Rapid differentiation of beeswaxes using EGA-MS and Py-GC/MS

[Background] Pyrolysis (Py)-GC/MS is widely used by forensic chemists to characterize and identify a wide range of materials based on the GC/MS profiles of the pyrograms. Definitive MS data can be obtained from the analysis of micrograms of material. A second feature that enhances the credibility of the data is that most samples can be analyzed directly, that is, without any sample pretreatment. This eliminates the possibility of introducing contamination and enhances data quality which ultimately elevates the credibility of the data. The third reason for using Py-GC/MS is that modern pyrolyzers are multi-mode systems: they can be configured to perform several analytical techniques, each one providing additional information about the sample. Flash pyrolysis (Py-GC/MS), evolved gas analysis (EGA-MS), and heart-cut GC/MS (HC-GC/MS), can be easily and quickly performed to characterize an unknown. This note illustrates how these techniques can be used to differentiate beeswaxes.

[Experimental] 150 µg of two different beeswaxes and a synthetic wax are used to illustrate the ease of differentiating two similar samples using EGA-MS and Py-GC/MS. The question being asked: Are the natural waxes being diluted by adding a synthetic wax? The samples are first analyzed using EGA-MS and then by Py-GC/MS (Multi-Shot Pyrolyzer: EGA/PY-3030D, Frontier Labs). A deactivated metal capillary tube (L=2.5 m, id.=0.15 mm) is used to connect the GC injector to the MS. The EGA tube, located in the GC oven is held at 300ºC. The EGA tube is replaced by a metal capillary column (Ultra ALLOY^{+}-5, L=30 m, i.d.=0.25 µm, Frontier Labs) when doing Py-GC/MS analysis. The furnace temperature profiles for the EGA and Py analysis are found in the figure captions.

[Results] EGA: Fig. 1 shows the EGA thermograms of the three samples. All three samples exhibit a series of straight-chain hydrocarbons (group A) eluting between 100 and 300ºC. The two beeswax samples also have a group of compounds (group B) eluting between 320 and 600ºC which are not present in the synthetic wax. The A/B area ratio is 0.65 for Beeswax C, and 2.3 for Beeswax J, indicating that there is a large difference in composition between these two beeswaxes. Beeswax C has more of peak group A than Beeswax J, suggesting that synthetic wax has been added to it.

Py-GC/MS: The pyrograms clearly show that the synthetic wax has a series of straight chain hydrocarbons, C24 to C36, see Fig. 2. And that the distribution of saturated hydrocarbons among these beeswaxes differs greatly. Compounds with an odd number of carbons are mostly observed in Beeswax J, while hydrocarbons with odd and even numbers of carbons are present, in almost equal amount, in Beeswax C which has a similar distribution to that of the synthetic wax. In addition, Beeswaxes C and J contain the palmitic acid esters of C24 to C36 alcohols. The results suggest that the synthetic wax probably has been added to Beeswax C. The Beeswax samples illustrate that using EGA-MS and Py-GC/MS to differentiate complex mixtures can be a simple yet definitive process.

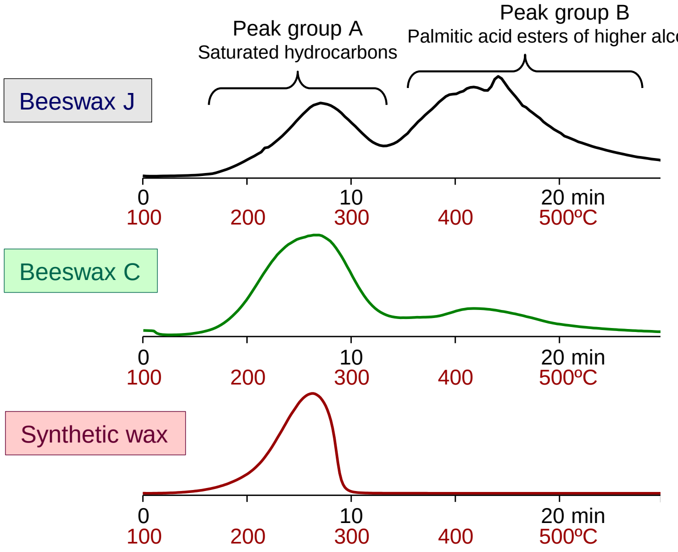

Fig. 1 EGA thermograms of three different types of waxes
Furnace temp.: 100~600ºC (20 ºC/min)

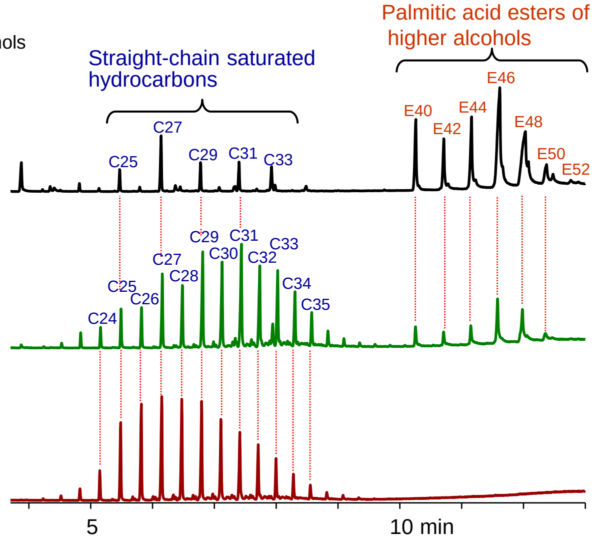

Fig. 2 Comparison of pyrograms
Furnace temp.: 600ºC, sample wt.: 150 µg

Keywords : Beeswax, Synthetic wax, Differentiation, EGA, Py-GC/MS

Products used : Multi-functional pyrolyzer, Vent-free GC/MS adapter, Deactivated metal capillary tube, UA+-5

Applications : General polymer analysis, Forensic differentiation

Related technical notes : PYA3-013E, PYA3-016E

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