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Effect of processing variables on the characteristics of persimmon liqueur

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Persimmon liqueur was prepared from fresh or dry fruit by: (1) extraction with alcohol, (2) fermentation of fresh fruit, and (3) extraction of dry fruit with distilled alcohol from an extract. Alteration in the ratio of raw and dry materials to a solvent, conditions of fermentation and degree of distillation resulted in a beverage with high aroma and taste and low amounts of polyphenols and proteins. Phenolic compounds were detected and characterized by ultraviolet (UV) spectroscopy in persimmon extracts as well as in liqueurs prepared from these extracts.

The final product contained 27% alcohol, 30% total sugars and 30% persimmon extract. Similar results were obtained with and without fermentation using processes developed in this study.

INTRODUCTION

Israel is a highly agricultural country, where the main fruits for local consumption are grapes, oranges, mandarines, pomelos and grapefruits, as well as specific tropical fruits such as persimmons. Dietetic surveys have shown that these specific tropical fruits can be used for every day food, as well as for industrial purposes (Choi & Sohn, 1977; Nagayama *et al.*, 1983).

Liqueur production is a common industry for many European countries. Israel produces only a small fraction of the total world production of liqueurs such as Sabra. The technological process of liqueur preparation is based on a mixture of alcohol, sugar, extracts of fruit, aromatic substances and colour.

Since the technological process to obtain extracts from fruits is now widely recognized, it was considered important to evaluate the possible agro-industrialization of persimmon for liqueur preparation.

The development of appropriate technology for fruit extraction is the most critical stage in liqueur preparation. Fruit aroma and flavour of liqueur content depend on many factors such as fruit variety, composition of fruit and technological processes (Ficca, 1983; Morgante & Cichelli, 1984; Ohba *et al.*, 1985). The technology of wine preparation from persimmons is described in some scientific reports (Junior & Aquarone, 1981; Takano *et al.*, 1981; Yagi & Kizaki, 1985). To our knowledge there are few references in the literature (Gyokusendo Sake Brewery, 1981) about the use of persimmons for the preparation of extracts for liqueurs.

This paper presents the results of different composition and sensory properties of persimmon liqueurs.

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MATERIALS AND METHODS

Persimmon (*Diospyros Kaki* L.) of two varieties: Fuio (with seeds) and Triumph (seedless) were used throughout all experiments. Pectinase (Novo-Industria/s, Denmark) was used as an enzyme, during the extraction. In the preparation of liqueurs the following processes were carried out: mashing, fermentation, distillation, extraction and aging. Fruit liqueur based on natural fruit–alcohol extracts was produced by three different processes. The first two used alcoholic extraction of fresh or dry fruits.

According to the first method, fruit was crushed and pectinase was added. After this treatment the fruit was soaked with 50% and 95% alcohol and matured to yield a fruit extract during 10 days, 20 days and 30 days as sampling times at room or specified temperatures. Persimmon was crushed, reacted with a macerating enzyme, pectinase, of 70 mg/kg of fruit during 24 h at room temperature. After this treatment persimmon was soaked with alcohol in tanks to yield an alcohol–fruit

extract. For preparation of alcohol–fruit extracts the following parameters were varied: concentration of alcohol (absolute 95% and 50%); temperature (20–25°C and 36°C); ratios (w/v) of material: solvent (fruit: alcohol) 1:1; 1:1·5; 1:2; 1:3; duration of extraction (10 days; 20 days; and 30 days).

After extraction the product was separated by decantation. The mixture of fruit was then pressed to obtain a maximum yield of extract. Extracts were then treated with bentonite (0·5 g/l) during one week to precipitate polyphenols and proteins. After precipitation, clarification was achieved using filtration with kieselguhr.

The second experiment involved extraction of dry fruit with 50% alcohol for 20 days at room temperature. Alcohol–fruit extracts were also obtained from dry fruit. Fresh persimmon was cut into pieces and dried in a lyophilizer. Unhydrolyzed and freeze-dried tissue was then extracted at room temperature with 50% ethyl alcohol for two weeks. Filtration was done under pressure. The third variation dealt with crushed fruit, treated with pectinase and fermented by *S. cerevisiae*. The fermentation of persimmons was done by the following process. Whole fruits were partly cut or crushed without seeds. Fermentation was conducted with *Saccharomyces cerevisiae* var. ellipsoides. The must was fermented for 21 days at 20–24°C. Sulfur dioxide (130 mg/l) was added in metabisulfite form. Fermentation was allowed to proceed to an ethanol concentration of 12–15% (v.v).

The fermented mash was distilled and, as a result, a distillate (extract) was obtained containing about 50% alcohol. Dried fruit was soaked with this distillate for 20 days and a final fruit double extract was used as a component for liqueur preparation. Optimal conditions for fruit extraction with alcohol were determined according to the detection of extracted substances by TLC. The analyses of standard liqueurs and liqueur musts were carried out using conventional methods (Amerine & Ough, 1980). A Hempel column packed with glass rings was used for fractional distillation.

The amount of total polyphenols was determined spectrophotometrically at 275 nm. Resorcinol was used as a standard (Gorinstein *et al.*, 1980). Determination of phenolic compounds of the resulting extracts and liqueurs was then achieved by UV spectroscopy (Tryon *et al.*, 1988). Three phenols were chosen as standards for UV analysis: flavonoids (catechin and quercetin) and tannic acid. Standards of phenolic compounds were purchased from Sigma Chemical Co.

Sample preparation

All determinations were carried out in triplicate. Samples were prepared according to Tryon *et al.*, (1988) with certain modifications based on the solubility of phenols (catechin and quercetin) and the origin of the liqueur. Liqueur (0·25 ml) was diluted to 5 ml with a solution of 24% (v/v) ethanol and 30% (v/v) dextrose. Quercetin was first dissolved in ethanol and then diluted with the ethanol–dextrose solution. Stock solutions (100 mg/l) of each phenol were prepared by dissolving the sample in ethanol–dextrose solution. The absorption of standard solutions (10 mg/l), as well as liqueurs was measured on a Wicon 930 Kautron UV spectrophotometer at scan range of 250–350 nm, at scan speed of 200 nm/min. All ultraviolet spectra were recorded using ethanol–dextrose solution as a blank.

Total proteins in fruit extracts were determined by the method of Bradford (1976) with some modifications. The sample was treated with Brilliant Blue G250, followed by colorimetry at 595 nm. The nitrogen content in each extract was determined by the micro-Kjeldahl method (AOAC, 1980; Nkonge & Ballance, 1982).

All fruit–alcohol extracts were subjected to taste panel analysis. It is known that aroma compounds contribute significantly to the importance of fruits in human sensory evaluation. We indentified the aroma scores by sensory evaluation. A trained sensory panel (including wine tasters and members of our department and producers of liqueurs in Israeli industry), evaluated the samples. Care was taken to keep the cover on the wine glass at all times except for the brief period of sniffing the head space. (Whitaker *et al.*, 1988). Panel members were supplied with bottles of standards. Fourteen final samples were tasted by 10 taste panel members. Taste and aroma were calculated from the results of panel tasting. The highest mark in this estimation was 10.

RESULTS AND DISCUSSION

Preliminary results on the extraction of persimmon (Fuio) are not reported in this paper, because the better taste and aroma were found in persimmon (Triumph) alcohol extracts. Therefore our research focused only on the preparation of liqueurs from persimmon (Triumph) alcohol extracts.

In Tables 1, 2 and 3 each number in samples 1–12 is the result of the analyses of variance comparing days and extraction procedures. The original protocols of each group are not presented in this research.

Tables 1 and 2 show the results of extracts obtained at room temperature. Nearly the same results were obtained at an extraction temperature of 36°C. The original Tables of all three experiments are not given in this report.

Based on the data of Table 1 it was found that extraction with 95% alcohol was not favourable in regard to taste and aroma.

The reviewed data showed that the best extraction was achieved with 50% alcohol, during 20 days at room temperature with a 1:1 ratio of fruit to alcohol (Table 2).

The second experiment was designed from the results of the first. Therefore the extraction was carried out at room temperature, with 50% alcohol during 20 days. Removal of water by drying resulted in a requirement

Table 1. Effect of extraction conditions on the chemical and sensory characteristics of persimmon extracts[a]

Sample number[b]	Extraction (days)	Fruit:alcohol (solid:solvent) (w/v)	Nitrogen content (% dry subst.)	Protein (%)	Phenolic compounds (% dry subst.)	Taste scores	Aroma scores
1	10	1:1	0·30	0·02	1·2	6·70	5·31
2	10	1:1·5	0·30	0·02	1·2	6·70	5·31
3	10	1:2	0·25	0·01	1·2	6·40	5·20
4	10	1:3	0·25	0·01	1·2	6·40	5·20
5	20	1:1	0·40	0·03	1·3	7·00	6·80
6	20	1:1·5	0·35	0·02	1·3	6·80	6·65
7	20	1:2	0·35	0·02	1·3	6·60	6·30
8	20	1:3	0·35	0·02	1·3	6·60	6·20
9	30	1:1	0·40	0·03	1·5	7·00	6·70
10	30	1:1·5	0·38	0·03	1·5	6·80	6·60
11	30	1:2	0·36	0·03	1·5	6·60	6·20
12	30	1:3	0·29	0·02	1·5	6·40	6·10

[a] Extraction at room temperature with 95% alcohol (v/v).
[b] Respectively group data of $n = 13 \times 2$ replications.

for a higher ratio of fruit to alcohol of 1:2, 1:2·5, and 1:3 in order to cover the dried fruit. The resulting data were very similar to those shown in Table 2. Results of chemical composition of the second experiment showed that the best extraction was achieved with 50% alcohol, during 20 days at room temperature and with a ratio of fruit to alcohol of 1:2·5. The taste (8·56) and aroma (8·52) scores were nearly the same as for sample 10 in Table 1.

The protocol of the third experiment involving the extraction of dry fruit was based on the results of the second one. During distillation the first and the last fractions were separated. The main distillate contained 60% by volume or more of alcohol. The first distillate fraction contained acetaldehyde, methanol and low boiling esters and comprised 0·1–2·0% of the total distillate volume. The second fraction comprised about 50–70% of the total distillate volume and the last fraction was about 30–50% of the total distillate volume. The major product consisted of 60–80% ethanol by volume. Therefore the raw ingredient in the preparation of liqueurs was the primary wine distillate that contained 52–86% ethanol by volume.

The extraction was done with dry persimmon at room temperature with 50% distillate, obtained after distillation of fermented persimmon mash, and over 20 days with ratios of fruit to distillate of 1:2; 1:2·5 and 1:3.

Chemical compositions from the third experiment showed that the best extraction was obtained with 50% distillate, during 20 days at room temperature and with a ratio of fruit to distillate of 1:2·5. The taste (8·95) and aroma (9·00) scores were the highest among all the data of the three different experiments.

Nitrogen, protein and phenolic compounds were found in moderate levels. Phenolic acids contribute to the aroma (or bouquet) and pigmentation of wines, beers and also fruit extracts. Unfortunately, the non-volatile phenolics can also cause unpleasant flavours, a lack of oxidative stability, and haze formation. It is known that the formation of turbidity results from the interaction between fruit proteins and phenols (Gorinstein, 1973; Yokotsuka *et al.*, 1983; Selimov, 1984). Flavonoids, including condensed tannins which are found in persimmon juice, have various biological activities (Piretti *et al.*, 1985; Torel *et al.*, 1986). All

Table 2. Effect of extraction conditions on the chemical and sensory characteristics of persimmon extracts[a]

Sample number[b]	Extraction (days)	Fruit:alcohol (solid:solvent) (w/v)	Nitrogen content (% dry subst.)	Protein (%)	Phenolic compounds (% dry subst.)	Taste scores	Aroma scores
1	10	1:1	0·43	0·05	0·9	7·80	7·63
2	10	1:1.5	0·43	0·05	0·8	7·80	7·50
3	10	1:2	0·38	0·04	0·9	7·60	7·42
4	10	1:3	0·38	0·04	0·9	7·45	7·23
5	20	1:1	0·57	0·06	1·0	8·72	8·72
6	20	1:1·5	0·59	0·06	1·0	8·62	8·49
7	20	1:2	0·47	0·05	0·9	8·40	8·33
8	20	1:3	0·45	0·05	0·9	8·15	8·22
9	30	1:1	0·64	0·08	1·1	8·54	8·47
10	30	1:1·5	0·62	0·07	1·0	8·52	8·40
11	30	1:2	0·59	0·07	1·0	8·22	8·04
12	30	1:3	0·57	0·07	1·0	8·05	7·93

[a] Extraction at room temperature with 50% alcohol (v/v).
[b] Respectively group data of $n = 13 \times 2$ replications.

Table 3. Characteristics of liqueurs

Sample number[a]	Preparation of extract[b]	Composition (%)			Taste scores	Aroma scores	Taste & aroma scores
		sugar	alcohol	fruit extract[c]			
1	experiment 1	30	24	20	5·85	5·65	5·750
2	experiment 1	30	24	30	6·25	7·50	6·865
3	experiment 1	30	27	20	5·25	6·25	5·750
4	experiment 1	30	27	30	6·75	7·00	6·875
5	experiment 2	30	24	20	6·00	5·25	5·625
6	experiment 2	30	24	30	6·50	6·80	6·650
7	experiment 2	30	27	20	5·75	5·30	5·525
8	experiment 2	30	27	30	6·30	6·90	6·600
9	experiment 3	30	24	20	6·00	6·00	6·000
10	experiment 3	30	24	30	6·25	6·00	6·125
11	experiment 3	30	27	20	6·20	5·70	5·950
12	experiment 3	30	27	20	6·75	7·25	7·000
13[d]	—	30	24	—	4·25	2·30	3·275
14[e]	—	30	24	—	6·15	5·60	5·875

[a] Respectively group data of $n = 13 \times 2$ replications.
[b] Experiment 1 = fresh persimmon (1) to 50% alcohol (1).
Experiment 2 = dry persimmon (1) to 50% alcohol (2·5).
Experiment 3 = dry persimmon (1) to 50% persimmon distillate (2·5).
[c] Persimmon extract prepared by three processes respectively.
[d] Afarsemon liqueur (control 1).
[e] Peachtree liqueur (control 2).

phenols in persimmon beverages can form complexes with proteins and metals (Kashiwada *et al.*, 1986; Aruome & Halliwell, 1987). Adsorbents can be added to the extracts to reduce the levels of polyphenols and proteins (Dukovic, 1979; Gorinstein *et al.*, 1984; Schneider, 1988). Determination and concentration of each phenolic present is important to the development of better and more stable fruit extracts. Persimmon extracts were treated with bentonite to decrease the level of phenolics which can cause turbidity. The content of phenolics was determined before and after treatment.

Figure 1 shows seven liqueur samples in comparison with catechin, quercetin and tannic acids, as the most representative acids in phenol composition of persimmon liqueur. The absorption maxima in all standards were between 350 nm and 250 nm. The content of phenols in the liqueur spectrum was similar to data shown by Piretti *et al.* (1985); Belitz & Grosch (1987) and Uchida *et al.* (1989). Absorbance of liqueur fining with bentonite was lower than in untreated samples, indicating the removal of phenols. The samples prepared from extracts with previous fermentation were higher in

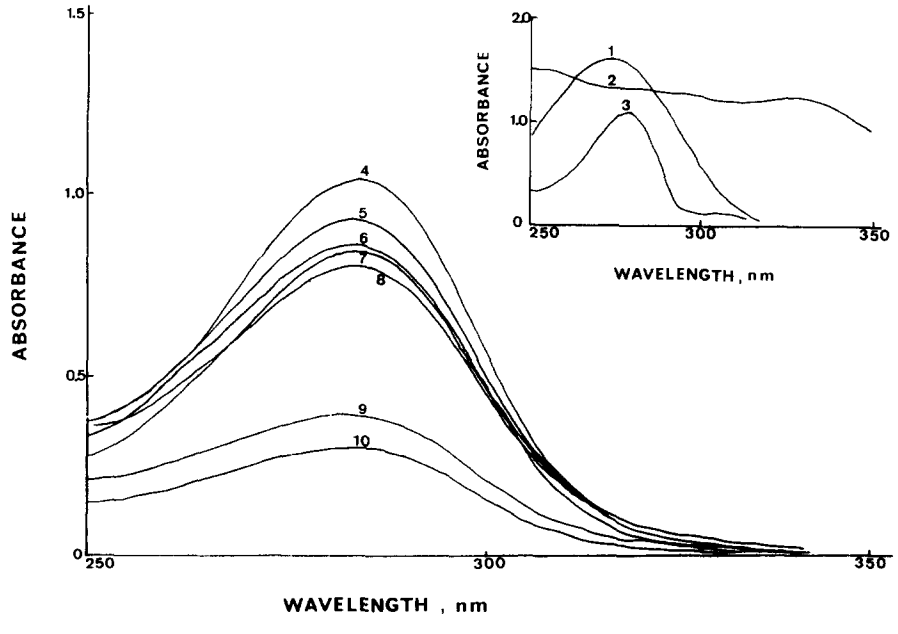

Fig. 1. UV spectra (1) tannic acid, (2) quercetin, (3) catechin, (4) Liqueur of 30% extract (corresponding to sample 4, Table 3), (5) Liqueur of 30% extract (corresponding to sample 2, Table 3), (6) Liqueur of 20% extract (corresponding to sample 1, Table 3), (7) Liqueur of 30% extract (corresponding to sample 6, Table 3), (8) Liqueur of 20% extract (corresponding to sample 12, Table 3), (9) Liqueur of 20% extract (corresponding to sample 7, Table 3), (10) Liqueur of 20% extract (corresponding to sample 5, Table 3).

these phenols (lines 4–6, Fig. 1) than without fermentation (lines 9–10). The numbers of peaks in liqueur samples were smaller than in pure standards, suggesting that the extracted compounds were a mixture of phenols.

In order to improve the quality of liqueur, sugar was added. In addition, treatment of liqueur with clarifying agents and filtration was done. Samples of liqueurs were blended on the basis of 30% sugar, 24% or 27% alcohol, and 20% or 30% extracts obtained by the three processes at room temperature during 20 days. The first experiment was conducted with fresh persimmon (1 w/v) to 50% alcohol (1 w/v); the second experiment with dry fruit (1 w/v) to 50% alcohol (2·5 w/v); and the third with dry fruit (1 w/v) to 50% persimmon distillate (2·5 w/v).

The results, including aroma and taste, are given in Table 3. The scores are the average of three taste panels. According to the data the best results were achieved in samples 12 and 4 which have 20% and 30% fruit extract, respectively. But in sample 4 taste score was very high while aroma was lower than in sample 12. Sample 13, an original Afarsemon liqueur produced from persimmon fruit at an Israeli winery, was used for tasting as control 1. According to the scores only sample 13, failed to exhibit typical taste and aroma characterization of this fruit. Sample 14, an original Peachtree liqueur, imported from Holland, was used as control 2 for comparison in liqueur estimation.

According to Table 3, samples of liqueur prepared from persimmon extracts had high aroma and taste scores. The results of taste and aroma scores with regard to sample 12 are in agreement with the literature (Gyokusenko Sake Brewery, 1981). The use of optimal conditions for fresh fruit extraction resulted in a similar chemical composition to that obtained with sample 4.

The process of preparing fruit extracts without previous drying, fermentation and distillation, must be faster and more effective than using all the stages of the technology.

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