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**Continuous Dyeing Using Radio
Frequency Energy**

**Part V: Laboratory Studies Involving Energy
Frequency and Dye Liquor Conductivity**

by
E. Garner and F.A. Barkhuysen

**SOUTH AFRICAN
WOOL AND TEXTILE RESEARCH
INSTITUTE OF THE CSIR**

**P.O. BOX 1124
PORT ELIZABETH
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CONTINUOUS DYEING USING RADIO FREQUENCY ENERGY PART V: LABORATORY STUDIES INVOLVING ENERGY FREQUENCY AND DYE LIQUOR CONDUCTIVITY

by E. GARNER and F.A. BARKHUYSEN

ABSTRACT

The dyeing of cotton fabric with high frequency (HF) energy was investigated and compared with conventional dyeing processes. It was found that cotton fabric was dyed successfully with reactive dyes using HF energy of frequencies 27,12 and 2450 MHz. When compared with pad-steam and pad-thermofix (dry heat) dyeing processes, dye fixations by both HF energy processes were excellent. For most of the thirty dyes examined, the highest dye fixations were obtained by one of the HF energy processes. Rubbing fastnesses of all the dyeings were good. Dye liquor electrical conductivity related well with ratios of HF energy and conventional process dye fixations.

INTRODUCTION

As a means of conserving energy, high frequency (HF) energy heating has become widely used by the textile industry in the U.K., Europe and the U.S.A.¹. HF energy has been used in the following applications in the textile industry: textile drying¹⁻³, heat treatment of plush fabric⁴, pleating garments⁵, welding of textile belts⁶, backing of carpets⁷, bonding of fabrics⁸, cleaning and preparation⁹, bleaching of flax¹⁰ and transfer printing¹¹. The popularity of HF energy processing is largely due to the significant energy savings associated with the system. Further advantages are better product quality, greater production speed, less dye migration and reduced floor space. Based on these advantages, predictions have been made that HF energy usage will grow more rapidly in the future^{1,2}.

Another important sector in which HF energy has great potential is in textile dyeing. In a recent review of developments in textile dyeing, a number of HF energy dyeing systems were described¹². These included the Lanapad (IWS) semi-continuous system for wool tops, the Fastran EDF (Dawson International) fully-continuous system for natural and synthetic fibres and the Apollotex Electron Reactor (Ichikin) open width machine.

An area which has received little attention to date is the HF energy dyeing of cotton with reactive dyes. In a previous study¹³, it was shown that cotton sliver could be dyed successfully with reactive and direct dyes using radio frequency energy. Very good dye fixations and wash fastness ratings were obtained. It was decided to extend this work and to study the effect of HF energy at two frequencies (27,12 and 2450 MHz) on dye fixation and fastness of cotton. The

results were compared with those obtained by conventional pad-steam and pad-thermofix process routes.

Reactive dyes were chosen because they are the fastest growing class of dyes for cotton¹⁴. From the many different types of reactive dyes which possess different functional groups with varying degrees of reactivity, vinyl sulphone dyes were chosen because they have a relatively moderate reactivity¹⁵.

One aspect of HF energy dyeing which has not previously been examined is the possible relationship between HF energy dyeing performance and some properties of the dye liquor such as electrical capacitance, dielectric constant and electrical conductivity. Such a study has now been carried out. In the present report only the study on electrical conductivity is described. The work on the other two properties will be reported elsewhere.

EXPERIMENTAL

Cotton Fabric

The cotton fabric used in this study was a scoured medium-weight (177 g/m²) plain weave fabric with a thickness of 0,44 mm . Fabric squares with a mass of 5,0 g were used in the dyeing treatments.

Dyes and Auxiliaries

These were all of commercial grade quality. Thirty vinyl sulphone dyes were chosen for study.

Treatments

Fabric samples were padded to an 80% wet pick-up in a Benz Laboratory Padder with a pad liquor containing:

X g/l Dye
50 g/l Urea
12 g/l Soda Ash
5 g/l Alcopol 650 (wetting agent)

Deionised water with an electrical conductivity of less than 2 μScm^{-1} was used. After padding, the fabric samples were placed in polyethylene plastic bags and then treated with HF energy at 27,12 MHz and 2450 MHz. For purposes of comparison, samples were also treated by pad-steam and pad-thermofixation processes.

HF Treatment at 27,12 MHz

Treatment at 27,12 MHz was carried out using a Fastran Electronic Reaction Chamber manufactured by Dawson International. A photograph of the machine is shown in Fig. 1 and a schematic diagram is given in Fig. 2.

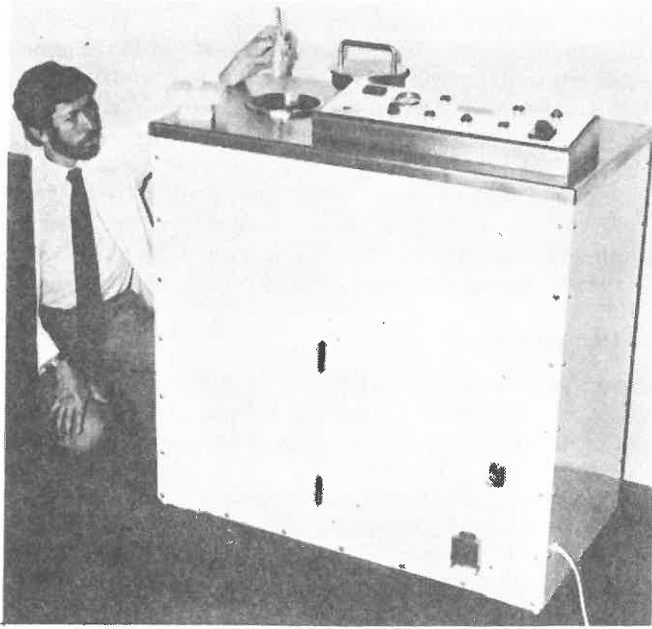


Fig. 1 - Fastran Electronic Reaction Chamber

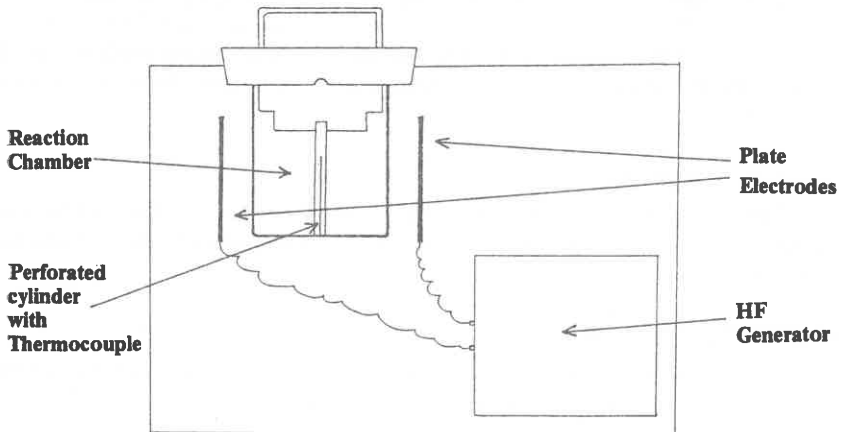


Fig. 2 - Fastran Electronic Reaction Chamber - Schematic Diagram (Not to scale).

The sample was wrapped around the hollow, perforated stainless steel cylinder which was then placed in the reaction chamber. A thermocouple probe inside the tube allowed dyeing temperature to be monitored continuously. Cotton sliver, having a moisture content of 100% was packed around the sample to allow energy absorption and steam generation. This meant that, during HF energy dyeing at 27,12 MHz, some dye fixation took place via steaming. The chamber was sealed during dyeing. Energy was supplied by a 1,5 kW generator operating at a frequency of 27,12 MHz. In the work reported, a generator output current of about 170 mA was used to maintain a dyeing temperature of 100° C within the chamber. Treatment times of 1 to 8 min. were used.

HF Treatment at 2450 MHz

Treatment at 2450 MHz was carried out using a domestic microwave oven. The power output of this machine was 0,5 kW and the generator operating frequency was 2450 MHz. The samples were placed on the base of the cooking area.

It was found preferable to use the "defrost" facility of the oven for dyeing experiments. Under these circumstances, the generator intermittently supplied power for 13 s of each 34 s cycle. Thus, during a 4 min. treatment, for example, there were 6 cycles involving a total power "on" time of 78 s. After the 6th cycle, the HF radiation was switched off and the sample left in the machine for the remainder of the 4 min. The dyeing temperature was monitored using thermal test papers placed in the plastic bags. Dyeings were carried out at 100° C.

It should be noted that, unlike the dyeing treatment involving the Fastran machine, no wet sliver was packed around the samples during treatments at 2450 MHz and consequently these treatments did not involve any introduction of steam from an external source during dyeing. The only steam present was that formed from the aqueous dye liquor padded on to the fabric. Treatment times of 1 to 8 min. were used.

Pad-steam Treatment

The pad-steam treatments were carried out in a Benz Laboratory Steamer. The steaming temperature was 100° C. Treatment times of 1 to 8 min. were used.

Thermofixation Treatment

The pad-thermofix treatment was carried out in a laboratory fan-circulation oven at 100° C. Treatment times of 1 to 8 min. were used.

Determination of Dye Fixation

Prior to padding, an aliquot of each pad liquor was used to prepare a reference standard. In this way, possible effects of the dyeing auxiliaries on the dye were taken into account. After dyeing, the dyed fabric samples were removed from the plastic bags and immediately placed into wash liquors containing 1 g/l ·[®]Alcopol 650.

In order to remove the unfixed dye from the samples, the wash liquors containing the dyed fabric were heated in sealed containers at 100° C for 20 min. After cooling, a known volume of wash liquor was diluted and its optical density compared with that of the reference using a Varian DMS 100 UV Visible Spectrophotometer. The optical densities were determined at the wavelengths of maximum absorbance and dye fixations were calculated in the usual manner. Values reported are the mean of at least two determinations.

Tests on Dyed Fabric

The Kubelka-Munk (K/S) values of some of the dyed samples were measured on an ICS Micromatch 2000 Spectrophotometer. The wet and dry rubbing fastnesses were determined on an AATCC Crockmeter using the SDC Standard Method.

Measurement of Electrical Conductivity

The Electrical conductivity of each of the thirty dye pad liquors was measured using a Metrohm E518 Conductometer. The conductivity cell was a dip-type with a cell constant of 0,815 cm⁻¹. All measurements were made at 25° C.

Measurement of Electrical Capacitance

Electrical capacitance of dye liquors was measured using a DIM DCM302 Capacitor Meter. The electrodes comprised copper plates coated with a plastic film. Measurements were made at 25° C.

Measurement of Dielectric Constant

Dielectric constants were derived by the method described by Le Fèvre¹⁶.

RESULTS AND DISCUSSION

Effect of Fixation Time

The dye manufacturer's recommendations were that dyeing times of up to 8 min. should be used for the pad-steam and thermofixation of these dyes at 100° C. In order to determine a suitable fixation time for the present study which

would show up any differences between the different dyeing treatments, cotton fabric samples were padded with three different dye liquors and treated for 1, 4 and 8 min. The dye fixation values obtained by the four treatments are shown plotted against dyeing time in Figure 3.

From the results obtained it may be seen that dye fixation generally increased with time and levelled off at about 8 min. The fixation values after 4 min. were in most cases relatively high (80% or more of the dye fixation which was obtained after 8 min.). The microwave treatment was found to be very severe and sometimes resulted in scorched samples, especially at longer treatment times. It was decided, therefore, to use a treatment time of 4 min. in all subsequent experiments.

Effect of Dye Concentration

The effect of dye concentration on dye fixation was also investigated. A further three dyes were selected and used to dye the cotton fabric at concentrations of 5, 10, 20 and 30 g/l. The results are shown graphically in Figure 4.

In general, dye fixations decreased with increasing dyeing concentration. This is consistent with conventional dyeing theory. The only exception appeared to be Reactive Orange 7, where dye fixation remained constant or increased slightly with increasing dye concentration. A dye concentration of 10 g/l was used in all subsequent experiments. At this concentration reasonable depths of shade were obtained.

Comparison of HF Energy and Conventional Dyeing Treatments

Each of the thirty reactive dyes was used to dye cotton fabric samples by the four dyeing treatments described above. In each case a dye concentration of 10 g/l and a dyeing time of 4 min. were used. Dye fixations were measured and are shown in Table 1.

From the results shown in Table 1 it is apparent that a very wide variation in dye fixation was obtained. Careful examination of the results revealed a number of trends. For example, thermofixation always resulted in the lowest dye fixation. HF energy dye fixation generally resulted in higher dye fixation than the pad-steam treatment. In order to make a firm comparison between the four treatments, the dye fixation values for each of the thirty dyes were placed in descending order of magnitude and then grouped into various classes. It was found that for 80% of the dyes, the best dye fixation was obtained by one of the two HF energy treatments.

Thus, despite the diversity of results shown in Table 1, it may be concluded that, under these conditions, the HF dyeing treatments resulted in better dye fixations than the conventional pad-steam and pad-thermofix treatments, in the majority of the cases.

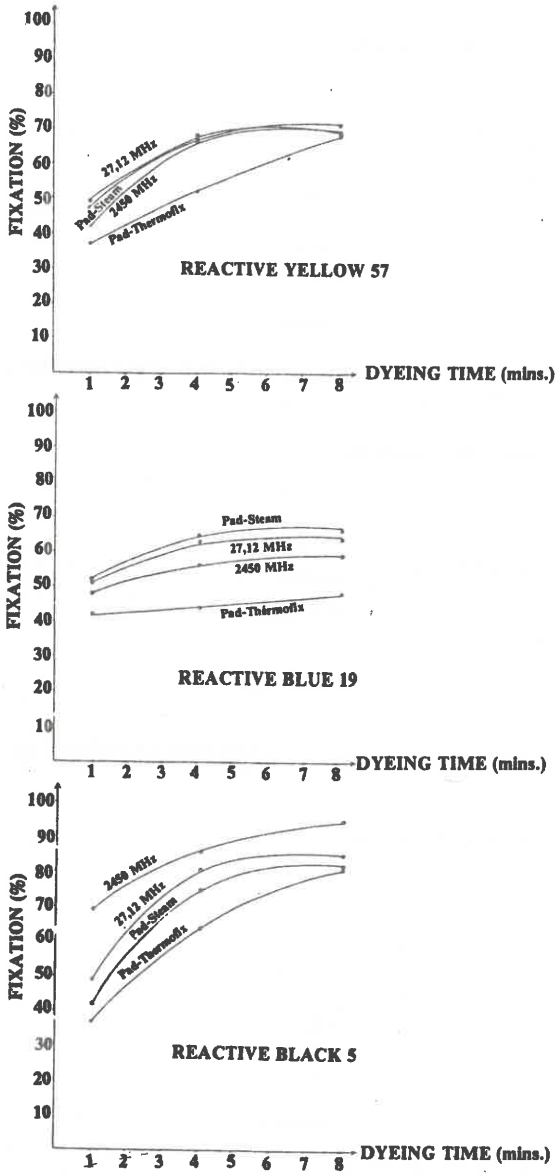


Fig. 3 - Effects of Dyeing Time on Fixation

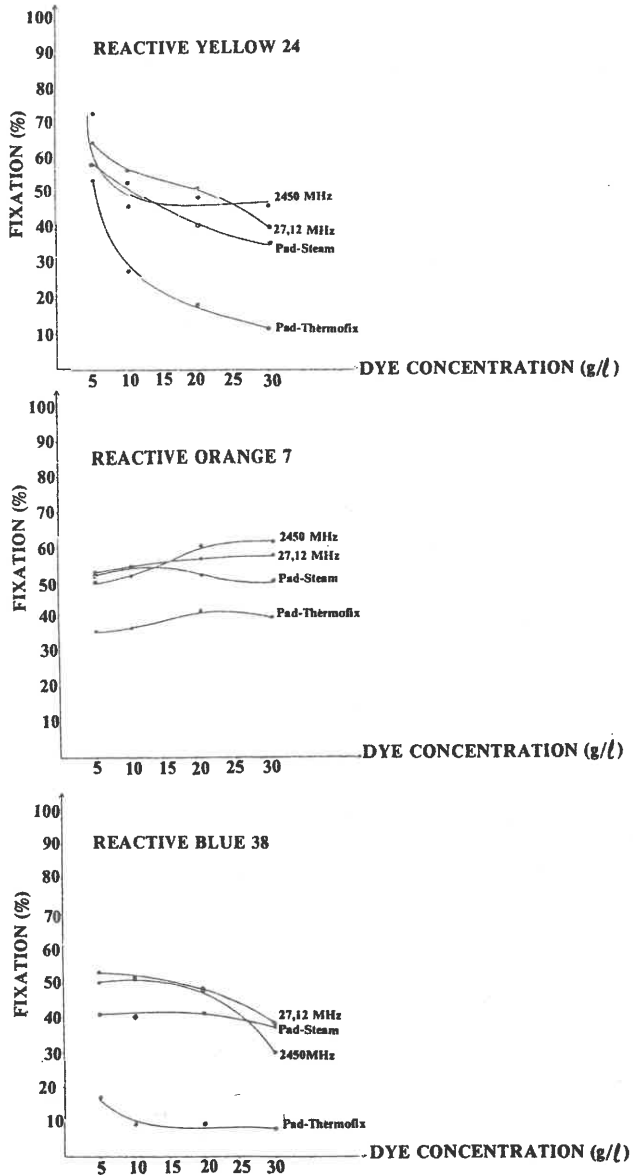


Fig. 4 - Effects of Dye Concentration on Fixation

TABLE 1
DYE FIXATIONS OBTAINED

DYE C.I. REACTIVE NUMBER	Pad Liquor Electrical Conduc- tivity (mScm ⁻¹)	DYE FIXATION (%)			
		HF (27,12 MHz)	HF (2450 MHz)	Pad-Steam	Pad- Thermofix
Yellow 57	17,2	68	66	67	52
Yellow 37	17,75	35	36	38	24
Yellow 42	18,1	38	26	34	13
Yellow 17	16,15	54	64	56	46
Yellow 15	17,8	57	55	58	40
Yellow 23	16,3	54	51	54	33
Yellow 24	17,45	57	46	53	28
Orange 15	18,1	42	45	39	27
Orange 82(S)	17,8	58	50	57	37
Orange 16	16,5	64	66	60	46
Orange 7	16,0	54	52	54	37
Orange 74(S)	17,6	50	44	46	33
Red 35	18,3	38	38	33	19
Red 21	16,8	43	42	37	29
Red 63	16,75	54	33	32	20
Red 106	17,2	49	53	49	31
Red 22	17,8	55	53	53	44
Red 23	18,6	56	85	50	33
Violet 5	17,1	43	43	41	29
Red 49	17,4	67	54	68	42
Blue 27	17,9	43	36	41	24
Blue 19	16,15	63	56	65	44
Blue 28	18,8	56	93	48	28
Blue 77	16,2	46	42	45	35
Blue 21	17,0	59	57	56	45
Blue 38	19,1	50	51	40	9
Brown 16	16,7	49	51	32	32
Brown 18	18,5	53	59	47	26
Black 5	17,5	81	86	75	64
Black 31	18,0	57	50	53	31

Comparing the two HF energy treatments themselves, the dye fixations were often similar. For about half the total number of dyes examined, the difference was 3% or less. However, there was some evidence to suggest that the dye fixations produced by 27,12 MHz frequency energy generally exceeded those by the 2450 MHz frequency energy. This was probably due to a larger steam content in the former cases, as referred to in "Experimental".

Colour Strength (K/S) of Dyed Fabrics

In order to study the effects of the four dyeing processes on the K/S values of the dyed fabrics, three dyes — a yellow, a red and a blue — were selected. These were also dyes which gave medium, low and high electrical conductivity dye liquors respectively. The properties which were measured for the three dye liquors are shown in Table 2. The K/S values obtained were as shown in Table 3.

In most cases, the K/S values were consistent with the dye fixations shown in Table 1. For each of the three dyes, the pad-thermofix process always produced both the lowest K/S value and the lowest dye fixation value. The K/S values did not appear to be closely related to the electrical properties of the dye liquors. For these three dyes, however, there was some degree of correlation between electrical conductivity and thermofix K/S values, between electrical capacitance and pad-steam K/S values and between dielectric constant and K/S values after HF treatment at 27,12 MHz.

TABLE 2
PROPERTIES OF DYE LIQUORS AT 25°C

Dye C.I. Reactive Number	Electrical Conductivity (mScm ⁻¹)	Electrical Capacitance (nF)	Dielectric Constant
Yellow 24	17,45	1,50	238
Red 21	16,80	1,06	173
Blue 38	19,10	1,08	177

TABLE 3
K/S VALUES OF DYED FABRICS

Dye C.I. Reactive Number	K/S VALUES AFTER DYEING			
	Pad-Thermofix	Pad-Steam	HF (2450 MHz)	HF (27,12 MHz)
Yellow 24	1,5	1,9	2,0	2,7
Red 21	1,9	2,1	2,6	2,2
Blue 38	1,3	2,1	4,1	2,3

Rubbing Fastness of Dyed Fabrics

The wet and dry rubbing fastnesses of the same three dyes were also assessed. The results were as shown in Table 4.

TABLE 4
RUBBING FASTNESS OF DYED FABRICS

Dye C.I. Reactive Number	RUBBING FASTNESS AFTER DYEING							
	Pad-Thermofix		Pad-Steam		HF (2450 MHz)		HF (27,12 MHz)	
	Dry	Wet	Dry	Wet	Dry	Wet	Dry	Wet
Yellow 24	5	4-5	5	4-5	5	4-5	5	4-5
Red 21	5	4-5	5	4-5	5	4-5	5	4-5
Blue 38	5	4	5	4-5	5	4-5	5	4-5

It is clear that excellent rubbing fastness ratings were obtained with no differences between the different fixation methods.

Effects of Pad Liquor Electrical Conductivity

The electrical conductivity of the dye pad liquors might be expected to influence the HF energy dyeing processes for two reasons. Firstly, it is a property which is dependent on ion mobility¹⁷. Secondly, ion mobility itself can affect the uptake of HF energy¹⁸. Water soluble dyes, such as reactive dyes, are themselves ions possessing solubilising groups such as sulphonic acid on the dye molecule. They might also, therefore, be expected to respond according to their respective mobilities in a HF energy field. Electrical conductivity provides a measure of such mobility.

HF energy dyeing has already been shown to be related to a "partial ionic" property of the dyestuff (dipole moment) in the case of disperse dyes on polyester¹⁹. It was therefore attempted to correlate the electrical conductivity of the thirty dye pad liquors and the dye fixations obtained during the two HF energy processes. No such correlation was found. The results showed that dye fixation by none of the four dyeing treatments correlated well with the electrical conductivity of the dye pad liquors, when plotted directly.

Better correlations were found when *ratios* of the dye fixations were considered. Figure 5 shows the dye fixation after HF energy treatment at 2450 MHz, divided by the dye fixation after thermofixation and plotted against

electrical conductivity. Similarly, Figure 6 shows dye fixation after HF energy treatment at 27,12 MHz divided by dye fixation after steaming and plotted against electrical conductivity. The high ratios for C.I. Reactive Brown 16 and Red 63 in Figure 6 have yet to be explained. The four treatments were paired off in this way, because steaming effects were more predominant in the HF (27,12 MHz) and steaming processes than in the other two treatments. (Dividing the dye fixation after HF energy treatment at 2450 MHz by steam fixation, gave a correlation inferior to that shown in Figure 5.)

Figures 5 and 6 suggest that there is a relationship between HF energy dye fixation and pad liquor electrical conductivity, but only after applying a correction for the other processes which take place simultaneously. Thus, during treatment with HF energy of frequency 2450 MHz, fixation probably takes place as a result of two processes, namely HF energy and thermofixation. Similarly, during treatment with HF energy of frequency 27,12 MHz, HF energy fixation and steam fixation occur concurrently. Dividing the HF energy fixations by

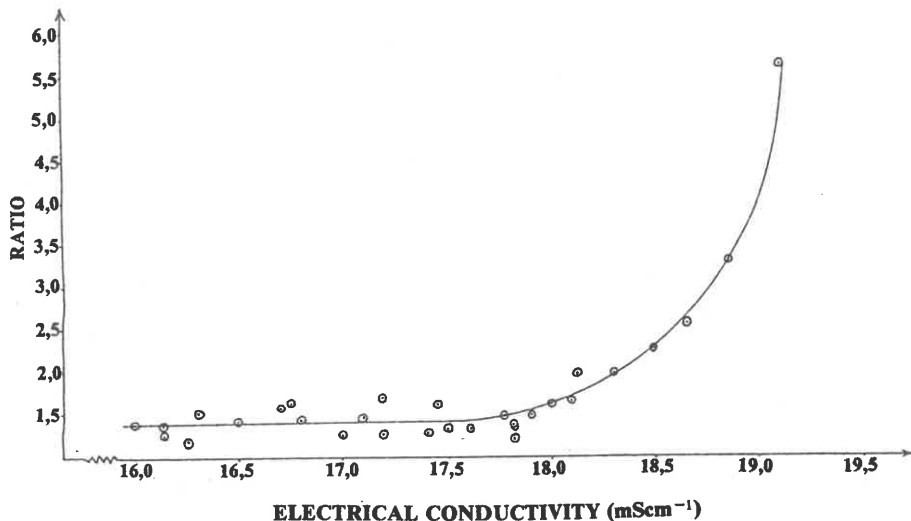


Fig. 5 - Ratio of 2450 MHz HF Dye Fixation to Thermofix Dye Fixation Plotted Against Electrical Conductivity.

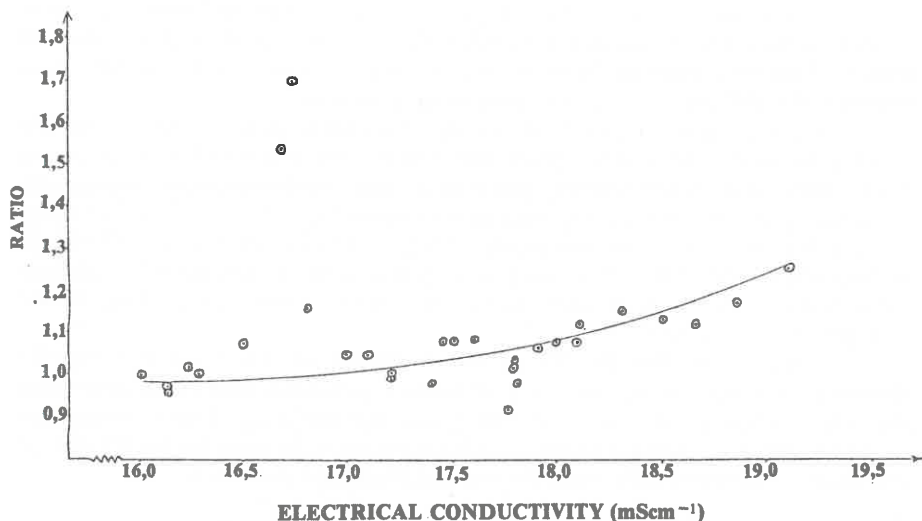


Fig. 6 - Ratio of 27,12 MHz HF Dye Fixation to Pad-Steam Dye Fixation Plotted Against Electrical Conductivity.

thermofixation and steaming fixations, therefore, gives parameters which allow for the dye fixation occurring via thermofixation and steaming. These parameters are therefore more dependent on pad liquor electrical conductivity. Electrical conductivity values could be identified for which these parameters appear to be extremely sensitive, for example, values higher than 17,8 mScm⁻¹ in the case of the 2450 MHz process.

Studies are currently underway at SAWTRI to ascertain the effects of altering the electrical conductivity of dye liquors by adding varying amounts of a number of different electrolytes. The above relationships and effects in other dye/fibre systems will also be studied.

SUMMARY AND CONCLUSIONS

A study was made of the use of high frequency (HF) energy in the dyeing of cotton fabric with reactive dyes. Thirty different vinyl sulphone reactive dyes were studied using HF energy with frequencies 27,12 MHz and 2450 MHz. These treatments were compared with those of conventional pad-steam and pad-thermofix (dry heat) processes.

It was found that, in general, dye fixations increased with increasing treatment time and decreased with increasing concentration of dye in the pad liquor. Excellent rubbing fastness ratings were obtained with no difference between the HF energy and the conventional treatments.

A comparison of the four dyeing treatments showed that, under the conditions used, HF energy produced higher dye fixation for most of the dyes, than did conventional pad-steam and pad-thermofix treatments. Comparing the two HF dyeing treatments themselves, HF energy of frequency 27,12 MHz more often than not produced higher fixation values than HF energy of frequency 2450 MHz. This was probably due to the experimental conditions which were such that more water was present in the former case leading, in turn to a greater steaming effect.

It was found that the HF energy dye fixations correlated well with the electrical conductivity of the dye pad liquors, provided that corrections were made for contributions due to steaming and thermofixing. These correlations could be used as a basis for explaining dye fixation behaviour by the HF energy processes, compared with those by the conventional processes.

THE 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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