

Rec. 139234

SAWTRI TECHNICAL REPORT

WU4/G/216

137

DYEING OF WOOL FROM A CHARGED SOLVENT SYSTEM

A PRELIMINARY REPORT

by

O. A. SWANEPOEL

and

LYNETTE ROESSTORFF

SOUTH AFRICAN WOOL TEXTILE RESEARCH INSTITUTE
P.O. BOX 1124
PORT ELIZABETH

DYEING OF WOOL FROM A CHARGED SOLVENT SYSTEM

A PRELIMINARY REPORT

by O. A. SWANEPOEL and LYNETTE ROESSTORFF

ABSTRACT

A charge consisting of a mixture of an anionic surfactant, an alkylolamide and water is described which can be used for the emulsification of a large number of wool dyestuffs in perchlorethylene medium. Dyeings of satisfactory wet-fastness and levelness can be carried out at 90°C and recovery of the solvent is facilitated by absorption of most of the water by the wool.

KEY WORDS

dyeing — dyestuffs — emulsion — perchlorethylene — solvents — surfactants — wool.

INTRODUCTION

As pointed out by Brunschweiler¹, the question of the advisability of dyeing from solvents is undeniably concerned with the water problem. The continuing decrease in, and the contamination of available fresh water, together with the problem associated with the purification of effluent, greatly increase the cost of using water. In addition to offering a solution to these problems several other factors also contribute towards making dyeing from organic solvents a confirmed, if yet an infantile, part of textile technology. For example, Sieber² has described how solvent processes could be employed in new methods of dyeing and finishing in which great time — and energy savings can be made. The more rapid processes give higher production figures whilst the low specific heat and low latent heat of evaporation of solvents such as perchlorethylene (respectively about five and 10 times lower than those of water) allow great savings in heating and drying energy.

Dyeing of synthetic fibres from organic solvent medium undoubtedly offers greater scope and more exciting possibilities than the dyeing of wool from this medium. In fact, it is quite impossible to dye wool in a medium from which water is completely excluded since the latter is essential for the swelling of natural fibres — a pre-requisite for the dyeing of these fibres. Wool can therefore at best be dyed only from what Mecheels³ refers to as the solvent/tenside/water system, also known as a "charged" system. Mecheels³ explains that if the water content is sufficiently low, this system is a homogeneous, true solution consisting of non-dialyzable micelles and with an effective relative humidity which is a function of the water content of the system. If the tenside/water mixture in the organic solvent is increased, however, the system changes into an emulsion with typical colloidal properties. The

system is then dialyzable and the relative vapour pressure of the water in the system is 1.0.

Treatment of wool in the emulsion type of charged system has definite disadvantages compared with treatments carried out in solvent systems of low vapour pressure. One disadvantage, for example, is that the wool is subject to normal relaxation⁴ and felting shrinkage, (if not shrinkproofed), in the former system. Another is that the presence of substantial quantities of water, with its high latent heat of evaporation, could seriously increase the distillation cost in the recovery of the solvent.

Because of the hydrophilic nature of existing wool dyestuffs, there appears to be little hope that these may be applied from the homogeneous type of charged system. The emulsion type of charged system offers greater promise in this respect and although it is the less attractive of the two types of systems, its potential usefulness should not be underrated. This paper describes some preliminary work in which the applicability of emulsion type of charged systems for the dyeing of wool with existing wool dyes was investigated.

MATERIALS AND METHODS

The all-wool fabrics used in this investigation were a woollen doctor flannel and an unbleached worsted serge.

All dyeings were carried out in tumbling capsules of a Liniset apparatus. The capacity of each stainless steel capsule was 450 ml and the weight of test specimen used was 20 g for each dyeing.

Chromaticity coordinates were calculated from reflectance readings taken at 10 nm intervals between 380 nm and 760 nm and the differences in colour, ΔE , between samples were calculated using the formula⁵

$$\Delta E = [(U_2 - U_1)^2 + (V_2 - V_1)^2 + (W_2 - W_1)^2]^{0.5}$$

EXPERIMENTAL AND DISCUSSION

A primary aim of this investigation was to find a charged system which, without alteration, would be suitable for the application of the maximum number of existing wool dyestuffs. It soon transpired that the widely different properties of various dyestuffs, would place severe demands on a "universal" charged system and that, at best, one could hope for a system which would be suitable for use with a reasonable portion of existing dyestuffs. It was also considered advisable that the amount of water present in the emulsion should be kept reasonably low to enable the wool to absorb a major portion of the emulsified water. This would not only facilitate rapid absorption of dyestuff by the wool but the thermo-economics of the solvent recovery process, which is greatly affected by the presence of water (high latent heat of evaporation), would also benefit.

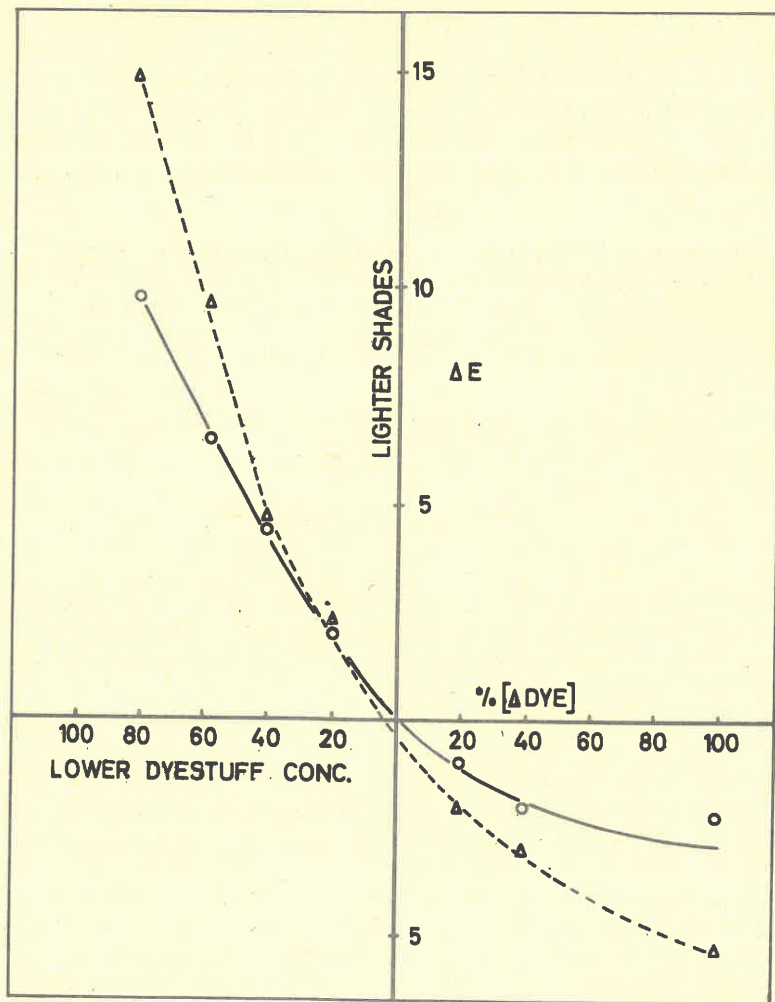


FIGURE 1
 Relationship between colour difference and difference in concentration of dye-stuff, Xylene Red B, used, using the dyeing of 2.5% as reference. See Table II for experimental details. The solid line depicts the Table II(a) dyeings and the broken line the Table II(b) ones.

Investigation showed that a combination of a linear alkyl sulphate or an alkyl aryl sulphonate with an alkalolamide offer some promise as a charge for the solvent dyeing of wool. Sodium dodecylbenzenesulphonate. (SDBS), an alkyl aryl sulphonate which is industrially available at low cost, and lauryl monoethanolamide (LEA), a readily available product used as a foam stabiliser in detergency, were then investigated in greater detail.

TABLE I
COMPOSITION OF CHARGE FOR SOLVENT DYEING OF WOOL
FROM PERCHLORETHYLENE AT A LIQUOR: WOOL RATIO OF
10:1 (v/w). PERCENTAGES CALCULATED ON THE WEIGHT
OF THE WOOL

	<u>%</u>
Sodium dodecylbenzene sulphonate (SDBS)	7.5
Lauryl monoethanolamide (LEA)	2.5
Water	50

Experiments showed that the dyeing of wool from perchlorethylene at a liquor ratio of 10:1, using the above tensides in the charge described in Table I to emulsify the dyestuff, justified further investigation. Employing the relative amounts of SDBS, LEA, and water shown in Table I, a number of dyeings were carried out in which both the charge and the dyestuff concentrations were varied. Thus, in the experiments described in Table II(a) the amount of charge was kept constant whilst the amount of dyestuff was varied. Table II(b) in turn, contains details of a series of dyeings in which both the concentrations of charge and of dyestuff were varied. It is noticed that the concentration of dyestuff used in each dyeing described in Table II(a) corresponded to one of the dyestuff concentrations used in Table II(b). The two dyeings in which 2.5% dyestuff was employed corresponded not only in terms of dyestuff concentration, but also in charge concentration and were therefore identical. The dyeings were carried out by entering the wool at 50°C, increasing the temperature to 90°C over 60 min, and dyeing at 90°C for 60 min. The samples were then centrifuged and dried. Chromaticity coordinates were calculated from reflectance spectrophotometric data and the difference in colour, ΔE , between each dyed sample and the dyeing carried out with 2.5% dyestuff was calculated. These values have been plotted in Fig. 1 against the difference in dyestuff concentration, Δ (Dye) expressed as a percentage of 2.5.

Fig. 1 shows that for the dyeings described in Table II(a) a linear relationship between the difference in shade and the difference in the amount of dyestuff applied was obtained at lower dyestuff concentrations. At a dyestuff concentration of 3.0% [Δ (dye) = 20%], however, the curve deviates from linearity and the deflection

TABLE II
CHROMATICITY COORDINATES OF WOOL SAMPLES DYED FROM
AQUEOUS AND FROM CHARGED SOLVENT SYSTEMS

	% (ON THE WEIGHT OF THE WOOL)															
	(a)								(b)							
SDBS	7.5	7.5	7.5	7.5	7.5	7.5	7.5	7.5	1.5	3.0	4.5	6.0	7.5	9.0	10.5	15.0
LEA	2.5	2.5	2.5	2.5	2.5	2.5	2.5	2.5	0.5	1.0	1.5	2.0	2.5	3.0	3.5	5.0
Water	50	50	50	50	50	50	50	50	10	20	30	40	50	60	70	100
Dyestuff	0.5	1.0	1.5	2.0	2.5	3.0	3.5	5.0	0.5	1.0	1.5	2.0	2.5	3.0	3.5	5.0

TABLE III
COMPOSITION AND AMOUNT OF CHARGE USED IN THE DYEING
OF WOOL WITH XYLENE RED B WITH VARIOUS CONCENTRATIONS
OF DYESTUFF AT A LIQUOR TO WOOL RATIO OF 10:1

DYESTUFF	AQUEOUS MEDIUM			SOLVENT MEDIUM			E
	x	y	Y	x	y	Y	
Kiton Green A	0.437	0.312	37.2	0.436	0.310	33.4	0.929
Irgalan Brown GRL	0.546	0.350	34.0	0.548	0.351	33.5	0.162
Cibalan Brilliant Yellow 3GL	0.592	0.387	25.9	0.590	0.386	26.1	0.202
Alizarine Brilliant Sky Blue GLW	0.386	0.248	41.0	0.380	0.239	41.6	0.146
Lanasol Yellow 4G	0.592	0.388	26.0	0.592	0.388	26.2	0.152
Xylene Blue AS	0.400	0.242	20.5	0.396	0.239	23.3	1.108
Alizarine Sky Blue FFB	0.408	0.260	26.8	0.400	0.255	25.6	0.379
Cibalan Blue BRL	0.476	0.308	20.0	0.478	0.310	26.3	2.609
Xylene Red B	0.526	0.317	80.2	0.534	0.322	79.5	0.253
Alizarine Light Green BT	0.471	0.320	21.6	0.471	0.318	24.0	0.910
Cibalan Grey BL	0.490	0.321	20.3	0.490	0.320	29.6	3.691
Neolan Blue 2G	0.456	0.303	22.1	0.466	0.307	29.4	2.551
Kiton Green B	0.431	0.307	42.5	0.429	0.303	44.3	0.392
Cibalan Yellow 2BRL	0.599	0.374	11.8	0.597	0.375	12.6	1.103

becomes more pronounced at higher dyestuff concentrations. A similar but very much smaller deflection from linearity at higher dyestuff concentrations is also observed for the dyeings described in Table II(b). However, curves of this type are known to deviate somewhat from linearity at high dyestuff concentrations. This deviation may have been accentuated by the poorer exhaustion at these values caused by the relatively high concentration of water in the liquor. The dyeings described in Table II(a), with their constant water concentration, gave a larger deflection because of the inability of the charge to emulsify all the dyestuff at the higher dyestuff concentrations. This means that the ratio of charge to dyestuff is important and that it must, at all times, be considered. At dyestuff concentrations lower than the reference value (2.5%), the dyeings referred to in Table II(a) behaved normally, but the Table II(b) dyeings were excessively pale at the very lowest dyestuff concentrations. This illustrates that not only is the ratio of charge to dyestuff important, but also the ratio of charge to perchlorethylene. This in turn implies that, in the solvent dyeing of wool from charged systems, the wool to liquor ratio is of very great importance.

Furthermore, Fig. 1 shows that up to 2.5% of the particular dyestuff, the behaviour of which is depicted in the graph could be used with a charge containing between 30% and 50% water and the appropriate quantities of surfactants as given in Table II(b). Experience had shown that more dyestuffs can be dispersed in a charge with a higher water content and the charge with 50% water was therefore considered to be of greater possible applicability. The wider applicability of the charge described in Table I was then investigated by attempting the dyeing of a number of commercial wool dyestuffs at the 2.5% dyestuff level from perchlorethylene with the aid of this charge. Reasonable results were obtained in about 60% of the cases investigated. A list of all the dyestuffs investigated, both suitable and unsuitable, are given in the appendix.

The chromaticity values of a number of fabrics which had been dyed with various dyestuffs, either by the conventional dyeing method from aqueous solution or from an emulsion of the dyestuff in a charged perchlorethylene medium, are given in Table III. The latter dyeings were carried out at a liquor ratio of 10:1 and were dyed at 90°C for 60 min after having entered the set bath at 50°C and taken to 90°C in 15 min. It is noticed that the appearance of the fabrics dyed in the solvent system was in most cases practically the same as that of samples dyed at the boil for 60 min from aqueous medium. The brightness, Y, was generally slightly higher for those fabrics dyed in the organic solvent.

The washing and perspiration fastness of wool dyed with several dyestuffs from the charged solvent system were compared with those of wool dyed from aqueous medium and differences were found to be slight. These values were usually somewhat lower than the published values for the particular dyestuff which can possibly be explained by the fact that neither the samples dyed from aqueous or from solvent medium were rinsed after dyeing.

The rate of exhaustion from the charged solvent system appeared to be more rapid than from aqueous solution, and also seemed to vary less from one dyestuff to

another, than is the case in dyeing from aqueous solution. This feature is probably associated with the small volume of water present and the fact that the water, in the process of being absorbed by the wool, carries with it the hydrophilic emulsified dyestuff.

The influence of added acetic acid to the charge was investigated for a few dyestuffs and did not appear to offer any special advantage either in the rate of dyeing, the levelness of the dyeing, the fastness properties of the dyed material, or in the degree of exhaustion attained. In fact, the latter parameter was sometimes adversely affected by the presence of acid which tended to increase the solubility of the dyestuffs in the organic solvent thus rendering it more difficult for the wool to extract the dyestuff from the solvent.

The equipment which would be required in full-scale application of this dyeing process, is either a solvent finishing or a drycleaning machine with facilities for heating the liquor in the drum compartment. This type of machine is currently becoming standard equipment in knitting factories and their use for the dyeing of wool presents the possibility that knitters may in future be able to knit up stocks of garments and to piece-dye consignments as the colour fashions become known. A further interesting possibility is that of combining shrinkproofing and dyeing operations. Shrinkproofing of knitwear from organic solvent medium is currently possible with solvent soluble resins such as Synthapret LKF (Bayer), Braxan AL (Pfersee) and Zeset (Du Pont). Application of these resins may conceivably be followed by emulsion dyeing from solvent medium and would involve a minimum of labour once the equipment has been programmed. Finally, Veldsman⁴ has shown that tumbling of wool in a charged system involving approximately 30% of water emulsified in perchlorethylene solution is an effective way of bringing knitwear to the fully relaxed state. The dyeings described above, therefore offer the added benefit of simultaneously producing the fully relaxed state.

Upscaled trials using the charge described in Table I and a liquor ratio of 10:1 are currently in progress and combination of the shrinkproofing and dyeing operations as speculated upon above are being attempted. The details of these experiments and the results obtained will be published in due course.

REFERENCES

1. E. Brunnschweiler, *Textilveredlung* 4, 745 (1969).
2. J. H. Sieber, *Textilveredlung* 4, 773 (1969).
3. J. Mecheels, *Textilveredlung* 4, 749 (1969).
4. D. P. Veldsman, *SAWTRI Bulletin* 4, (1), 10 (March, 1970).
5. See E. Coates and B. Rigg, *J. Soc. Dyers Col.* 81, 469 (1965).

APPENDIX

DYESTUFFS INVESTIGATED FOR COMPATIBILITY WITH CHARGED
PERCHLORETHYLENE SOLVENT SYSTEM. COMPOSITION OF CHARGE
WAS THAT GIVEN IN TABLE I AND LIQUOR TO WOOL RATIO
WAS 10:1

COMPATIBLE

INCOMPATIBLE

LANASOL DYES

Yellow 4G
Blue 3R
Orange G

Red 6G
Scarlet 2R
Blue 3G
Red G
Red 5B^c

CIBACROLAN DYES

Yellow 2G

Blue 8G
Blue 8GK
Green G
Green VM

CIBALAN DYES

Yellow 2BRL
Blue BRL
Bordeaux GRL
Bordeaux RL
Blue FBL
Brilliant Blue RL
Bordeaux 3BL
Green GL
Brown BL
Brown 5RL
Brown VRL
Brilliant Yellow 3GL
Brilliant Blue GL
Orange RL
Scarlet GL
Grey 2GL
Grey BL

Blue 3GL
Brilliant Scarlet RL
Brilliant Red BL
Yellow FGL
Brown TL
Violet RL
Brilliant Yellow 3GL
Yellow GRL

COMPATIBLE**INCOMPATIBLE**

IRGALAN DYES

Yellow 2GL
Blue RL
Blue GL
Bordeaux 2BLN
Black GBL
Black RBL
Dark Brown 5R
Bordeaux 2BL
Brown GRL
Brown 2RL
Navy Blue 5RL
Grey RL
Olive BGL
Navy Blue 2GL
Red 4GL
Red 3G
Red 2BL
Violet 5RL
Yellow GL
Yellow 2GL
Yellow 2RL

Violet 4BL
Brown 7RL
Grey BRL
Brown 3BL
Brown Violet DL
Rubine RL

REMAZOLAN DYES

Yellow GL
Brilliant Blue R

Brilliant Blue B
Red F3B
Orange GG
Orange GR
Red R
Yellow RR
Yellow G

XYLENE DYES

Fast Yellow P
Fast Blue BL
Brilliant Cyanine
Blue AS
Blue VS
Brilliant Blue B
Brilliant Blue 5GM
Fast Violet R
Fast Yellow ES
Fast Yellow P140
Fast Violet 4RN
Light Yellow 2GP
Milling Blue BCN
Yellow S
Milling Yellow G
Milling Yellow 3GL
Red B

Fast Blue FF
Acid Milling Orange 2G
Black 4BP
Black 4B
Brilliant Black BN
Fast Violet B
Fast Red VP
Fast Violet P3R
Fast Red P3B
Fast Yellow RPN
Light Yellow R
Milling Blue 6G
Milling Red 3BLN
Milling Red B
Milling Orange R
Violet RL
Milling Yellow GG
Turquoise Blue G

ALIZARINE DYES

Brilliant Blue PFN
Chrome Green VSNN
Chrome Grey BS
Brilliant Blue OCR
Brilliant Green 6EW
Brilliant Violet RW
Brilliant Sky Blue BLW
Brilliant Sky Blue GLW
Brilliant Sky Blue RLW
Cyanol ZEF
Direct Blue AR Conc.
Direct Blue AGG
Cyanine Green GLA
Green N

Direct Blue 3FL
Direct Grey GL
Direct Green BL
Direct Blue 3GL
Direct Violet EBB
Direct Brown L3RP
Direct Brown RMW
Fast Blue 3R
Fast Blue R
Light Blue AR
Light Blue B
Light Blue 5GL
Light Blue 4GL
Light Blue FG

ALIZARINE DYES

Green 5G	Light Blue BRP
Cyanine Green G	Light Grey BLL Conc.
Direct Brown GP	Light Grey GLL
Fast Blue BE	Light Grey G
Fast Grey G	Light Blue RG
Fast Violet FRL	Light Blue R
Fast Green G	Light Violet RS
Fast Blue G	Light Violet RCN
Light Blue FF	Light Violet 3RL
Light Blue BGA	Light Red RNN
Light Blue HRL	Light Grey RLL
Fast Green 2GW	Red A 120
Light Green GS	Yellow GD
Light Green BT	Red SZ
Light Green GNS	
Night Blue AG	
Milling Green B	
Light Violet 2RC	
Sky Blue SGLW	
Sky Blue FFB	

KITON DYES

Fast Blue BGA	Black 4BNN
Fast Yellow G	Black HA
Fast Blue FB Con.D	Fast Red 2G
Green A	Fast Yellow 3RL
Violet 4RN	Fast Blue FGL
Green B	Fast Rubine GRL
Green V	Fast Red BGL
	Fast Red 4BLN
	Fast Blue GB

NEOLAN DYES

Blue 2G	Black 2G
Black SR Conc.	

Published by
The South African Wool Textile Research Institute
P.O. Box 1124, Port Elizabeth, South Africa
and printed in the Republic of South Africa
by Nasionale Koerante Beperk, P.O. Box 525, Port Elizabeth