[Reprinted from the Journal of Pharmaceutical Sciences,
Vol. 59, No. 2. February, 1970. © 1970 by the American Pharmaceutical Association]

Polyamide–Silica Gel Layer Chromatography of Yellow Food Dyes

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Abstract ☐ The separation of five yellow food dyes and three toxic yellow dyes by mixed polyamide–silica gel thin-layer is described. The method shows good separation and sharp spots. For comparison, the thin-layer chromatography (TLC) on only polyamide and on only kieselguhr is also carried out.

Keyphrases ☐ Yellow dyes—analysis ☐ Dyes, yellow—separation, identification ☐ Polyamide–silica gel chromatography—analysis

The separation of synthetic food dyes on a thin-layer of cellulose (1), silica gel (2), aluminum oxide (3), starch (4), polyamide (5), and paper chromatography (6) has been reported, but none of these techniques gave entirely satisfactory results. Recently, the separation of 11 red food dyes on polyamide (12%)–silica gel G (88%) mixed thin-layers has been successfully applied by Chiang (7). Therefore, further application of this method was tried. In this note, the separation of five yellow food dyes and three toxic yellow dyes (auramine, metanil yellow, and picric acid) by mixed polyamide–silica gel TLC is described. For comparison, the TLC of only polyamide and of only silica gel is also reported.

EXPERIMENTAL

Material—The solvents and chemicals are the reagent grade of Wako Pure Chemical Industries, Ltd., Osaka, Japan.

Preparation of Polyamide–Silica Gel Mixed Layer—Eight grams of polyamide chip (Nylon 6,[1] type 1022B of UBE Industrial Ltd., Osaka, Japan) was dissolved in 80 ml. of 90% formic acid, and then 20 ml. of distilled water was added. After gentle warming (below 40°) and stirring, a homogeneous solution was obtained; this was cooled to the room temperature and 52 g. of silica gel G (E. Merck) was added. Two hundred milliliters of the above mentioned solution was poured into a dish (14.5 × 19.5 × 2.5 cm.) and a glass plate (12 × 14 × 0.1 cm.) was dipped into it. Both sides of the glass were covered homogeneously. The glass was placed over the dish for 2 min. to let the excess solution drain back. It was then air dried for 3 hr. and heated at 100° for 30 min.

Preparation of Polyamide Layer—Twenty grams of polyamide was dissolved; then the procedure as described in the previous method, but without adding silica gel G, was followed.

Preparation of Silica Gel Layer—Dilute slurries of silica gel G (45 g. in 100 ml. of water) were sprayed at 2 kg./cm.^{3} pressure from a distance of 20 cm. onto eight sheets of glass plates (12 × 14 cm.) in a horizontal position, then dried at 100° for 30 min. The thickness of the layers was about 250 μ.

Chromatographic Procedure—One microliter of 0.3% alcoholic solution of yellow AB, yellow OB, and auramine, and 0.3% water solution of other dyes was applied to the start line 1.5 cm. 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.

Table I—Chromatographic Data

		—Solvent I^a—			—Solvent II^b—		
No.	Dyes	P–S^c	S^d	P^e	P–S	S	P
1	Naphthol yellow S	0.48	(0.95,	0.37^f)	0.10	(0.74,^{f}	0.02)
2	Yellow AB	0.14	(0.62,	0.22)	0.74	(0.97,	0.48)
3	Yellow OB	0.10	(0.52,	0.22)	0.72	(0.97,	0.45)
4	Tartrazine	0.63	(0.98,	0.85)	0.01	(0.37,^{f}	0.01)
5	Sunset yellow FCF	0.56	(0.98,	0.66)	0.14	(0.56,^{f}	0.08)
6	Metanil yellow	0.28	(0.80,	0.14)	0.47	(0.83,	0.23)
7	Auramine	0.37	(0.71,	0.36)	0.62	(0.71,	0.89)
8	Picric acid	0.40	(0.97,	0.22)	0.53	(0.84,	0.22)
Time required,^{g} (min.)		50	30	200	90	70	600

^{a} Solvent I: methanol–23% ammonium chloride solution–chloroform (30:20:1.3). ^{b} Solvent II: isobutanol–ethanol–0.45% sodium chloride solution (3:5:1). ^{c} P-S, R_f value on polyamide-silica gel mixed layer. ^{d} S, on silica gel layer. ^{e} P, on polyamide layer. ^{f} Tailing. ^{g} Time required to ascend 10 cm. from origin.

RESULTS AND DISCUSSION

R_f values obtained with two solvent systems are given in Table I. It has been found that the results obtained by the mixed polyamide–silica gel layers show better separation and sharper spots than that obtained by polyamide and silica gel layers. Also the time required to ascend 10 cm. from origin for the mixed layers is shorter than that for the polyamide layers. Separation mechanism on the mixed layers is based on the formation of hydrogen bonds between the CONH group of polyamide and the sample and adsorption or partition between the silica gel and the sample. In the mixed layer, polyamide also serves as a strong binder and makes the layer very durable and easy to handle. Also the layer did not crack or peel and could be stored easily. Both sides of the glass are independent of each other and chromatography can be performed simultaneously on both sides. The addition of a small amount of salt (about 0.05% sodium chloride or 0.4% ammonium chloride) in the solvent mixture is essential to break hydrogen bonding between the polyamide and the dyes. Oil-soluble dyes of yellow AB and yellow OB are rather difficult to separate because of the close similarity of their structures (different only in one methyl group). The content of polyamide (13.6%) in this mixed layer was above that of the previous report (12%) (7) in order to obtain a more durable layer

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ACKNOWLEDGMENTS AND ADDRESSES

Received July 14, 1969, from the *Department of Pharmacy, Taipei Medical College, Taipei, Taiwan, China.*
Accepted for publication September 15, 1969.

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[1] U. S. manufacturer: American Enka Corp.