Precise Compositional Analysis of Industrially-Used Natural Wax by Reactive Py-GC in the Presence of TMAH

[Background] Reactive Py-GC in the presence of an organic alkali, such as tetramethylammonium hydroxide (TMAH), has been utilized as a practical method for characterization not only of intractable condensation polymers but also of complex natural organic materials. By using this technique, precise compositional analysis of various natural organic materials can be often carried out without doing any cumbersome sample pretreatments. As an example, the reactive Py-GC technique applied to compositional analysis of an industrially-utilized natural wax is presented in this note.

[Experimental] A crude carnauba wax sample collected from the leaves of Cerifera palm tree in Brazil was used. About 30 µg of the cryo-milled wax sample was subjected to reactive Py-GC at 500 ºC in the presence of 4 µl of 25 wt% TMAH methanol solution. The column temperature was initially set at 50 ºC, secondly heated to 200 ºC at a rate of 10 ºC/min, then to 250 ºC at a rate of 5 ºC/min, and finally to 300 ºC at a rate of 10 ºC/ min.

[Results] Fig.1 shows a typical pyrogram obtained from the carnauba wax sample at 500 ºC in the presence of TMAH. This pyrogram showed a series of peaks assigned mainly to the methyl derivatives of straight-chain fatty acids (C_{16}-C_{32}), ω-hydroxy fatty acids (C_{16}-C_{30}), alcohols (C_{22}-C_{34}), and α, ω-diols (C_{22}-C_{32}), which were derived from the constituents of the wax sample through the reactive pyrolysis. Then the chemical composition of these components was successfully determined from their peak intensities observed on the pyrogram, corrected using the concept of the effective carbon number (ECN), with less than 2 % of the relative standard deviation (*n* = 3).

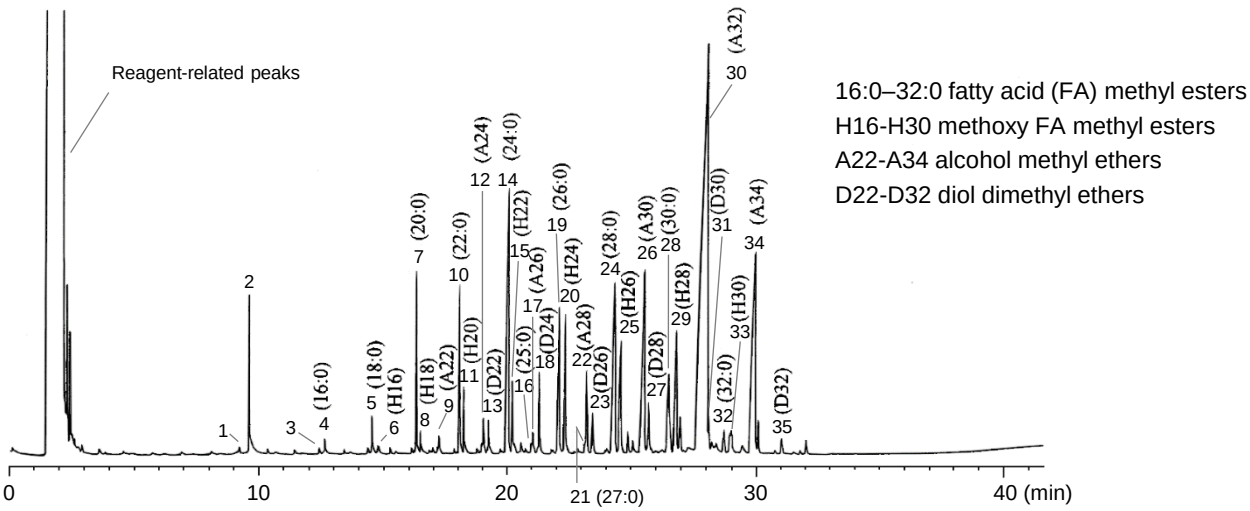

Fig. 1 Typical pyrogram of carnauba wax sample obtained in the presence of TMAH at 500 ºC.

Pyrolysis temp. : 500 ºC, GC oven temp. : 50 ºC - (10 ºC/min) - 200 ºC - (5 ºC/min) - 250 ºC - (10 ºC/min) - 300 ºC
Separation column : Ultra ALLOY+-1 (100 % dimethylpolysiloxane), Length 30 mm, 0.25 mm i.d., Film thickness 0.25 µm
Carrier gas flow : 50 mL/min, Column flow : 1.0 mL/min

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Keywords : Natural wax, Reactive Py-GC, TMAH, Compositional analysis, Effective carbon number

Products used : Multi-functional pyrolyzer, UA-1

Applications : Analysis of natural organic materials

Related technical notes : PYA2-012E

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