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Part VIII: RF Dyeing of Polyester**

by

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# CONTINUOUS DYEING USING RADIO FREQUENCY ENERGY PART VIII: RF DYEING OF POLYESTER

by F.A. BARKHUYSEN, N.J.J. VAN RENSBURG and R.J. HARWOOD\*

## ABSTRACT

*The radio frequency (RF) energy dyeing of different types of polyester fibres with various disperse dyes and carriers was investigated. It was found that the polyester could generally not be dyed to deep shades in the absence of carriers, with the exception of the carrierless polyester type. The addition of carrier to the dye liquor significantly improved dye fixation. Carrier constitution had a large effect on the dye fixation, with n-alkyl phthalimide and aromatic carboxylic acid ester based carriers producing the best results. Dye fixation generally increased with increasing carrier concentration and fixation values higher than 90% were obtained even at relatively deep shades.*

*The washfastness and sublimation fastness ratings of the dyed polyester depended on the amount of carrier used and were generally good. The surface trimer content of RF dyed polyester was generally very low and similar to that found on polyester exhaust dyed at 130 °C under pressure.*

## INTRODUCTION

Polyester is one of the most important textile fibres and it constitutes about 50% of the total world production of synthetic fibres<sup>1</sup>. Polyester fibres are rather difficult to dye, mainly due to the low rate of diffusion of the dye molecules into the fibre. The movement of the dye through the fibre is governed by the frequency with which sections of the polymer chains change their position during dyeing. The segmental motion of the chains starts at the glass transition temperature ( $T_g$ ) of the fibre and increases with increasing temperature. This facilitates the diffusion of the dye into the fibres<sup>2</sup>. Due to the stiffness imparted to the polymer chains by the phenyl residues of the terephthalate group, polyester has a relatively high glass transition or second order transition temperature<sup>3</sup>. Furthermore, the spaces which become available for diffusion as a result of the segmental motion, appear to be little larger than the size of the dye molecules themselves, and the dye molecules therefore remain within the range of forces responsible for their binding to the fibre.

In practice several methods are available for the dyeing of polyester<sup>4</sup>. These can be divided into three main groups, namely atmospheric dyeing at the boil in the presence of carriers (or accelerating agents), dyeing under pressure at high temperatures (120°C to 140°C) or baking at temperatures in the range 190°C to 220°C (thermosol dyeing). The most common approach is to dye the

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polyester at elevated temperatures ( $> 120^{\circ}\text{C}$ ), but in the cases where the required pressure or thermosol dyeing machines are not available, dyeing is carried out at atmospheric temperature in the presence of carriers.

The mechanism of polyester dyeing in the presence of carriers has been studied in great detail and some nine different mechanisms have been proposed to describe the function of carriers<sup>5</sup>. There are still a number of unsolved questions in this field, however, and it is presently accepted that several of these mechanisms may operate simultaneously during dyeing. Although possible interactions between the carrier and dye cannot entirely be eliminated, available experimental evidence suggests that the most likely action of the carriers is associated with their effect on the physical properties of the fibre<sup>6</sup>. Recent investigations have emphasized the importance of the plasticizing effect of carriers on the fibre structure, thereby reducing the  $T_g$  of the fibre<sup>7,8</sup>. Furthermore, it was postulated that carriers induced folding of the polymer chains and that the extent of chain folding was related to the ease of dyeing<sup>9</sup>. To date a very large number of products have been evaluated in a search for a suitable dyeing accelerant<sup>5</sup>. In general substances with effective 'carrier action' fall mainly within 4 groups of compounds, namely phenolics, primary amines, hydrocarbons and ethers.

The dyeing of polyester by conventional carrier, pressure of thermosol dyeing techniques is well established. In all these techniques the temperature of the material is increased by indirect means, i.e. the heat is generated externally and transferred to the product by convection, conduction or radiation. It is also possible, however, to generate heat within the product by radio frequency (RF) energy. A number of manufacturers have developed dyeing machines which operate on this principle and wool as well as acrylic fibres are already dyed commercially by RF techniques. Scant information, however, exists about the dyeing of polyester with RF energy. Preliminary studies at SAWTRI indicated that polyester could be successfully dyed to pale shades in a pilot scale Fastran RF dyeing machine<sup>10</sup>. In the case of deep shades, however, the dye fixation dropped to unacceptably low values. This is mainly due to the fact that the maximum temperature attainable in the Fastran machine is only about  $110^{\circ}\text{C}$  which is still some  $10^{\circ}\text{C}$  to  $30^{\circ}\text{C}$  below the temperature required for pressure dyeing. Clearly, the only way in which the dye fixation could be improved would be to use carriers. A study was therefore initiated to investigate the RF dyeing of polyester in the presence of various carriers and to evaluate the effect of parameters such as the carrier constitution and concentration on the fixation of various dyes on different types of polyester, as well as the fastness properties of the dyed fibres.

## EXPERIMENTAL

### Materials:

Various polyester fibre types with linear densities ranging from 1,6 to

16,0 dtex were used in this study.

### Chemicals:

All the dyestuffs, carriers and auxiliaries were of commercial grade quality. Samaron Yellow 4GSL 400 (C I Disperse Yellow 22), Samaron Yellow Brown HRSL 150 (C I Disperse Orange 29) and Samaron Red HGL 150 (C I Disperse Red 65), all azo class dyes, were used. Sixteen carriers, with different chemical constitutions, were evaluated. De-ionised water was used for all the experiments.

### Treatments:

#### (i) RF Dyeing

RF dyeing was carried out on a pilot scale Fastran Continuous Top Dyeing machine described previously<sup>11</sup>. The anode current was set at 400 mA to produce a temperature of 110°C in the dyeing tube. The dwell time of the samples in the RF field was about 5 minutes while the total dwell time in the tube was about 35 minutes. The polyester tops were padded with dye solution containing:

x% dye

y% carrier

1 g/l Lameprint 651 (thickener).

The pH of the dye liquor was adjusted to 5,0 with CH<sub>3</sub>COOH.

The dyestuff concentration was varied from 0,4 to 3,2% while the carrier concentration was varied from 0 to 8% (o.m.f.). To compensate for differences in the wet pick-ups obtained on the various polyester tops, the dyestuff concentration for each particular set of experiments was adjusted in order to apply the same amount of dye to all the different polyester lots.

#### (ii) Exhaust Dyeing

Exhaust dyeings were carried out on an Ahiba Turbomat dyeing apparatus at 130°C for 30 minutes according to the recommendations of the dyestuff manufacturers.

### Tests:

The dyed samples were soaped at 100°C for 20 min with 0,5 g/l ©Ultravon HD. Aliquots of the soap solutions were diluted with ethanol in a ratio of 1 : 3 and the dye fixation determined spectrophotometrically. The depth of shade ( $K/S$ ) of the dyed samples was measured on a Micromatch 2000 spectrophotometer. The fastness to washing and the sublimation fastness of the dyed substrates were determined according to the ISO 3 and BS 1006:PO1:1978

**TABLE 2**  
**EFFECT OF CARRIER TYPE ON DYE UPTAKE**

CARRIER	TYPE	FIXATION (%)*			
		®Samaron Red HGL 150 (Low energy dye)		®Samaron Yellow 4GSL 400 (High energy dye)	
		Trevira 340	Trevira 820	Trevira 340	Trevira 820
—	—	58,1	75,0	52,9	41,0
®Colcar PCS	Chlorinated Benzene	93,0	94,5	87,9	65,9
®Invalon HTC	Trichloro Benzene	85,9	79,8	73,3	52,4
®Remol HT	Diphenyl	84,5	66,2	73,5	57,7
®Invalon TA-ZA	Trichloro Diphenyl	83,5	72,4	74,0	64,4
®Colcar HC New	Hydrocarbon	97,1	92,3	92,3	79,9
®Remol LF	Aromatic Hydrocarbon	92,6	73,1	79,5	62,0
®Remol LE	Aromatic Ester Hydrocarbon	96,2	89,0	86,3	65,5
®Colcar EB	Ester Based	98,5	91,1	88,6	70,3
®Levegal PT	Aromatic Carboxylic Acid Ester	97,5	87,3	95,0	85,2
®Viscavin GR	Aromatic Carboxylic Acid Ester	97,8	87,6	82,0	64,6
®Colcar LEV	Blended Aromatic	81,0	79,4	79,9	66,3
®Levegal DTE	Halogenated Aromate	91,3	78,4	76,0	58,7
®Matexil CA-MN	Methyl Naphthalene	97,0	72,6	94,0	64,9
®Levegal PEW	n-Alkyl Phthalimide	98,9	97,0	95,9	83,5
®Carolit ELFC	O-Phenyl Phenol	96,9	90,4	91,1	60,8
®Invalon HTB	Eccelerant/ Emulsifier	98,2	93,4	73,0	50,3
AVERAGE		93,1	84,0	83,9	65,8

\*0,4% Dye/1% o.m.f. Carrier

more noticeable for the high energy dye, especially in the case of Trevira 820, where the dye fixation obtained for the different carriers ranged from about 50% to 85%. In general, the n-alkyl phthalimide, aromatic carboxylic acid ester and the hydrocarbon type carriers produced the highest dye fixation values while the diphenyl and trichlorobenzene based carriers produced the lowest values. Obviously the carriers behaved in a different manner during RF dyeing than during conventional exhaust dyeing, where it is generally accepted that carrier structure has little effect on its effectiveness. It is known, however, that different carriers exhibit widely different behaviour both as far as rate of uptake of dyes by fibres as well as the dye equilibrium values are concerned. It is possible therefore, that differences in the diffusion coefficients of the carriers could have affected dye fixation values especially in view of the relatively short dyeing times which prevailed during RF dyeing.

### **Effect of carrier and dyestuff concentration on dye fixation**

In some further work the n-alkyl phthalimide carrier, which produced the highest average dye fixation values, was selected to dye the two polyester types with the high energy dye. In this particular case, the concentration of both the dye and carrier was varied. From the results, given in Table 3, it is clear that the percentage dye fixation generally decreased as the dye add-on level increased. Furthermore, an increase in the concentration of carrier resulted in an increase in the dye fixation. This is in agreement with the findings of various research workers on the effect of carrier concentration on dye fixation during conventional exhaust dyeing<sup>17,18</sup>. It is clear that 1% to 2% carrier produced acceptable dye fixation values when the polyester was dyed with 0,4% dye, while 4% carrier was required for 0,8% dye and at least 6% carrier was required when the polyester was dyed to very deep shades. When the polyester was dyed with 3,2% dye, the use of 6% carrier increased the dye fixation by about 60%. It must be pointed out that the concentration of dye needed to produce an ISO 1/1 Standard Depth of shade for this particular dye is 0,4%. It would therefore normally not be used at concentrations much higher than 1%.

### **Depth of shade of RF dyed polyester**

The effect of increasing carrier and dyestuff concentration on the depth of shade ( $K/S$ ) of RF dyed polyester is depicted in Fig. 1.

It is clear that the depth of shade of the polyester increased only slightly with increasing dye concentration in the absence of carrier. When dyeing was carried out in the presence of carrier, however, a significant increase in the depth of shade was found. In the case of the high concentrations of dye (1,6% and 3,2%), the  $K/S$  values increased with increasing carrier concentration, up to the highest value investigated, whereas the  $K/S$  values appeared to reach an equilibrium in the case of 0,8% dye and showed a maximum followed by a progressive decrease in the case of 0,4% dye. As far as the two types of polyester

**TABLE 3**  
**EFFECT OF DYE AND CARRIER CONCENTRATION ON DYE FIXATION**

Dye Concentration (%)	FIXATION (%)*											
	0% Carrier		1% Carrier		2% Carrier		4% Carrier		6% Carrier		8% Carrier	
	Trevira 340	Trevira 820	Trevira 340	Trevira 820	Trevira 340	Trevira 820	Trevira 340	Trevira 820	Trevira 340	Trevira 820	Trevira 340	Trevira 820
0,4	52,9	41,0	95,9	83,5	96,8	88,6	97,2	94,3	98,1	95,9	98,9	95,8
0,8	41,5	26,1	53,7	61,0	78,0	78,3	93,7	93,2	97,7	96,7	98,3	97,5
1,6	28,9	7,6	47,8	47,9	55,2	45,4	63,2	50,5	80,0	86,6	88,8	90,0
3,2	19,4	6,0	28,7	11,1	30,7	17,4	61,6	39,7	70,8	61,3	76,3	65,3

\*@Samaron Yellow 4GSL 400/@Levegal PEW carrier



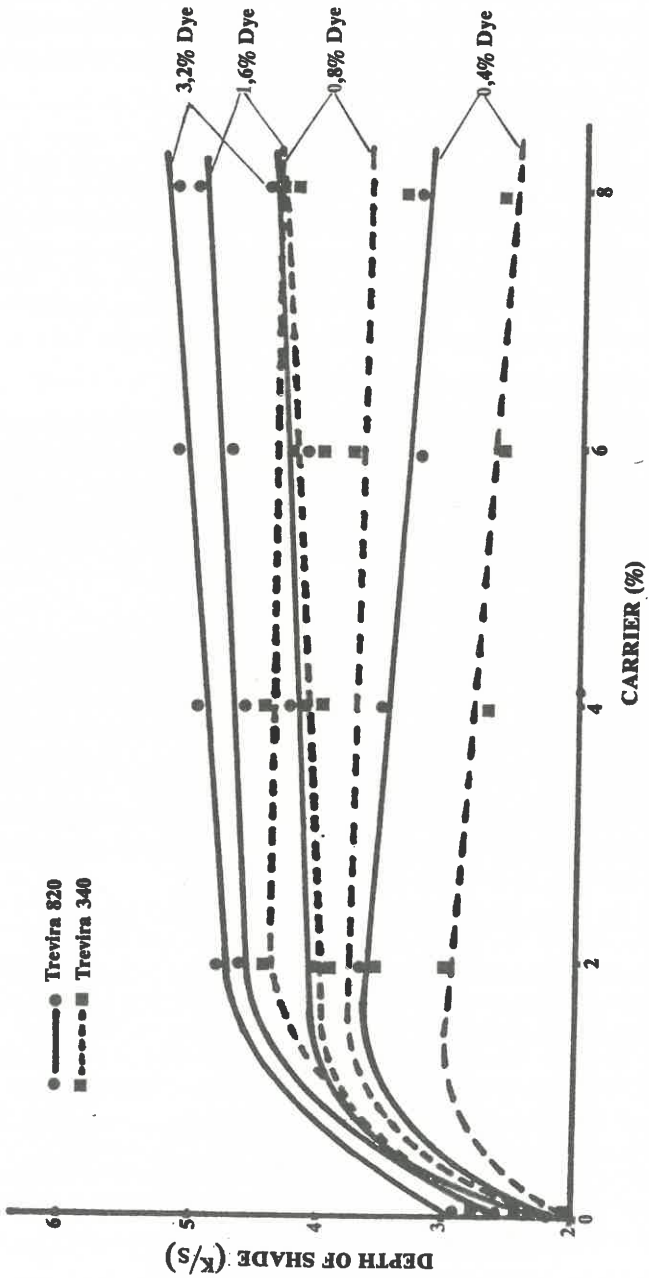


Fig. 1 - Effect of Increasing Dye\* and Carrier\*\* Concentration on Depth of Shade.

\* ©Samaron Yellow 4GSL 400  
 \*\* ©Levegal PEW

were concerned, it is clear that for the same concentration of dye and carrier, a deeper shade was obtained on the coarse polyester (Trevira 820) than on the finer fibres (Trevira 340). This is in agreement with the findings of research workers during the conventional dyeing of polyester<sup>19</sup>.

### **Washfastness of RF dyed polyester**

The washfastness ratings (ISO 3) of the dyed polyester samples are given in Table 4. The fastness ratings of the dyed polyester generally decreased somewhat with an increase in the dye add-on level. In the case of the carrier dyeings the ratings were lowest for the lowest carrier concentration (1%), but improved with increasing carrier concentration. Similar trends were observed for both polyester lots. The finer fibres (Trevira 340) showed marginally lower ratings, especially as far as the staining ratings were concerned.

### **Sublimation fastness of RF dyed polyester**

Table 5 gives the sublimation fastness ratings of the RF dyed polyester. It is clear that, for this specific dye, high sublimation fastness ratings were obtained for all the dye concentrations studied. The "effect on shade" ratings were 5 in all cases but the "staining of polyester" ratings deteriorated somewhat when the dye or carrier concentration was increased.

### **Oligomer content of RF dyed polyester**

It is well-known that the presence of oligomers on the fibre surface can cause problems during the processing of polyester fibres. Dyeing generally leads to the migration of oligomers (monomers, dimers and trimers) to the surface of the fibres and this can aggravate the problem. Furthermore, it is known<sup>12</sup> that when dyeing is carried out in the presence of carrier, especially the surface trimer content of the fibres can be increased significantly. In view of the fact that relatively high concentrations of carrier were required to dye polyester to deep shades in the RF dyeing machine, it was considered important to establish the surface oligomer content of the dyed fibres.

Table 6 gives the surface trimer content of polyester which was RF dyed in the Fastran machine in the presence of increasing concentrations of carrier. For purposes of comparison the surface trimer content of polyester which was dyed by a conventional exhaust dyeing process (130°C/30 min.) is also given. It is clear that dyeing in the absence of carrier had no effect on the surface trimer content of the polyester. When the dyeing was carried out in the presence of carrier, however, there was a slight increase in the surface trimer content of the fibres. The surface trimer content did not seem to depend on the actual amount of carrier used during dyeing. Furthermore, there was little difference between the RF and exhaust dyeing processes. The surface trimer content of the fibres was generally very low and it is unlikely that any of these fibre lots would have caused problems associated with oligomers.

**TABLE 4**

**EFFECT OF DYE AND CARRIER CONCENTRATION ON WASHFASTNESS RATINGS**

% Dye ( <sup>o</sup> Samaron Yellow 4 GSL 400)	% Carrier ( <sup>o</sup> Levegal PEW)	ISO 3 WASHFASTNESS RATING													
		Trevira 820							Trevira 340						
		Change in Shade	Staining						Change in Shade	Staining					
			Acetate	Cotton	Nylon	Polyester	Acrylic	Wool		Acetate	Cotton	Nylon	Polyester	Acrylic	Wool
0,4	0	5	4-5	4-5	5	5	5	5	4	4-5	4-5	5	4-5	5	4-5
	1	5	4	5	5	5	5	4	4	4-5	5	4-5	5	4	
	2	4	4	4-5	5	5	5	5	5	5	5	5	5	5	
	4	5	4-5	5	5	5	5	4	4-5	4-5	5	4-5	5	4-5	
	6	5	4-5	5	5	5	5	5	5	5	5	5	5	5	
	8	5	4-5	4-5	5	5	5	5	5	4-5	5	5	5	5	
0,8	0	4	4-5	5	5	5	5	3	4-5	4-5	5	4-5	5	4-5	
	1	4	2-3	4	5	5	4	4	4	4-5	5	4-5	5	4	
	2	4	3-4	4-5	5	5	4	4	4-5	4-5	5	5	5		
	4	5	4	5	5	5	5	5	4-5	5	5	5	5		
	6	5	4-5	5	5	5	5	5	5	5	5	5	5		
	8	5	4-5	5	5	5	5	5	5	5	5	5	5		
1,6	0	4	4-5	5	5	5	5	4	4-5	4-5	5	5	5	4-5	
	1	3	2-3	4	5	5	4	4	3	3-4	5	5	5	3	
	2	4	2-3	4	5	5	3-4	3	3	3-4	5	4-5	5	3	
	4	4	3-4	4-5	5	5	4	3	2-3	3-4	5	5	5	2-3	
	6	5	3-4	4-5	5	5	4	4	4	4-5	5	5	5	4	
	8	4	3	4	5	5	5	4	4	4-5	5	5	5	4	
3,2	0	4	4	5	5	5	5	4	4	4-5	5	5	5	4-5	
	1	3	2-3	4	5	4-5	5	3	3	2	3-4	5	5	2-3	
	2	4	2-3	4	5	5	5	3	3	2-3	3-4	5	4-5	2-3	
	4	3	3	4	5	5	5	3-4	3	1-2	3	5	4-5	1-2	
	6	4	3	4-5	5	5	5	3-4	4	2-3	3-4	5	5	3	
	8	4	3	4	5	5	5	4	4	3	4	5	5	3-4	

**TABLE 5**  
**EFFECT OF DYE AND CARRIER CONCENTRATION ON SUBLIMATION FASTNESS RATINGS**

% Dye <sup>a</sup>	SUBLIMATION FASTNESS RATING (100 °C / 30 sec.)																							
	0% Carrier				1% Carrier				2% Carrier				4% Carrier				6% Carrier				8% Carrier			
	Trevira 340	Trevira 820	Trevira 340	Trevira 820	Trevira 340	Trevira 820	Trevira 340	Trevira 820	Trevira 340	Trevira 820	Trevira 340	Trevira 820	Trevira 340	Trevira 820	Trevira 340	Trevira 820	Trevira 340	Trevira 820	Trevira 340	Trevira 820	Trevira 340	Trevira 820	Trevira 340	Trevira 820
0.4	5	4-5	5	4-5	5	4-5	5	4-5	5	4-5	5	4-5	5	4-5	5	4-5	5	4-5	5	4-5	5	4-5	5	4-5
0.8	5	4-5	5	4-5	5	4-5	5	3-4	5	4-5	5	4	5	4-5	5	4-5	5	4-5	5	4-5	5	4-5	5	4-5
1.6	5	4-5	5	4-5	5	4	5	4	5	3	5	4	5	4-5	5	4	5	4-5	5	4	5	4-5	5	4
3.2	5	4	5	4-5	5	3-4	5	3-4	5	2-3	5	3-4	5	3-4	5	2-3	5	3-4	5	2-3	5	3-4	5	2-3

<sup>a</sup>Samaron Yellow 4GSL 400 (High energy dye).

**TABLE 6**  
**SURFACE TRIMER CONTENT OF POLYESTER DYED\* WITH**  
**INCREASING CONCENTRATIONS OF CARRIER\*\***

(%) Carrier	SURFACE TRIMER (%)			
	RF		Exhaust (130°C/30 min.)	
	Trevira 340	Trevira 820	Trevira 340	Trevira 820
0	0,01	0,01	0,01	<0,01
1	0,04	0,01	0,01	0,03
2	0,03	0,03	0,03	0,03
4	0,04	0,01	0,03	0,01
6	0,04	0,01	0,04	0,04
8	0,04	0,01	0,01	0,01
Untreated Control	Trevira 340 0,1		Trevira 820 0,1	

\* 1,6% ®Samaron Yellow 4GSL 400

\*\* Levegal PEW

### SUMMARY AND CONCLUSIONS

The RF dyeing of different polyester types was studied, especially as far as the effect of carrier, dyestuff and polyester type on dye fixation, fastness properties and surface trimer content of the fibres were concerned.

It was found that the different polyester lots showed large differences in dye fixation during RF dyeing. The dye fixation values depended on the specific dyestuff used. Relatively low dye fixation values were obtained when polyester was dyed in the absence of carrier but it was found that carriers increased the fixation values significantly. Some 16 different carriers were evaluated and it was found that they varied widely in their effect on the dye fixation. In general the alkyl-phthalimide, carboxylic acid ester and hydrocarbon based carriers produced the highest dye fixation values. The effect of carrier was more pronounced in the case of the high energy dye where the dye fixation was increased from about 40% to about 85%. In some further studies, where the concentration of both dye and carrier were varied, it was found that an increase in carrier concentration generally resulted in an increase in dye fixation. For

relatively high dye concentrations this was associated with an increase in the depth of shade of the fibres, but for lower concentrations of dye the depth of shade in fact decreased above certain concentrations of carrier.

The washfastness ratings of the dyed polyester were good and it was found that an increase in carrier concentration improved the washfastness slightly. The sublimation fastness ratings of the RF dyed polyester were high.

The surface trimer content of RF dyed polyester was very low and on average, similar to that of fibres dyed by the high temperature exhaust dyeing method.

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### USE OF PROPRIETARY NAMES

The names of proprietary products where they appear in this report are mentioned for information only. This does not imply that SAWTRI recommends them to the exclusion of other similar products.

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