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Lipolytic Effects of Citrus Peel Oils and Their Components

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This study was conducted to determine the lipolytic effects of eight kinds of citrus peel oils and their components. All of the citrus peel oils revealed lipolytic effects on olive oil model solution ranging from 10.9 to 73.8%. Hakyul (*Citrus natsudaidai* Hayata) showed the highest lipolytic effect (73.8%), followed by yuza (*Citrus junos* Sieb. ex Tanaka, 68.1%) and lemon (*Citrus limonium*, 63.4%), and their effects were comparable with or stronger than that of 5 mM raspberry ketone ($p < 0.05$). Among 17 authentic compounds relating to citrus peel oils, octanal (78.6%) showed the highest lipolytic effect, followed by γ-terpinene (76.3%), limonene (75%), terpinen-4-ol (70.7%), nerol (69.9%), *p*-cymene (67.7%), and geranyl acetate (67.2%), and their effects were stronger than that of 5 mM raspberry ketone ($p < 0.05$). Ethyl acetate, α-pinene, myrcene, citronellal, linallyl acetate, and citronellol exhibited poor lipolytic effect in the model solution. Lipolytic effect was found to be high when the oils included a higher content of γ-terpinene and *p*-cymene. Limonene showed potential lipolytic effect, and its effect is likely to be enhanced by the presence of γ-terpinene and *p*-cymene. It is considered that monoterpene hydrocarbons consisting of one or two double bonds would have stronger lipolytic effect than those having three double bonds.

KEYWORDS: Lipolytic effect; citrus peel oils; hakyul (*C. natsudaidai* Hayata); octanal; γ-terpinene and *p*-cymene

INTRODUCTION

There is increasing interest in biological effects of plant resources. Edible plants may play a role in preventing cardio- and cerebrovascular diseases, carcinogenesis, obesity, and various chronic diseases (*1*−*6*). In recent years many researchers have focused on natural substances with antiobese effects such as capsaicin (*7*−*10*) and conjugated linoleic acid (*11*, *12*). The pungent principle of red pepper is capsaicin, which has been reported to elevate body temperature (*13*), stimulate the secretion of catecholamines (*14*), promote energy expenditure (*15*), and suppress body-fat accumulation (*16*) in experimental animals. However, the administration of capsaicin is strongly pungent and neurotoxic to humans (*17*). Conjugated linoleic acid has been shown to inhibit carcinogenesis (*18*, *19*) and atherosclerosis (*20*) and to have an antiobese effect (*11*, *12*). Ushiki et al. (*21*) reported that raspberry ketone, 4-(4-hydroxyphenyl)-2-butanone, accelerates lipolysis. Raspberry ketone is largely responsible for the raspberry fruits' aroma and is very popular as a synthetic flavor in foods and cosmetics (*22*). Takeshi (*23*) reported that the molecular structure of raspberry ketone is similar to that of capsaicin, and the essential oil of raspberries melts human fat >3 times as much as capsaicin.

Many aromatic plants and their essential oils are used as flavoring agents in a wide range of food, beverage, and confectionary products and fragrance applications. Especially, citrus aroma is popular in most parts in the world. A number of different types of citrus aroma essences are important in the food flavor, cosmetics, and perfume industries. Citrus fruits are rich in essential oils including aroma compounds such as terpene hydrocarbons, alcohols, aldehydes, and esters and bioactive substances such as phenolic compounds, limonoids, carotenoids, and tocopherols. Citrus essential oils have been known to support various biological activities such as antioxidation, anticarcinogenesis (*24*), and free radical-scavenging effect (*25*). However, there is no information in the literature on the lipolytic effects of citrus essential oils. Consequently, the objective of this study was to investigate the lipolytic effects of citrus peel oils and their components to increase the knowledge about substances responsible for biological effects of citrus flavor.

MATERIALS AND METHODS

Materials. Eight kinds of citrus fruits and 17 kinds of authentic compounds were used in this study. In detail, the citrus samples were Valencia orange (*Citrus sinensis* Osbeck forma *Valencia*) from South Africa, navel orange (*C. sinensis* Osbeck forma *Navel*) and lemon (*Citrus limonium*) from America, and yuza (*Citrus junos* Sieb. ex Tanaka), hakyul (*Citrus natsudaidai* Hayata), satsuma mandarin (*Citrus unshiu* Marcov. forma *Miyagawa-wase*), hallabong ([*Citrus unshiu* Marcov × *C. sinensis* Osbeck] × *Citrus reticulata* Blanco), and kumquat (*Fortunella japonica* Swingle) from Korea.

Seventeen kinds of authentic compounds were used in this study. Ethyl acetate was obtained from EM Industries Inc. (Gibbstown, NJ). Nerol, geraniol, geranyl acetate, citronellal, *p*-cymene, and limonene

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were obtained from Wako Pure Chemical Industries (Osaka, Japan). Citral (a mixture of neral and geranial) and citronellol were obtained from Tokyo Kogyo Co., Ltd. (Tokyo, Japan). Myrcene and terpinen-4-ol were obtained from Aldrich Chemical Co. (Milwaukee, WI). γ-Terpinene was provided by Seoul Perfumery Co., Ltd. (Seoul, Korea), and α- and β-pinenes, linalool, linalyl acetate, and octanal were provided by French-Korean Aromatics (Youngin, Korea). Oleic acid was obtained from Wako Pure Chemical Industries, and olive oil was obtained from Daejung Chemicals & Metals Co., Ltd. (Incheon, Korea). Chloroform was obtained from Sigma Chemical Co. (St. Louis, MO). 4-(4-Hydroxyphenyl)-2-butanone was obtained from Aldrich Chemical Co.

Preparation of Citrus Peel Oils. Valencia and navel oranges, lemon, yuza, and kumquat were obtained from a market in Seoul. Satsuma mandarin, hallabong, and hakyul were provided by the National Institute of Subtropical Agriculture of Korea. The peel oil samples were prepared by cold-pressing method. Each fruit (~5 kg) was sliced, and the albedo layer was separated from the flavedo. The peel oils were extracted by hand-pressing from the flavedo and were collected in brine solution on ice. The oil extract was centrifuged at 4000*g* for 15 min at 4 °C. The supernatant was dehydrated with anhydrous sodium sulfate at 5 °C for 24 h and filtered. The oil was stored at −25 °C until analyzed.

Identification of Citrus Peel Oil Composition by GC and GC-MS. The major volatile components of cold-pressed oils were determined by GC and GC-MS. An Agilent 6890N gas chromatograph equipped with a DB-Wax (60 m × 0.25 mm i.d., film thickness = 0.25 μm) fused-silica capillary column (J&W Scientific, Folsom, CA) and a flame ionization detector was used for quantitative determination. A Varian Saturn 2000R 3800 GC (Walnut Creek, CA) linked with a Varian Saturn 2000R MS was used for the identification of the peaks. The analytical conditions were the same as those in the previous paper (*26*). Data calculation was based on the relative peak area percent method. Individual components were identified by comparing their mass spectra with those of reference compounds in the data system of the Wiley library and NIST Mass Spectral Search Program (ChemSW Inc., NIST 98 version database) connected to a Varian Saturn 2000R MS. Other identifications were made by comparison of both mass spectrum and their GC retention data with those of authentic compounds previously analyzed and stored in the data system.

Measurement of Lipolytic Effects. The determination of lipolytic effects of citrus peel oils and authentic compounds was carried out by an Agilent 6890N GC. To determine the sample concentration in the model solution, citrus peel oils at different concentrations (25, 50, 100, 150, and 200 μL) were mixed with 50 μL of olive oil and then made up to volume with chloroform to 1 mL in a brown vessel (3 mL) with a tightly screwed cap with an inner seal made of Teflon. The mixture was shaken with a mechanical shaker and then allowed to stand for 60 min at 37 °C in an incubator. After incubation, 1 μL of the mixture was injected to the GC, and the peak area percentage was evaluated. The lipolytic effects of most of the citrus peel oils were strong in the contents of 50 and/or 100 μL of the oils in the model solution. No significant difference was found between the lipolytic effects of 50 and 100 μL of tested peel oils. From this preliminary experiment results, 50 μL of citrus peel oils was determined to be the optimal concentration in the model solution.

Each sample (50 μL) of 8 kinds of citrus peel oils and 17 kinds of authentic compounds was mixed with 900 μL of chloroform and 50 μL of olive oil in a brown vessel (3 mL) with a tightly screwed cap with an inner seal made of Teflon. The control sample was prepared using choloform instead of peel oils or authentic compounds. 4-(4-Hydroxyphenyl)-2-butanone (raspberry ketone) was employed as a standard compound to examine the lipolytic effects. Fifty microliters of 20 mM 4-(4-hydroxyphenyl)-2-butanone in chloroform was added to the reaction mixture (final concentration of 1 mM) (*21*). The mixture was shaken with a mechanical shaker and left to stand for 60 min at 37 °C in an incubator. After 60 min, the sample solution was shaken with a mechanical shaker for 30 s prior to GC analysis. The lipolytic effect was investigated by evaluating the increase of peak area percent of oleic acid at the gas chromatogram.

Determinations of lipolytic effects of citrus peel oils and their components were carried out by GC. An Agilent 6890N gas chromatograph equipped with a FID was used. A polar (DB-Wax) column (60 m × 0.25 mm i.d., film thickness = 0.25 m, J&W Scientific) were used for GC analysis. The oven conditions and injector and detector temperatures were the same as those given above for the Agilent 6890N GC, and 1 μL of the mixture was injected. The relative peak area percent was used for data calculation.

Statistical Methods. All tests and analyses were run in duplicate and averaged. Results were tested by one-way analysis of variance ($p < 0.05$) using the Statistical Analysis System (*27*) software package. Significant differences between means were determined by Duncan's multiple-range tests.

RESULTS AND DISCUSSION

Volatile Flavor Components of Citrus Peel Oils. The major compositions of citrus peel oils are shown in **Table 1**. The common characteristic of the citrus peel oils used in this study was their high content of monoterpene hydrocarbons. Among these compounds, limonene was the most abundant compound, followed by γ-terpinene and myrcene. Limonene is a major terpene in most citrus oils, its content in kumquat (96.53%) being higher than those in other citrus fruits such as Valencia orange (95.59%), navel orange (95.27%), hakyul (91.01%), hallabong (90.45%), satsuma mandarin (87.11%), lemon (75.11%), and yuza (73.16%). The contents of γ-terpinene of yuza, lemon, and hakyul peel oils were higher than those of other citrus oils, whereas the contents were lower in Valencia orange and kumquat. β-Pinene was present in considerable quantity in lemon. The content of myrcene was higher in navel orange, satsuma mandarin, and hallabong.

Lipolytic Effects of Citrus Peel Oils. The lipolytic effects (percentage of oleic acid produced from olive oil) of eight kinds of citrus peel oil were determined in this study. According to calculation based on the increase of peak area percent of oleic acid, all of the citrus peel oils used in the present experiment revealed lipolytic effects on olive oil ranging from 10.9 to 73.8%, as shown in **Figure 1**. On the basis of breakdown of olive oil into oleic acid and glycerol, significant differences among these fruits were observed. Hakyul showed the highest lipolytic effect (73.8%), followed by yuza (68.1%) and lemon (63.4%), and their effects were comparable with or stronger than that of 5 mM raspberry ketone ($p < 0.05$). The peel oils presenting a lipolytic ratio between 30 and 60% were as follows: Valencia and navel oranges, satsuma mandarin, and kumquat. Hallabong, a new hybrid citrus, showed a weak lipolytic effect.

Lipolytic Effects of Authentic Compounds. The results are shown in **Figure 2**. Octanal (78.6%) showed the highest lipolytic effect, followed by γ-terpinene (76.3%), limonene (75%), terpinen-4-ol (70.7%), nerol (69.9%), *p*-cymene (67.7%), and geranyl acetate (67.2%), and their effects were stronger than that of 5 mM raspberry ketone ($p < 0.05$). The lipolytic ratio was ~31% in β-pinene and ~18% in linalool. Ethyl acetate, α-pinene, myrcene, citronellal, linalyl acetate, citral, citronellol, and geraniol showed a slight lipolytic effects, and these eight compounds showed lower lipolytic effects than that of 1 mM raspberry ketone ($p < 0.05$). The lipolytic effects of α-pinene and myrcene were almost 0%.

Lipolytic Effects of Citrus Peel Oils and Authentic Compounds. Among eight kinds of citrus peel oils, the lipolytic effect of hakyul was the highest, and yuza also showed strong lipolytic effect on olive oil, as shown in **Figure 1**. The lipolytic effect of lemon oil was comparable with that of 5 mM raspberry ketone ($p < 0.05$). Octanal, γ-terpinene, and limonene had strong lipolytic effects on olive oil, whereas ethyl acetate, α-pinene, myrcene, citronellal, linalyl acetate, and citronellol showed poor lipolytic effects.

Table 1. Volatile Components[a] of *Citrus* Peel Oils

compound	RI	Valencia orange[b]	navel orange[c]	lemon[d]	yuza[e]	hakyul[f]	satsuma mandarin[g]	hallabong[h]	kumquat[i]
ethyl acetate	900	*[j]	*	*	*	*	0.01	*	1.13
α-pinene	1035	0.49	0.51	0.59	0.84	0.98	0.73	0.48	0.32
camphene	1082	*	0.01	0.74	0.16	0.02	*	0.01	*
undecane	1112	0.07	0.17	0.02	0.81	0.02	0.01	–	0.01
β-pinene	1123	0.10	0.24	6.34	0.29	0.43	0.48	0.10	0.04
myrcene	1168	1.11	2.26	1.01	0.77	0.82	2.11	1.92	0.09
limonene	1235	95.59	95.27	75.11	73.16	91.01	87.11	90.45	96.53
γ-terpinene	1261	0.03	0.11	7.56	9.62	6.03	4.81	0.51	0.05
p-cymene	1282	0.03	0.11	0.39	0.44	0.48	0.25	0.02	0.01
terpinolene	1294	0.01	0.01	0.02	0.02	0.13	0.22	0.11	0.02
octanal	1297	*	0.03	*	*	0.37	0.06	–	–
tridecane	1312	*	0.06	*	*	0.03	*	0.01	0.02
tetradecane	1396	0.01	0.01	0.05	0.02	0.02	0.02	0.06	0.01
linalool oxide	1433/1451	0.17	0.01	0.11	0.19	*	0.10	0.02	0.06
citronellal	1488	0.15	0.02	0.17	2.58	*	0.03	0.19	*
linalool	1553	0.12	0.19	0.43	0.18	0.12	0.63	0.53	0.09
octanol	1566	*	0.01	0.30	0.04	*	0.01	0.01	0.01
linalyl acetate	1579	0.03	0.01	0.01	0.10	0.01	*	*	–
β-elemene	1595	0.01	0.01	0.02	0.01	0.01	*	0.01	0.03
terpinen-4-ol	1612	0.04	*	0.04	0.76	0.01	0.12	*	0.01
citronellyl acetate	1666	0.05	0.01	0.03	0.04	*	0.03	0.04	0.01
citral	1688/1733	0.02	0.04	0.30	0.14	0.04	0.16	*	*
geranyl acetate	1768	0.04	0.01	0.01	0.01	0.04	0.02	*	0.01
citronellol	1774	0.03	0.02	0.07	0.01	*	0.01	0.09	0.01
geraniol	1852	0.01	*	0.01	0.02	*	–	0.01	0.02
nerol	1858	0.02	*	0.02	0.04	*	0.02	–	–
octanoic acid	2083	0.02	0.01	*	0.10	*	*	0.01	0.02

[a] Given in relative peak area percent. [b] *C. sinensis* Osbeck forma *Valencia.* [c] *C. sinensis* Osbeck forma *Navel.* [d] *C. limonium.* [e] *C. junos* Sieb. ex Tanaka. [f] *C. natsudaidai* Hayata. [g] *C. unshiu* Marcov. forma *Miyagawa-wase.* [h] [*C. unshiu* Marcov × *C. sinensis* Osbeck] × *C. reticulata* Blanco. [i] *Fortunella japonica* Swingle. [j] *, <0.005%.

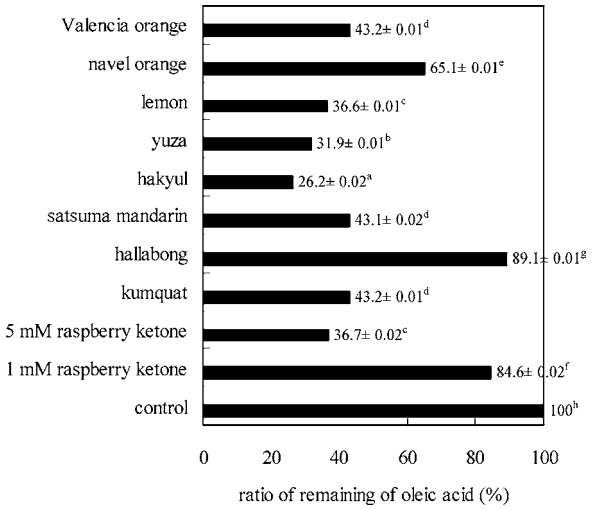

Figure 1. Lipolytic effects of citrus peel oils on olive oil: means of the ratio of remaining oleic acid in olive oil model solution (percnet). Values with the same letters are not significantly different by comparison to control ($p < 0.05$).

Lipolytic effects of citrus peel oils and their components were significantly different, as shown in **Figures 1** and **2**. A possible explanation for the difference in efficiencies found in this study may be the substantial variation in the composition of the citrus peel oils (**Table 1**). The higher efficiency may have been caused by the composition of peel oils having a higher content of octanal and terpenes with the exception of myrcene and α-pinene. Lipolytic effect was found to be high when the terpenes included a higher content of γ-terpinene and *p*-cymene. The higher efficiency of hakyul may be caused by the composition of 0.37% octanal, 6.03% γ-terpinene, and 0.48% *p*-cymene; these compounds were abundant in hakyul compared with the other fruits. In hakyul, the combined percent of octanal,

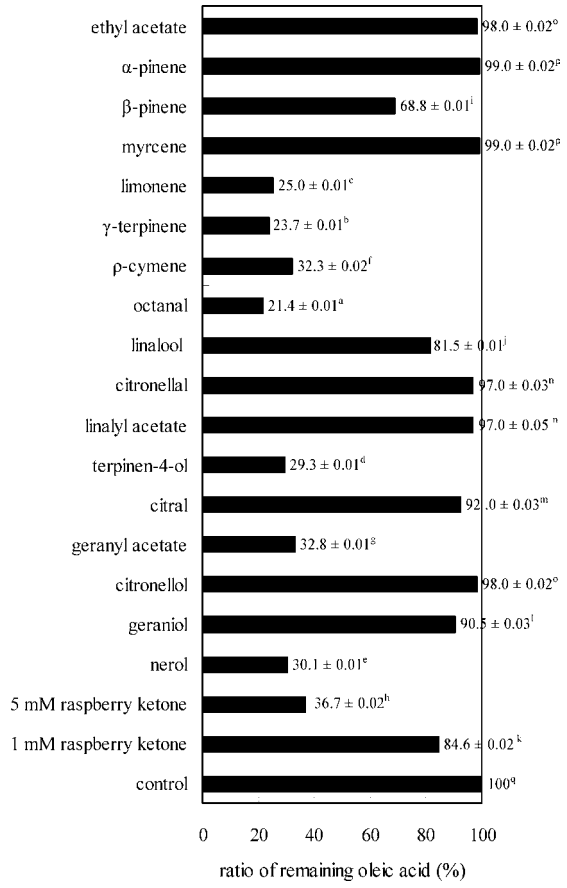

Figure 2. Lipolytic effects of the authentic aroma components relating to citrus peel oils on olive oil: means of the ratio of remaining oleic acid in olive oil model solution (percent). Values with the same letters are not significantly different by comparison to control ($p < 0.05$).

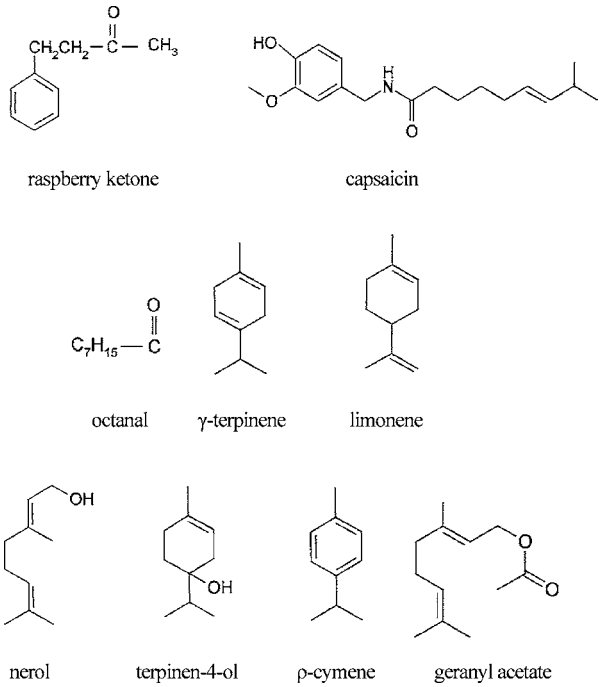

Figure 3. Citrus peel oil components showing high lipolytic effect.

γ-terpinene, limonene, and *p*-cymene was the highest, at 97.89%. Yuza and lemon were also abundant in γ-terpinene and *p*-cymene, and their combined percent ranged from 7.95 to 10.06%. The higher efficiency of yuza and lemon may be caused by the composition of 9.62 and 7.56% γ-terpinene and 0.44 and 0.39% *p*-cymene, respectively.

As the peel oils of kumquat and Valencia and navel oranges were mainly composed of limonene in a proportion of >95%, limonene would play the principal role in the breakdown of olive oil. In addition to limonene content, the lipolytic effect of lemon, yuza, hakyul, and satsuma mandarin peel oils is likely to be further enhanced by a considerable amount of γ-terpinene and *p*-cymene. Satsuma mandarin and hallabong were relatively abundant in linalool compared with others as 0.63 and 0.53%, respectively. However, there was no direct correlation between linalool content and lipolytic effect. Kumquat showed a relatively strong lipolytic effect, although the effective components content, such as γ-terpinene and *p*-cymene, was low. In each citrus peel oil except yuza, terpinen-4-ol and nerol contents were low; nevertheless, these compounds showed high lipolytic effect. Hallabong, which had poor lipolytic effect, contained little or none of the more effective compounds such as octanal, γ-terpinene, and *p*-cymene. Hallabong peel oil was mainly composed of limonene in a proportion of >90%. Factors other than peel oil components that contribute to the lipolytic effect may exist.

The structures of volatile compounds showing high lipolytic effect are shown in **Figure 3** together with the structures of raspberry ketone and capsaicin. Octanal and monoterpenes having one or two double bonds in the ring would be expected to show high lipolytic effect. It is considered that monoterpene hydrocarbons consisting of one or two double bonds such as γ-terpinene and limonene would be stronger to break the lipid than those of three double bonds such as *p*-cymene. Nerol, an open-chain monoterpene with two double bonds, also presented higher lipolytic effect than geranyl acetate, an open-chain monoterpene with three double bonds.

Monoterpenes are predominant plant product and are most often isolated as the major components of the essential oils from plant material. The general compounds found in *Citrus* oils are limonene, γ-terpinene, terpinolene, and so on. The *p*-menthane skeleton such as limonene and γ-terpinene appears to represent the most stable monoterpene structure (*28*). Most of the naturally occurring acyclic monoterpenes have pleasant odors. The simplest acyclic monoterpenes such as nerol can be formed directly from geranyl pyrophosphate or geraniol (*29*). The flavor compounds showing high functional properties have the isopentenyl group in their chemical structures (*30*). Recently, it has been discovered that citrus essential oils and their components such as limonene, consisting of terpene hydrocarbons, have various functional and chemopreventive properties in model solution. The hydrophobicity and conjugated double bond of flavor components seem to be important to the functionality. However the mechanism among these terpene compounds is not yet clear (*24*, *30*). Although the present study was carried out in a given reaction medium, lipolytic function of citrus peel oils and their components was found. This positive result will contribute to the improvement of application for food processing. Further in vivo studies are required to know the mechanisms of lipolytic effect of citrus peel oils.

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