WU4/6/2/1

# SAWTRI TECHNICAL REPORT



No. 495

Electroflotation of Dye Liquors
Containing Wool Grease
Using Aluminium Electrodes

by

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# ELECTROFLOTATION OF DYE LIQUORS CONTAINING WOOL GREASE USING ALUMINIUM ELECTRODES

by E. WEIDEMAN and N. J. J. VAN RENSBURG

## **ABSTRACT**

The effect of electroflotation using aluminium electrodes on the C.O.D., D.O.C. and colour values of liquors containing acid, disperse, mordant and basic dyes in the presence and absence of wool grease was investigated. In general electroflotation followed by sieving, filtering or centrifuging reduced the C.O.D., D.O.C. and colour values of the liquors considerably. The presence of wool grease affected some improvement in colour in the case of basic dyes.

# INTRODUCTION

Vast amounts of water are used for the preparation, dyeing and finishing of textiles. There are various processes for the treatment of dyehouse effluent, either to facilitate its re-use by the textile mill or to comply with effluent regulations laid down by the local authorities. These processes can be grouped into: physical processes (sedimentation, filtration, distillation, etc.) biological processes (activated sludge, aerobic treatment, anaerobic digestion, etc.), physico-chemical processes (osmosis, electrolysis, etc) and chemical processes (chlorination, ozone treatment). Although in most cases it is technically possible to purify wastewater sufficiently to be circulated in a textile mill, the cost of such effluent treatments frequently is prohibitive. Basically this is due to the fact that relatively expensive processes¹ such as adsorption (using active carbon), ion exchange and osmosis have to be employed to remove the last traces of organic material and inorganic salts from the water.

Parish<sup>2</sup> discussed the various water treatment processes which would allow the re-use of water in textile mills and he grades each process in relation to water quality, versatility, cost, ease of operation, current plant size, etc. He concluded that the re-use of water in textile mills should be introduced stepwise, by extended pilot scale testing. Finally there are numerous patents describing various processes and commercial systems for the re-use of water such as Liracat, Scholl, Advanced Water Services, etc. Despite all these processes, however, it was stated in 1978 that the re-use of water in the UK Textile Industry was not yet widely practised<sup>3</sup>.

Dürig¹ reviewed the ecological aspects of water usage in the textile industry and concluded that biological degradation had been found the most effective way thus far of coping with organic substances in effluent streams. He suggested that a mill should not install its own activated sludge unit, but should rather treat the textile waste water by combining it with domestic

effluent. Consequently only cooling and rinse liquors should be collected separately and re-used in the mill, while the rest of the waste water should be diluted with public sewage.<sup>4</sup>

In view of the proposal by Dürig in 1976 that textile waste water and domestic effluent be mixed it was interesting to note that a textile mill recently patented a process whereby mixed wool scouring and dyeing effluent streams are purified using an electroflotation technique<sup>5</sup>. It is well-known that textile waste waters can be purified by electroflotation using iron and titanium cathodes and anodes<sup>6,7</sup>. In the patent mentioned above, however, the electrodes are made of aluminium, and it has been claimed that these electrodes offer several advantages over iron electrodes for the continuous electroflotation of effluent streams.

Since at present a limited amount of information is available about this novel process, a study was initiated to investigate the effect of electroflotation using aluminium electrodes, on the colour, chemical oxygen demand (C.O.D.) and dissolved organic carbon (D.O.C.) values of dye liquors containing different types of dyes, such as acid, basic, disperse and mordant dyes, as well as dye liquors containing wool grease.

# **EXPERIMENTAL**

## Chemicals

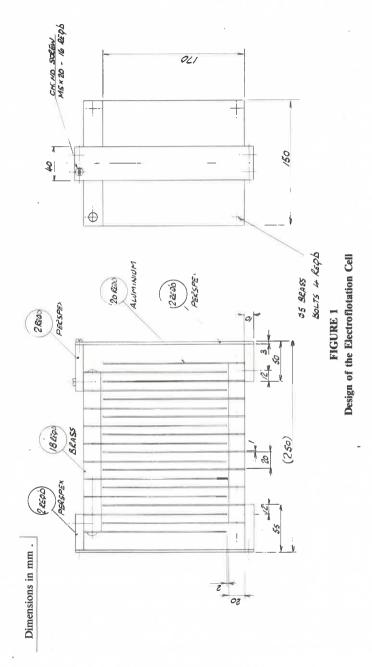
Commercial dyes and laboratory grade chemicals were used. Mains water was used to prepare the simulated dye-house "effluents", i.e. liquors containing dyes and auxiliaries in the concentrations normally encountered with spent liquors.

#### Electroflotation

An electrolytic cell was constructed (see Fig. 1). The dye solutions were treated in a batch-wise manner by the electroflotation technique in a polypropylene vessel (25 $\ell$ ) by passing a current through the cell at a voltage of 11 V for 30 minutes. The temperature of the solution during electroflotation was kept at 60°C. All dye liquors contained ®Alcopol 650 (10 mg/ $\ell$ ) as wetting agent. In the cases where the effect of wool grease was studied 150 mg/ $\ell$  of grease was used except where otherwise indicated. Prior to the addition of the wool grease to the dye liquors the grease was emulsified with ®Berol Lanco, a non-ionic detergent.

## **Tests**

Aliquots were taken from the various liquors before and after electroflotation and diluted with distilled water prior to the analysis being



SAWTRI Technical Report, No. 495 — February 1982

THE EFFECT OF ELECTROFLOTATION ON THE C.O.D. AND D.O.C. VALUES OF DIFFERENT **SOLUTIONS OF CI ACID BLACK 24** TABLE I

Wool grosse Dv	Composition of Liquor	)	C.O.D. (mg $\theta_2/\ell$ )		Dissolved	Dissolved Organic Carbon (mg/l)	(mg/l)
	Dye concentra- tion (mg/l)	Before electroflotation	After electro- flotation/ Sieving	After electro- flotation/ Filtering	Before electro- flotation	After electro- flotation/ Sieving	After electro- flotation/ Filtering
	0	159	122	120	9	9	-
	10	171	138	122	6	7	2
	20	177	189	125	21	29	48
	150	286	203	123	51	31	15
0	200	587	270	142	198	38	65
	1000	1100	009	192	272	43	42
	2000	5750	3200	1200	1500	381	358
	10000	10900	5100	2200	1840	450	327
	0	545	200	124	17	2	-
	10	557	200	120	34	12	2
	20	290	200	118	19	5	4
9	150	602	231	127	9/	4	4
061	200	834	509	158	155	45	7
	1000	1448	1064	292	252	22	9
	2000	8400	2200	1600	1720	209	326
	10000	14100	10200	2900	3300	066	870

carried out. All the samples were filtered either through a 50  $\mu$ m sieve or through Whatman GFC 3 Glassfibre filter paper retaining particles bigger than about 1,2  $\mu$ m. In one case the liquor was centrifuged at 12 500 rev/min for one minute in a MSE 25 centrifuge. The chemical oxygen demand (C.O.D.) of the solutions was determined according to a standard method<sup>8</sup>. The dissolved organic carbon (D.O.C.) was measured on a Technikon Auto Analyser<sup>9</sup>.

The colour of the solutions was determined by measuring the absorption at the wavelength of maximum absorption.

## RESULTS AND DISCUSSION

The effect of electroflotation on the C.O.D. and D.O.C. values of liquors containing various concentrations of an acid wool dye (C.I. Acid Black 24) in the presence and absence of wool grease can be seen in Table I. It is obvious that electroflotation reduced the C.O.D. and D.O.C. values of the liquors considerably. Since clear solutions are required for the D.O.C. analyses, all

TABLE II

THE EFFECT OF ELECTROFLOTATION ON THE COLOUR
(ABSORBANCY) OF DIFFERENT SOLUTIONS OF C I
ACID BLACK 24

Composition of Liquor		Absorbancy (at 590 nm)		
Wool Grease concentration (mg/l)	Dye concentration (mg/l)	Before electroi	After lotation	
	0	0,03	0,03	
	. 10	0,32	0,04	
	. 50	0,56	0,01	
	150	1,55	0,01	
150	500	>2,00	0,03	
	1000	>2,00	0,03	
	5000	>2,00	0,86	
	10000	>2,00	2,00	
	0	0,04	0,04	
	10	0,26	0,03	
	50	0,54	0,01	
	150	1,48	0,02	
0	500	>2,00	0,02	
	1000	>2,00	0,03	
·	5000	>2,00	2,00	
	10000	>2,00	2,00	

the samples were filtered immediately after electroflotation prior to being analysed. The samples which had been filtered through the fine glass fibre filter (1  $\mu$ m) generally gave lower values than those which had been passed through the coarser 50  $\mu$ m sieve, which was to be expected. Table I shows that the C.O.D. of the solutions containing dye, as well as that of the solutions containing dye and wool grease was reduced considerably by electroflotation, indicating that not only dye but also grease was removed from the solution. The dissolved organic carbon content of the samples was also reduced by electroflotation with in general, tendencies similar to those observed with the C.O.D values.

The effect of electroflotation on the colour of the dye solutions containing different concentrations of Acid Black 24 (from 10 to 1 000 mg/l) is shown in Table II. It is clear that electroflotation reduced the colour of the

TABLE III

THE EFFECT OF ELECTROFLOTATION ON THE C.O.D. OF DIFFERENT CLASSES OF DYES\*

		Wool Grease	C.O.D. (mg 0 <sub>2</sub> / $\ell$ )	
Dye Class	C.I. Number	Concentration (mg/ $\ell$ )	Before electrof	After lotation
	Disperse Brown 1	0	379	168
	"	150	651	294
Disperse	Disperse Blue 19	0	378	128
	"	150	660	182
	Disperse Orange 38	0	380	135
	"	150	579	180
	Acid Black 24	0	296	110
	"	150	696	124
Acid	Acid Red 249	0	227	97
	"	150	344	121
ъ :	Basic Blue 1	0	255	160
Basic	"	150	542	173
Mandani	Mordant Blue 1	0	235	71
Mordant	"	150	413	107
		0	170	120
	_	150	543	126

<sup>\*150</sup> mg/ℓ dye was used in all cases

All samples were filtered through fibre glass before being analysed

liquors significantly. Solutions containing up to 1 000 mg/ $\ell$  dye had practically no colour after electroflotation for 30 minutes, irrespective of whether wool grease was present in the liquors or not. In view of the fact that dyehouse effluents would seldom, if ever, contain dyes in concentrations exceeding 500 mg/ $\ell$ , it is quite clear that the electroflotation process holds considerable promise as a method of reducing the colour of dye-containing effluents.

The electroflotation of different classes of dyes was examined next and the results are represented in Table III. It can be seen that the C.O.D. values of all the different types of dyes, namely disperse, acid, basic and mordant dyes were reduced considerably by the treatment. This also applied where wool grease was added to the dye liquors. The corresponding D.O.C. values are given in Table IV and it can be seen that in all cases the D.O.C. values were reduced considerably by electroflotation. The colour of all the dye solutions

TABLE IV

THE EFFECT OF ELECTROFLOTATION ON THE D.O.C. OF DIFFERENT CLASSES OF DYES

		Wool Grease	<b>D.O.C.</b> (mg/ $\ell$ )	
Dye Class	C.I. Number	Concentration (mg/l)	Before electrof	After lotation
	Disperse Brown 1	0	57	10
	" "	150	79	11
	Disperse Blue 19	0	72	7
Disperse	"	150	129	10
	Disperse Orange 38	0	72	14
	"	150	129	13
	Acid Black 24	0	46	5
Acid	"	150	70	8
Aciu	Acid Red 249	0	39	7
	"	150	53	7
	Basic Blue 1	0	16	9
Basic	"	150	56	9
	Mordant Blue 1	0	26	4
Mordant	"	150	10	3
	_	150	10	1

was reduced significantly by the treatment (see Table V) and practically colourless solutions were obtained in all cases, with the exception of C I Basic Blue 1. Furthermore it is interesting to note that the addition of wool grease to the C I Basic Blue solution resulted in a considerable improvement in the colour of the solution after electroflotation.

In view of the fact that all the dyes generally produced satisfactory results with the exception of C I Basic Blue 1, it was decided to study the effect of increased electroflotation times on the C.O.D. and colour of liquors containing C I Basic Blue 1. The results, given in Table VI, show that an increase in the time of electroflotation resulted in a decrease in the C.O.D. and the colour values of the liquors. In this particular case equilibrium seems to have been reached only after a reaction time of three hours.

During the electroflotation studies it was noticed that the aluminium electrodes dissolved gradually. This is in agreement with the findings reported in the patent<sup>5</sup> describing the electroflotation of waste liquors using aluminium

TABLE V
THE EFFECT OF ELECTROFLOTATION ON THE COLOUR (ABSORBANCY) OF DIFFERENT CLASSES OF DYE

		Wool Grease	Absorbancy	
Dye Class	C.I. Number	Concentration (mg/l)	Before electro	After flotation
	Disperse Brown 1	0	1,07	0,04
	"	150	1,06	0,04
Disperse	Disperse Blue 19	0	1,51	0,02
	"	150	1,54	0,02
	Disperse Orange 38	0	1,54	0,02
	"	150	1,54	0,03
	Acid Black 24	0	1,65	0,01
	"	150	1,82	0,01
Acid	Acid Red 249	0	1,60	0,02
	"	150	1,66	0,02
Davis	Basic Blue 1	0	2,00	1,80
Basic	"	150	2,00	0,51
Mondons	Mordant Blue 1	0	0,51	0,01
Mordant	"	150	0,87	0,00
		0	0,01	0,01
_		150	0,25	0,01

electrodes. In fact, the inventor mentions that these electrodes slowly dissolve thus releasing aluminium ions in solution *in situ*, which are known to assist flocculation. It was decided, therefore, to evaluate the effect of a chemical flocculation treatment using aluminium sulphate on the C.O.D. value of various dye solutions, and to compare these results with those obtained with electroflotation. Two dose levels were evaluated, namely, one using practical levels of aluminium sulphate and one using excessive amounts. The results are given in Table VII. It is clear that the C.O.D. of the liquors was reduced by the

TABLE VI
THE EFFECT OF TIME OF ELECTROFLOTATION ON THE COLOUR
(ABSORBANCY) AND C.O.D. OF C2 BASIC BLUE 1

Time of Electroflotation hr	Absorbancy	C.O.D. (mg $0_2/\ell$ )
0	>2,00	283
0,5	>2,00 >2,00	169
1,0	0,81	82
2,0	0,10	67
3,0	0,09	20
4,0	0,08	24
5,0	0,09	20

TABLE VII

THE EFFECT OF ALUMINIUM SULPHATE ON THE C.O.D. OF VARIOUS DYE SOLUTIONS

D *	Wool Grease	C.O.D. (mg 0 <sub>2</sub> /ℓ)			
Dye*	Conc. (mg/l)	Untreated	50 mg/l A1 <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub>	2200 mg/l/ A1 <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub>	
	0	163	134	_	
Disperse Brown 1	0	378	198	_	
Acid Black 24	0	218	136	113	
Basic Blue 1	0	250	196	168	
Mordant Blue 1	0	230	101		
-	150	551	251	!	
Disperse Brown 1	150	667	405		
Acid Black 24	150	676	308	154	
Basic Blue 1	150	574	295	172	
Mordant Blue 1	150	427	218		

<sup>\*150</sup> mg/ℓ dye

addition of aluminium sulphate, although not to quite the same degree as that obtained by the electroflotation process, except when a very high concentration of aluminium sulphate was employed. Table VIII shows the effect of chemical flocculation with aluminium sulphate on the colour of the liquors. Although the colour of the solutions was reduced considerably by aluminium sulphate, the treatment seemed less effective than electroflotation, especially in the case of the basic dye.

Finally, it was considered important to establish the effect of centrifuging instead of filtration on the C.O.D. values of the liquors which had been treated in the electroflotation cell. The results, given in Table IX, show that centrifugation of the treated liquors produced results almost as good as those of samples which had been filtered through a fibre glass filter, and considerably better than those of samples which had been filtered through a relatively coarse sieve. The centrifuging of the liquors after electroflotation therefore seems to have some potential as an aid to improving the quality of the treated water.

TABLE VIII

THE EFFECT OF ALUMINIUM SULPHATE ON THE COLOUR
(ABSORBANCY) OF VARIOUS DYE SOLUTIONS

	Wool Grease	Absorbancy		
Dye*	Conc (mg/l)	Untreated	50 mg/l A1 <sub>2</sub> (SO <sub>4</sub> ) <sub>3</sub>	
	0	0,02	0,03	
Disperse Brown 1	0	1,50	0,09	
Acid Black 24	0	1,40	0,05	
Basic Blue 1	0	>2,00	2,00	
Mordant Blue 1	0	1,55	0,01	
<del></del>	150	0,03	0,03	
Disperse Brown 1	150	1,70	0,12	
Acid Black 24	150	1,50	0,12	
Basic Blue 1	150	>2,00	2,00	
Mordant Blue 1	150	1,90	0,01	

<sup>\* 150</sup> mg/l dye

THE EFFECT OF FILTERING, SIEVING OR CENTRIFUGING ON THE C.O.D. OF SOLUTIONS OF TABLE IX

150 mg/\ell dye | 5000 mg/\ell dye 1 120 290 270 130 130 160 D.O.C. (mg/l) 53 75 48 47 50 CI ACID BLACK 24 AFTER ELECTROFLOTATION No dye 38 10 11 12 S 50 00 150 mg/l dye | 5000 mg/l dye | 5 300 3 800 1 970 2 500 1 950 830 8 150 5 700 C.O.D. (mg  $0_2/\ell$ ) 270 230 230 148 580 230 190 128 No dye 156 128 102 102 550 190 115 108 Wool Grease Concentration (mg/l) 150 150 150 150 0000 Centrifuge Centrifuge Electroflotation, Sieve Electroflotation, Sieve Filter Filter Treatment None None

## SUMMARY

The effect of electroflotation using aluminium electrodes on the C.O.D., D.O.C. and colour values of liquors containing different dyes in the presence and absence of wool grease was investigated. It was found that liquors containing up to  $1\,000\,$  mg/ $\ell$  dye had practically no colour after electroflotation. In general acid, disperse and mordant dyes gave acceptable results, but it was not the case with the basic dye studied. It appeared, however, that the addition of wool grease to the dye considerably improved the colour of the liquor after electroflotation. Alternatively, an increase in the time of electroflotation also reduced the colour of the liquors to acceptable levels.

For purposes of comparison some of the liquors were treated with aluminium sulphate as flocculant. Although the C.O.D. and D.O.C. values of the liquors were reduced considerably by this treatment, the levels were not as low as those obtained with electroflotation. Futhermore, flocculation with aluminium sulphate also seemed less effective in reducing the colour of the liquors than did electroflotation.

It was found that centrifuging of the liquors after electroflotation instead of sieving or filtering appears to offer some promise in improving the quality of the treated water.

# **ACKNOWLEDGEMENTS**

The authors wish to thank Miss W. Black and Mr S. A. Musmeci for valuable technical assistance.

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Published by
The South African Wool and 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.

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ISBN 0 7988 1975 8