Original article

Essential oils obtained by flash vacuum-expansion of peels from lemon, sweet orange, mandarin and grapefruit

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Abstract — **Introduction**. Essential oils represent an increasing economic importance in the citrus by-product industry. Flash vacuum-expansion, a process leading to the disintegration of plant tissues, was applied to the recovery of essential oils from fruits of four *Citrus* species. **Materials and methods**. The flash vacuum-expansion process (steam-heating, then rapid introduction into a vessel under vacuum) was applied to peels of lemon, sweet orange, mandarin and grapefruit. Essential oils were recovered with a condenser, then separated by centrifugation. Oil volatile compounds were separated and identified by coupled capillary gas liquid chromatography/mass spectrometry. **Results and discussion**. Peel essential oil yields were (2.41, 1.43, 0.64 and 0.73) kg × t^{-1} of fruit for lemon, sweet orange, mandarin and grapefruit, respectively. Oils were enriched in limonene and other monoterpenes, while relative concentrations of monoterpene alcohols (linalool, α-terpineol, β-citronellol, nerol, geraniol) and aldehydes (neral, geranial) were lower in comparison with the volatile compounds of native peels. **Conclusion**. The flash vacuum-expansion process allowed production of citrus peel essential oils with yields comparable to the Food Machinery Corporation (F.M.C.) process. Oils were enriched in monoterpene hydrocarbons and correlatively impoverished in oxygenated volatile constituents.

citrus / agroindustrial sector / methods / extraction / essential oils / plant extracts / peel

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Huiles essentielles obtenues par flash détente sous vide d’écorces de citrons, oranges, mandarines et pomelos.

Résumé — **Introduction**. Les huiles essentielles ont une importance économique croissante dans l’industrie des sous-produits d’agrumes. Le procédé de flash détente sous vide, un processus provoquant la désintégration de tissus végétaux, a été appliqué à l’extraction d’huiles essentielles des fruits de quatre espèces de *Citrus*. **Matériels et méthodes**. Le procédé de flash détente (chauffage à la vapeur, puis introduction rapide dans un récipient sous vide) a été appliqué à des écorces de citrons, oranges, mandarines et pomelos. Les huiles essentielles ont été récupérées à l’aide d’un condenseur, puis séparées par centrifugation. Les composés volatils ont été séparés et identifiés par chromatographie en phase gazeuse sur colonne capillaire couplée à la spectrométrie de masse. **Résultats et discussion**. Les rendements en huiles essentielles des écorces ont été respectivement de (2,41, 1,43, 0,64 et 0,73) kg × t^{-1} de fruits pour le citron, l’orange, la mandarine et le pomelo. Les huiles ont été enrichies en limonène et autres monoterpènes, alors que les concentrations relatives en alcools monoterpéniques (linalol, α-terpinéol, β-citronellol, nérol, géraniol) et en aldéhydes (néral, géranial) ont été abaissées par rapport aux composés volatils des écorces brutes. **Conclusion**. Le procédé de flash détente sous vide a permis d’extraire des huiles essentielles de l’écorce d’agrumes avec des rendements comparables à ceux obtenus par le procédé de la *Food Machinery Corporation* (F.M.C.). Les huiles ont été enrichies en monoterpènes et corrélativement appauvries en composants volatils oxygénés.

agrumes / secteur agro-industriel / méthodes / extraction / huiles essentielles / extraits d’origine végétale / écorce

1. Introduction

Aside from single strength and concentrated juices, massive amounts of peels are recovered from the citrus juice industry. Valorisation of these by-products is achieved in diverse manners: production of pectins, feed for livestock, recovery of essential oils, waxes and flavonoids, and also minor uses such as lemon (*Citrus lemon* L.) and citron peel (*Citrus medica* L.) candying, bitter orange peel (*Citrus aurantium* L.) marmalade and liquors [1–3]. Citrus essential oils are of major concern to the food (soft drinks), detergent, cosmetic and perfume industries. Most of the citrus essential oils are nowadays recovered simultaneously with the fruit juice extraction as cold-pressed oils by the Food Machinery Corporation (F.M.C.) process [4, 5]. Yields of essential oils depend on the *Citrus* species, cultivar, area and period of production; typical yields with the F.M.C. procedure are: 3 kg $\times$ t^{-1} of lemons, 1.5 kg $\times$ t^{-1} of sweet oranges and 1 kg $\times$ t^{-1} of grapefruits (Riverbend[1], pers. commun.).

In the flash vacuum-expansion process, plant materials are first steam-heated for (5 to 10) min at (70 to 90) °C before being instantly introduced into a vacuum chamber at (2 to 5) kPa. This process has occasionally been used for a decade in the wine making industry [6]. When heated grapes enter the vacuum chamber, the grape skin disintegrates due to the instant evaporation of 10% of water and volatile components, thus facilitating the diffusion of skin anthocyanins. After fermentation, red wines from vacuum-expanded grapes are more coloured than those from traditionally processed grapes. Thus, this process can be viewed as an extraction process [7] which could serve for the recovery of volatile compounds from agro-industry wastes.

Since citrus peel essential oils are contained in balloon-shaped vesicles located in the flavedo of citrus peel, we thought that the flash vacuum-expansion process would lead to rupture of the vesicle walls, thus releasing oils which would be recoverable through a condenser. We tested this process on lemon, sweet orange, mandarin and grapefruit peels.

2. Materials and methods

2.1. Fruits

Sound mature lemons [*Citrus limon* (L.) Burm, cv. Primafiori], sweet oranges [*Citrus sinensis* (L.) Osb, cv. Naveline], mandarins [*Citrus reticulata* Blanco, cv. Villalate] and grapefruits [*Citrus paradisi* Macfayden, cv. Ruby Red] were obtained from a local market, stored in cold conditions (4 °C) and processed a week after. Peels were obtained by gently hand-pressing halved fruits in a Santos® juicer.

2.2. Solvents and chemicals

The solvents (*n*-pentane, dichloromethane) were of analytical grade. Reference compounds, if available, and *n*-alkanes (C_5–C_{22}) standards were from Aldrich Chimie (Saint-Quentin Fallavier, France).

2.3. Flash vacuum-expansion experiment

Peels (100 kg) were introduced through a loading funnel into a horizontal stainless steel steam-heating chamber (*figure 1*) (L = 230 cm, l = 40 cm, h = 50 cm) equipped at its bottom with a rotating twin screw (L = 220 cm, ∅ = 15 cm) for both conveying and steam-heating the peels at normal pressure through steam injection holes (*figure 2*). Peel exudates and condensed steam were collected through pipes at the heating chamber bottom and discarded. After having passed through the steam-heating chamber (7 min), heated peels (85 °C to 90 °C) were introduced through an airtight feeding pump into a large vacuum vessel (*figure 3*) (r = 30 cm, h = 150 cm, v = 424 L) where a vacuum (3 kPa) was generated by a vacuum pump cooled by a closed cooling water circuit [8]. Instantly evaporated aromatic liquors (water phase) and essential oils were collected after passage into a condenser (h = 210 cm, ∅= 23 cm); oils were then recovered by centrifugation (9000 *g*, 5 min) and kept in the cold (–20 °C) under argon.

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[1] S.A. Riverbend, Santomera, Spain, 2001.

2.4. Hydrodistillation

Extraction yields of essential oils by flash vacuum expansion (*table I*) were determined by submitting native and vacuum-expanded peels to hydrodistillation. Peels (200 g) were added to distilled water (500 mL), thoroughly mixed at high speed for 3 min in a Waring blender (Waring, USA), and submitted for 3 h to hydrodistillation in a Clevenger-type apparatus.

2.5. Extraction of volatile compounds

Aliquots (100 g) of native and vacuum-expanded peels were cryomilled in liquid nitrogen with a Dangoumill 300 freezer-mill (Prolabo, Paris, France) for 3 min (top impact frequency). An aliquot of the finely pulverised peel (0.5 g) was stirred into 30 mL of azeotropic (pentane/dichloromethane, 2:1) mixture. Phase separation was achieved by centrifugation at 9000 *g* for 5 min. The upper organic phase was recovered, dried over anhydrous sodium sulphate and finally concentrated at 37 °C with a 25 cm Vigreux distillation column to a volume of 2 mL. Prior to GC and GC-MS analysis, solvent extracts were diluted (1/10, v/v) in the above azeotropic mixture.

2.6. GC analysis

A Varian 3380 gas chromatograph was used with a flame ionisation detector (FID), an on-column injector, a DB-Wax (column A, J & W Scientific, Folsom, CA, USA) fused silica capillary column (60 m × 0.32 mm i.d. × 0.25 μm film) or a DB-1 (column B, J & W Scientific, Folsom, CA, USA) fused silica capillary column (30 m × 0.32 mm i.d. × 0.25 μm film). Oven temperature was increased from 40 °C to 245 °C at a rate of 3 °C × min^{-1} where it was held for 20 min. Injector temperature was raised from 20 °C to 245 °C at 180 °C × min^{-1} where it was held for 90 min. Detector temperature was 245 °C. Hydrogen was the carrier gas at 2.0 mL × min^{-1}. Injected volumes were 2 μL of concentrated extract. Response factors were taken as 1.0 for all compounds, except for limonene (1.3), with reference to *n*-hexanol as internal standard. Linear retention indices were calculated with reference to *n*-alkanes (C_5–C_{22}). Concentrations are given as the average of triplicate analyses.

Figure 1.
Steam-heating chamber of the flash vacuum-expansion process (with the permission of FABBRI SA, 84320 Entraigues, France).

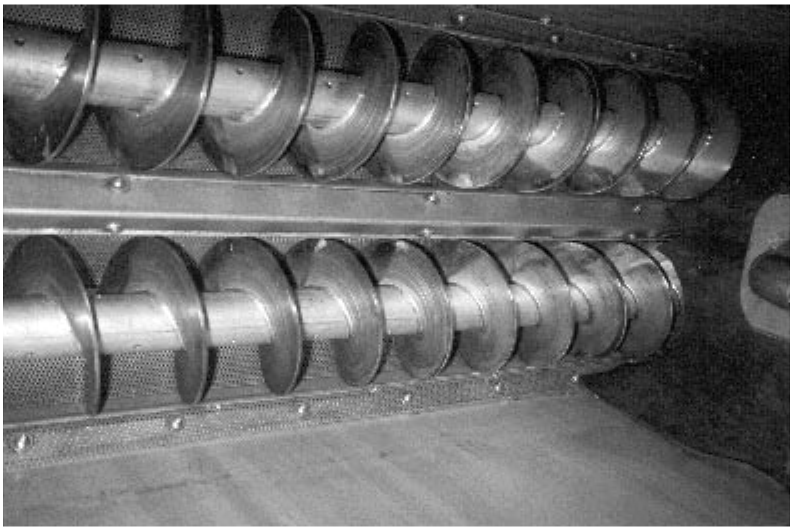

Figure 2.
Steam-heating injection screw (with the permission of FABBRI SA, 84320 Entraigues, France).

Figure 3.
Vacuum chamber and oil recovery system (with the permission of FABBRI SA, 84320 Entraigues, France).

2.7. GC-MS analysis

A Varian 3300 gas chromatograph coupled to a Fisons Trio-1000 quadrupole mass spectrometer with an electron ionisation mode (EI) generated at 70 eV was used. The ion source temperature was 200 °C and the filament emission current was 1 mA. The same columns as above were used for separation. Oven temperature was increased from 40 °C to 220 °C at a rate of 3 °C × min^{-1} where it was held for 20 min. The injector was heated from 20 °C to 245 °C at 180 °C × min^{-1}. Detector temperature was 245 °C. Helium was the carrier gas at 1.1 mL × min^{-1}. Electron impact mass spectra were recorded in the (40 to 600) amu (atomic mass unit) range at 1 s interval. Injected volumes were 1 µL of concentrated extract.

Compounds were identified on the basis of linear retention indices on both columns (DB-Wax and DB-1) and EI mass spectra (Electronic impact mass spectra library, Wiley, 275 L and [9]) from the literature or from authentic standard compounds.

3. Results and discussion

3.1. Extraction of essential oils by flash vacuum-expansion

The essential oil contents of native and vacuum-expanded peels were determined using hydrodistillation and the difference was compared to the oil weight actually recovered in the condensor after vacuum-expansion (*table I*).

Amounts of peel essential oils recovered by vacuum-expansion process ranged from 2.41 kg × t^{-1} of fruit for lemon to 0.64 kg × t^{-1} for mandarin (*table I*). Considering efficient

Table I.
Theoretical and efficient yields in citrus peel essential oils obtained by flash vacuum-expansion (kg essential oils × t^{-1} fruits).

Origin of the essential oils	Lemon	Sweet orange	Mandarin	Grapefruit
Native peels[1]	5.50	4.96	1.91	2.70
Vacuum-expanded peels[2]	1.89	3.09	0.98	1.85
Theoretical recoverable amount[3]	3.62	1.87	0.93	0.85
Vacuum-expanded essential oil[4]	2.41	1.43	0.64	0.73
Yield[5] (%)	44	29	33	27

[1] Maximum recoverable oil amount (obtained by hydrodistillation).
[2] Residual oil in processed peels (obtained by hydrodistillation).
[3] Difference between native and vacuum-expanded essential oil peel contents obtained by hydrodistillation.
[4] Amount actually recovered from the condenser.
[5] Yield = [(amount actually recovered / maximum recoverable oil amount) × 100].

yields calculated as the ratio between actually recovered essential oils by flash vaccuum-expansion *versus* the theoretical recoverable amount, they ranged from 67% for lemon to 86% for grapefruit, showing the good efficiency of the condensing system. Overall extraction yield (= oil amount actually recovered / oil amount in native peels) varies from 27% of oils effectively contained in the grapefruit peels to 44% for lemon. These yields were comparable to the yields attainable by the F.M.C. process, (3, 1.5 and 1) $\text{kg} \times \text{t}^{-1}$ for lemons, sweet oranges and grapefruits, respectively (Riverbend[1], pers. commun.).

3.2. Composition of citrus essential oils

Quantitative analysis of volatile compounds were firstly performed on native peels and peels exiting the steam-heating chamber: similar amounts and relative distributions were observed in both cases showing that oils were neither lost, nor damaged by the first steam-heating step.

With regard to the native peel volatile compounds, the four citrus oils obtained by vacuum-expansion were relatively enriched in limonene, the major citrus peel monoterpene hydrocarbon [5], especially in the case of lemon (*table II*). The same trend was observed for β-pinene while other monoterpenes remained more or less constant. The behaviour of the sesquiterpenes [*cis*- and *trans*-α-bergamotene, β-caryophyllene, α-humulene, (*Z*)-β-farnesene, valencene, β-bisabolene, and δ-cadinene] was variable, either remaining constant, or decreasing after vacuum-expansion. This relative increase in limonene content is accompanied by a relative decrease in oxygenated compounds.

The relative distribution of volatile components obtained from lemon native peels lay in the range of those reported for Italian lemon peel oils [5] with ~ 65% limonene, ~ 9% β-pinene, ~ 8% γ-terpinene, ~ 1.5% β-myrcene and ~ 1.5% α-pinene. Monoterpene aldehydes, neral and geranial, were present at high levels, ~ 1% and ~ 3%, respectively. It must be mentioned that two monoterpene alcohols, nerol and geraniol, were present in very high relative concentrations as compared to Mediterranean or American oils [5, 10]. After vacuum expansion, oil was enriched in monoterpene hydrocarbons (limonene, β-pinene, γ-terpinene, sabinene, α-pinene and β-myrcene). Monoterpene alcohols (linalool, α-terpineol, β-citronellol, nerol and geraniol) and their acetates were drastically reduced while concentrations of monoterpene aldehydes (citronellal, neral and geranial) were divided by a factor of 1.5 to 2 (*table II*). Aliphatic aldehydes (hexanal, octanal, nonanal and decanal) were not affected.

The relative distribution of volatile components obtained from sweet orange native peels exhibited a very high proportion of mono- and sesquiterpene hydrocarbons (> 98%) in agreement with previously reported compositions [10]. Apart from valencene which was found in high relative concentration, other compounds (monoterpene and sesquiterpene alcohols and aldehydes) were also found in typical proportions [10]. Vacuum-expansion yielded an essential oil impoverished in monoterpene alcohols, while monoterpene aldehydes and acetates remained unaltered.

The relative distribution of volatile compounds from the native mandarin peels was similar to those from the limonene chemotype of mandarins (*Citrus reticulata* Blanco group) [11], showing a high proportion of limonene (~ 95%) followed by β-myrcene (~ 2%) and sabinene (~ 1%), and having a very low γ-terpinene content. It must be noted that two monoterpene aldehydes (neral and geranial) and acetates of monoterpene alcohols were not detected. As in the three other citrus species, relative proportions of monoterpene alcohols (linalol, α-terpineol, β-citronellol and nerol) were lowered by a factor of 2 to 3 in the oils produced by vacuum-expansion. An aliphatic aldehyde, decanal, was × 4 concentrated in this oil. Sesquiterpene aldehydes (α- and β-sinensal) were lowered by a factor of 3.

The relative distribution of volatile compounds from the native grapefruit peels was typical [10, 12], exhibiting a high limonene

Table II.
Relative percentage of volatile components in essential oils from lemon, sweet orange, mandarin and grapefruit native and vaccuum-expanded peels.

Compound	Linear retention index		Lemon		Sweet orange		Mandarin		Grapefruit	
	on DB-Wax	on DB-1	native oil	ve essential oil	native oil	ve essential oil	native oil	ve essential oil	native oil	ve essential oil
α-Pinene	1028	918	1.53	1.63	0.50	0.50	0.51	0.18	0.48	0.49
α-Thujene	1032	923	0.33	0.41	0.01	0.01	0.01	0.01	0.01	nd[5]
Camphene	1066	934	0.05	0.05	0.01	0.01	nd	nd	0.01	nd
Hexanal	1083	780	nd	nd	0.04	nd	0.01	nd	0.05	nd
β-Pinene	1106	960	8.97	10.77	0.02	0.04	0.06	0.18	0.03	0.06
Sabinene	1119	960	1.52	1.78	0.35	0.46	1.13	0.28	0.28	0.30
δ-3-Carene	1139	996	nd	nd	0.64	0.44	nd	nd	nd	nd
β-Myrcene	1163	976	1.45	1.68	1.91	2.00	1.76	1.50	1.79	1.91
α-Terpinene	1174	998	0.17	0.22	nd	nd	nd	nd	nd	nd
Limonene	1198	1022	66.23	70.30	94.41	95.32	94.59	95.70	94.55	95.27
β-Phellandrene	1201	1009	0.28	0.28	nd	nd	nd	nd	nd	nd
1,8-Cineole	1203	1010	0.02	0.02	nd	nd	nd	nd	nd	nd
(*E*)-2-Hexenal	1211	824	0.01	nd	0.02	nd	0.03	nd	0.06	nd
(*Z*)-β-Ocimene	1232	1023	0.06	0.05	0.01	nd	0.01	0.01	0.01	0.01
γ-Terpinene	1241	1045	8.07	8.85	0.01	0.02	0.01	0.24	0.05	0.05
(*E*)-β-Ocimene	1247	1038	0.09	0.02	0.03	0.03	0.07	0.05	0.08	0.16
p-Cymene	261	1001	0.07	0.09	nd	nd	nd	0.01	0.01	0.01
α-Terpinolene	1274	1072	0.38	0.39	0.13	0.08	0.04	0.03	0.02	0.01
Octanal	1282	985	0.02	0.05	0.10	0.15	0.05	0.05	0.17	0.35
Nonanal	1384	1077	0.07	0.07	0.02	0.03	0.01	0.01	0.04	0.05
cis-Sabinenehydrate	1460	–	0.07	0.01	0.02	nd	0.01	nd	nd	nd
Citronellal	1467	1128	0.10	0.07	0.06	0.04	0.07	0.10	0.08	0.06
Decanal	1486	1182	0.04	0.03	0.15	0.16	0.18	0.46	0.31	0.18
Isopinocamphone	1523	–	0.01	nd	0.02	0.02	0.04	0.07	0.09	0.11
p-Menth-2-en-1-one	1542	–	0.12	0.09	nd	nd	nd	nd	0.01	nd
Linalool	1544	1085	0.26	nd	0.38	0.18	0.35	0.09	0.12	0.05
cis-α-Bergamotene	1556	1431	0.08	0.01	0.07	0.02	0.05	0.03	0.10	0.15
trans-α-Bergamotene	1571	1417	0.52	0.24	0.02	0.01	0.03	0.18	0.04	nd
β-Caryophyllene	1576	1393	0.33	0.15	0.04	0.05	0.10	0.09	0.30	0.38
Terpinen-4-ol	1590	1160	0.13	0.02	nd	0.01	0.01	nd	nd	nd
β-Santalol	1633	–	0.02	0.01	nd	nd	nd	nd	nd	nd
α-Humulene	1648	1424	0.03	0.02	0.01	0.01	0.02	0.03	0.05	0.04
Citronellyl acetate	1653	1333	0.03	0.02	nd	nd	nd	nd	nd	nd
(*Z*)-β-Farnesene	1658	1209	0.11	0.02	0.01	nd	nd	nd	0.03	0.04
Neral	1665	1208	1.13	0.58	0.05	0.05	nd	nd	0.05	0.03
α-Terpineol	1687	1170	0.63	0.10	0.07	0.03	0.09	0.01	0.07	0.02
Dodecanal	1695	1384	0.03	0.01	0.04	0.02	0.08	0.06	0.08	0.02
Valencene	1696	1240	0.09	0.04	0.39	0.12	nd	nd	0.02	nd
β-Bisabolene	1712	1500	0.48	nd	nd	nd	0.04	0.23	0.05	nd
Neryl acetate	1713	1341	0.16	nd	0.01	0.01	nd	nd	0.05	0.03

Table II.
(Condinued).

Compound	Linear retention index		Lemon		Sweet orange		Mandarin		Grapefruit	
	on DB-Wax	on DB-1	native oil	ve essential oil	native oil	ve essential oil	native oil	ve essential oil	native oil	ve essential oil
Geranial	1716	1240	2.90	1.66	0.13	0.09	nd	nd	0.15	0.04
δ-Cadinene	1739	1516	nd	nd	0.05	0.03	0.11	0.15	0.17	nd
Geranyl acetate	1747	1357	0.32	0.14	0.02	0.01	nd	nd	0.07	0.05
β-Citronellol	1763	1208	0.19	0.01	0.04	nd	0.05	0.02	0.03	0.02
Nerol	1793	1208	1.07	0.02	0.05	0.01	0.01	0.02	0.02	0.01
Geraniol	1843	1240	1.70	0.02	0.07	0.01	0.02	0.02	0.05	0.02
(*E*)-Nerolidol	1013	1547	0.03	0.01	0.01	nd	0.02	0.02	0.02	0.01
γ-Eudesmol	2151	–	0.03	0.01	nd	nd	nd	nd	nd	nd
α-Bisabolol	2200	1662	0.05	0.01	nd	nd	nd	nd	nd	nd
β-Sinensal	2201	1673	nd	nd	0.01	0.02	0.11	0.04	0.04	0.01
α-Sinensal	2272	1726	nd	nd	0.01	nd	0.33	0.16	0.05	nd
Nootkatone	2280	1802	0.01	0.05	0.05	0.02	nd	nd	0.33	0.05

ve: vacuum-expanded; nd: not detected.

content (~ 95%) and decreasing proportions of β-myrcene (~ 1.9%), α-pinene (~ 0.5%) and sabinene (~ 0.3%). Linalool and α-terpineol were the major monoterpene alcohols (~ 0.1% to 0.15%) of the native peel oil, and their relative levels were divided by a factor of 2 after vacuum-expansion like other monoterpenols, β-citronellol, nerol and geraniol. Finally, the concentration of nootkatone, a typical grapefruit volatile component [13], decreased from (0.3 to 0.05)% after vacuum-expansion (*table II*).

4. Conclusion

The flash vacuum-expansion process, when applied to peels from different *Citrus* species (lemon, sweet orange, mandarin and grapefruit), afforded essential oils in yields equivalent to those attainable by the F.M.C. process. Oils were relatively enriched in limonene with a correlative impoverishment in oxygenated compounds (monoterpene alcohols, aldehydes and esters) while aliphatic aldehydes either remained stable or proportionally increased.

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Aceites esenciales obtenidos por expansión súbita al vacío de cortezas de limones, naranjas, mandarinas y toronjas.

Resumen — **Introducción**. Los aceites esenciales tienen una importancia económica que va en aumento dentro de la industria de subproductos de cítricos. La técnica de la expansión súbita (*flash vacuum-expansion)*, un proceso que provoca la desintegración de tejidos vegetales, se ha aplicado a la extracción de aceites esenciales de cuatro especies de *Citrus*. **Material y métodos**. La técnica de expansión súbita al vacío (vapor caliente seguido de la introducción rápida en un recipiente al vacío) se aplicó a cortezas de limones, naranjas, mandarinas y toronjas. Se recuperaron los aceites esenciales mediante un condensador y, luego, se separaron por centrifugación. Los compuestos volátiles de aceite se separaron e identificaron, seguidamente, mediante espectrometría por cromatografía gas-líquido con columna capilar. **Resultados y discusión**. Los rendimientos en aceites esenciales de las cortezas fueron de (2,41, 1,43, 0,64 y 0,73 kg) × t^{-1} de fruta para el limón, naranja, mandarina y toronja, respectivamente. Los aceites fueron enriquecidos con limoneno y otros monoterpenos, mientras que se empobrecieron concentraciones relativas de alcoholes monoterpénicos (linalool, α-terpineol, β-citronellol, nerol, geraniol) y de aldehídos (neral, geranial) con respecto a los compuestos volátiles de las cortezas naturales. **Conclusión**. La técnica de la expansión súbita al vacío ha permitido la extracción de aceites esenciales de cortezas de agrios con rendimientos semejantes a los que se obtienen mediante el procedimiento de la *Food Machinery Corporation* (F.M.C.). Los aceites fueron enriquecidos con hidrocarburos de monoterpeno y correlativamente empobrecidos en componentes volátiles oxigenados.

***Citrus* / sector agroindustrial / métodos / extracción / aceites esenciales / extractos vegetales / piel (vegetal)**