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Note

Rapid analysis of coffee flavour by gas chromatography using a pyrolyzer

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Gianturco[1] discussed the formation and rate of production of volatile compounds during the roasting coffee beans. As the roasting time was increased, both the number and size of the chromatographic peaks increased. This phenomenon indicates that most components of coffee aroma arise from the roasting process[1,2]. Baltes[3] has recently described phenol formation during coffee roasting.

The Curie-point principle has been widely used as a method for analyzing non-volatile materials[4,5]. First a sample is rapidly heated to a temperature that is sufficient to decompose it to volatile substances. These are then analyzed by gas chromatography (GC). Colenutt and Thorburn[6] adopted a conventional pyrolysis GC system for the analysis of volatile compounds. In their system, desorption is achieved in the pyrolysis unit, but no pyrolysis occurs.

This paper presents a simple and rapid method for analyzing the flavour of coffee grounds using a gas chromatograph equipped with a pyrolyzer. The results obtained with this method were compared with those from conventional methods, *i.e.*, headspace analysis and simultaneous distillation–extraction (SDE).

EXPERIMENTAL

Gas chromatography

A 50-m fused-silica capillary column (0.22 mm I.D.) coated with Carbowax 20M was used to separate volatiles transferred to the column through the splitter injection port. The splitting ratio was adjusted to 1:50 with carrier gas (nitrogen) at 22 p.s.i. The linear flow-rate was 42 cm/s and the column flow-rate was 2.0 ml/min. The injection port temperature was 200°C. The column temperature was increased from 80 to 200°C at 2°C/min. The gas chromatograph used was an Hitachi Model 163 equipped with a flame ionization detector heated to 250°C.

Gas chromatography–mass spectrometry (GC–MS)

An Hitachi Model M-80B mass spectrometer–gas chromatograph (Hitachi Model 663) equipped with an Hitachi Model 0101 data system was used under the following conditions: ionization voltage, 70 eV; emission current, 80 μA; ion acceleration voltage, 3100 V; ion-source temperature, 200°C.

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Curie-point pyrolyzer

Volatiles were removed from a *ca.* 10-mg sample of Colombian Arabica coffee grounds by heating at 235°C in a ferromagnetic sample support in the pyrolyzer. The total heating time was 3 s. The pyrolyzer can easily be connected to the original injection port of the gas chromatograph or the gas chromatograph–mass spectrometer unit.

Headspace sampling

A 150-g amount of coffee grounds was placed in a 500-ml headspace vessel equipped with an Allihn condenser. The trapping column (170 mm × 6.4 mm I.D.) containing *ca.* 2 g of Porapak Q (60–80 mesh) was fixed to the top end of the Allihn condenser. A 400-ml volume of water at 70°C was poured into the vessel. The extraction of the coffee grounds and the trapping of headspace volatiles were carried out in a water-bath at 70°C stirred at a constant rate. The headspace vessel was purged for 2.5 h by the passage of nitrogen at a flow-rate of 240 ml/min. The entrapment column was reversed[7] and heated at 180°C for 0.5 h. The volatiles collected in the trapping were then backflushed to a glass trap chilled with solid carbon dioxide[7]. The trap was rinsed with 30 μl of diethyl ether. This permitted efficient transfer of the trapped volatiles into a vial for storage until analysis.

Simultaneous distillation–extraction (SDE)

A Nickerson-Likens extractor as modified by Schultz *et al.*[8] was used. A 400-g amount of coffee grounds in 1 l of water was added to a 2-l flask, and 150 ml of dichloromethane were added to a 300-ml flask. The distillation head was attached and then both flasks were heated to boiling. The distillation was carried out for 6 h at atmospheric pressure. The extract was dried over anhydrous sodium sulphate for 12 h and then evaporated to *ca.* 0.5 ml for instrumental analysis.

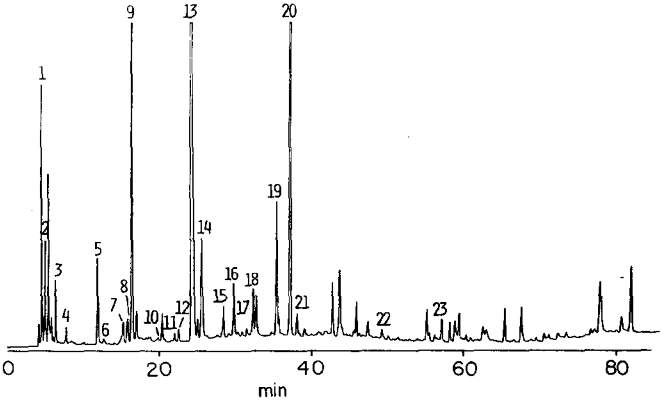

Fig. 1. Gas chromatogram of the volatiles from coffee grounds desorbed in the Curie-point pyrolyzer at 235°C. Peaks: 1 = acetaldehyde; 2 = acetone; 3 = 2,3-butanedione; 4 = 2,3-pentanedione; 5 = pyridine; 6 = pyrazine; 7 = 2-methylpyrazine; 8 = acetoin (3-hydroxy-2-butanone); 9 = acetol (2-ketopropyl alcohol); 10 = 2,3-dimethylpyrazine; 11 = 2-ethyl-6-methylpyrazine; 12 = 2-ethyl-5-methylpyrazine; 13 = acetic acid; 14 = furfural; 15 = 2-acetylfuran; 16 = propionic acid; 17 = furfuryl acetate; 18 = 5-methylfurfural; 19 = γ-butyrolactone; 20 = furfuryl alcohol; 21 = isovaleric acid; 22 = guaiacol; 23 = phenol.

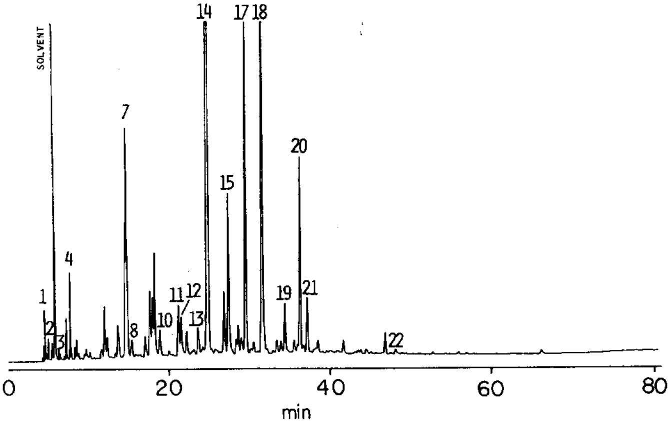

Fig. 2. Gas chromatogram of the headspace volatiles from a coffee brew. See Fig. 1 for peak identification.

RESULTS AND DISCUSSION

Fig. 1 shows a gas chromatogram of the volatiles from coffee grounds obtained in the Curie-point pyrolyzer at 235°C. When the pyrolyzer was used at different heating temperatures ranging from 170 to 235°C (total heating time per sample: 3 s), the qualitative composition of the volatiles from the coffee grounds did not change. The effluent gas from the outlet of the pyrolyzer had a coffee-like odour. Since the above heating conditions are much milder than the roasting conditions used for coffee beans (180–215°C for 15–17 min or 260°C for 5 min)[2], the influence of the pyrolysis of the coffee grounds may be neglected in the analysis of the volatiles. The mixture of compounds in Fig. 1 was analyzed by GC–MS and identified by comparing MS and retention time data with those of individual compounds.

The headspace analysis (adsorbent trap) (Fig. 2) and SDE methods (Fig. 3)

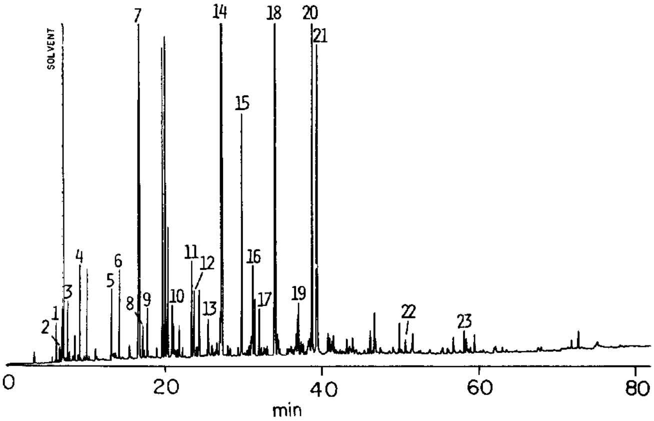

Fig. 3. Gas chromatogram of the volatiles obtained from coffee grounds by SDE. See Fig. 1 for peak identification.

were chosen in order to compare the results for the qualitative analysis of volatile compounds from coffee grounds. The dependence of the headspace method on compound volatility is obvious. The peak area % drops off quite quickly as the vapour pressure of the compound decreases.

The SDE method gave very good recoveries[9]. Its disadvantages include the problems of the solvent front overlapping with low boiling compounds and artifact formation due to thermally induced change. Changing to SDE had a very negative effect on the recovery of polar substances, *e.g.*, acetic acid (peak 13).

The pyrolyzer method offers a very simple and efficient means of isolating flavours from a very small amount of coffee grounds. It is possible to analyze both low boiling compounds, *e.g.*, acetaldehyde (peak 1) and acetone (2) and high boiling compounds, *e.g.*, guaiacol (22) and phenol (23) using the pyrolyzer method, as shown in Fig. 1. Consequently, this method is very simple and useful for the analysis of flavour compounds in food.

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