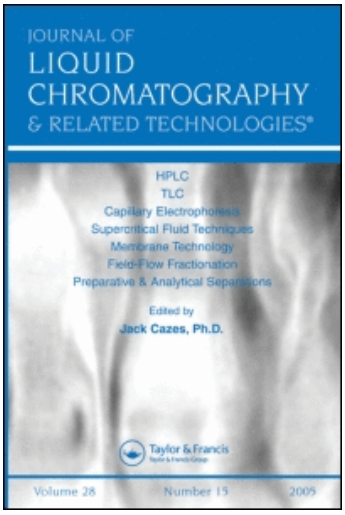

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Chromatographic Studies of 34 Organic Acids on Papers Impregnated with Hydroxides of Aluminium and Cadmium

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Chromatographic Studies of 34 Organic Acids on Papers Impregnated with Hydroxides of Aluminium and Cadmium.

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SUMMARY

Papers impregnated with aluminium hydroxide and cadmium hydroxide have been used for the chromatographic separation of organic acids exist in various biological materials, soil and water. The following important separations: cinnamic acid from hippuric acid; benzoic and m-nitrobenzoic acids from gallic, β-naphthalene acetic, β-naphthoxy acetic, phthalic, quinic and salicylic acids; and salicylic acid from citric, cis-aconitic, malic, quinic, tartaric and trans-aconitic acids can be achieved in common electrolytes ($Cd(NO_3)_2$, KI, NaCl, NH_4Cl) solution.

Hydroxides[1] show amphoteric behaviour i.e. they may exchange either cations or anions depending upon the pH

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of the solution, and this may be shown by the following ionic equilibria.

$$Al(OH)_2^+ + OH^- \rightleftharpoons Al(OH)_3 \rightleftharpoons AlO(OH)_2^- + H^+$$

Due to ion exchange behaviour and stability in aqueous solutions/organic solvents these materials have been used for the separation of anions as well as cations. However they have not been tested for the separation of organic acids so far. Therefore, in continuation to our previous work[2,3,4] now an attempt has been made to test the separation potential of papers impregnated with hydroxides of aluminium and cadmium for organic acids. As a result many binary separations have been achieved in ethanolic solution of electrolytes.

Experimental

Material used: Whatman No: 1 chromatographic paper strips (15 x 3.5 cm) and glass jar (20 x 5 cm) were used for chromatography. All chemicals used were of analytical grade.

Aqueous/ethanolic solution (1%) or saturated solution of acids was used.

Preparation of Impregnated Paper: Paper strips were impregnated in 1M sodium hydroxide solution for 30 sec. The excess reagent was removed by placing the strips over a filter paper sheet and allowing to dry at room temperature. The strips were then immersed in 1M cadmium nitrate

solution. The excess solution was drained off and the strips were placed over a filter paper sheet. Then strips were dried at 100°C, washed with distilled water three times to remove the excess reagent and dried again at 100°C. The strips were impregnated with $Al(OH)_3$ by the same procedure given above and 1M aluminium nitrate solution in the place of cadmium nitrate was used.

Spotting of Test Solution: Test solution was spotted on the papers with the help of a fine capillary. For tailing acids the front limit (R II) and the rear limit (RT) were measured while for other Rf is taken as usual.

Test Solutions and Their Detection: The acids on the strips were detected by the reported procedure summarized below:

(1) Ascorbic and (2) Uric acids are detected by ammonical silver nitrate; (3) Gallic acid is detected by ammonium vanadate; (4) Adipic, (5) Barbituric, (6) Benzoic, (7) Cinnamic, (8) Citric, (9) Cis-aconitic, (10) Fumaric, (11) Hippuric, (12) α-Ketoglutaric, (13) Maleic, (14) Malic, (15) Malonic, (16) β-Naphthalene acetic, (17) β-Naphoxy acetic, (18) m-Nitrobenzoic, (19) Nicotinic, (20) Oxalic, (21) Oxaloacetic, (22) Phthalic, (23) Quinic, (24) Salicylic, (25) Succinic, (26) Sulphamic, (27) Tartaric and (28) Trans-aconitic acids are detected by bromophenol blue; (29) Alanine, (30) Arginine HCl, (31) l-Aspartic, (32) l-cystine, (33) Glutamic and (34) Glycine acids are detected by ninhydrin; Aqueous alkaline bromophenol blue

was used for the location of acids on cadmium hydroxide paper while ethanolic alkaline bromophenol blue was used for aluminium hydroxide papers.

Discussion

The chromatographic behaviour of 34 organic acids has been studied in fifteen solvent-systems: Acetone; aqueous solutions of ammonium chloride, cadmium nitrate, potassium iodide and sodium chloride; benzene, carbon tetrachloride; chloroform; distilled water; ethyl alcohol; ethanolic solutions of ammonium chloride, cadmium nitrate, potassium iodide and sodium chloride; and propanol. In distilled water and aqueous solutions of electrolytes, acids spread over entire strips. In benzene, carbontetra chloride, chloroform and propanol acids do not move. Thus these solvent-systems can not be used for separating acids on strips impregnated with hydroxides of aluminium and cadmium. However, acetone, ethyl alcohol and ethanolic solution of common electrolytes have been proved to be good solvent-systems. Tables I,II and III show that these papers can be used to separate acids present in various biological mixtures. For example uric, oxalic, nicotinic, citric etc. exist in blood and uric acid can be separated from nicotinic acid (Table II). Similarly benzoic, malic, citric, oxalic, succinic, aconitic, α-ketoglutaric etc. exist in orange juices and benzoic acid can be separated from rest of the acids (Table I). Acids used as plant growth regulators (β-napthalene acetic and β-naphthoxy

Table I : Separation Achieved on Papers Impregnated with $Cd(OH)_2$

Sl.No.	Acids	Separated from	Solvent
1.	Benzoic (0.9); Cinnamic (1); and m-Nitrobenzoic (1).	Adipic (0-1.5); Alanine (0); Arginine HCl (0); Ascorbic (0-3.6); l-Aspartic (0); Barbituric (0); Cis-aconitic (0-1.4); Citric (0); l-Cystine (0); Fumaric (0); Gallic (0-3.9); Glutamic (0); Hippuric (0-1.5); α-Ketoglutaric (0); Maleic (0-4.4); Malic (0); Malonic (0-1.5); β-Napthalene acetic (0), β-Naphthoxy acetic (0-2.8); Nicotinic (0); Oxalic (0); Oxaloacetic (0); Phthalic (0); Quinic (0); Salicylic (0); Succinic (0-4); Sulphamic(0); Tartaric (0); Trans-aconitic (0-2) and Uric (0).	Acetone
2.	Benzoic (0.75-0.9); cinnamic (0.73-1); m-Nitrobenzoic(0.8-2) and Salicylic (0.73-0.8).	Adipic (0-5); Alanine (0-4.3); Arginine HCl (0-2.5); l-Aspartic (0-4); Barbituric (0); Cis-aconitic (0-5.5); Citric (0); l-Cystine (0); Fumaric (0-2); Glutamic(0-4.5); Glycine (0-2.4); Maleic (0-5.5); Malic (0-4); β-Naphthalene acetic (0-5.2); Nicotinic(0-5.6); Oxalic (0); Oxaloacetic (0-2.2); Phthalic(0-4.5); Quinic (0-2.2); Sulphamic (0-3.2); Tartaric (0); Trans-aconitic (0-2.5) and Uric (0).	Ethanol; Ethanolic $Cd(NO_3)_2$ (1%) Ethanolic KI (1%); Ethanolic NaCl (1%) and Ethanolic NH_4Cl (1%).

Table II : Separations Achieved on Papers Impregnated with $Al(OH)_3$

Sl.No.	Acids	Separated from	Solvent
1.	Benzoic (1); Cinnamic (1); m-Nitrobenzoic (1) and Salicylic (1).	Adipic (0); Alanine (0); l-Aspartic (0); Ascorbic (0); Arginine - HCl (0); Barbituric (0); Cis-aconitic (0); Citric (0); Hippuric (0); α-Ketoglutaric (0); Maleic (0); Malic (0); Malonic (0); Nicotinic (0); Quinic (0); Sulphamic (0); Trans-aconitic (0) and Uric (0).	Acetone
2.	Adipic (1); Benzoic (1); Cinnamic (1); Fumaric (0.85); Malonic (0.85); Nicotinic (0.85); m-Nitrobenzoic (1); Phthalic(0.85); and Salicylic (0.9).	Arginine HCl*(0-3); l-Cystine*(0);Glutamic* (0-3.6); Glycine*(0-2.4); and Uric (0-2.2).	Ethanol or Ethanolic NaCl (1%)
3.	Adipic (1); Fumaric (0.85); Nicotinic (0.85); m-Nitrobenzoic (1); Phthalic(0.85); and Salicylic (0.9).	l-Cystine (0); Glycine (0-3); and Uric (0-2.8).	Ethanolic $Cd(NO_3)_2$ (1%)
4.	Adipic (1); Benzoic (1); Cinnamic (1); Fumaric(0.85); Malonic (0.85);Nicotinic(0.85); m-Nitrobenzoic (1);Phthalic (0.85); and Salicylic (0.9).	Alanine (0-6); l-Cystine (0); Glutamic(0); Glycine (0); and Uric (0).	Ethanolic KI (1%)
5.	Adipic (1); Benzoic (1); Cinnamic(1); Fumaric (0.85); Malonic (0.85); Nicotinic (0.85); m-Nitrobenzoic (1); Phthalic (0.85); and Salicylic(0.9).	l-Cystine (0); Glycine (0-7.2) and Uric (0-2.8).	Ethanolic NH_4Cl (1%).

Separation marked with asterisk (*) were achieved 90%.

Table III : Some of the Important Separations Achieved

Sl.No.	Acids	Separated from	Paper Impregnated with	System
1.	Among Unsaturated Acids: Cinnamic	Hippuric	$Cd(OH)_2$ or $Al(OH)_3$	Acetone
2.	Among Aromatic Acids: Benzoic and m-Nitro-benzoic acids.	Gallic; α-Naphthalene acetic; β-Naphthoxy acetic; Phthalic; Quinic and Salicylic.	$Cd(OH)_2$	Acetone, Ethanol, Ethanolic $Cd(NO_3)_2$ (1%), Ethanolic KI (1%), Ethanolic NaCl (1%), and Ethanolic NH_4Cl (1%).
3.	Among Hydroxy acids: (a) Salicylic	Cis-aconitic; Citric; Malic; Quinic; Tartaric and Trans-aconitic.	$Cd(OH)_2$	Ethanol, Ethanolic $Cd(NO_3)_2$ (1%), Ethanolic KI (1%), Ethanolic NaCl (1%) and Ethanolic NH_4Cl (1%).
	(b) Salicylic	Cis-aconitic; Citric; Malic; Quinic, Tartaric and Trans-aconitic.	$Al(OH)_3$	Acetone

acetic acids) can be separated from acid (benzoic, cinnamic) used as defoliants (Table II). Generally formic, acetic, succinic, lactic, fumaric, malic, benzoic etc. are present in aerobic soil and benzoic acid can be separated from rest of the acids (Table II). These impregnated papers can also be used for the difficult separations i.e. the separation of similar acids (Table III).

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