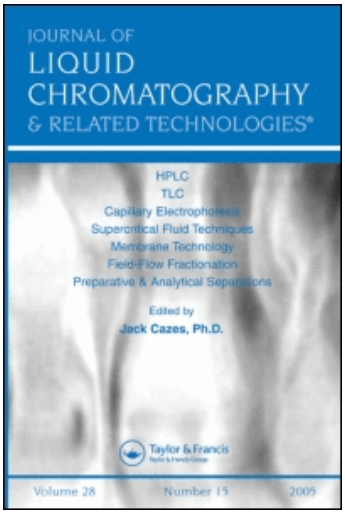

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Chromatographic Separation of Closely Related Dyes on Metal Salt-Impregnated Thin Layers

S. P. Srivastava[a]; L. S. Chauhan[a]; V. K. Dua[a]
[a] Department of Chemistry, University of Roorkee, Roorkee, India

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CHROMATOGRAPHIC SEPARATION OF CLOSELY RELATED DYES ON METAL SALT-IMPREGNATED THIN LAYERS

S.P.Srivastava, L.S.Chauhan
and V.K.Dua

Department of Chemistry, University of
Roorkee, Roorkee-247672(India)

ABSTRACT

The chromatographic characteristics of 42 dyes belonging to different groups have been studied on metal salt-impregnated plates. A suitable separation scheme for the dyes studied and suitable adsorbent system as well as solvent system have been developed.

INTRODUCTION

The chemical identity of a dye is of interest to the colour user, since two dyes giving the same shade on dyeing may differ substantially in fastness properties, but chemical identity is often more difficult to establish than identity or similarity in dyeing and fastness properties. TLC studies on different groups of synthetic dyes have been reported[1-4]. Recently Arsov and coworkers[5] have reported a suitable method for the separation of fat dyes while a review on the TLC of acid dyes, reactive dyes, disperse dyes, metal complex dyes, mordant dyes, basic dyes, solvent dyes has been given by Venkataraman[6]. Recently impregnation of TLC plates with different substances has been tried to improve the separation of various class of compounds[7-13]. The present paper reports our studies on the use of metal salt-impregnated silica gel

plates for the separation and identification of 42 closely related dyes.

EXPERIMENTAL

Glass plates 20x20 cm^2 were coated with a mixture of silica gel (50 g) and 5 per cent metal salt solution (100 ml) by means of Stahl-type applicator. The thickness of the layers was 0.5 mm. The coated plates were activated at 60 $\pm$ 1° for 24 h. 2-3 ul of 0.1 per cent alcoholic or alcohol: water (1:1, V/V) solution of the dyes was used for spotting. After development, the dyes were visualised as such.

RESULTS & DISCUSSION

The chromatographic behaviour of various dyes on impregnated plates can be divided into two groups (i) dyes moving appreciably in neutral solvent i.e. n-butanol-ethyl acetate-benzene termed as group A dyes and (ii) dyes showing little or no movement in this neutral solvent system but moving in n-butanol-H_2O-formic acid solvent system termed as group B dyes. The impregnants tried were cadmium sulphate, cadmium acetate, zinc acetate, zinc sulphate, manganese sulphate and manganese acetate. It was found that on increasing the amount of metal salt, the hR_f value decreases for most of the dyes and after 7 per cent of metal salt as impregnant, the hR_f value remains practically constant and the best results for all the dyes reported have been obtained with 5 per cent metal salt as impregnant. The most suitable impregnant found was cadmium acetate. Further, it is worthwhile to report that for the solvent system n-butanol-water-formic acid, the hR_f value increases as the formic acid concentration increases but the hR_f values become closer on increasing formic acid concentration. Similarly for the solvent system

n-butanol-ethyl acetate-benzene, the hR_f value increases as the percentage of ethyl acetate is increased while increase in benzene concentration decreases the hR_f value. The suitable composition for this solvent system found was n-butanol:ethyl acetate-benzene (40:25:35). The results with this solvent system are given in table 1, while for the other solvent system, satisfactory results were obtained by using solvent composition n-butanol:water:formic acid (35:10:5) (table 2). The results obtained only with $CdAc_2$ or $CdSO_4$ as impregnant are reported here along with the hR_f values on plain silica gel (for comparison), because of the various impregnants tried they gave the best results.

The difference of $\pm$ 3 units in hR_f has been taken as the criterion of satisfactory separation. Some of the typical separations on silica gel-cadmium acetate layer, using n-butanol-water-formic acid as developing solvent where the dyes of group A have been divided into three sub groups A_1, A_2 and A_3, are listed in table 3. With the solvent system, n-butanol-ethyl acetate-benzene, the typical separations obtained are given in table 4. Here again the group B dyes have been divided into three sub groups B_1, B_2 and B_3.

The chromatographic behaviour of dyes on metal salt-impregnated silica gel plates suggested that the interaction of the metal ion with N of the NH_2 group should be an important factor influencing chromatographic behaviour. Similar view was also put forward by Yasuda[7-10] for the TLC separation of aromatic amines on metal salt-impregnated plates. However, this cannot be the sole factor because the hR_f values on cadmium sulphate and cadmium acetate impregnated plates are different. Further study in this direction is in progress.

TABLE I

Solvent System: Butanol: Ethyl Acetate: Benzene (40:25:35)

Dyes	hR_f		
	Silica Gel	Silica gel + $Cd.Ac_2$	Silica gel + $CdSO_4$
Rosaniline hydrochloride	71^{ST}	58	54^{ST}
Chrysodine	34	23	20
Malachite green	21^{ST}	12	33^{T}
Methyl red	85^{T}	46	82
Crystal violet	45^{T}	38	59
Fuchsine basic	67^{ST}	62	64^{ST}
Orange G	20^{ST}	11	12
Auramine O	57	49	50
Bromocresol green	44^{T}	59	60^{T}
Bromophenol blue	42^{ST}	40	37
Bromothymol blue	82^{T}	80	80
Phenol red	54	50	54
Thymol blue	75	72	75
Acridine orange	52	23	30
Cadion 2B	95^{T}	95	94
Dichlorofluorescein	55	14	13
Rhodamine B	56	4	6
Eosine bluish	54	15	8
Eosine yellowish	53	10	15
Naviline yellow	98	96	93
Naviline brilliant pink	96^{T}	92	93^{ST}
Methyl violet	58	45	55
Aniline blue	96^{T}	90	90^{ST}
Bromocresol purple	74	65	62
Gentian violet	76^{ST}	52	60

ST = slight tailing ; T = Tailing

TABLE II

Solvent System: Butanol: Water: Formic Acid (35:10:5)

Dyes	hR_f		
	Silica gel	Silica gel+ $Cd.Ac_2$	Silica gel+ $Cd.SO_4$
Alizarin red S	62^{ST}	48	51
Alizarine blue	36	30	26^{ST}
Bismarck brown	77^{T}	72	35
Erichrome black T	32	12	20
Aluminon	93	65	93^{T}
Xylidine ponceau	11	5	9
Benzopurpurine 4B	52	57	51
Methylene blue	37^{T}	37	31^{ST}
Nigrosin	0	0	0
Fuschsinacid	21^{ST}	9	19
Light green	13	17	12
Purpurin	95^{ST}	76	95
Haematoxylin	70^{T}	35	85
Diamond blue	78^{ST}	80	80
Dimethyl yellow	95^{T}	94	94^{T}
Titan yellow	80	76	74
Congo red	76^{T}	74	74

ST = Slight tailing; T = Tailing

TABLE III

Solvent System: Butanol: Ethyl Acetate: Benzene (40:25:35)

A_1 Dyes	hR_f	A_2 Dyes	hR_f	A_3 Dyes	hR_f
Alizarine red S	48	Alizarine red S	48	Alizarine red S	48
Alizarine blue	30	Alizarine blue	30	Alizarine blue	30
Bismarck brown	72	Congo red	74	Bismarck brown	72
Eriochrome black T	12	Diamond blue	80	Eriochrome black	12
Aluminon	65	Light green	17	Aluminon	65
Nigrosin	0	Xylidine ponceau	5	Xylidine ponceau	5
Fuchsineacid	9	Methylene blue	37	Methylene blue	37
Diamond blue	80	Benzopurpurine	57	Nigrosin	0
Dimethyl yellow	94	Aluminon	65	Light green	17
Titan yellow	76	Dimethyl yellow	94	Purpurin	76
				Diamond blue	80
				Dimethyl yellow	74

TABLE IV

Solvent System: Butanol-Water-Formic Acid (35:10:5)

B_1 Dyes	hR_f	B_2 Dyes	hR_f	B_3 Dyes	hR_f
Rosaniline hydrochloride	58	Fuchsine basic	62	Gentian violet	52
Chrysoidine	23	Orange G	11	Bromocresol purple	65
Malachite green	12	Bromocresol green	49	Aniline blue	90
Methyl red	46	Bromophenol blue	40	Methyl violet	45
Crystal violet	38	Eosine bluish	15	Navilene yellow	96
Bromothymol blue	80	Navilene yellow	96	Dichloro fluorescein	14
Phenol red	50	Navilene brilliant pink	92	Chrysoidine	23
Thymol blue	72	Methyl violet	45	Crystal violet	38
Cadion 2B	95	Bromocresol purple	65	Methyl violet	45
Rhodamine B	4	Rhodamine B	4	Rosaniline hydrochloride	58
Bromocresol purple	65	Acridine orange	23	Thymol blue	72
		Thymol blue	72	Bromothymol blue	80
		Bromothymol blue	80		

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