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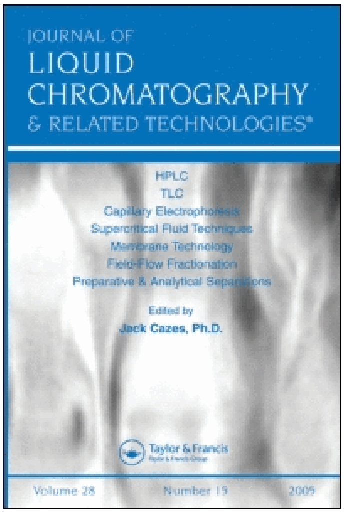

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THE BEHAVIOUR OF SOME PHENOLIC ACIDS AND ALDEHYDES ON THIN LAYERS OF SILICA GEL IMPREGNATED WITH Fe(III)

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ABSTRACT

The behaviour of phenolic aldehydes and acids commonly found in lacustrine sediments as degradation products of lignine was tested by thin layer chromatography on silica gel plates plain and impregnated with Fe(III) in different solvent systems. The differences expressed as R_i values can be used as one of the parameters in the identification of some phenolic aldehydes or acids.

INTRODUCTION

A number of papers has appeared considering the chromatographic behaviour of phenolic acids and aldehydes on thin layers of silica gel and cellulose (1-5). Improvement in resolution has been achieved by impregnating the adsorbents with inorganic or organic substances (5-8). Ferric chloride and ferric nitrate have also been used as impregnants in paper chromatography of some phenolic compounds and drugs (9,10). Here we investigate the separation of some phenolic acids and aldehydes, commonly found in sediments as degradation products of lignin (11,12), on thin layers of si-

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lica gel impregnated with ferric nitrate (in different solvent systems) and compare their chromatographic properties with those on plain plates.

MATERIALS AND METHODS

Chemicals

Phenolic acids and aldehydes were purchased from Fluka A.G. Switzerland, Merck Germany, and B.D.H. England.

Commonly available analytical grade chemicals were used in all other cases.

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Abbreviations

p-HPhAcac - p-Hydroxy phenyl acetic acid
m-HBac - m-Hydroxy benzoic acid
p-HBac - p-Hydroxy benzoic acid
Vac - Vanillic acid
Sac - Salicylic acid
Syrac - Syringic acid
p-HBal - p-Hydroxy benzaldehyde
Val - Vanilline
Sal - Salicylaldehyde

Sorbent layers

Precoated plates of silica gel $60F_{254}$ (Merck), plain and impregnated with $Fe(NO_3)_3$ (1%, by spraying) were used.

Solvent systems

I Ethyl acetate
II Ethyl acetate - benzene (15 : 5) v/v
III Ethyl acetate - benzene (9 : 11) v/v (13)
IV Ethyl acetate - benzene (5 : 15) v/v
V Benzene
VI Chloroform (14)

VII Chloroform -acetic acid (9 : 1) v/v (13)
VIII Chloroform - acetic acid (6 : 4) v/v
IX Chloroform - acetic acid - water (5 : 4 : 1) v/v
X Benzene - dioxane - acetic acid (90 : 25 : 4) v/v (15)
XI Isopropanol - ammonia - water (8 : 1 : 1) v/v (16)

Detection

Phenolic acids and aldehydes on plain plates were detected by spraying with methanolic 1% $FeCl_3$ or saturated solution of o-dianisidine in acetic acid or by UV absorbance.

Procedure

The chromatograms were developed by the ascending technique. The ascent of solvent was 8-11 cm. R_f values were determined using the arithmetic mean of 2-4 runs. R_f values on impregnated plates were compared with those obtained on plain plates and the parameter $R_i = (R_f \text{ impregnated} - R_f \text{ plain})$ was calculated.

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RESULTS AND DISCUSSION

Phenolic acids and aldehydes were chromatographed on impregnated silica gel plates in different solvent systems, chosen on the basis of the published data. Some of the recomended solvent mixtures were modified by varying the component ratios. The R_f values obtained are presented in Table 1. From these data it is apparent that all substances can be separated by using suitable solvents.

In non-polar and medium-polar solvent systems phenolic acids hardly move, whereas aldehydes show appreciable mobility, especially Sal; Sac is the only acid having relatively higher R_f's in rather non-polar solvents (IV-VI). In solvent systems containing acetic acid all the compounds tested show increased mobility which is proportional to the amount of acid. The weakly basic solvent system is the only one in which Sal moves rather slowly in comparison with other solvents.

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TABLE 1

R_f Values of Phenolic Acids and Aldehydes on Fe-(III) Impregnated Silica Gel Thin Layers

Compound	R_f/Solvent System										
	I	II	III	IV	V	VI	VII	VIII	IX	X	XI
p-HPhAcac	0.85	0.76	0.59	0.24	0.00	0.03	0.35	0.84	0.85	0.52	0.41
m-HBac	0.89	0.81	0.62	0.28	0.00	0.05	0.35	0.89	0.85	0.57	0.47
p-HBac	0.86	0.81	0.62	0.31	0.00	0.03	0.40	0.89	0.85	0.53	0.38
Sac	0.79	0.80	0.72	0.42	0.00	0.20	0.72	0.98	0.95	0.74	0.60
Syrac	0.79	0.67	0.46	0.21	0.00	0.09	0.67	0.98	0.95	0.54	0.23
Vac	0.85	0.76	0.59	0.26	0.00	0.06	0.73	0.95	0.95	0.57	0.30
p-HBal	0.75	0.69	0.67	0.47	0.10	0.14	0.54	0.87	1.00	0.60	0.68
Sal	0.95	1.00	0.81	1.00	0.72	0.94	0.87	1.00	1.00	0.89	0.51
Val	0.91	1.00	0.67	1.00	0.17	0.30	0.74	0.94	1.00	0.67	0.57

TABLE 2

R_i Values of Phenolic Acids and Aldehydes in Different Solvent Systems

Phenolic acid/ aldehyde	Solvent System					
	I	II	III	IV	V	VI-X	XI
p-HPhAcac	+0.25	+0.30	+0.25	+0.09	-	-	-
m-HBac	+0.14	+0.22	+0.33	+0.12	-	-	+0.09
p-HBac	+0.07	+0.19	+0.19	+0.07	-	-	-
Sac	+0.37	+0.38	+0.49	+0.17	-	-	-
Syrac	+0.19	+0.21	+0.27	+0.06	-	-	-
Vac	+0.18	+0.20	+0.24	+0.10	-	-	-
p-HBal	-	-0.03	-0.02	-0.02	-	-	-
Sal	+0.08	-	+0.10	-	-0.15	-	-0.17
Val	+0.07	-0.03	+0.14	-	-0.12	-	+0.07

- no or very small differences

The R_i values of phenolic acids and aldehydes are presented in Table 2. It can be seen that all phenolic acids have positive R_i in neutral solvents (except when they do not move at all), the ortho-hydroxyphenolic acid (Sac) having the largest value i.e. it moves faster on impregnated layers. This may possibly be explained as a consequence of masked silanol groups of silica gel by the Fe(III)-impregnation or by better solubility of the Fe(III)-complexes in the solvent system applied. The largest R_i values are obtained in solvent systems I-III ; increasing the amount of benzene (IV,V) causes retardation of all the compounds and R_i values become smaller. In solvent systems VI-X there are no significant differences in the behaviour of acids and aldehydes on plain and impregnated plates.

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To explain the observed behaviour of the hydroxy acids or aldehydes on impregnated layers in different solvent systems, different interactions would have to be taken into account : those between impregnant and silica gel, between silica gel and the substances and between the impregnant and the substances, and all this depending on the solvent. Concerning the interaction of the impregnant and the silica gel, it is known that the composition and structure of iron(III)hydr/oxyde precipitates depend on many parameters (pH, temperature, Fe(III) concentration, time of aging etc) (17).

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