Quality control of pigment formulations

In cooperation with the company Siegwerk one of the leading international manufacturers of printing inks for packaging and other kinds of printing material, this method was developed at the Chair of Food Science, Justus Liebig University Giessen [1].

Introduction

Printing ink formulations are complex mixtures, which consist of pigments and/or colorants, solvents, resins, and additives such as UV absorbers or plasticizers. Chromatographic methods have not been considered for substances of low solubility. However, HPTLC seems to be a promising analytical tool, due to the single use of the plate. Pigment components of poor solubility stay at the starting zone without disturbing the separated zones. Also sample preparation can be kept minimal because of the one-time use of the plate.

Sample preparation

Depending on the respective pigment formulation 5, 10 or 30 mg of each sample were dissolved in 1.0 mL tetrahydrofuran, methanol, dimethylformamide or a mixture of these solvents [1], sonicated (15 min), and centrifuged (5 min, 10000 g).

Sample application and layer

Bandwise with Automatic TLC Sampler (ATS 4) HPTLC plates silica gel 60 F_{254} (Merck), 20 × 10 cm, band length 6 mm, track distance 9.5 mm, distance from lower edge 9 mm, application distance from left edge 14 mm, application volume 1–10 µL.

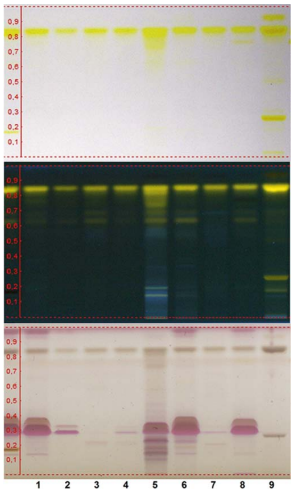
Chromatography

In the AMD 2 system using a 9-step gradient with ethyl acetate, methanol, water, and toluene with alkaline conditioning (1 N aqueous ammonia solution), AMD time 80 min, migration distance 56 mm.

Derivatization and documentation

The plate was immersed into a 10% aqueous sulfuric acid solution using the Chromatogram Immersion Device (immersion time 0 s, immersion speed 3 cm/s) and heated at 110 °C for 5 min using the TLC Plate Heater, documentation with the TLC Visualizer under UV 254 nm, 366 nm, and white light.

Note: This derivatization is now also possible with the Derivatizer.



AMD 2 chromatograms of 9 different batches of pigment yellow 12 under white light, UV 366 nm, and white light after derivatization; tracks 1 and 2 same supplier; tracks 3–9 different suppliers

Results and discussion

The developed generic method allowed the industrial quality control of pigment formulations (p. 16). There were not only differences between pigment formulations of different manufacturers but also between different batches of the same supplier. A total of 124 samples (18 different pigment formulations and up to 20 batches of the same pigment formulation) were investigated. It was possible to compare 18 pigment samples against a benchmark during one run. The analysis time for one sample was less than 5 min and the solvent consumption was below 10 mL. Due to the use of multi-detection, differences between pigment batches were detectable. Post-chromatographic derivatization with sulfuric acid reagent gave further information and showed particular differences regarding the binder and coating materials in the samples.

[1] C. Stiefel, S. Dietzel, M. Endress, G. Morlock, J. Chromatogr. A 1462 (2016) 134–145

Contact: Prof. Dr. Gertrud Morlock, Justus Liebig University Giessen, Heinrich-Buff-Ring 26–32, 35392 Giessen, Germany
Gertrud.Morlock@uni-giessen.de