Microwave Heating of Grapeseed: Effect on Oil Quality

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The effects of microwave and air-drying of grapeseeds on the physical and chemical parameters of their oils were investigated. Microwave treatment improved oil yield and increased viscosity, conjugated dienes, and peroxide values while reducing pigment contents (K_{410} and K_{670} values) and *p*-anisidine and saponification values. Removal of tannin by washing grapeseed with alcohol resulted in oil with low pigment content (K_{410} and K_{670} values), peroxide value, and tocopherol contents and high diene, *p*-anisidine, and saponification values. γ-Tocotrienol, the major tocopherol of grapeseed oil, increased while δ-tocopherol concentrations decreased on heat treatment of grapeseed. The negative effect of tannin removal was confirmed by the low tocopherol contents of its oil. The results demonstrate the impact of using microwave heating in producing oil from grapeseed.

Keywords: *Microwave; heating effect; grapeseed oil; chemical and physical parameters; tocopherols; oil quality*

INTRODUCTION

Grapeseed, a major component of grape pomace, is a waste residue of the wine industry. About 1000−2000 tonnes of grape pomace is produced annually from wineries in the Okanagan Valley of British Columbia. This byproduct, along with grapeseed, is currently underexploited. Economic disposal of the solid residues by the wine and juice industries has been a constant concern since the early 1970s. Increasing production trends and stringent environmental pollution abatement programs led to investigations into the potential byproduct recovery from these solid residues (Kinsella, 1974; Rice, 1976). Amerine et al. (1967) reviewed byproduct recovery from winery wastes, including tartrates, grapeseed oil, tannin, stock feed, and fertilizer. Grapeseeds constitute a significant proportion of dried pomace (38−52%) and may be 3−5% of the weight of the grapes (Larrauri et al., 1996; Kinsella, 1974). A variety of nutraceutical and functional food products including procyanidins, phenolic compounds, tannins, dietary fiber, oil, and resveratrol (a cancer chemopreventive agent) can be extracted from grapeseeds (Bourzeix et al., 1986; Fuleki and Ricardo da Silva, 1997; Igartuburu et al., 1997; Mazza, 1995; Pezet and Cuenat, 1996; Girard and Mazza, 1998).

The oil content of grapeseed ranges from 11 to 22% depending on variety and environmental growing conditions (Miric et al., 1977; Rice, 1976; Tarandzhiiska and Stamenov, 1989). Lipid contents of the seeds from red grapes were reported to be higher than those from seeds of white grapes (Izzo and Muratore, 1993). Grape varieties with elevated sugar content have been associated with high grapeseed oil content (Teodorescu et al., 1974). The processing, properties, composition, and application of grapeseed oil as a frying oil were briefly summarized by Kinsella (1974), who suggested its inclusion in foods with low saturated fatty acid content designed for lowering serum cholesterol. Grapeseed oil has been touted as a good anticholesteremic edible or dietetic oil, especially for atherosclerotic patients (El-Zeany et al., 1982). It is increasingly being used as a traditional specialty oil in personal care products, aromatherapy, and the rapidly growing cosmeceutical industry.

Our aim is to propose the transformation of grape pomace, grapeseed in particular, into economically valuable ingredients for the food and nonfood industries. This study involved the extraction of oil from grapeseed, its improvement, and the effect of these processes on oil quality. Although grapeseed recovery from pomace has been investigated before, microwave conditioning, to our knowledge, has not been used for the extraction of grapeseed oil.

MATERIALS AND METHODS

Grapeseeds. Grape pomace was from mechanically harvested and crushed Merlot, which was fermented on the skin for 14 days and pressed in a horizontal bladder press (Bucher-108) at Black Sage Vineyards, Oliver, BC, in 1996. Grapeseed was separated from the pomace by sieving through 5 and 3 mm sieves on a RO-Tap (Tyler RX-29, Tyler, PQ, Canada) for 10 min. The seed was further cleaned by air separation in a laboratory aspirator (Cuthbert Co. Ltd., Winnipeg, MB). The yield of grapeseed was 22.1 ± 1.1% of the pomace.

Processing Treatments. Three different thermal treatments were applied to grapeseed samples. In the first treatment, samples were air-dried in a fluid bed dryer (Lab-Line Instruments Inc., Melrose Park, IL) for 2 h at 50 °C. For the second treatment, grapeseed samples were dried in a Panasonic home microwave oven, model NN-S766 WC (Matsushita Electric of Canada Ltd., Mississauga, ON) with maximum heating power output of 950 W at 60 Hz for 24 min with intermittent cooling and mixing every 3 min. The third treatment consisted of drying the grapeseed continuously for 9 min in the same Panasonic microwave oven. To remove the

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tannins, a sample of untreated grapeseed was rinsed with cold tap water for 2 min and then stirred (12000 rpm, Caframo RZR50, Wiarton, ON) for 8 h with 3 volumes of 95% ethanol. The ethanol was removed by vacuum filtration, and the grapeseed was further extracted twice with 95% ethanol. After the last filtration, the seed was air-dried at 25 °C for over 48 h. All grapeseed samples were ground (Thomas Wiley Mill, Philadelphia, PA) to pass a 1 mm screen.

Oil from all milled grapeseed samples was extracted using petroleum ether as described by Oomah et al. (1996). A sample of whole untreated grapeseed was hydraulically pressed (Carver Press, 280 kg/cm^2) to extract cold-pressed oil. A commercial grapeseed oil produced and packed in Spain (Aceites Borges Pont, S.A., Catalonia, Spain) purchased from a local food store was used as control.

Analytical Procedures. Official methods (AOCS, 1993) were used for the determination of the saponification value (method Cd 3-25), *p*-anisidine value (method Cd 18-90), and conjugated dienoic acid (method Ti 1a-64) of oils. The peroxide value of the oils was determined using the PeroXOquant quantitive peroxide assay kit (Pierce, Rockford, IL). As an index of color, the absorbance at 410 and 670 nm of a 10% v/v solution of oil in hexane was measured with a spectrophotometer (DU-640B, Beckman Instruments Inc., Fullerton, CA). The viscosity of the oil was measured with a controlled stress Bohlin rheometer CVO (Bohlin Instruments Ltd., Gloucestershire, U.K.). Measurements were performed at 25 °C with a steel cone-plate geometry (20 mm, 2°) under a ramping shear of 2.5−10 Pa.

Tocopherols in grapeseed oils were analyzed by an HPLC system (Waters 840 system, Milford, MA) consisting of a pump (model 510), an autosampler (model 712), and a fluorescence detector (McPherson SF-749 spectrofluorometer, Acton, MA) interfaced with a personal computer. A normal phase column (4.6 × 150 mm, Primesphere 5 silica, 5 μm) with a guard column (4.6 × 30 mm) (Phenomenex, Torrance, CA) was used with hexane/2-propanol/dimethylpropane (1000:5:1, v/v/v) as mobile phase. The system was operated isocratically at a flow rate of 1 mL/min. Separations were carried out at 25 °C (Waters TCM temperature controller) with the fluorescence detector excitation and emission wavelengths set at 297 and 325 nm, respectively. Typically, a 10 min equilibration period was used between samples, requiring ~40 min/sample. Quantitation was based on an external standard method; the calibration curves ranged from 3.97 to 15.87, from 5.41 to 21.63, and from 6.0 to 24.0 μg/mL of reference compounds α-, δ-, and β-,γ-tocopherols, respectively (Sigma Chemical Co., St Louis, MO). Because pure standards of tocotrienols are not available commercially, palm oil (Nutrolein, an enriched tocopherol and tocotrienol fraction from golden palm oil, a gift from the Malaysian Palm Council of America, Inc., Chicago, IL) was used for estimating the tocotrienol contents of grapeseed oils. Prior to HPLC analysis, grapeseed oil was diluted with hexane to obtain a concentration of ~160 g/L and filtered (0.45 μm, Gelman Science Inc., Ann Arbor, MI) and a 20 μL sample was injected. The oil content of the ground grapeseed samples was determined by Soxhlet extraction with petroleum ether for 6 h. The moisture content was determined according to an AOAC (1984) method.

At least three determinations were made for all assays. Analysis of variance by the general linear models (GLM) procedure, means comparison by Duncan's test, and Pearson correlation were performed according to the Statistical Analysis System (SAS Institute, 1990).

RESULTS AND DISCUSSION

Moisture and oil contents of the original grapeseed sample were 12.6 ± 0.1 and 14.6 ± 0.1%, respectively. Exposure of seeds to microwave-drying resulted in progressive increase in weight loss and seed temperature (Figure 1). A loss of 102.5 g/kg of the original weight was attained after 9 min of drying; after that time, little additional weight loss occurred. Increase in seed temperature occurred up to 9 min of drying (25−110 °C) and remained stable thereafter. The stability in temperature achieved at 9 min suggests that all free water has been removed, thereby drastically decreasing further microwave energy absorption. Because limited weight loss occurred on drying beyond 9 min, a seed sample was continuously exposed to microwave-drying for 9 min. The parallel increase in weight loss and temperature with drying time suggested a linear weight loss−temperature relationship, which was found to be highly significant ($r^2 = 0.968$). The weight loss curve fitted the power model ($y = ax^b$, $r^2 = 0.981$) when regressed against drying time as reported previously for oilseeds (Oomah and Mazza, 1992). Similar temperature and weight loss effects have been observed during microwave heating of soybeans (Yoshida et al., 1997; Yoshida and Takagi, 1997) and several other seeds (Cavalcante and Muchovej, 1993). Moisture and oil contents of grapeseed samples were significantly different among treatments (Table 1). Moisture content decreased upon treatment, with the continuously microwave-dried sample having the lowest moisture content (2.5%). Oil contents of the seeds (mean = 14.8 ± 0.5%) were comparable to those from Merlot grape samples from Sicily (13.9%) (Izzo and Muratore, 1993). Microwave treatments improved the yield of oil from the seeds (Table 1). This finding is consistent with results reported for roasted sesame seed (Yoshida and Takagi, 1997). Cold-pressing of grapeseeds extracted only 36% of the oil content compared to petroleum ether extraction. The quality of the oil, however, was superior to that of oil produced by solvent extraction as indicated by its lighter color (Table 1) and low peroxide value (Table 2).

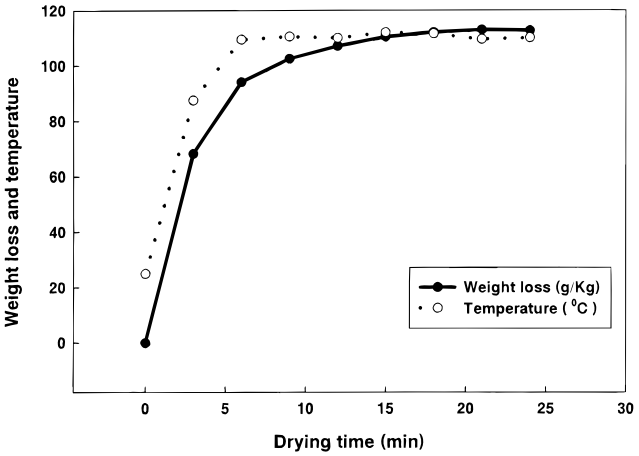

Figure 1. Relationship between heating time and weight loss and temperature of grapeseed in a microwave oven.

Table 1. Physicochemical Properties of Grapeseed and Grapeseed Oil

	grapeseed[a]		grapeseed oil[a]		
sample	moisture (%)	oil yield (%)	K_{410}	K_{670}	viscosity (m·Pa)
untreated	12.6[a]	14.6[b]	1.185[a]	0.439[a]	22.2[e]
air-dried	3.5[c]	14.2[b]	0.882[b]	0.335[b]	35.5[d]
microwaved (24 min)	2.9[d]	15.4[a]	0.819[c]	0.299[c]	44.1[b]
microwaved (9 min)	2.5[e]	15.3[a]	0.805[d]	0.283[d]	44.3[b]
tannin-removed	6.7[b]	14.5[b]	0.428[e]	0.154[e]	41.6[c]
cold-pressed	NA[b]	5.3[c]	0.179[f]	0.060[f]	46.3[a]
commercial	NA	NA	0.082[g]	0.022[g]	41.9[c]

[a] Means in a column followed by the same letter are not significantly different by Duncan's multiple-range test at the 5% level. [b] NA, not applicable.

Table 2. Chemical Properties of Grape Seed Oil

sample	diene value[a]	triene value[a]	AV[a]	saponification value[a]	peroxide[a] (mequiv/kg)
untreated	0.428[d]	0.015[c]	8.5[bc]	187.1[e]	4.8[c]
air-dried	0.424[d]	0.009[e]	9.1[bc]	195.9[b]	4.5[d]
microwaved (24 min)	0.486[c]	0.015[c]	7.2[c]	192.4[d]	5.6[a]
microwaved (9 min)	0.491[c]	0.020[b]	7.5[c]	196.0[b]	5.4[b]
tannin-removed	0.778[a]	0.013[d]	10.4[b]	199.3[a]	5.6[a]
cold-pressed	0.518[b]	0.014[cd]	3.2[d]	194.4[bc]	1.9[f]
commercial	0.412[d]	0.243[a]	18.2[a]	192.9[cd]	2.1[e]

[a] Means in a column followed by the same letter are not significantly different by Duncan's multiple-range test at the 5% level.

Treatment of grapeseed produced a significant decrease in chlorophyll (absorbance at 670 nm) and carotenoid (410 nm) pigments of the oil (Table 1). The commercial and cold-pressed oils were the lightest in color as indicated by their low absorbance at both wavelengths. Similar loss of pigments due to heat treatments of vegetable oils has recently been reported (Albi et al., 1997). However, darkening of oil is also known to result from microwave-drying of soybean and sesame seeds (Yoshida et al., 1997; Yoshida and Takagi, 1997). The decrease in absorbance may be due to the degradation of chlorophylls, commonly observed during oil processing, to derivatives that are much harder to remove during bleaching (Eskin et al., 1996). Absorbance values of oil from treated grapeseed were similar to those of oil extracted from microwave-treated soybeans (Yoshida et al., 1997).

The viscosity of the oil increased significantly upon treatment of grapeseed (Table 1). There was an increase as a consequence of conventional heating (fluid bed dryer) and a more pronounced 2-fold increase when the seeds were subjected to microwave-drying. The increase in viscosity may be related to the formation of dimers and polymers as a result of an increase in the length of the carbon chain (Albi et al., 1997), but it may also be related to differences in saturated fatty acids with consequent higher melting points (Eskin et al., 1996). Cold-pressed grapeseed oil had the highest viscosity, which was comparable to that of a commercial safflower oil (47.5 ± 1.5 mPa·s) purchased locally and assayed under identical conditions. The viscosity of the commercial oil was similar to that of oil from tannin-removed grapeseed. Our viscosity values were slightly lower than those ranging from 51 to 60 cP at 25 °C reported by Kinsella (1974).

Conjugated dienes increased significantly with seed drying by microwave energy, and this was independent of exposure time (Table 2). Similar increases in conjugated dienes have been observed upon toasting of grapeseed (Gattuso et al., 1983). Removal of the seed tannins resulted in a much larger increase in conjugated diene content of the oil. This may partly be due to changes in pigment constituents that can affect the absorption considerably. It may also be an indication of extensive linoleate oxidation of the oil as a consequence of removal of antioxidants such as tocopherols (Table 3) by this treatment. Conjugated diene content of the cold-pressed oil was significantly higher than those from microwave-treated, air-dried, and untreated seeds. The commercial grapeseed oil had a conjugated diene value comparable to those of oil from air-dried and untreated seeds. Conjugated triene values of oils extracted from untreated, microwave treated for 24 min, and cold-pressed grapeseed were similar (Table 2). The similar triene values suggest very small changes in linolenate oxidation in most of the treatments that were applied. Oil from air-dried seeds had the lowest conjugated triene value. Continuous microwave-drying significantly increased the triene value. The commercial grapeseed oil had the highest triene value, indicating that the oil had undergone some physicochemical refining treatment.

p-Anisidine values (AV) of oils from the heat-treated seeds were <10 (recommended value for fresh fully refined oil) and not significantly different from that of untreated seeds (Table 2). Tannin removal by alcohol washing of the seed produced an increase in AV. Oil from the cold-pressed seed had very low AV, indicating low levels of carbonyl compounds in the absence of heat. The very high AV of the commercial grapeseed oil may be a reflection of a long storage period undergone by the oil. Increases in AV have been observed during roasting of sesame seeds in an electric oven (Yoshida and Takagi, 1997).

Saponification values of oil increased significantly upon treatment of seeds (Table 2), tannin removal having the greatest effect. The increase in saponification value of the oil from the ethanol-washed seeds may be due to the increase in the free fatty acids in the seeds as a result of triglyceride breakdown. High temperature is also known to result in considerable free fatty acid formation from triglycerides (Molero-Gomez et al., 1996). Oils from air-dried, continuously microwave-treated, and cold-pressed seeds had similar saponification values. Microwave heating with intermittent cooling of the seed resulted in a reduction in saponification value of the oil, which was similar to that of the commercial grapeseed oil. The saponification values of the grapeseed oils were within the standards for refined oil (185−196) (Molero-Gomez et al., 1996).

The peroxide value was 1.9 mequiv/kg of oil for cold-pressed grapeseed, and this value increased significantly to reach the maximum of 5.6 for oil extracted from grapeseed continuously heated in the microwave for 24 min (Table 2). Microwave treatment of grapeseed significantly increased the peroxide value of extracted oil ($P < 0.0001$), irrespective of heating time, compared to those extracted from the untreated seeds. However, continuous microwave heating resulted in significantly lower peroxide values than microwave treatment for 24 min. Air-drying of the seed led to a reduction in peroxide value compared to the untreated seeds. The slightly elevated peroxide value of the tannin-removed seed may reflect some linoleate degradation of the sample. Increases in peroxide value have previously been observed when grapeseed was toasted (Gattuso et al., 1983). Changes in peroxide values of grapeseed oil were similar to those reported for sesame seeds roasted at 160 °C (from 1.4 to 5.4 mequiv/kg of oil) (Yoshida and Takagi, 1997). Although significant differences were observed in peroxide values among treatments, the changes were minor and peroxide values were lower than those generally recommended for grapeseed oil (<10).

Tocol contents of oil differed significantly ($P < 0.05$) among treatments (Table 3). γ-Tocotrienol was the major tocol of grapeseed oil, representing 38−72% of the total tocopherols. γ-Tocotrienol ranged from 5.3 to 48.8 mg/100 g of oil for oil extracted from tannin-free grapeseed and from seed heated continuously in the microwave for 9 min, respectively. α-Tocopherol, α-to-

Table 3. Total Tocopherols, Tocotrienols, and Vitamin E Contents of Grapeseed Oil (Milligrams per 100 g)

	tocopherol[a]				tocotrienol[a]				
sample	α	β	γ	δ	α	γ	δ	total[a]	vitamin E[a]
untreated	0.54[e]	0.21[d]	0.84[cd]	6.97[a]	0.67[e]	15.23[e]	0.00[c]	25.43[d]	1.06[e]
air-dried	1.19[d]	0.94[b]	1.09[c]	5.87[b]	4.99[c]	44.77[b]	0.38[b]	59.66[b]	3.62[d]
microwaved (24 min)	2.88[c]	0.68[c]	1.71[bc]	2.17[d]	2.95[d]	41.36[c]	0.47[b]	53.06[c]	4.63[c]
microwaved (9 min)	3.39[b]	1.00[b]	2.15[b]	1.00[e]	5.48[b]	48.82[a]	0.78[a]	63.82[a]	6.14[b]
tannin-removed	0.00[f]	0.00[d]	0.00[d]	2.47[c]	0.00[f]	5.26[f]	0.00[c]	8.69[e]	0.05[f]
commercial	5.59[a]	2.31[a]	3.31[a]	0.00[f]	15.70[a]	28.48[d]	0.00[c]	55.40[c]	11.84[a]
palm oil[b]	20.7 ± 1.3	1.1 ± 0.2	2.6 ± 0.3		18.8 ± 1.0	46.6 ± 1.6	6.4 ± 0.2	96.2 ± 4.3	27.6 ± 1.7

[a] Means in a column followed by the same letter are not significantly different by Duncan's multiple-range test at the 5% level. [b] Mean and standard deviation, $n = 8$.

Table 4. Correlation Coefficients for Oil Quality of Grapeseed

				tocopherols				
	yield	AV	triene value	α	α_3	β	γ	vitamin E
peroxide value	0.967[c]	−0.197	−0.589	−0.665	−0.899[a]	−0.862[a]	−0.703	−0.781
anisidine value			0.866	0.589	0.826[a]	0.753	0.567	0.691
triene value				0.792	0.924[b]	0.876[a]	0.779	0.854[a]
α-tocopherol					0.898[b]	0.919[b]	0.985[c]	0.966[c]
α-tocotrienol						0.992[d]	0.904[b]	0.966[c]
β-tocopherol							0.932[b]	0.980[c]
γ-tocopherol								0.978[c]

[a] $P < 0.05$. [b] $P < 0.01$. [c] $P < 0.005$. [d] $P < 0.0001$ ($n = 6$).

cotrienol, and γ-tocotrienol levels increased significantly, resulting in higher vitamin E equivalents with increased severity of heat treatment. The concentration of β-tocopherol of oil extracted from air-dried seed was not significantly different from that extracted from continuously microwave-treated seeds. δ-Tocopherol was susceptible to degradation and decreased sharply (from 7 to 1 mg/100 g of oil) with continuous microwave treatment. Air-drying and microwave-drying for 24 min resulted in 15 and 68% decreases, respectively, in δ-tocopherol content compared to the untreated seeds. The commercial grapeseed oil contained no δ-tocopherol. Decrease in δ-tocopherol and no changes in β-tocopherols due to microwave treatments have previously been reported in soybeans (Yoshida and Kajimoto, 1989).

The biologically active vitamin E content relative to that of α-tocopherol, calculated by using the formula proposed by McLaughlin and Weihrauch (1979), ranged from 0.05 to 11.8 mg/100 g of oil for the tannin-removed grapeseed sample and the commercial grapeseed oil, respectively. The latter had tocopherol levels comparable to those of virgin grapeseed oil reported by Dionisi et al. (1995). Oil from heated grapeseed contained >40 mg of γ-tocotrienol/100 g of oil, similar to that found in palm oil but ~1.5 times higher than that of commercial grapeseed oil (Dionisi et al., 1995). Total tocol contents of heated grapeseed oil (53−64 mg/100 g of oil) were similar to that of safflower oil (51.6 mg/100 g of oil) and higher than that of peanut oil (37.9 mg/100 g of oil) (McLaughlin and Weihrauch, 1979). The vitamin E content of the commercial grapeseed oil was similar to those of peanut oil, olive oil, and soybean oil (Ensminger et al., 1993). The low tocopherol content of oil from tannin-free grapeseed is consistent with the antioxidative effects of polymeric grapeseed tannins in vivo, negating the effects of vitamin E (Tebib et al., 1997).

The vitamin E content showed strong association with tocopherol isomers (Table 4). The Pearson correlation coefficients of α-, β-, and γ-tocopherols and α-tocotrienols ranged from 0.854 to 0.978 for vitamin E equivalents. The tocopherol isomers were significantly correlated with each other (r values between 0.898 and 0.992) (Table 4). The analysis also showed that peroxide value was negatively associated with tocopherols, but significantly ($P < 0.05$) only with α-tocotrienol and β-tocopherol. Anisidine and triene values were positively correlated with α-tocotrienol ($r = 0.826$ and 0.924, respectively). The triene value was also positively associated with β-tocopherol and vitamin E equivalents. The high positive correlation of peroxide value with oil yield from grapeseed and negative association with tocopherols suggest that oil stability may be reduced with increased oil extraction.

The data presented indicate that microwave conditioning of grapeseed produces changes in the quality of its oil. Some positive effects, such as decrease in chlorophyll level and increase in α-tocopherol and α- and γ-tocotrienols, were observed as a result of microwave treatments. The elevated tocotrienol concentration of the oil from the microwave-treated seeds provides an added benefit for its use as a nutraceutical because tocotrienols have been reported to have positive biological and health effects (Watkins et al., 1998). Microwave-drying of grapeseed is rapid and with proper controls can be used to produce good-quality oil that may have applications in the foods and growing phytoceutical industries.

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