COMMUNICATIONS

Comparison of Batchwise and Continuous Steam Distillation-Solvent Extraction Recovery of Volatiles from Oleoresin Capsicum, African Type (*Capsicum frutescens*)

Batchwise and continuous steam distillation–liquid-liquid extraction methods were used to recover volatiles from the oleoresin of capsicum, African type (*Capsicum frutescens*). In the continuous system, with the oleoresin pot temperature maintained at ca. 225 °C, a higher yield of volatile material was obtained than with the pot at ca. 150 °C. The increased yield at the higher temperature was found to be due largely to higher yields of free acids, predominantly acetic acid, and methyl esters of lauric, palmitic, and stearic acids—compounds which are not distilled over in appreciable quantities with the batch steam distillation system. With the oleoresin pot at the lower temperature, the mixture of volatiles obtained was similar in composition to that obtained in the batch method.

A system of recovering volatiles from fats and oils was described by Teranishi et al. (1977) in which steam distillation and liquid–liquid extraction are combined in a continuous manner. A batchwise simultaneous steam distillation–extraction apparatus was described by Likens and Nikerson (1964). A modified version of this latter apparatus was evaluated by Schultz et al. (1977), using a synthetic mixture. The continuous method was used to obtain volatiles from fats and oils, but no data have been presented as to the effectiveness of such a system. This paper gives a comparison of continuous and batchwise methods as applied to a practical problem, the isolation of volatiles from capsicum oleoresin.

Volatiles from Californian green bell peppers *Capsicum annuum*) have been analyzed by Buttery et al. (1969), and the characterisitic odor has been attributed to 2-methoxy-3-isobutylpyrazine. Hot peppers (*Capsicum frutescens*) are used to enhance the flavor of many foods. Pungency of the capsaicinoid components have been known for many years, and analytical methods for these compounds have been described by Masada et al. (1971), Todd et al. (1977), Sticher et al. (1978), and others. Volatiles of Tabasco peppers have been studied by Haymon and Aurand (1971); no single component was correlated with Tabasco aroma. Jalapeno pepper volatiles have been studied by Huffman et al. (1978), and fresh Jalapeno pepper flavor has been attributed to 2-methoxy-3-isobutylpyrazine. This report of the comparison of the continuous and batchwise steam distillation–liquid-liquid extraction methods evolved from our attempts to isolate sufficient volatile materials to characterize hot pepper odor.

EXPERIMENTAL SECTION

Materials. Oleoresin of capsicum, African type (*Capsicum frutescens*), was purchased from Kalamazoo Spice Company, Kalamazoo, MI. Distilled water was used in the steam generators. Ethyl ether, reagent grade, was purchased from J. T. Baker Chemical Co., Phillipsburg, NJ. Pentane and heptane were purchased from Burdick and Jackson Laboratories, Inc., Muskegon, MI.

Equipment. The batchwise equipment has been previously described by Schultz et al. (1977), and the continuous system also has been described previously by Teranishi et al. (1977). In the continuous system, the overflow tube from the oleoresin pot had to be enlarged from $^1/_4$ in. o.d. to $^1/_2$ in. o.d. and was wrapped with a heating tape to prevent solidification of the oleoresin. With this modification the overflow tube did not become plugged, and the continuous system worked smoothly with the oleoresin.

Gas chromatographs used were Hewlett/Packard Model 5830A or 5840A series. Pyrex glass and stainless steel capillary open tubular columns were made in our laboratory.

Component identifications were based on gas chromatographic–mass spectrometric (GC–MS) data obtained with a quadrupole mass spectrometer (Electronic Associates Quad 300 mass filter, Finnigan Corporation 3000–1B electronics). The GC–MS identifications were further checked by GC retention time comparisons with authentic samples.

Procedure. Details of the batchwise operation have been described previously by Schultz et al. (1977) and of the continuous operation by Teranishi et al. (1977). Organic acids were isolated from the extracted distillates with 5% aqueous sodium bicarbonate solution. Acids were isolated by acidification of the bicarbonate solution and extraction with ethyl ether. The acids were methylated with diazomethane, and the methyl esters were analyzed by GC–MS. Neutrals were also analyzed by GC–MS.

RESULTS AND DISCUSSION

The major difference between the batchwise and the continuous systems is that in the continuous system there is an additional experimental parameter, the temperature of the pot in which spent oil, oleoresin in this case, is kept before it goes out the overflow (see Teranishi et al., 1977). In the case of the batchwise method, oil temperature cannot exceed approximately 100 °C since it is intimately mixed with water in the pot from which volatiles and steam are generated. In the case of the continuous system, the oil pot must be maintained at a temperature of at least 100 °C in order to prevent condensation of the steam to liquid water, but the oil pot temperature can be raised easily to a 200–250 °C range. Steam, generated in another pot, sweeps through the oil pot and carries volatiles up the spinning band column.

A representative batchwise recovery of volatiles is as follows: From 1.48 kg of oleoresin of capsicum, African

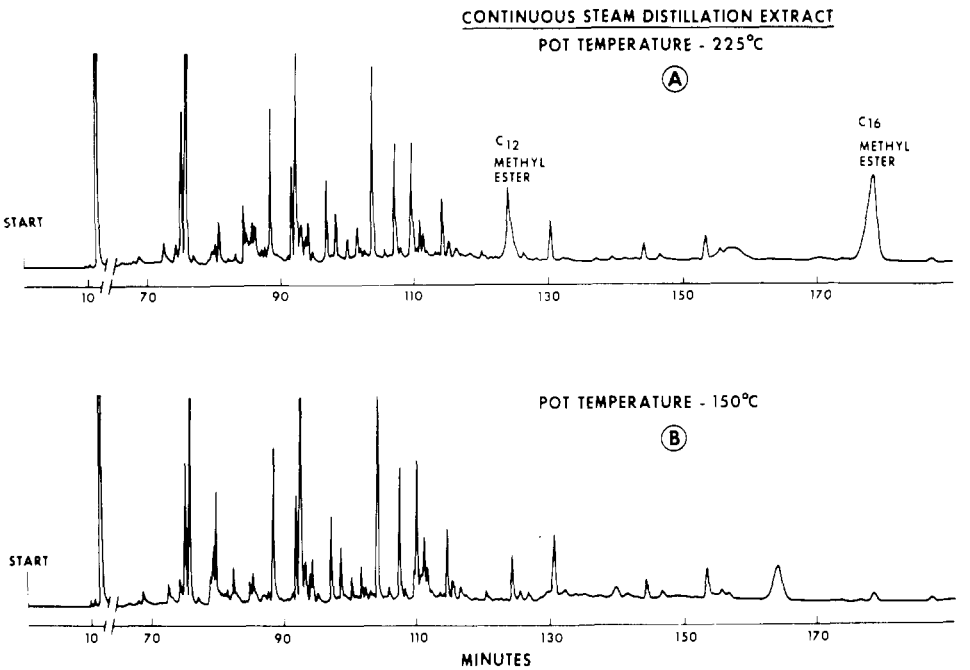

Figure 1. Gas chromatograms of volatiles from oleoresin capsicum. Column: 150 m, 0.75 mm i.d. stainless steel open tubular column coated with methyl silicone oil, SF 96 (50), containing 5% Igepal CO-880. Temperature program: initial temperature, 60 °C; rate of 1 °C/min was started at time of injection; final temperature, 170 °C until chromatogram finished. Chromatogram A: volatiles from continuous steam distillation-extraction with the oleoresin pot at ca. 225 °C. Chromatogram B: volatiles from continuous steam distillation-extraction with the oleoresin pot at ca. 150 °C.

type (*Capsicum frutescens*), the amount of 2.49 g of volatiles (0.168% yield) was obtained by a batchwise steam distillation-extraction at atmospheric pressure, followed by solvent stripping. A gas chromatographic analysis showed that the concentrated extract still contained 10% solvent. Thus, the corrected yield was 0.151%.

A representative run employing the continuous extraction apparatus produced the following: From 2.90 kg of oleoresin, the amount of 5.46 g (0.188% yield) was obtained with the oleoresin pot at 180–200 °C. A gas chromatographic analysis showed that the solvent-stripped extract had 5% solvent remaining. Thus, the corrected yield was 0.179%. With the oleoresin pot maintained at about 150 °C, the volatiles obtained were similar qualitatively and quantitatively (approximately 0.15% yield) to those obtained by the batchwise method.

Two gas chromatograms, A and B, are shown in Figure 1. Chromatogram A is that of volatiles obtained with the oleoresin pot temperature maintained at ca. 225 °C. The last two peaks in chromatogram A represent methyl laurate and methyl palmitate. Methyl stearate is not shown in this chromatogram because its retention time is about 5 h under these conditions. Chromatogram B is that of volatiles obtained with the oleoresin pot temperature maintained at about 150 °C. Chromatograms of volatiles obtained by the batchwise method are very similar to chromatogram B.

Volatiles from the continuous distillation-extraction method, with the oleoresin pot temperature at ca. 225 °C, were chromatographed with a glass capillary open tubular column. Free fatty acids were observed. The acids were extracted with a sodium bicarbonate solution, regenerated back to the free acids, extracted with ethyl ether, and converted to methyl esters with diazomethane. GC–MS analyses showed that the predominant methyl ester was that of acetic acid, with traces of butyric, isobutyric, valeric, isovaleric, hexanoic, and isohexanoic methyl esters.

Thus, this study shows that by maintaining the oil pot, or oleoresin in this case, at higher temperatures, compounds such as free fatty acids and higher molecular weight methyl esters can be isolated with the continuous system, compounds which are not isolated in appreciable amounts by the batchwise system.

Sensory evaluation of volatiles isolated from hot peppers will be reported upon completion.

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