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学位論文全文に代わる要約
Extended Summary in Lieu of Dissertation

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学位論文題目：
Title of Dissertation

A method for extracting aroma compounds from edible oils/fats and high-fat foods
（食用油脂および高脂肪食品からの香気成分抽出法）

学位論文要約：
Dissertation Summary

In this study, a new concept, the oiling-out effect, was demonstrated and defined using a model study. The oiling-out effect dramatically improved the efficiency of extracting aroma compounds from edible oils/fats and high-fat foods using liquid-liquid extraction (LLE), which consists of hexane and methanol. An effective method for extracting aroma compounds in edible oils/fats and high-fat foods based on the oiling-out effect was proposed.

Chapter 1: The importance of understanding the native aroma profile in edible oils/fats and high-fat foods was described. Fat is an essential nutrition for us and is mainly used for energy and a component of cell membrane. The fat-rich foods are palatable for us in general. Volatile compounds in food affect the palatability and eating behavior of the food. Conventional methods, head-space solid-phase microextraction (HS-SPME), solvent extraction (SE), and solvent assisted flavor evaporation (SAFE), for extracting volatile compounds in oily samples were introduced and then their pros and cons were organized. A large amount of fat in food interferes with the extraction and concentration of volatile compounds. To reveal the native profile of aroma compounds in edible oils/fats and high-fat foods, an effective approach is desired. I focused on a big gap of the partition coefficient (log *P*) between medium- and long-chain triacylglycerols and volatile compounds and hypothesized that the volatile compounds and triacylglycerols could be separated by LLE.

Chapter 2: To demonstrate the oiling-out effect, a model study was conducted. It was revealed that the distribution ratio of the hexane layer and methanol layer on LLE was changed with increasing the amount of cocoa butter, which consists of long-chain triacylglycerols. In addition, the cocoa butter was in the hexane layer, and the volatile compounds were collected in the methanol layer. The recovery test showed that decanoic acid was pushed out from the hexane layer contained cocoa butter to methanol layer. Although the log *P* value of decanoic acid is closer to hexane than methanol, the content of decanoic acid in the methanol layer increased significantly. These results indicate that the polarity of the hexane layer decreased dramatically with added cocoa butter, resulting in pushing out decanoic acid from the hexane layer to the methanol layer. These phenomena described above were defined as the oiling-out effect.

Chapter 3: To establish an effective method for extracting volatile compounds in high-fat foods, the extraction method based on the oiling-out effect was demonstrated using dark chocolate. The hexane and methanol bilayers named the oiling-out assisted liquid-liquid extraction (OA-LLE) allowed us to separate triacylglycerols and volatile compounds in dark chocolate. As a result, the aroma extract was obtained from only 5.0 g dark chocolate. Compared with SE and HS-SPME, many and various kinds of volatile compounds were extracted using OA-LLE. The recovery of semi-volatile compounds (exceeding retention index 2000) were especially improved.

Chapter 4: To establish an effective method for extracting volatile compounds in edible oils/fats, OA-LLE was demonstrated using extra virgin coconut oil (EVCO). This method consists two small scale LLE, that is, it is a simple, rapid, no heating, and easy performing method. As with the dark chocolate, the aroma extract was obtained by only 5.0 g of EVCO. Sensory analysis revealed that the aroma extract from OA-LLE maintained similar aroma of the reference sample (EVCO). The volatile compounds in EVCO were listed and

the enantiomer ratio of δ-lactones was revealed using a gas chromatography-mass spectrometry (GC-MS). Using odor activity value, 14 of potent odorants for EVCO were also revealed. It was also demonstrated that the aroma extracts of olive oil and beef tallow were successfully obtained using OA-LLE. These results indicate that OA-LLE is a powerful method for isolating volatile compounds from edible oils/fats (100% oil matrix), which have the strongest matrix effect.

Chapter 5: Further application of OA-LLE was proposed using extra virgin olive oil (EVOO). Some oily foods contain non-volatile compounds such as pigment and flavonoids in addition to triacylglycerols. When the foods are extracted by OA-LLE, the aroma extracts are contaminated by non-volatiles in some cases due to a solvent-based extraction. Indeed, the OA-LLE extracts from EVOO showed dark green. To completely remove the non-volatiles from the aroma extracts, OA-LLE followed by SAFE (OA-LLE + SAFE) was performed. The aroma extracts obtained by OA-LLE + SAFE showed no color. Using OA-LLE + SAFE, 41 of volatile compounds were identified in the aroma extracts obtained by only 5.0 g of EVOO (cv. Hojiblanca). For gaining a deeper insight into the aroma profile of EVOO, OA-LLE × 3 + SAFE was conducted. Using 15.0 g of EVOO (cv. Hojiblanca), 59 volatiles were identified by GC-MS. A wide range of volatile compounds in EVOO was revealed using OA-LLE × n + SAFE, and this approach should help to characterize the aroma quality of EVOOs.

Chapter 6: Overall summary, discussion, and conclusion were given. The oiling-out effect is similar to the salting-out effect of chemicals in aqueous solution by adding sodium chloride. For the oiling-out effect, adding the medium- and/or long-chain triacylglycerols, changes the polarity of the hexane layer (low-polar organic phase) and pushes out relatively hydrophilic compounds into the methanol layer (polar organic phase). In terms of polarity, the oiling-out effect is a similar concept to the salting-out effect. Further studies in the field of physical chemistry will be needed to fully explain the oiling-out effect. The salting-out effect is used for many researches and industries. Here, the oiling-out effect focused on the use for extracting aroma compounds in oily foods. In the near future, the oiling-out effect should be useful for other researches and industries as with the salting-out effect.

The current extraction method has overcome the strong matrix effect of oil, resulting in that an extraction efficiency of volatile compounds was dramatically improved. The method based on the oiling-out effect is useful to understand the aroma profile of edible oils/fats and high-fat foods. The findings should help to investigate the native aroma profile of edible oils/fats and high-fat foods and will encourage researches in the field of not only food science and its industry but also health science.