Coriander Spice Oil: Effects of Fruit Crushing and Distillation Time on Yield and Composition

Bruce M. Smallfield,† John W. van Klink,‡ Nigel B. Perry,*,‡ and Kenneth G. Dodds§

New Zealand Institute for Crop and Food Research Ltd., and New Zealand Pastoral Agriculture Research Ltd., Invermay Agriculture Centre, Private Bag 50034, Mosgiel, New Zealand, and Plant Extracts Research Unit, New Zealand Institute for Crop and Food Research Ltd, University of Otago, P.O. Box 56, Dunedin, New Zealand

Crushing intensity and distillation time were evaluated for their effects on the oil yield and composition of steam-distilled essential oil from fruits of *Coriandrum sativum* var. *microcarpum* L. A comparison of oils produced by laboratory- and pilot-scale stills showed that the two still types gave comparable yields and oil composition. The laboratory still was then used to compare oil yields and compositions from fruits crushed at three different intensities, at intervals during a distillation period of 60 min. Both crushing intensity and distillation time had significant ($P < 0.05$) effects on the yield and composition of the oil. The maximum oil yield was less from the light-crushed fruits, but the rate of oil recovery was significantly ($P < 0.05$) higher. From the light-crushed fruits, 95% of the maximum yield was extracted in 22.5 min compared with 32 and 39 min for the standard and heavy-crushed fruits, respectively. The effect of crushing intensity on oil composition was most pronounced on the low-boiling-point α-pinene and on the higher-boiling-point geranyl acetate. Crushing had little effect on linalool content, but distillation time could be manipulated to alter the linalool content of the oil.

Keywords: *Coriander; Coriandrum sativum; essential oil composition; yield; steam distillation*

INTRODUCTION

Coriander, *Coriandrum sativum* L. (family Apiaceae or Umbelliferae), is one of the essential oil plants being investigated by the New Zealand Institute for Crop and Food Research Ltd as a new crop for New Zealand. The main objective of this research was to assess the feasibility of commercial production of coriander spice oil (also called fruit or seed oil). In addition, we have reported some results on the production of coriander herb oil (*1*) and near-critical CO_2 extraction of coriander seed (*2*).

Coriander spice oil is steam-distilled from mature, dried coriander fruits (*3*). The commercial value of a coriander spice oil depends on its physical properties, chemical composition, and aroma. The current International Standard for "oil of coriander" describes these physical properties and requires a minimum linalool (or linalol) content of 70% (*4*). However, the components responsible for the characteristic coriander oil aroma have not yet been fully identified (*5*).

Because of the structure and location of the oil receptacles (commissural vittae) inside the fruits of Umbelliferae (*6*), crushing is necessary to obtain the maximum yield of essential oil and reduce the distillation time (*3, 7*). Purseglove et al. (*3*) gave examples of 8−17% yield improvement and an 8−25% reduction in operational times from the crushing of coriander fruits, but did not discuss the effects of crushing on oil composition. Perineau et al. (*8*) studied the effect of various treatments, including crushing, on the yield and composition of oils from coriander fruits. They found that the oil yield after 1 h was tripled by crushing, but the oils from crushed fruits were enriched in hydrocarbons at the expense of the linalool content. However, these results were for hydro distillation (fruits in boiling water) rather than for steam distillation, which is standard commercial practice (*3*). Marotti and Piccaglia (*9*) have shown that the degree of grinding of raw material has a significant effect on yield and composition of hydro distilled oil from fruits of fennel, another Umbelliferae. However, we could not find any published results on the optimum crushing conditions for coriander fruits, nor on the optimum time for steam distillation. Clearly these parameters are crucial for commercial production of coriander spice oil.

We describe here detailed studies of the yield and composition of coriander oils from pilot- and laboratory-scale steam distillations. The laboratory-scale apparatus was used to study the effects of crushing intensity and distillation time on oil yield and composition.

MATERIALS AND METHODS

Coriander Supply and Charge Preparation. The coriander fruits, *Coriandrum sativum* var. *microcarpum* L., were from plants of Russian origin, grown at Rakaia, Canterbury, New Zealand, harvested in March 1992, and dried using a cereal grain drying system (to below 14% moisture content). The coriander was stored in polythene grain bags at room temperature and humidity until distillation in May 1993. The whole fruits had a mean thousand weight of 7.59 g.

A commercial grain crusher consisting of two rotating, smooth rollers was used to crush the fruit immediately before

* To whom correspondence should be addressed. Telephone: 64-3-479-8354. Fax: 64-479-8543. E-mail: perryn@crop.cri.nz.
† New Zealand Institute for Crop & Food Research Ltd.
‡ Plant Extracts Research Unit.
§ New Zealand Pastoral Agriculture Research Ltd.

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Table 1. Still Operating Conditions and Charge Details for Experiments 1 and 2

exp.	still type	boiler pressure (kPa)	steam flow (mL/min)	charge weight (kg)	dry matter content (%)	distillation time (min)
1	pilot	20	460	49	87.9	40
1	lab	50	9.5	1.4	87.9	40
2	lab	50	11.9	1.6	88.5	60

Table 2. Identification of Coriander Spice Oil Components

	RI			identification method			component level (%)		
peak	DB1	DBWax	identity	GC[a]	RI	GC−MS	mean[b]	min	max
1	935	1054	α-pinene	+	+	+	6.8	1.8	31.5
2	951		camphene	+	+	+	1.4	0.4	6.1
3	958		sabinene?	−	+	−	0.3	0	1.2
4	971		β-pinene	+	+	+	0.3	0.1	1.2
5	981	1154	myrcene	+	+	+	1.1	0.4	3.4
6	1016	1265	p-cymene	+	+	+	0.7	0.2	1.6
7	1025		limonene	+	+	+	2.7	1	7.3
8	1053	1242	γ-terpinene	+	+	+	6.1	2.5	15.4
9	1088	1552	linalool	+	+	+	65.8	33	81.6
10	1133	1513	camphor	+	+	+	5.1	1.9	6.6
11	1156		unknown	−	−	−	0.2	0	0.3
12	1172		α-terpineol	−	+	−	0.3	0	0.7
13	1178	1498	decanal	+	+	+	0.3	0	0.9
14	1242	1854	geraniol[c]	+	+	+	2.6	0	8.2
15	1258	1752	decanol	+	+	−	0.3	0	1
16	1290		undecanal?	−	+	−	0.2	0	0.7
17	1361	1762	geranyl acetate	+	+	+	4	0.2	10.2
18	1439		*E*-2-dodecenal	−	−	+	0.1	0	0.5

[a] Co-injection on DB-1. [b] Experiment 2. [c] Coelutes with *E*-2-decenal.

distillation. Three different crush intensity treatments were achieved by altering the compression on the axle load spring. To quantify the degree of physical disruption caused by each treatment, triplicate 100-g sub-samples of the uncrushed and crushed material for each distillation were sieved through a graduated (4 mm, 2.36 mm, 1.4 mm, and 0.6 mm) sieve series (1 min shaking). In addition, a sub-sample of the material from each crush treatment was examined under a binocular microscope (× 16). Increasing the intensity of crush increased the degree of separation of the pericarp from the mericarp and the degree of rupturing of the endosperm and the epithelial wall of the commissural vittae (*6*), but did not cause a major reduction in crush particle size. For the uncrushed fruits, 94% of the sample had particles with a diameter range of 2.36 to 4 mm. The proportion of particles in this range reduced to 91% with the light-crush and to 84% with standard- and heavy-crush. The packing density of the light-crush fruits averaged 0.283 g/cm^3 compared with 0.276 g/cm^3 and 0.277 g/cm^3 for the standard and heavy-crush fruits, respectively.

Distillations. Details of pressures, steam flow rates, charge weights, fruit dry matter (DM) contents, and distillation times are presented in Table 1. A full description of the pilot still is given in Smallfield et al. (*1*). The lab-still charge vessel consisted of a 700 mm × 110 mm i.d. glass column with a 100 mm i.d. flat flange and a single-socket flat flange lid. The charge material was supported on a 1-mm thick stainless steel (SS) mesh positioned on a lip 600 mm below the top flange. Steam was piped (8 mm i.d. SS tube) in at the center of the column's dome base through a threaded socket with a screw cap. The steam source was a modified 6 L SS pressure cooker heated on a thermostatically controlled hot plate, with steam flow rate controlled through a 12-mm i.d. SS needle valve. A barrel tap with Teflon O rings in the base of the column was used to drain condensate to prevent the steam pipe from becoming flooded. To reduce condensation and heat loss, the column was wrapped in a 2.5-mm flexible insulation sheet. A double surface (Davies) condenser (200 mm × 30 mm o.d.) was connected to the column lid by a distillation bridge (300 mm × 22 mm o.d.). A 500-mL cylindrical separating funnel was used to collect the distillate. Oil floating on the surface of the condensed water was removed at the end of each run, and the oil yield was measured in a 25-mL graduated pipet to within 0.1 mL.

Comparison of Pilot and Lab Stills. Experiment 1 consisted of comparative steam distillations of standard-crush fruits in our lab and pilot stills. The height of the charge was 500 mm in both stills and the charge volume was 155 510 cm^3 for the pilot still and 4320 cm^3 for the lab still, giving a volume ratio of 36:1. Other details are given in Table 1. Total oil yield and chemical composition were compared for three replicate runs, with the stills being operated simultaneously.

Effects of Crushing Intensity and Distillation Time. Experiment 2 compared three intensities of crushing (light, standard, and heavy) with five replicates of each treatment distilled in the lab still on separate days (treatment order was randomized within each day). Other distillation details are given in Table 1. Preliminary runs were used to select suitable sampling intervals of 0−1, 1−2, 2−3, 3−5, 5−7.5, 7.5−10, 10−15, 15−25, 25−35, 35−45, and 45−60 min for collection of separate condensate fractions during each distillation.

GC Analyses and Component Identification. Coriander fruit oils were analyzed as 1% solutions in hexane, using a Perkin-Elmer autosystem gas chromatograph under the control of Perkin-Elmer Omega (version 5.2) software. The column was a 9.5 m × 0.25 mm i.d., J&W DB-1 (0.25 μm film thickness), with H_2 carrier gas (linear velocity 38 cm/s). Injections (0.5 μL) were made into a split (100:1) injector at 260 °C. The flame ionization detector was at 310 °C. A temperature program of 5 °C /min, from 50 to 325 °C, was assumed to show the maximum number of components that could be resolved on this column.

Initial peak identifications were performed by co-injection of standard compounds (on the DB-1 column) and retention indices (Table 2). DB-1 Kovats retention indices (RIs) of major peaks were measured by co-injection of a fruit oil with *n*-alkanes (C8, 10, 12, 14, 16, 18, 20, 22, and 24) on this temperature program. RI's were also measured on a 30 m × 0.25 mm i.d., J&W DB−Wax column (0.25 μm film thickness), with similar operating conditions.

GC−MS analyses were carried out using a Perkin-Elmer 8420 capillary gas chromatograph attached to a Perkin-Elmer ion trap detector (ITD). Injections (0.6 μL) were made into a split injector (30:1, 260 °C). A BP-1 column (12 m × 0.25 mm i.d., 0.25 μm film thickness) was used with helium as the carrier gas (4.5 psi). The column oven was temperature-programmed from 50 to 200 °C at a rate of 30 °C/min, and

Table 3. Effect of Still Type on the Yield and Composition (%) of Coriander Spice Oil

still type	oil yield (mL/100 g DM)	α-pinene (%)	γ-terpinene (%)	linalool (%)	geranyl acetate (%)
pilot	1.98	7.50	6.97	67.00	2.94
lab	2.09	7.38	6.73	67.03	2.85
$LSD_{(P = 0.05)}$	0.13	0.59	0.17	1.09	0.15

held at 200 °C for 15 min. Peaks were detected by an ITD (250 °C) and correlated with a GC profile of the same sample on the DB-1 column. Mass spectra were acquired over a mass range of 40−300 at 1 scan per second. Components were identified by matching MS data with a library (*10*) and RI data with published values (*11*) (Table 2).

The same number of components were resolved on the DB-1 column using a faster temperature program (50 to 200 °C at 45 °C/min with a hold at 200 °C for 1 min), so this program was used to analyze all of the oils. GC peaks of area greater than 0.2% in the oil were labeled peak 1 to peak 18. Peaks due to linalool and geranyl acetate were used as references to correct retention time fluctuations between runs.

Statistical Analysis. For experiment 1, the effect of still type was tested by analysis of variance with distillation run as the blocking factor. For experiment 2, the effect of crushing intensity on oil yield was tested by comparing the regression parameters of a fitted double exponential equation:

$$Y = \alpha\left(1 - \left(\frac{\rho_1^t + \rho_2^t}{2}\right)\right)$$

where Y = oil yield after time t (mL/100 g dry matter), t = distillation time (min), α = maximum oil yield (asymptote), and ρ_1 and ρ_2 = curvature parameters. This double exponential gave a significantly ($P < 0.01$) better fit than the single exponential form. In particular, the estimates of maximum yield appeared better. However, both regressions were highly significant ($P < 0.01$). A more complicated model, with variable weighting on the two curvature (ρ) parameters, did not significantly improve the fit. The effect of crushing intensity on the percentage of a particular oil component was tested by analysis of variance for each sampling fraction. Oil yield data were used to calculate the change in composition with distillation time.

RESULTS AND DISCUSSION

We identified most of the major monoterpenes listed by Lamparsky and Klimes (*5*) in our coriander spice oils (Table 2). We also identified low levels of C10 and C12 aldehydes typical of coriander herb oils (*1*) and sometimes found in spice oils (see *12* and references therein). The combination of a rapid GC analysis method (about 10 min per analysis), automated sample injection, and computerized data handling enabled us to analyze the 171 oils involved in this study in an acceptable time.

The results of the comparison of lab- and pilot-scale stills (Table 3) showed that the two still types gave comparable oil yields and compositions. The only significant difference ($P < 0.05$) was the slightly lower γ-terpinene level in the oils produced from the lab still. Therefore, the results from the lab still in the experiment described below should be a good guide to results from the pilot still, and also to full-scale production. Being able to use the lab still for this experiment saved hundreds of kilograms of both coriander fruit and steam that would have been needed for replicated treatments on the pilot scale.

The effects of crushing intensity and distillation time on the oil yield are plotted in Figure 1. The estimate of maximum yield (α) obtained from the light-crush fruits

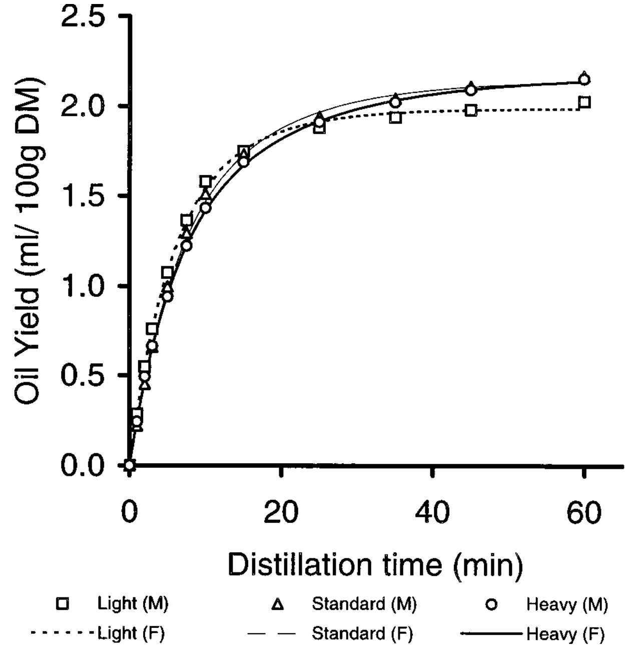

Figure 1. Effect of crushing intensity and distillation time on yield of coriander oil: comparison of mean values (M) and fitted curves (F).

Table 4. Regression Parameters for the Fitted Double Exponential Curve of Time and Oil Yield

crushing treatment	α[a]	ρ_1[b]	ρ_2[b]
light	1.99	0.795	0.900
standard	2.15	0.829	0.929
heavy	2.17	0.822	0.942
SED[c]	0.047	0.023	0.015

[a] Estimate of maximum oil yield (mL/100 g dry matter). [b] Curvature parameters. [c] Mean SED for treatment comparisons standard versus light or heavy and light versus heavy.

was significantly ($P < 0.05$) less than the maximum yields from the standard- or the heavy-crush fruits (Table 4, Figure 1). Also, the first curvature parameter (ρ_1) of the double exponential equation of oil yield for the light-crush fruits was significantly ($P < 0.05$) less than that for the heavy-crush fruits. Since the value of ρ_1 was smaller than ρ_2 it tended to dominate the early stages (approximately the first 10 min) of the time versus oil yield relationship. Therefore, 95% of the maximum oil yield from the light-crush fruits had distilled after 22.5 min compared with 32 min for the standard-crush fruits and 39 min for the heavy-crush fruits. If the distillations were stopped after 22.5 min, the oil yields from the light- and heavy-crush fruits would be 1.89 and 1.87 mL/100 g DM respectively and the yield from the standard-crush fruits would be slightly higher at 1.93 mL/100 g DM. However, if the distillations were run to 95% of maximum yield, the additional 9.5 min for the standard-crush and 16.5 min for the heavy-crush treatments would only produce additional yields of 0.16 mL/100 g DM and 0.11 mL/100 g DM, respectively. Steam consumption required for these additional yields would increase by 42.2% for the standard-crush fruits and 73% for the heavy-crush fruits. Based on a 24-h production cycle and distillation time to extract 95% of maximum yield, 22% less oil would be produced from the standard-crush fruits and 36% less from the heavy-crush fruits than would be produced from light-crushed fruits distilled for 22.5 min. Purseglove et al. (*3*) described a continuous distillation

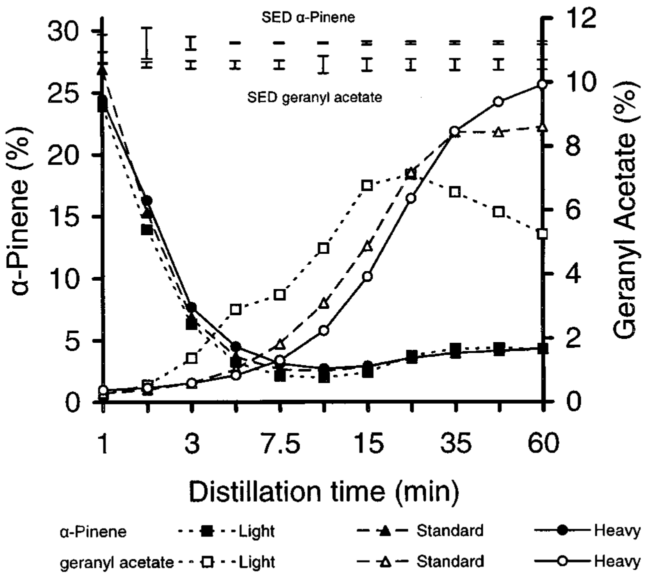

Figure 2. Effect of crushing intensity on the α-pinene and geranyl acetate contents (%) in coriander oils for each time fraction.

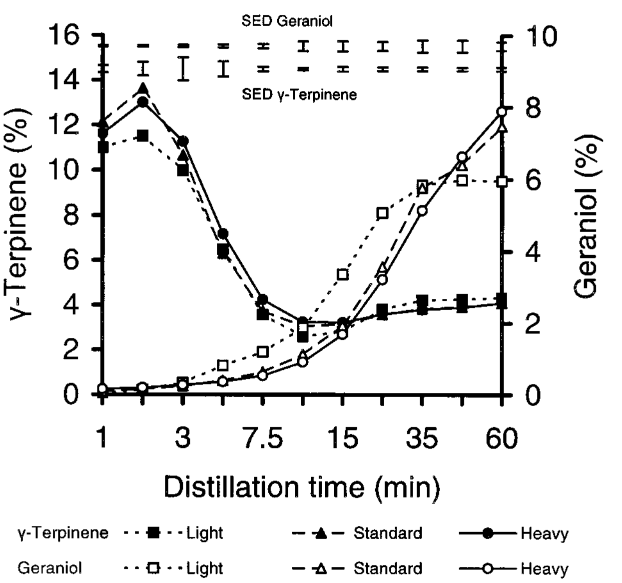

Figure 3. Effect of crushing intensity on the γ-terpinene and geraniol contents (%) of coriander oils for each time fraction.

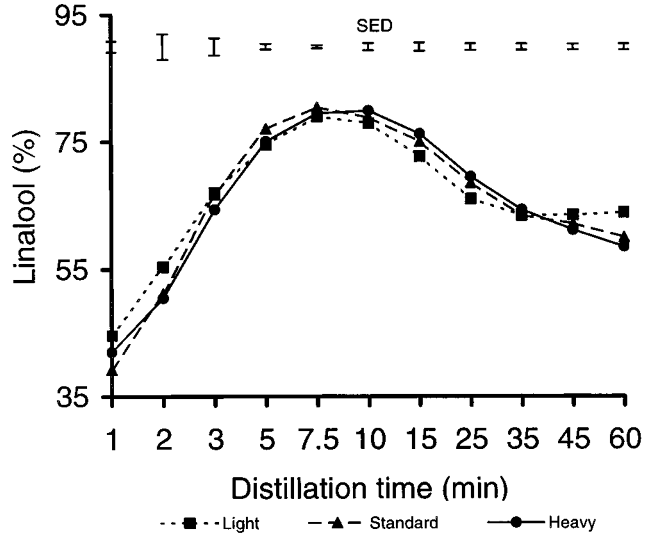

Figure 4. Effect of crushing intensity on the linalool content (%) of coriander oils for each time fraction.

process for coriander in the former USSR in which the contact time for the ground material in the apparatus was approximately 30 min.

The pattern of compositional change of the coriander spice oils during the 60-min distillations for the three crushing treatments are shown in Figures 2, 3, and 4. We have chosen α-pinene and γ-terpinene, linalool, and geraniol and geranyl acetate to illustrate the contrasting effects of crushing and time on the respective hydrocarbon (lowest boiling-point), tertiary alcohol (medium boiling-point) ,and primary alcohol and ester (highest boiling-point) components of these oils. The α-pinene and γ-terpinene contents declined rapidly in fractions collected during the first 10 min of distillation, then leveled out to 2.7% for α-pinene and 4% for γ-terpinene. The decline in α-pinene and γ-terpinene contents was mirrored by a rapid increase in the level of linalool, which peaked at about 80% in the fractions collected at 5−7.5 and 7.5−10 min and then declined steadily for the remainder of the distillation. The contents of geraniol and geranyl acetate increased steadily with time during the distillation. The time-course of recovery of coriander spice oil components from crushed fruits was, therefore, predominantly controlled by boiling point.

However, crushing intensity did have a significant ($P < 0.05$) effect on relative levels of the major components (Figures 2, 3, and 4). Increasing the intensity of crushing significantly ($P < 0.05$) increased the α-pinene content in fractions removed at 5, 7.5, and 10 min, but after 10 min the effect was no longer significant ($P > 0.05$) (Figure 2). Oils from light-crush fruits had a significantly ($P < 0.05$) lower γ-terpinene content than those from the standard- and heavy-crush fruits for the first 10 min of the distillation, except for the fraction removed at 3 min (Figure 3). Intensity of crushing also had a significant ($P < 0.05$) effect on linalool content (Figure 4). In the oil fractions removed at 1, 45, and 60 min, the light-crush fruits produced oil with the highest linalool content. However, in the fractions removed at 5, 7.5, and 25 min, the light-crush fruits produced oil with the lowest linalool content. For all oil fractions where there was a significant effect of crushing intensity on linalool content, differences between the highest and lowest treatment were never large, ranging from 2 to 14%. In contrast, the effect of crushing intensity on the higher boiling-point components was proportionally much larger (Figures 2 and 3). In the oil fractions that were removed between 3 and 15 min into the distillation, the light-crush fruits produced oils with geraniol and geranyl acetate levels 0.57 to 3.3 times higher than those from the other two crushing treatments. From 25 min until the end of distillation the oil fractions from the light-crush fruits had lower geraniol and geranyl acetate levels than the fractions from the standard- and heavy-crush fruits.

These effects of crushing intensity on oil composition can be explained in terms of the effects Guenther (*7*) reported for crushed and uncrushed oils of caraway seed, and those that Marotti and Piccaglia (*9*) reported for three grinding intensities of fennel seed. In the oil from the light-crush fruits, the lower levels of α-pinene and γ-terpinene and higher levels of geraniol and geranyl acetate suggested that there was a higher

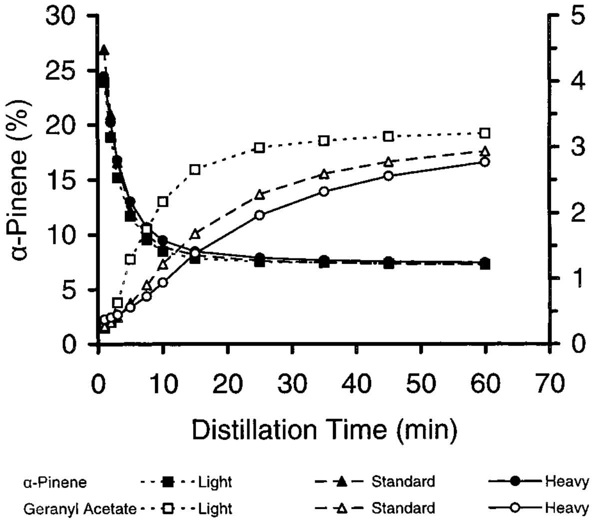

Figure 5. Effect of crushing intensity and distillation time on the predicted α-pinene and geranyl acetate contents of coriander oil.

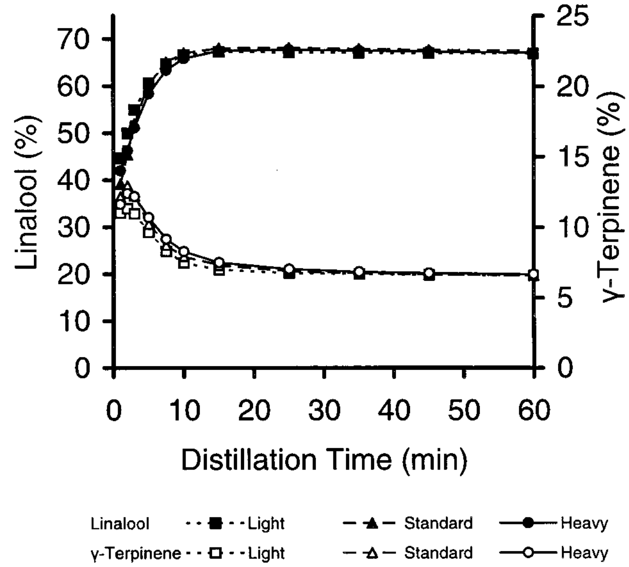

Figure 6. Effect of crushing intensity and distillation time on the predicted linalool and γ-terpinene contents (%) of coriander oil.

proportion of the commissural vittae that had not been ruptured and, therefore, the recovery of oil components from these vittae was controlled by hydro diffusion rather than by boiling point. In hydro diffusion, oil components must dissolve in water before they can diffuse to the surface of the fruit where they can enter the vapor phase and be distilled. Primary alcohols and esters, such as geraniol and geranyl acetate, are more water-soluble than hydrocarbons such as α-pinene and γ-terpinene. Microscopic examination of samples from the different crush treatments confirmed that even though the light-crushing appeared to produce a high degree of physical disruption to the coriander fruit, it was easy to find unruptured commissural vittae (but in every case only one of the two was intact). All vittae had ruptured in the heavy-crush sample and only one unruptured vittae was located in the standard-crush sample.

Using the cumulative oil yield and oil composition data for each sample fraction, it was possible to predict the character of the oil for each crushing intensity if the distillations were stopped at different times (Figures 5 and 6). The effects of crushing intensity on the low (α-pinene and γ-terpinene) and middle boiling point components (linalool) disappeared with increased distillation time. The most pronounced effects of crushing intensity occurred with the high boiling-point component (geranyl acetate) (Figure 5). Levels would be highest in oils from light-crush fruits and lowest in oils from heavy-crush fruits. Therefore, crushing intensity could be used to alter the balance of the high and low boiling point components in coriander spice oil. Crushing intensity could not be used to markedly alter the linalool content of the oil (to achieve the greater than 70% linalool content set down in the ISO standard, *4*) (Figure 6). However, in a batch distillation process, if the oil fraction that condensed from 4 min until the end of a 60 min distillation was collected separately the resulting oil would have a natural linalool content of 72% and would comprise between 53% (light-crush) and 60% (standard- and heavy-crush) of the calculated maximum oil yield. The α-pinene content of this fraction would be 0.45 times lower and the geranyl acetate content would be 1.7 times higher than that of the complete oil.

CONCLUSIONS

We have shown that a small-scale laboratory still can be used to predict the results of pilot-scale distillations of coriander spice oil. Therefore, we believe that our results on the effects of crushing and distillation time readily translate into full-scale production.

Different degrees of crushing of coriander fruits gave slight but significant differences in oil yields. Light-crush fruits, with some unruptured vittae, gave lower maximum oil yields than fruit samples in which all of the vittae ruptured. On the other hand, 95% maximum yield from light-crush fruits could be extracted in a shorter time and, therefore, could result in one-quarter to one-third more oil being produced in a 24-h production cycle.

Manipulating the intensity of crushing could be used to alter the balance of the high- and low-boiling-point components of coriander spice oil, but could not be used to influence the linalool content. Using a batch distillation process, the linalool content could, however, be altered by partitioning of the oil during the distillation, because linalool content peaked at about 7 min into a 60-min distillation.

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