

MICROWAVE DYEING OF REGULAR AND CARRIERLESS  
DYEABLE POLYESTERS WITH DISPERSE DYES

by

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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.

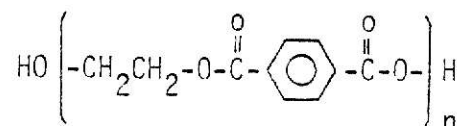
The purpose of this research project was to develop methods suitable for dyeing regular and carrierless dyeable polyester fabrics with selected disperse dyestuffs and to compare the results with conventional dyeing techniques in terms of uniformity, dye penetration or diffusion, color yield, and changes 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 pad-applying 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°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, method 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  $915 \pm 25$  and  $2450 \pm 50$  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 generation 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 categories: 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].

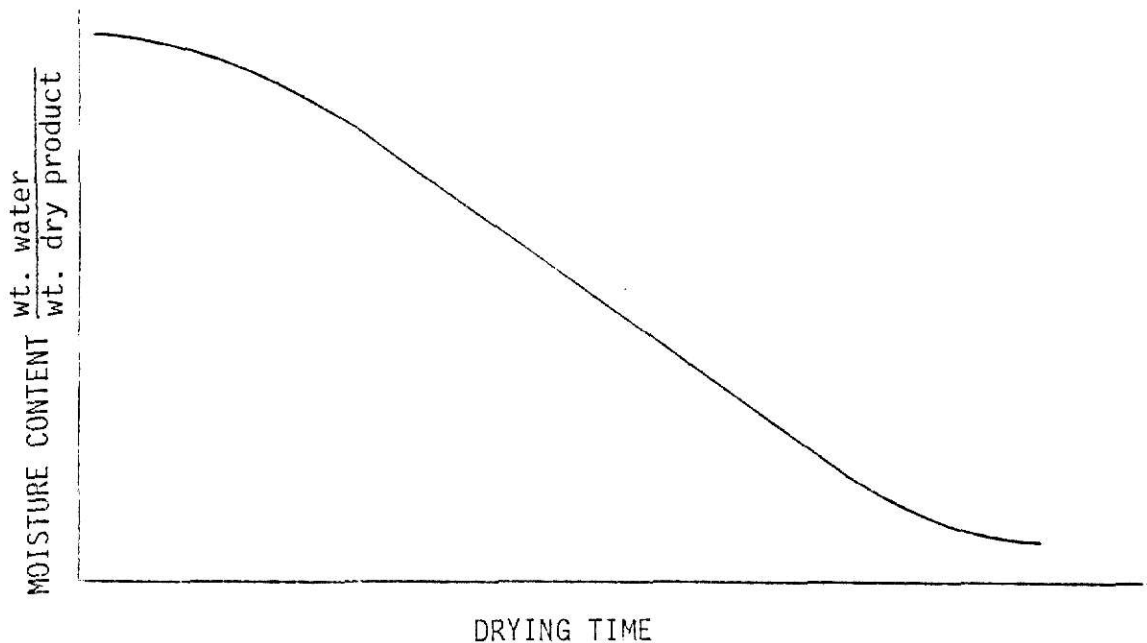


Figure 1: Typical Drying Curve Using Conventional Heating (Taken from D. W. Lyons's Ph.D. Thesis, Atlanta, Georgia, Georgia Inst. Technol., 1966).



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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-

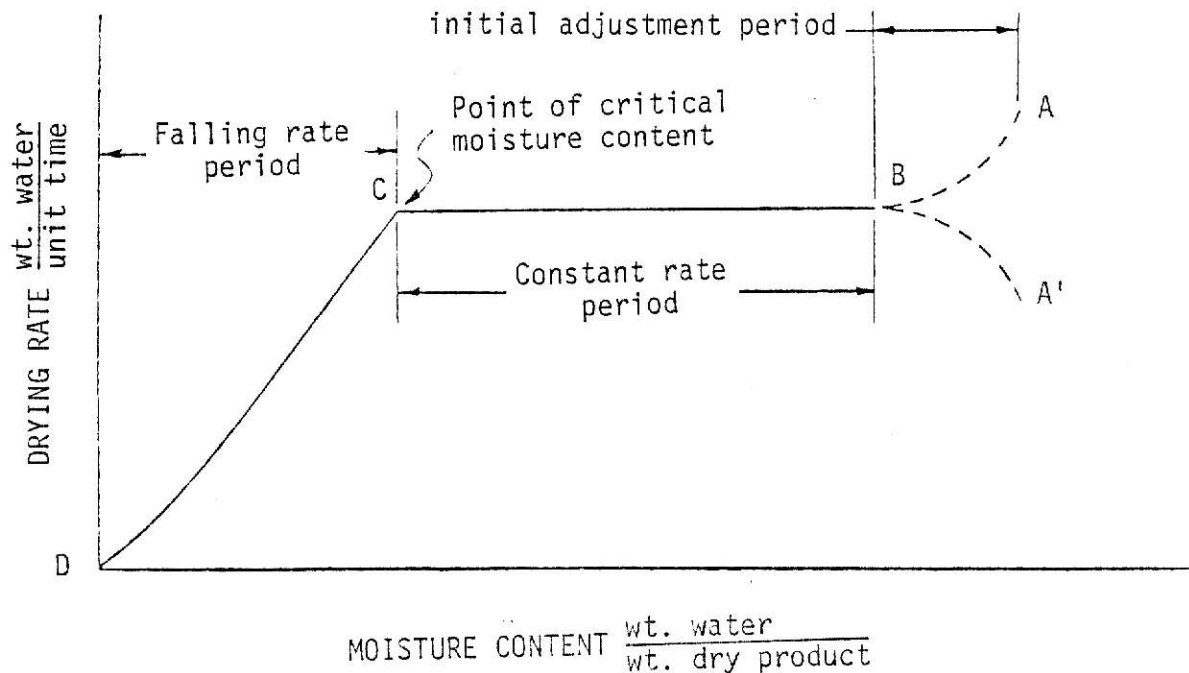


Figure 2: Typical Drying Rate Curve for Drying Using Conventional Heating (Taken from D. W. Lyons's Ph.D. Thesis, Atlanta, Georgia, Georgia Inst. Technol., 1966).

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<sub>2</sub>O, EG, and H<sub>2</sub>O). Few studies have investigated the feasibility of using microwaves to dye polyester fibers with disperse dyes, and only one study attempted 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  $\Delta E$ , (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 structural 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 obtained 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.