# Lemon Oil to β-Cyclodextrin Ratio Effect on the Inclusion Efficiency of β-Cyclodextrin and the Retention of Oil Volatiles in the Complex

Bhesh R. Bhandari,* Bruce R. D'Arcy, and Lien Le Thi Bich

School of Land and Food, Food Science and Technology, University of Queensland, Gatton, QLD 4345, Australia

Microencapsulation of lemon oil was undertaken with β-cyclodextrin using a precipitation method at the five lemon oil to β-cyclodextrin ratios of 3:97, 6:94, 9:91, 12:88, and 15:85 (w/w) in order to determine the effect of the ratio of lemon oil to β-cyclodextrin on the inclusion efficiency of β-cyclodextrin for encapsulating oil volatiles. The retention of lemon oil volatiles reached a maximum at the lemon oil to β-cyclodextrin ratio of 6:94; however, the maximum inclusion capacity of β-cyclodextrin and a maximum powder recovery were achieved at the ratio of 12:88, in which the β-cyclodextrin complex contained 9.68% (w/w) lemon oil. The profile and proportion of selected flavor compounds in the β-cyclodextrin complex and the starting lemon oil were not significantly different.

**Keywords:** *Microencapsulation; lemon oil; β-cyclodextrin; flavor volatiles*

## INTRODUCTION

Microencapsulation of flavors in a β-cyclodextrin molecule is one of the most effective methods for protecting flavors against oxidation, heat degradation, and evaporation (Hedges et al., 1995; Szente and Szejtli, 1988; Reineccius, 1989; Pagington, 1985). This protection is due to the fact that the flavor molecules are tightly held within the β-cyclodextrin molecule. The interaction between β-cyclodextrin (host) and flavor molecules (guests) may involve total inclusion or association with only the hydrophobic part of the molecule (Shahidi and Han, 1993; Szejtli et al., 1979). Various flavor compounds have different degrees of polarity, molecular size, and chemical compositions. Therefore, flavoring compounds may possess a particular configuration of complex formation with β-cyclodextrin. Szente and Szejtli (1988) reported that short-chain esters and aldehydes are not suitable for complexation with β-cyclodextrin. In a similar way, Reineccius and Risch (1986) found that smaller molecules are less retained than larger molecules. Additionally, they found zero inclusion of the flavor compound isoeugenol in β-cyclodextrin. The variable retention property of β-cyclodextrin may sometimes produce an unbalanced flavor profile for certain flavor powders that are prepared using β-cyclodextrin, particularly when small molecules are involved.

There have been reports of numerous flavor compounds being encapsulated using β-cyclodextrin (Pagington, 1986). However, most of the studies are limited to total flavor retention and stability during storage. As each flavoring material is composed of individual flavor compounds in various proportions, the final note offered by the product will depend on maintaining the original flavor composition during processing. The aim of this study was, therefore, to microencapsulate lemon oil using β-cyclodextrin, and to investigate the overall characteristics, including the profile of flavor volatiles, of the complex as affected by the ratio of lemon oil to β-cyclodextrin used during the complexation process.

* Address correspondence to this author. E-mail: bb@burger.uqg.uq.edu.au. Facsimile: +61 7 5460 1171.

## MATERIALS AND METHODS

**Raw Materials.** Cold pressed lemon oil stored at 4 °C and β-cyclodextrin (Japan Food and Chemical Pty. Ltd., Tokyo) were used as raw materials in the microencapsulation process. The concentration of volatiles in the lemon oil was 97.63% as determined by GC-MS analysis using the internal standard tetradecane. The moisture content of the β-cyclodextrin was 9.94% as determined by the vacuum-drying method (AOAC, 1990).

**Complexation Process.** A precipitation method used by Reineccius (1989) was used to prepare the lemon oil−β-cyclodextrin complex. Fifty grams (±0.01) of β-cyclodextrin was dissolved in 500 mL of an ethanol to water (1:2) mixture maintained at 55 °C (±2 °C) on a hot plate. A predetermined quantity of lemon oil dissolved in ethanol (10% w/v) was then slowly added to the warm β-cyclodextrin solution. During addition of the lemon oil solution, the β-cyclodextrin solution was continuously stirred (magnetic stirrer) and the temperature maintained at 55 °C (±2 °C). The heating was stopped following this addition, and the resultant mixture was covered and stirred for 4 h. The final solution was refrigerated overnight at 4 °C. The precipitated β-cyclodextrin−oil complex was recovered by filtration. This precipitate was dried in a convection oven at 50 °C for 24 h. The powder was then removed from the oven and allowed to air-dry at 25 °C for an additional 24 h in order for the powder to reach its equilibrium moisture content. The final powder at equilibrium was weighed. The amount of powder recovered (dry basis) was calculated by deducting its moisture content. Finally, the lemon oil powder was stored at 25 °C in an airtight bottle.

The following five starting ratios of core material (lemon oil) to β-cyclodextrin were used: 3:97, 6:94, 9:91, 12:88, 15:85. Each starting ratio was prepared and investigated in triplicate. Subsequently, each of the investigated parameters (e.g., total oil, surface oil) were studied in duplicate for each prepared sample.

**Moisture Determination.** The moisture content of the lemon oil powder was analyzed by drying a powder sample (3−4 g) in a vacuum oven at 70 °C for 24 h, under pressure <6.7 kPa (AOAC, 1990).

**Capillary GC-MS Analysis.** The concentrated extracts were analyzed by a standard GC-MS procedure using a

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**Table 1. Recovery of Lemon Powder (Complex) at Various Lemon Oil to β-Cyclodextrin (B-CD) Ratios**

| treatment (starting ratio of lemon oil to B-CD) | initial material (B-CD + lemon oil) (g, db)[a] | | recovered powder (complex) (g, db*) | | |
|---|---|---|---|---|---|
| | mean | STD[b] | mean | STD | recovery[c] (%) |
| 3:97 | 46.557 | 0.02 | 38.150 | 0.15 | 81.94[a] |
| 6:94 | 48.057 | 0.01 | 40.613 | 0.45 | 84.51[b] |
| 9:91 | 49.544 | 0.01 | 41.960 | 0.09 | 84.69[b,c] |
| 12:88 | 51.058 | 0.02 | 48.290 | 0.04 | 94.58[d] |
| 15:85 | 52.567 | 0.03 | 49.286 | 0.22 | 93.76[d,e] |

[a] db*: dry weight basis. [b] STD: standard deviation. [c] Treatments having the same letter are not significantly different ($P > 0.05$).

**Table 2. Effect of Complexation on Co-Crystallization of β-Cyclodextrin (B-CD) and the Recovery of the Powder Complex**

| treatment (starting ratio of lemon oil to B-CD) | B-CD used (g, db) | theoretical co-crystallized B-CD[a] (g, db) | lemon oil used (g) | lemon oil + theoretical co-crystallized B-CD[a] (g, db) | actual recovered powder (g, db) | difference[b] (g, db) |
|---|---|---|---|---|---|---|
| 3:97 | 45.034 | 36.034 | 1.522 | 37.557 | 38.150 | +0.593 |
| 6:94 | 45.050 | 36.050 | 3.007 | 39.057 | 40.613 | +1.556 |
| 9:91 | 45.031 | 36.031 | 4.513 | 40.544 | 41.960 | +1.416 |
| 12:88 | 45.038 | 36.038 | 6.020 | 42.058 | 48.290 | +6.232 |
| 15:85 | 45.036 | 36.036 | 7.532 | 43.567 | 49.286 | +5.719 |

[a] Assumed amounts of co-crystallized B-CD, result from total B-CD used minus its soluble amounts in 500 mL of water ($1.8 \times 5 = 9$ g). [b] Difference in weight between the actual recovered powder and the theoretical amounts of lemon oil used plus amounts of co-crystallized B-CD.

Hewlett-Packard (HP) 5890 Series II gas chromatograph interfaced to a HP 5970 mass selective detector operating in the scan mode ($m/z$ 30−300). Also, the GC-MS system was interfaced to a fused silica capillary column (50 × 0.22 mm i.d. coated with 5% phenylpolysilphenylenesiloxane; BPX-5, SGE Ltd., Melbourne, Australia). A splitless injection system was used together with a helium carrier gas at a flow rate of 0.57 mL/min (column head pressure of 21 psi). Additionally, the column oven was temperature-programmed to rise from 50 °C (1 min initial hold) to 200 °C at 3 °C/min. The injector temperature was 250 °C, and electron impact mass spectral analysis was carried out at an ionization energy of 70 eV and an ion source temperature of 300 °C. Retention indices were determined by interpolation of the GC-MS retention times to those of *n*-alkanes ($C_5$−$C_{22}$ mixture) under identical conditions. One microliter of each concentrated volatile extract was analyzed by GC-MS.

**Total Oil Extraction.** The total amount of oil in the powder was determined using a solvent (hexane) extraction method, followed by GC-MS analysis of the concentrated extract (Yoshii et al., 1992). A typical extraction involved initially mixing a sample of the powder (0.15−0.20 g) with distilled water (8 mL) and hexane (4 mL) in a glass tube (13 × 100 mm), which was then sealed. The solution was then heated in a heating block (BH 5701) at 85 °C for 20 min, with intermittent shaking. The organic phase containing the volatile compounds was decanted, and the aqueous phase was exhaustively extracted with hexane 3 times (3 × 4 mL) at 85 °C using the above method. Three consecutive extractions were considered enough to recover all volatiles, as the fourth extract did not contain any volatiles when analyzed by GC. To the combined hexane extracts was added a solution of the internal standard tetradecane (0.991 mg/mL of hexane): 1 mL for the 3:97 treatment, and 2 mL for the remaining treatments. For each treatment, this final extract was concentrated to approximately 1 mL using a nitrogen stream. Finally, the concentrated extract was transferred to a 2 mL vial, and stored at 0 °C until required for GC-MS analysis.

**Surface Oil Extraction.** The volatile compounds present on the surface of the powder were determined by washing a sample of powder (3−5 g) with hexane (20 mL) using the method of Bhandari et al. (1992). This solvent−powder mixture was gently shaken manually for 20 min. The mixture was then filtered, and the residue was further washed with hexane (10 mL). For each treatment, hexane (1 mL) containing the internal standard tetradecane (0.991 mg) was added to the filtrate, which was then concentrated using a nitrogen stream to approximately 1 mL. This extract of the surface oil was stored at 0 °C until required for GC-MS analysis.

**Identification of Lemon Flavor Compounds.** The structural assignments of lemon oil volatiles were accomplished by comparing the mass spectra and retention indices of compounds with published data (Lis-Balchin et al., 1996; Widner and Collins, 1991), and with the *NBS Registry of Mass Spectral Data* using a computer system.

**Quantitative Analysis of Lemon Oil Volatiles.** Quantitative analysis (100% recovery factor) of the volatile compounds present in the original lemon oil, the total oil volatiles extracted from the powder, and the surface oil washed from the powder involved using the GC-MS instrument and an internal standard (tetradecane), including consideration of response factors (limonene response factor). The weight of each powder sample (β-cyclodextrin and complex) was calculated on a dry weight basis.

**Statistical Analysis.** This experiment was designed based on a completely randomized block design with equal replication. There were five ratios of lemon oil to β-cyclodextrin. Analysis of variance for the retention of flavor volatiles, flavor load, and the amount of surface flavor volatiles from the five treatments were done using SPSS for Windows (6.1). Pair comparisons of the investigated parameters between treatments were done using the least significant difference (LSD) test at the 5% level ($P < 0.05$).

## RESULTS AND DISCUSSION

**Complexation.** The data from this study were obtained over a 10−15 day period from the day of lemon powder production. The recovery of the lemon powder at the equilibrium state is presented Table 1.

As can be seen in Table 1, the lemon oil powder that was recovered is less than the amount of β-cyclodextrin and lemon oil originally used. There was a significantly large increase ($P < 0.05$) in the recovered powder for the 12:88 and 15:85 treatments compared to the 3:97, 6:94, and 9:91 treatments. Additionally, there was a significant increase ($P < 0.05$) in powder recovery for the 6:94 and 9:91 treatments compared to the 3:97 treatment. Examination of the data in Table 2 suggests that this increase was not directly proportional to the amounts of lemon oil added. Therefore, the increase in recovered powder is more likely to be due to increasing

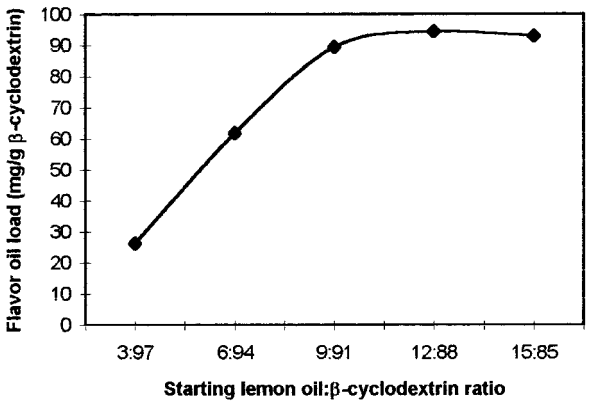


**Figure 1.** Flavor oil load of $\beta$-cyclodextrin as a function of the starting lemon oil to $\beta$-cyclodextrin ratio (average of three replications); treatment difference highly significant ($P < 0.0001$); $LSD_{0.05}$ not significantly different for 12:88 and 15:88 treatments.

amounts of $\beta$-cyclodextrin co-crystallizing from the solution as the initial ratio of lemon oil to $\beta$-cyclodextrin is increased, since the co-crystallized product would be expected to be less soluble than pure $\beta$-cyclodextrin. Starting ratios of lemon oil to $\beta$-cyclodextrin that were greater than 12:88 did not significantly affect ($P > 0.05$) the amount of powder recovered. This result may suggest that here, the maximum co-crystallization of $\beta$-cyclodextrin with lemon oil has been reached.

Such a conclusion is supported by the results in Table 2 where for the first three treatments (3:97, 6:94, and 9:91) the amount of co-crystallized product is not much greater than that for the theoretical product. However, for the 12:88 and 15:85 treatments, there is a sizable increase in the amount of recovered product relative to the theoretical amount of product. Clearly, at these greater concentrations of lemon oil in the starting solution, the lemon oil is being included more strongly by the available $\beta$-cyclodextrin, and the noncomplexed $\beta$-cyclodextrin in the solution is now at its least level. This conclusion is supported by the data presented in Figure 1 where maximum inclusion of lemon oil occurs for these two treatments. In conclusion, it appears that high starting ratios of lemon oil to $\beta$-cyclodextrin produce maximum inclusion of lemon oil, minimum noncomplexed $\beta$-cyclodextrin, and maximum recovery of the lemon powder.

Some other factors which may contribute to the lower recovery of lemon oil powder (Table 1) might be an operational loss as some of the lemon oil was observed to be left in the solution after forming complexes, and some evaporation may occur during the long complexation process. In a solution, there will be an equilibrium of flavors between the liquid and the complexed states which means that there will be some loss of flavors in the liquid phase. A significant amount of surface oil is also expected to be lost by evaporation during the drying step.

Statistical comparison indicated that there was no significant difference ($P > 0.05$) between the 6:94 and 9:91 treatments, and between the 12:88 and 15:85 treatments (Table 1). Such data indicate that an optimum ratio of lemon oil to $\beta$-cyclodextrin during complexation existed at around 12:88.

**General Observations.** The color taint of the lemon oil powder was found to be different from that of the pure $\beta$-cyclodextrin powder. First, all of the treatments produced powders that showed a reflection of a light greenish yellow color relative to the white color of pure $\beta$-cyclodextrin. This difference in color is due to the inclusion of lemon oil pigments into $\beta$-cyclodextrin. In the case of the 15:85 treatment, before the filtration of the final co-crystallized, some colorless droplets of oil were noticed on the surface of the solution. This result indicated that all the pigments present in the commercial lemon oil may have been completely included into the $\beta$-cyclodextrin molecules, even though some of the lemon oil was not included. Additionally, in a subjective observation, the smoothness of the final powder was found to increase as the starting amount of lemon oil was increased. Thus, the pourable property of the lemon powder was increased at higher ratios of lemon oil to $\beta$-cyclodextrin. Finally, although the density of the final powder was not determined, an increase in the volume of the recovered powder was observed from the 3:97 to 15:85 treatment.

**Analysis of Total Oil in the $\beta$-Cyclodextrin Complex.** The initial question that needed to be addressed was the choice of analysis method. Westing et al. (1988), who studied the complexation of orange oil, Reineccius and Risch (1986), who worked with artificial flavors, and Szente and Szejtli (1986), who encapsulated coffee flavor, all chose to isolate flavors from $\beta$-cyclodextrin complexes using steam distillation/solvent extraction and analyze the isolated volatile compounds by gas chromatography (GC). Harangi and Nanasi (1984) and Yoshii et al. (1992) determined the essential oil and $\delta$-limonene, respectively, by extraction of the flavor content of the complexes at high temperature using chloroform, followed by GC analysis of the flavor extract.

Our study used the method of Yoshii et al. (1992) but with hexane as the extracting solvent. Experimentally, trials were done using both hexane and chloroform. It was observed that the extracts obtained using hexane separated more rapidly from the aqueous layer than did those obtained using chloroform. In addition, since hexane is lighter than water, it was experimentally easier to work with since the hexane extract needs only to be decanted from the lower aqueous layer in which the $\beta$-cyclodextrin had precipitated. It is also easy to repeat the extraction process in order to recover the maximum amount of flavor compounds.

A suitable volume (approximately 1 mL) of a concentrated extract was made for each trial. Limonene accounts for about 75% of the chemical composition of lemon oil, while the percentage of other flavor volatiles ranged from 0.7 to 7.0%. This distinction made it difficult to determine the standard volume for each treatment. In fact, an optimum volume of extract was made for each treatment on the basis of the original lemon oil solution (e.g., 19 mg of lemon oil in 2 mL of hexane). The actual volume of each extract does not need to be consistent or accurately known since an internal standard was used to quantify the volatile component of each extracted oil.

According to Shaw (1977), lemon oil contains a significant amount of nonvolatile material that will not be eluted from a GC column. Therefore, an amount of the original lemon oil used to prepare the encapsulated product was analyzed by GC-MS, and the total volatile content was quantified using an internal standard. The volatile content was then incorporated in the above quantifications.

**Total Oil Retention in the Complex.** The results

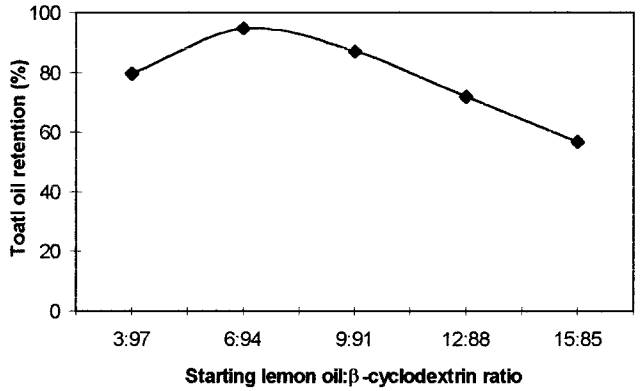


**Figure 2.** Retention of total flavor volatiles (as determined by GC) as a function of the starting lemon oil to β-cyclodextrin ratio (average of three replications); treatment difference highly significant ($P < 0.0001$); $LSD_{0.05}$ not significantly different between 12:88 and 15:85 treatments.

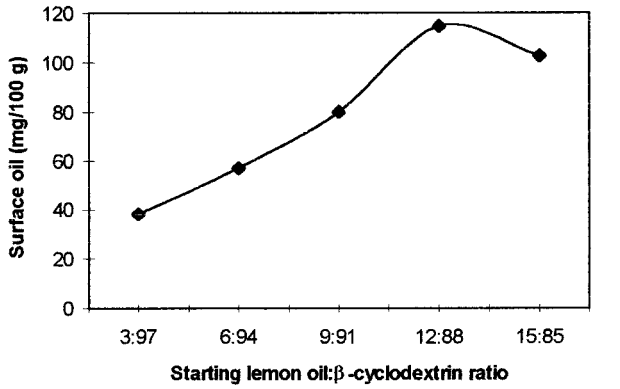


**Figure 3.** Surface retention of flavor volatiles (as determined by GC) as a function of the starting lemon oil to β-cyclodextrin ratio (average of 3 replications); treatment difference highly significant ($P < 0.0001$); $LSD_{0.05}$ not significantly different for 12:88 and 15:85 lemon oil treatments.

in Figure 2 indicate that the retention of lemon oil volatiles (total extracted volatiles as a percentage of the volatile content of the lemon oil used) reached a maximum (94.85%) for the 6:94 treatment. This result was significantly different ($P < 0.05$) from the retention found for the other four treatments. The maximum retention of δ-limonene, the predominant flavor compound of lemon oil, was reported to be 95% when kneading δ-limonene with a mixture of β-cyclodextrin and maltodextrin (Furuta et al., 1994); however, here the flavor retention involves the amount of δ-limonene included into the β-cyclodextrin cavity and absorbed on the maltodextrin, rather than molecular inclusion alone as occurs in our study. Thus, our result compares more than favorably with that of Furuta et al. (1994).

**Inclusion Efficiency of β-Cyclodextrin.** As shown in Figure 1, the maximum load of oil volatiles occurred at the 12:88 treatment. It was 94.51 mg of volatiles/g of β-cyclodextrin, that is, 9.451 g of oil volatiles (or 9.68 g of lemon oil) per 100 g of β-cyclodextrin. This value was significantly different ($P < 0.05$) when compared with the other treatments except 15:85. This capacity is in the range of the theoretical maximum loading for β-cyclodextrin with essential oil of 8−12% (Pagington, 1986). In other studies using β-cyclodextrin, Westing et al. (1988) reported that the total oil content of orange oil−cyclodextrin complexes prepared with an initial oil content of 10% (w/w) was 8.2% (w/w). The lower inclusion capacity of β-cyclodextrin in this study compared to our experiment is probably due to the addition of lower amounts of essential oils, and shorter stirring times after completion of the orange oil addition. It could also be that components of orange oil do not complex as readily with B-CD compared to the components of lemon oil. However, in another study, Szente and Szejtli (1988) have reported the lemon oil content in the complex to be 9.8%, which is close to our result.

**Surface Oil.** The amount of surface oil volatiles as determined by washing the powder with hexane ranged from 35 to 115 mg/100 g of dried powder (Figure 3). The value significantly increased ($P < 0.05$) from the 3:97 treatment through to the 12:88 treatment. The content of surface oil volatiles in the 15:85 treatment was slightly decreased compared to the 12:88 treatment; however, this decrease was not significant ($P > 0.05$). The highest value was found for the 12:88 treatment (115 mg/100 g of dried powder). Westing et al. (1988) reported that the surface oil content for orange oil−cyclodextrin complexes, determined using Soxhlet extraction with pentane, was 431 mg/100 g of dried powder, which is much greater than the level found by us for lemon oil−β-cyclodextrin complexes. In this regard, it should also be noted that the Soxhlet method may extract higher amounts of oil than by simply washing with a solvent.

**Profile of Lemon Oil Volatiles for the β-Cyclodextrin Complex.** Identification of the flavor compounds in the lemon powder and on the surface of this powder was accomplished by GC-MS analysis. Twenty-one flavor compounds were identified in the original lemon oil, the total included lemon oil, and the surface oil.

All the flavor compounds in the original lemon oil were included into β-cyclodextrin, except for α-terpinyl acetate which was not detected in all the total oil extracts for the 12:88 and 15:85 treatments. However, α-terpinyl acetate was detected in the surface oil

**Table 3. ANOVA of the Eight Main Volatiles in the Original Lemon Oil and in the Total and Surface Oil Extracts of the Lemon Oil to β-Cyclodextrin Complex Powders**

| volatile | mol wt[a] | concn (%) in original lemon oil | mean concn of extracts (%) | | concn (%) difference of extracts from original lemon oil[b] | |
|---|---|---|---|---|---|---|
| | | | total oil | surface oil | total oil | surface oil |
| α-pinene | 136.24 | 7.09 | 6.14 | 1.32 | −0.95* | −5.77* |
| β-pinene | 136.24 | 2.89 | 2.83 | 0.35 | $-0.06^{ns}$ | −2.54* |
| β-myrcene | 136.24 | 1.66 | 1.50 | 1.51 | −0.16* | −0.15* |
| limonene | 136.24 | 78.25 | 79.64 | 84.84 | $+1.39^{ns}$ | +6.59* |
| γ-terpinene | 136.24 | 2.20 | 2.17 | 2.51 | $-0.03^{ns}$ | +0.31* |
| linalool | 154.26 | 0.70 | 0.76 | 0.25 | $+0.06^{ns}$ | −0.45* |
| neral | 152.24 | 1.78 | 1.67 | 1.34 | $-0.11^{ns}$ | $-0.44^{ns}$ |
| geranial | 152.24 | 3.66 | 3.76 | 3.52 | $+0.10^{ns}$ | $-0.14^{ns}$ |

[a] Weast and Astle (1979). [b] Where * = significant at 5%, ns = not significant.

extracts for these treatments, suggesting a possible concentration effect.

Additionally, a number of peaks were absent in all surface oil extracts, particularly in those for the 3:97 and 6:94 treatments. Such a finding might result from a loss of certain volatiles during the oven drying process or be due to a concentration effect, or both. The eight main flavor compounds found in commercial lemon oil, namely, α-pinene, β-pinene, β-myrcene, limonene, γ-terpinene, linalool, neral, and geranial, were present in all of the extracts, but in different proportions between the total and surface oil extracts (Table 3).

A comparison was made of the proportion of each volatile compound included into the B-CD molecule. Though the amount of lemon oil encapsulated might be less in a particular treatment, our purpose was to study whether the volatiles exist in the same proportion in the complex as in the original lemon oil. Table 3 compares the proportion of volatiles of all treatments with the original lemon oil. As can be seen in Table 3, the β-cyclodextrin encapsulation of lemon oil produces a lemon oil powder that was not much different in proportion of the major volatiles to that for the original lemon oil. Statistical analysis confirmed that the compositions (percent of total volatiles) of six of the eight major flavor volatiles in the total oil extracts were not significantly different ($P > 0.05$) from the compositions of these volatiles in the original lemon oil (the two exceptions being α-pinene and β-myrcene) (Table 3). In contrast, for the surface oil extracts, only the compositions of neral and geranial were not significantly different ($P > 0.05$) compared with those for the original lemon oil.

Disregarding minor components whose proportions in lemon oil are less than 0.70% of the total oil composition, the molecular weights of the major volatiles (Table 3) suggest one volatile molecule could be included into one β-cyclodextrin molecule (Pagington, 1986). This conclusion is based on the work of Reineccius and Risch (1986), who demonstrated 100% inclusion occurs for linalool (molecular weight of 154). The similarity in composition for the eight main volatiles between the original lemon oil and the lemon oil powder is likely to be due to the small size of all the volatile molecules studied. Therefore, an unbalanced flavor profile of the microencapsulation product using β-cyclodextrin may not occur for a flavoring material such as lemon oil. But, it should also be noted that some minor volatiles which may have an important influence on the total flavor profile have not been compared in this study. A sensory analysis of the flavored product is desirable to assist in this investigation.

## CONCLUSIONS

It was found that a lemon oil powder can be successfully produced by a microencapsulation technique using β-cyclodextrin. The product was similar to the original lemon oil in the proportions of major flavor volatiles. The flavor volatile composition on the lemon powder surface was different from the original lemon oil in both the profile and compound proportions.

Retention of oil volatiles was maximum for a starting ratio of lemon oil to β-cyclodextrin of 6:94 (fresh weight basis). With respect to the cost, the inclusion capacity of β-cyclodextrin is more important than the retention of the volatiles in the product since starting oil not included by the β-cyclodextrin could be recovered. Interpretation of the experimental results suggests that a maximum inclusion capacity of β-cyclodextrin with lemon oil occurs for a starting ratio of lemon oil to β-cyclodextrin of 12:88 or greater, with the resultant maximum recovery of co-crystallized product. The product produced from a starting ratio of lemon oil to β-cyclodextrin of 12:88 was a free-flowing β-cyclodextrin−lemon oil complex containing 9.68 g of lemon oil/100 g of β-cyclodextrin, a commercially acceptable outcome.

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