Dyeing Polyester With Microwave Heating Using Disperse Dyestuffs

By K. Haggag, H.L. Hanna, B.M. Youssef, N.S. El-Shimy, Textile Research Division, Nat'l Res.Centre, Dokki, Cairo, Egypt

Introduction

An investigation was undertaken to study the effect of microwave heating on aqueous and solvent-pretreatment (perchloroethylene), as well as dyeing polyester fibers with disperse dyes. Microwave irradiation and solvent-pretreatment allow a high increase in dye-uptake and dyeing rate acceleration. Performance of dye levelling and color homogeneity was achieved, which was found to be better than that obtained by conductive heating. In addition, an assessment of the physical changes in polyester structure brought about by solvent and aqueous pretreatment heated by microwave irradiation using x-ray analysis and electronic microscopic investigation was reported.

In textile processing it is necessary to apply heat as in dye fixation, heat setting or drying the product. Heat can be transferred to the material by radiation, conduction and convection. These three ways of transferring can be used either separately or in combination.

The saving of time and energy is of immediate interest to the textile industry. The introduction of new techniques which will allow less energy to be user: is a highly important area of activity tc consider.

Great advances in the fields of radar and communications have been effected. As a result of this a new technology has been born. Microwave has entered the early stages of development. The investigation of the 900 MHz (Mc/second) frequency band and the development of a magnetron valve operating at this frequency and capable of producing 25 kW. of output power have

Figure 1–Effect of dyeing bath temperature on dyeing of polyester samples treated in PCE by microwave heating for different times and dyed with conductive heating.

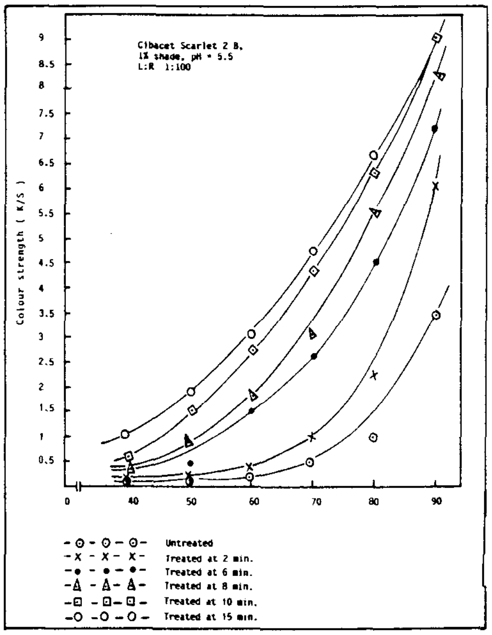
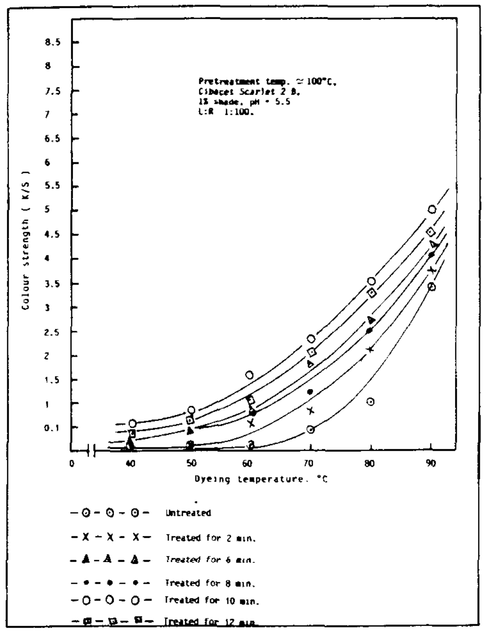


Figure 2– Effect of dyeing bath temperature on dyeing of polyester samples treated in water for different time intervals using microwave heating and dyed with conductive heating.



placed microwaves on the industrial map as well as made available microwave units suitable for modern material processing techniques.[1]

The textile industry has investigated many uses for microwave energy from heating, drying, dye fixation, printing and curing of resin-finished fabrics, to disinvesting wool fabrics.[2-7] In 1966, Ciba-Geigy obtained one of the earliest patents for using microwave heating in dyeing and printing fibrous material with reactive dyes.

Since then many authors have[8-15] investigated the feasibility of using microwaves for a variety of dyeing and finishing processes. However, the potential of using microwave radiation as a heating source in textile dyeing until now has not been investigated widely. Few studies have focused on investigating the feasibility of using microwaves to dye polyester fibers with disperse dyes. Only one study attempted to elucidate the effects of microwave irradiation on the fiber structure.[16]

The present work was undertaken with a view toward studying the rate of dyeing and characteristics of the dyeing process of solvent-pretreated and untreated polyester fabrics by microwave heating with disperse dyes. The study also included the physical changes in polyester structure brought about by microwave irradiation using x-ray analysis and scanning electron microscopic examination.

Experiment

Materials

Substrate

Unfinished polyester fabric 100% (150 g/m², 70/2 denier) was used. The fabric was first treated with a solution containing 5 g/l soap and 1 g/l sodium carbonate at a temperature of 70 °C for about 1 hour. Then it was thoroughly washed with water and air dried at room temperature.

Dyestuffs

Disperse dyestuffs were applied and included:

Cibacet Scarlet 2 B (Ciba-Geigy), C.I. Disperse Red 1, 11110, Terasil Brown 3R (Ciba-Geigy), C.I. Disperse Brown 1, 11152; and Palanil Red GE (BASF), C.I. Disperse Red 65.

Dispersing agents:

Sodium lignine sulphonate (ISMA Dye, Kafr El-Dawar, Egypt).

Carriers:

Matexil CA -MN (I.C.I.)

Organic Solvents:

Tetrachloroethylene (B.P. 121 °C), and water.

Acids and alkalies:

Acetic acid, sodium carbonate and sodium hydroxide. All the chemicals and the solvents used were of laboratory grade.

Treatment:

The beakers containing the solvents and/or water were introduced into a laboratory microwave oven, "Moulinex model FM 3935 QT with an output of 1.5 kW, operating at 2450 MHz. After reaching the boiling point of the solvents, the sampels were treated for different intervals of time (2-15 minutes). After treatment, the samples were picked up from the solvents, sucked into a Buckhner flask, centrifuged and then dried at room temperature for a lengthy period.

Dyeing:

Both untreated and solvent pretreated samples were dyed by the discontinuous dyeing method. For an accurate comparison the untreated and pre-

Photo shows electronic microscope micrographs of (1) untreated polyester fiber, (2) treatment in H_2O at 100 degrees C. in PCE for 15 min.

treated samples were also dyed together in one vessel, i.e. competition dyeing. Dyeing was at pH 5-5.5, liquor ratio l:lOO, l-1.5% shade and for different times 2-15 minutes. After dyeing, they were recovered from the solution washed with cold water, soaped at 70 *D for 15 minutes (5 g/l soap), then washed again with cold water, rinsed and dried at room temperature. After drying they were subjected to different measurements.

Color *measurements*

Reflectance measurements of dyed samples (relative color strength, K/S values) were determined using Perkin Elmer, Lambda 3B, UV/VIS spectrophotometer and by applying the Kubelka-Munk equation.17

On the other hand, the extracted dye by dimethylformamide/water (1\1, v/v) was measured spectrophotometrically using spectrophotometer, Carl Zeiss Jena. the dye concentration in extracted liquor was calculated from standard curves.

Shrinkage test:

Shrinkage due to solvent treatment was estimated by measuring the changes in the dimensions of the piece sample:

$$\% \text{ area shrinkage} = \frac{\text{area before treatment - area after treatment}}{\text{area before treatment}} \times 100$$

Weight loss:

Also weight loss due to solvent treatment was determined as:

$$\% \text{ Weight loss} = \frac{\text{weight before treatment - weight after treatment}}{\text{Weight before treatment}} \times 100$$

X-Ray *analysis:*

X-ray diffraction patterns were run using an X-ray diffractometer model Shimadzu XRD 610, with a software system, DP 61, Cu K, Cu-tube radiation at 30 kV and 20 *mA,* directly on the polyester fabric. All of the patterns were run under the same experimental conditions of instrument setting.

Microscopic examination

A scanning electron microscope JSM T-20 from JEOL-Japan, with a magnification range 35-10,000 and resolution of 200 A*, and accelerating voltage 19 kV, was used. Before examination the filaments were prepared on an appropriate disk and coated with a cold spray.

Tensile strength:

The tensile strength and elongation at break (warp) were determined by the strip method according to ASTM, D-225666 T.18

Fastness *properties:*

An evaluation of the overall fastness of the dyed fabrics was performed by determining washing, rubbing, perspiration and light fastness according to the standard methods.18

Results and Discussion

It's important to note that the study included a comparison between three substrates of polyester: polyester treated in perchloroethylene (PCC), poly ester treated in water, and an untreated one. The study also contained the effect of microwave heating on both pretreatment and the dyeing process itself, made up by conductive and microwave heating.

Pretreatment heating

The solutions containing the different types of polyester fabric were heated for varying lengths of time in a microwave oven. Immersion of sample solutions were first brought to the boil (121 *C). After pretreatment by microwave heating, the samples were dyed conventionally by conductive heating (Cibacet Scarlet 2 B, 1% shade, pH = L:R 1:lOO).

Figures 1 and 2 represent the effect of pretreatment in PCE and water heated by microwave respectively. It is obvious that the curves of polyester samples treated in water and PCE have a similar shape, and the relative color strength (K/S) increases by increasing the dye bath temperature and reaches a maximum at 90°C. Also, as the time of pretreatment increases, the magnitude of dye uptake increases.

As expected, PCE-treatment causes a significant rise in polyester dyeability. Upon exposure to microwave radiation the additional localized energy from oscillation of the PCE molecules inside the polymer may increase the breaking of interaction bonds, causing the internal structure to be opened to a greater dye diffusion.

Polyester dyeing rates:

The study of dyeing rates included four procedures:

1. H2O-pretreatment for different periods (2-15 minutes) in an aqueous solution. Pretreatment and dyeing were heated by microwave energy. Figure 3 (Cibacet Scarlet 2 B, 1% shade, pH = 5.5, L:R 1:lOO at lOO"C).

2. PCE-pretreatment for different times (2-15 min.) then dyed in aqueous solution for different periods of time (2-15 min.), pretreatment and dyeing were heated by microwave radiation, Figure 4 (Cibacet Scarlet 2 B, 1 % shade, pH = 5.5, L:R 1:lOO at 100 *C temperature.

3. H2O-pretreatment by microwave irradiation (2-15 min.), then conventional dyeing by conductive heating from an aqueous solution (15 him. - 3 hours), Figure 5 (Cibacet 2 B, 1% shade, pH = 5.5, L:R 1:lOO and at 90°C).

4. PCE-pretreatment with microwave irradiation (2-15 min. at 121 *C). then conventional dyeing by conductive heating from an aqueous solution (15 min. - 3 hours), Figure 6 (Cibacet 2 B, 1 % shade, pH = *5.5,* L:R 1:lOO at 90°C).

From the analysis of results obtained with procedures 1 and 2 it can be seen that the specimens are subjected to the same conditions of pretreatment and dyeing, except pretreatment medium When using PCE as a pretreatment media, the dye uptake is much higher and dyeing rate is much faster than the corresponding ones with an aqueous pretreatment.

Water and PCE are solvents that are known to heat up readily under microwave radiation. This heating takes place easily, internally and instantly, through out the substrate (e.g.-localized heating). In the case of water it can not exceed the evaporation temperature of 100°C but in the case of PCE, it is 121 *C.

Beside the elevated temperature of pretreatment (PCE), as stated, microwave radiation increases the amount of plasticization and Tg decrement of the polymer, so that the molecules of the dye could more readily penetrate the interior.

The specimens used in procedure 3 and 4 are pretreated under the same conditions of procedures 1 and 2, but dyed by conventional conductive heating.

The rate of dyeing with microwave heating is much faster than the rate of dyeing with conductive heating. In the case of microwave heating equilibrium can be reached within a few minutes, where in the case of conductive heating equilibrium is established after a few hours. Of course, this depends upon conditions of pretreatment (time, temperature and nature of solvent, as Well as temperature of the dyeing bath). in other words, the heating technique is the main factor in this respect.

Additionally, the effect of microwave radiation on the polymer itself suggests that microwave-induced oscillation of the dye molecules did occur, and that such oscillation aided the diffusion of the dyes into the polyester fiber.

The oscillation of water molecules in the dye bath could promote dye diffu-

Figure 3 – The dyeing curves of polyester fiber treated and dyed by microwave heating (medium of treatment = water)

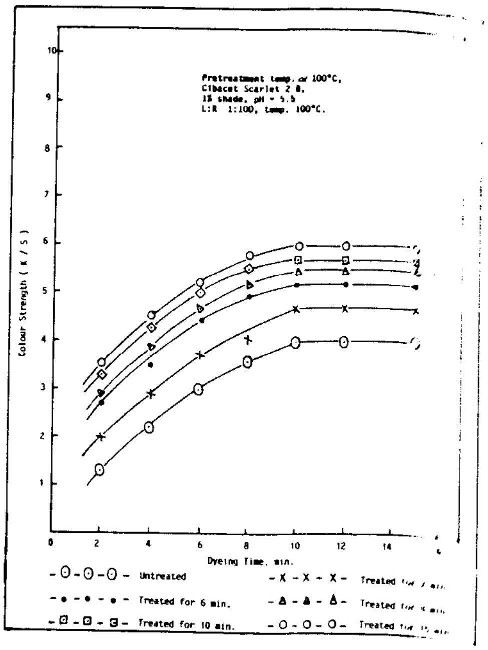
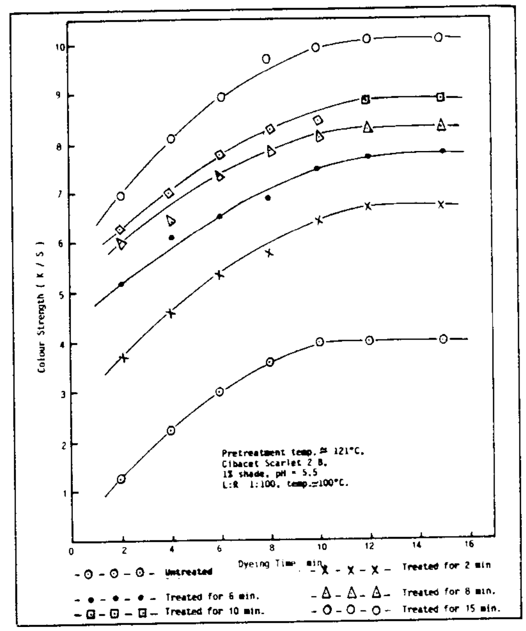


Figure 4– The dyeing curves of polyester fiber treated and dyed by microwave heating (medium of treatment = PCE)



sion. At room temperature, the mole cules in liquid water are believed to be highly ordered in layers or clusters.[IS] Even at the boil clusters of molecule: still exist. This aggregation limits solute migration through water. Microwave radiation-induced oscillation of the water molecules in boiling water ma! result in the break-up of these cluster! to some extent, thus facilitating migra tion of solutes, and, in general, increas ing the movement of any solute through water and, ultimately, increasing the rate of diffusion.

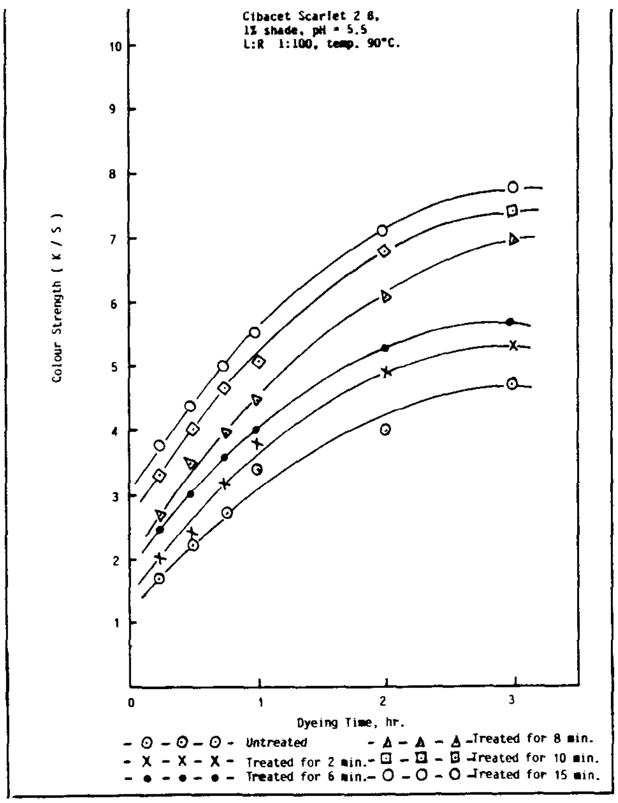
Microwave equilibrium dyeing:

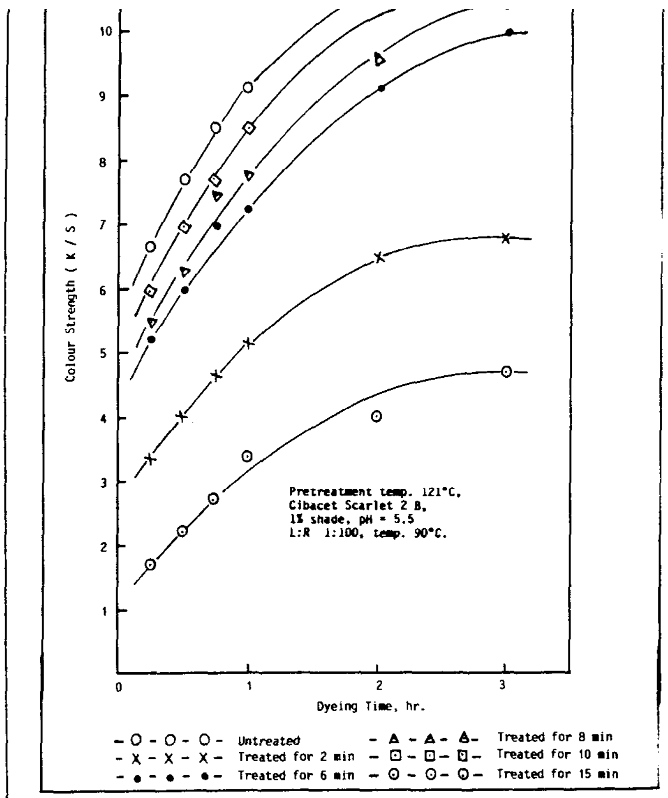
Untreated and treated polyester samples in PCE and water using microwave heating (8-15 min.) were dyed wit disperse Cibacet 2 B until reaching equilibrium. From Figure 7 (1 % shade pH = 5.5, L:R 1:100 and at 97-100°C it can be clear that:

a) The untreated samples can undergo equilibrium faster than the untreated ones, and PCE treatment accelerates dyeing more than water treatment.

b) As the time of treatment i creases, equilibrium is also reach ed faster.

c) The half dyeing time (t 1/2) is at





fected by the specific character of both treatment and dyeing, and in contrast to conductive heating, it occurs within a few seconds; unfortunately, it can not be calculated from the graph.

Microwave dyeing

The action of a carrier addition (Matexil CA-MN, 0.3 ml/100 ml) on polyester microwave dyeing was studied. From Figure 8 (Cibacet Scarlet 2 B, 1 % shade, pH = 5.5, L:R 1:lOO at 97-lOO*C), it is obvious that the addition of carrier significantly enhances the dye absorption for both untreated and PCE-treated, also using microwave heated (15 minutes) polyester samples.

Scanning electronic micrographs:

Untreated and treated polyester fibers in PCE and water at the boil and heated by microwave irradiation were examined under an electronic microscope. From the pictures (scale of illustration is 2000) two facts can be noticed.

-First is that the morphological shape undergoes some changes, which lead to an increase in the active surface (e.g.-increase in dye moles adsorption).

-Second, there's an increase in fiber diameter (e.g.-an increase in diffusion rate inside the fiber).

Treatment with boiled water did not cause any increase in fiber diameter, while with treatment in PCE for 15 minutes an increase of 79% was observed.

X-Ray examination:

X -ray diffraction patterns of polyester fabric before and after water and solvent pretreatment, as shown in Figure 9, revealed that the degree of crystallinity is increased by PCE-treatment. However, when microwave heating was applied the degree of crystallinity is much lighter than conductive heating.

Because the solubility parameter principle of perchloroethylene approaches that of polyester, the perchloroethylene is able to influence crysta linity by increasing the mobility of the polymer chains. Naturally, the degree of crystallinity depends on the time and temperature of treatment, and on the nature of the solvent in general. The interaction between polyester, perchloroetheylene and heat generated internally during microwave heating give rise to an appreciable increase in crystalization.

Figure 7– Rate of dyeing curves for polyester fiber treated and dyed by microwave heating (medium pf treatment = water and PCE)

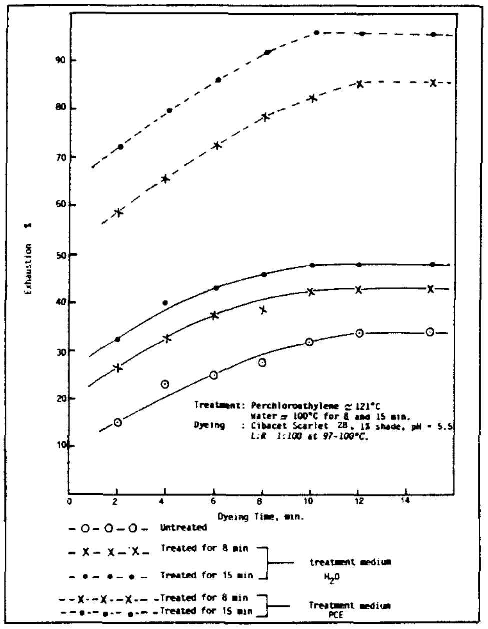
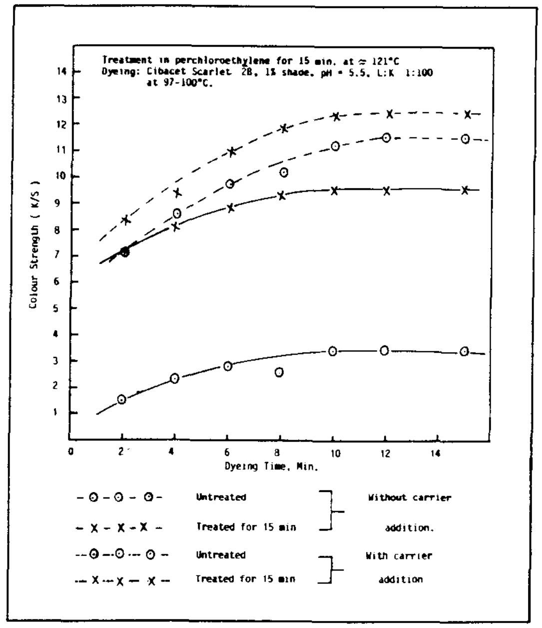


Figure 8– Effect of carrier (Matexil CA-MN) addition in microwave dyeing of polyester fiber.



Now as the degree of crystallinity increases, the dye uptake decreases, but in our study the reverse holds true. This may be due to the following reasons:

1. The interaction between perchloroethylene and polyester causes plasticization, lowering the glass transition temperature Tg about 30-50 °C, a change which would enhance the rate of dye diffusion.
2. Breaking down a certain amount of hydrogen bonds and separating molecular chains will thereby increase spacing, size and pores and voids in polyester structure, which improves dyeability.
3. The shape and morphological structure of the fiber is partially destroyed, which increases the surface of dye adsorption.
4. An increase in fiber diameter facilitates penetration of dyeing solution inside the fiber.

All of these deformations persist even after almost complete removal of the solvent.

Shrinkage:

The tendency of polyester used was to shrink when subjected to treatment in perchloroethylene (121 °C) and water (= 100 °C) for different time (2-15 minute) periods. The area shrinkage percent increases with increasing time but not seriously. In all cases, it does not exceed the normal value; it is around 5.7 after 15 minutes.

Weight loss:

Compared were the accurate weights of polyester fabrics before and after PCE-pretreatment and subjected to microwave heating; it was found that there is no difference at all. The weight loss percent equals zero. This means that perchloroethylene has no solubilizing effect on the used polyester fabric.

Tensile changes:

Changes were studied in tensile strength and elongation at break of polyester substrates subjected to treatment in PCE and water for different temperatures and different time intervals by microwave heating. It was observed that tensile properties and elongation of polyester substrates subjected to boiling in water by microwave heating for different periods (2-15 minutes) are nearly unchanged. Treatment in PCE is accompanied by an increase in tensile strength and elongation of polyester fabrics (after 15 minutes); it was 4.07 % adn 14.5 %, respectively. As the temperature and time of treatment in the solvent increase the tensile properties also increase.

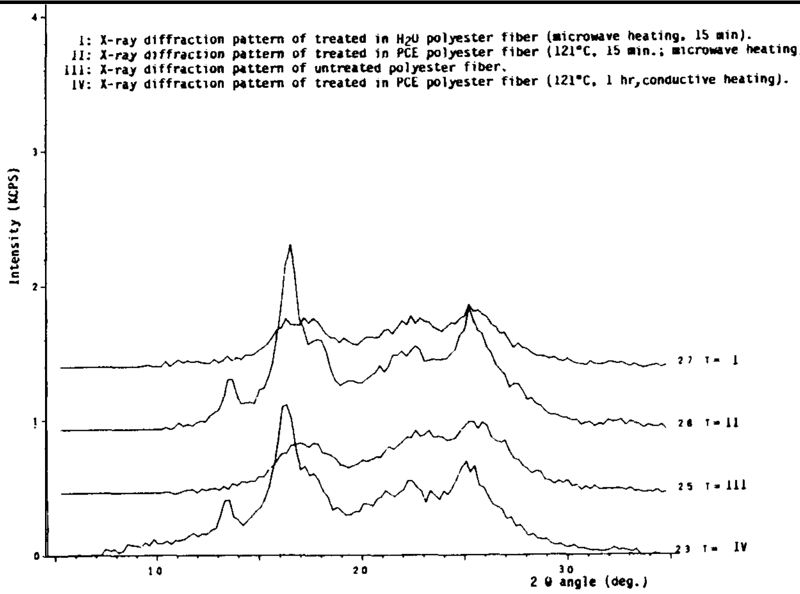
Increases in strength is usually accompanied by an increase in elongation—e.g.-the yield point is not yet reached.

Fastness properties:

The fastness measurements (washing, rubbing, perspiration and light) of polyester fabrics dyed by both conductive and microwave heating in experimental disperse dyestuffs indicate the following:

Polyester fabrics pretreated in PCE (15 min., 121 °C) and dyed at the boil for 15 minutes, pH 5, L:R 1:100, shade 1.5% and without any carrier addition yield satisfactory values. The overall fastness properties of untreated dyed fabrics are lower than the pretreated ones; and, the untreated samples using microwave heating technique acquires higher washing fastness properties than using conductive heating. □ □ □

Figure 9-



References

(1) Belton, J.G.; Dyer, 43, 662 (1970).

(2) Englert, RD., Mar, C.D. and Berriman, I.P.; U.S. Pat, 3, 845, 12 (1974).

(3) Pepperman, A.B., Vail, S.L. and Lyons, D.W.; Textile Chemist and Colourist, 9, 137 (1977).

(4) Metaxas, A.C.. Catlow, N. and Evans, D.G.; Journal of Microwave Power, 13, 4, 341 (1978).

(5) Gourdenne, A.A., Maassarani, H. and Monchaux, P.; Polymer, 22, 2, 471 (1979).

(6) Regan. B.M., Chioo-Cheng, J.H. ar:.; Streict, N.J.; Journal of Food Protection, 43, 658 (1980).

(7) Haggag, K.; Tinctoria. 88, 3, 66 (1991).

(8) Adams, R.G. *et* al., U.S. Pat., 3,484,17g, 16 (1969).

(9) Dawson, T.I.; Textile Month, 47 (1972).

(10) Delaney, M.J.; Textile Chemist and Cot-ourist, 4. 5. 119 (1972).

(11) Evans, D.G. and Skell, J.K.; Journal of Society of Dyers and Colourists. 88, 423 (1972).

(12) Pendergrass, J.E., Hatcher, J.D. and Lyons, D.W.; Journal of Microwave Power, 7, 3. 207 (1972).

(13) Needles, H.I. et al.. Book of Papers National Technical Conference, AATCC, 201 (1977).

(14) Berns. R.S. and Needles, H.I.; Journal of the Society of Dyers and Colourists 95, 207 (1979).

(15) Haggag, K.; Amer. Dyest, Reptr.. 79. -5 (1990).

(16) Holme and Mataxas, A.C.; J. Microwave Power, *14. 4,* (1979).

(17) Judd, D.B. and Wyszinki, G.; "Cola in Business", Science and Industry. 3rd Ed. John Wiley and Sons (1975).

(18) The Society Dyers and Colourists: Standards Methods for Assessment of Colour Fastness of Textiles, Third Report of Fastness Test Coordinating Committee, Yorkshire, England, 24. 27. 63, 71 (1955).

(19) Franks, W.; A. Comprehensive Treatise, 2, New York, Plenum Press (1972).