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Polyamide Mixed Layer Chromatography of Food Dyes

Hong-Yen Hsu*, Hung-Cheh Chiang** and Pi-Chu Yang**

*Bristol Research Laboratory of Taiwan

**School of Pharmacy, Taipei Medical College

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The thin-layer chromatography of food dyes has been studied by numerous investigators, The separation of synthetic food dyes by thin layers of cellulose[1], starch[2], silica gel[3], aluminum oxide[4], and polyamide[5,6] has been reported. Recently, a better separation of red food dyes on polyamide-silica gel mixed layer and yellow food dyes on polyamide-kieselguhr mixed layer were obtained by Chiang[7,8]. Therefore, further modification of this method was tried. In this studies, the separation of seven kinds of orange, green and blue food dyes by mixed polyamide-silica gel and polyamide-kieselguhr thin layer chromatography were described. For comparison, thin layer chromatography on only polyamide, kieselguhr and silica gel is also described.

EXPERIMENTAL

Materials. The polyamide chip was Nylon 6, type 1022B, of UBE Industrial Ltd. (Osaka, Japan). The solvents were reagent grade of Wako Pure Chemical Industries, Ltd. (Osaka, Japan).

Preparation of polyamide-silica gel mixed layer. Eight grams of polyamide chip were dissolved in 100ml of 90% formic acid. After standing for overnight, a homogeneous solution was obtained, then 52g of silica gel G(E. Merck) were added. Three hundred ml of above mentioned solution were poured into a dish (15×20×2.5cm), and a glass plate (12×14×0.1cm) was dipped into it. Both sides of the glass were covered homogeneously. The glass was hung for two min over the dish to let the excess solution drain off. It was then air dried for 3h. and heated at 100°C for 30 min.

Preparation of polyamide-kieselguhr mixed layer. Before proceeding as described in the previous method, 10g of polyamide was dissolved; then 50g of Kieselguhr G(E. Merck) were added.

Preparation of polyamide layer. Dissolve 20g of polyamide in 90ml of 90% formic acid, then 10ml of distilled water were added. After stirring, a homogeneous solution was obtained. The other steps are like the method described in the preparation of polyamide-silica gel mixed layer, but without adding silica gel G.

Preparation of silica gel layer. Dilute slurries of silica gel G (45g to 120ml of water) were sprayed at 1.5kg/cm^2 pressure from a distance of 20cm onto 8 sheets of glass plate (12×14cm) in a horizontal position, then dried at 100° for 30 min. The thickness of layers were about 250μ.

Preparation of kieselguhr layer. The above mentioned method was employed but useing kieselguhr G instead of Silica gel G.

Chromatographic procedure. A 0.5% solution of these food dyes was applied to the starting line 1.5cm from the bottom of the layer, and the plate was developed by ascending techniques,

The chamber had been equilibrated with the respective solvent for 30 min before use.

RESULTS AND DISCUSSION

R_f values of polyamide–silica gel mixed layers, silica gel layers and polyamide layers with two solvent systems are given in Table I. Also these of polyamide–kieselguhr layers, kieselguhr layers, and polyamide layers with two other solvent systems are given in Table II.

It has been found that the best results is obtained on the polyamide–kieselguhr layer with the solvent system IV. The spots on silica gel layers are rather tailing and diffusing and on kiselguhr layers show higher R_f values. In the mixed layer, polyamide serves as a strong binder and make the layers very durable and easy to handle. A 10cm ascent from the origin is more rapid using the mixed layers than using polyamide layers. Brilliant Blue and Indigocarmine are rather difficult to separate because of the close similarity of their structure.

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TABLE I

Solvent I: methanol–5% NH_4Cl solution–5% sodium citrate solution–glacial acetic acid (25:11:20:0.4);

Solvent II: 28% ammonia water–5% sodium citrate solution (2:1).

P–S, R_f value obtained on polyamide–silica gel layer; S, silica gel layer; p, polyamide layer.

No. Dyes	Solvent I			Solvent II		
	P–S	S	P	P–S	S	P
1 Orange I (Orange no. 1)	0.12	0.82	0.04	0.14	0.97	0.20
2 Acid Violet 6B (Violet no. 1)	0.19	0.38	0.13	0.01	0.09	0.02
3 Guinea Green B (Green no. 1)	0.34	0.49	0.31[d] 0.24	0.00	0.12[t]	0.05
4 Light Green SF Yellowish (Green no. 2)	0.31[d] 0.53	0.85[t]	0.43	0.12[t]	0.71[t]	0.12
5 Fast green FCF (Green no. 3)	0.48	0.71	0.36	0.22	0.58[t]	0.59

6 Brilliant Blue FCF (Blue no. 1)	0.54	0.64	0.51	0.14[t]	0.66[t]	0.21
7 Indigo Carmine	0.54	0.64	0.51	0.07[t]	0.56[t]	0.17
Time required(min)[a]	130	30	460	50	15	260

[a] Time required to asend 10cm from origin., [d] Double spots,
[t] Tailing, ( ) Chinese food color no.

TABLE II

Solvent III: ethanol–5% NH_4Cl solution (25:4); Solvent IV: methanol–5% NH_4Cl solution (38:23). P–K, R_f value obtained on polyamide–kieselguhr layer; K, kieselguhr layer; P, polyamide layer.

No. Dyes	Solvent III			Solvent IV		
	P–K	K	P	P–K	K	P
1 Orange I	0.49	0.97	0.53	0.29	0.97	0.18
2 Acid Violet 6B	0.80	0.95	0.86	0.68	0.97	0.52
3 Guinea Green B	0.80	0.95	0.85	0.75	0.97	0.69
4 Light Green SF Yellowish	0.37	0.84	0.83	0.76[d] 0.58	0.97	0.76
5 Fast Green FCF	0.65	0.91	0.84	0.78	0.97	0.72
6 Brilliant Blue FCF	0.76	0.91	0.86	0.81	0.97	0.79
7 Indigo Caramine	0.75	0.90	0.86	0.85	0.96	0.79
Time required(min)[a]	120	40	960	150	40	480

中 文 摘 要

食用色素之多醯胺混合薄層分析

許鴻源 姜宏哲 楊碧珠

臺灣必治妥研究所 臺北醫學院藥學系

對七種水溶性食用色素（食用橙色一號，黃色一號，藍色一號及二號，綠色一，二及三號）應用 Polyamide-Silica gel 混合薄層，Polyamide-Kieselguhr 薄層，Silica gel 薄層，Kieselguhr 薄層，Polyamide 薄層等五種薄層分析法分別進行鑑別並檢討所得之結果。