disperse Violet 33 (Dispersol Rubine C-B)
Y- C. I. Disperse Yellow 126 (Dispersol Yellow D-7G)

MICROWAVE DYEING OF REGULAR AND CARRIERLESS DYEABLE POLYESTERS WITH DISPERSE DYES

by

JAW HUA CHIAO-CHENG

B. S., Fu Jen University, 1977

AN ABSTRACT OF A MASTER'S THESIS

submitted in partial fulfillment of the

requirements for the degree

MASTER OF SCIENCE

Department of Clothing, Textiles, and Interior Design

KANSAS STATE UNIVERSITY
Manhattan, Kansas

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ABSTRACT

A substantial portion of the energy required to produce a textile is consumed in the wet processing area. Therefore, considerable research has been directed towards reducing energy consumption in wet processing. Microwave dyeing and finishing techniques are being explored because they can provide a more uniform and more rapid method of heating and drying textiles than conventional heating techniques with considerable energy savings.

In order to develop a suitable microwave dyeing method for regular dyeable and carrierless dyeable polyesters, various dyeing procedures were explored with different solvent systems, water temperatures, and methods of exposure. The dyeing procedures involved (1) two Thermosol dyeing methods with conventional predrying or microwave predrying and (2) six microwave-fixation methods with 100% water, 10% urea, or 45% urea pad-baths in which the padded specimens were exposed over a heated or boiling water bath in order to maintain the sufficient moisture content during microwave irradiation. The results obtained with microwave dyeing methods were compared to that obtained with the conventional thermosol method in terms of depth of shade, evenness of color, dye penetration and changes in the physical properties of the polyester fabrics after microwave irradiation. In this study, density gradient analysis, breaking strength and elongation, and small-angle light scattering tests were used to assess structural changes within the fiber after dyeing.

Microwave predrying in thermosol dyeing eliminated premature dye penetration and reduced the drying time without causing a loss in color yield. K/S values were significantly affected by the type

of polyester, dyeing method, and urea concentration in the pad-bath.

The eight dyeing methods evaluated in this study did not cause significant changes in breaking strength or elongation, whereas the density values and photographs from SALS test indicated that different structural changes were occurring in the fiber with conventional thermofixation, microwave predrying, and microwave-fixation.