

## Study on Non-Enzymatic Lactone Release from Dehydrated Butter Oil

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## **Study on Non-Enzymatic Lactone Release from Dehydrated Butter Oil**

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Butter oil is mainly used to enhance the buttery and milky flavor in production of recombined milk and butter, manufacture of ice cream, confectionary, and fat spreads. The flavor of butter oil directly affects the quality of the end product. Thus, it is important to use the high quality butter oil which can be indicated by the extraction and analysis of volatile flavor compounds (VFC). Since the existing extraction technique prior to GC-MS analysis has deficiency in VFC extraction from oily sample, the first part of this research was devoted to the low-density polyethylene (LDPE) membrane pouch extraction. This method was developed to extract VFC from butter oil prior to GC-MS analysis. Butter oil represented for oily sample with complex flavor compounds. The butter oil was placed in the pouch and heat-sealed for extraction in diethyl ether. The extraction was taken at 40°C for 1 h and followed by GC-MS analysis. The result showed the wide spectrum of VFC including 12 fatty acids and 7 lactones, which characterized butter oil aroma. The achievement of separation was explained by the molecular sieve effect of LDPE membrane. The relatively small and medium molecules such as VFC could diffuse through the amorphous region of the LDPE membrane while the relatively large molecules such as triglycerides in butter oil could not diffuse through the region. Thus, the VFC were extracted from butter oil to diethyl ether. The percent recoveries of fatty acids and lactones by this method were 1.00-1.74% and 10.1-11.2%, respectively.

Sensory evaluation of aroma recombine and reference butter oil was assessed to indicate the accuracy of the extraction. The aroma recombine was prepared from 12 odorants in the same concentration as determined in butter oil. The average sensory scores and standard deviations from 10 panelists showed that creamy odor and buttery odor had high scores in both butter oil and aroma recombine. These odors characterized the main aroma of butter oil while sweet odor was undertone of the creamy odor. The sensory scores showed that the components extracted by LDPE membrane pouch method were matched the odor character in butter oil.

The next part is specific lactone release from dehydrated butter oil by non-enzymatic treatment. The effect of moisture, temperature and storage time on lactone release in butter

oil was studied. The accumulated lactone concentrations were determined along with moisture content in butter oil samples during storage. The dehydrated samples had moisture content reduced from 948 ppm to 280 ppm and were kept at 5°C or 20 °C for 4 weeks. After storage, the samples were weekly extracted by LDPE membrane pouch method and analyzed by GC-MS. The lactone concentration highly increased in the first two weeks and remained at high level while there was no increase of free fatty acids. Lactones potential were also manipulated to determine the ability of the specific lactone release.

The lactone formation mechanism was proposed with the intermediate structure of 6-membered-ring, which composed of 4- or 5-hydroxy fatty acid glyceride ester and water molecule. Lactone potentials indicated that butter oil with 750 ppm moisture content and kept at 20°C for 3 weeks gave the highest lactone release. The result showed that  $\delta$ -tetradecalactone and  $\delta$ -dodecalactone were predominance in the dehydrated butter oil which gave a pleasant and rich butter flavor after storage without enzyme application.

In conclusion, LDPE membrane was recognized for its permeability and act as molecular sieve. In cooperated with organic solvent, LDPE membrane can separate small molecules such as VFC from large molecules of triglycerides. The method was developed to establish the efficient methodology for VFC extraction from butter oil prior to GC-MS analysis without depending on specific apparatus. The generated lactones along with the former fatty acid were expected to characterize the buttery and creamy aroma for butter oil samples. The specific lactone release is the alternative method for lactone generation in flavor industry.

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### 論 文 内 容 の 要 旨

Butter oil is mainly used to enhance the buttery and milky flavor in production of recombined milk and butter, manufacture of ice cream, confectionary and fat spreads. The flavor of butter oil directly affects the quality the end product. Thus, it is important to use the high quality butter oil which can be indicated by the extraction and analysis of volatile flavor compounds (VFC). Since the existing extraction technique prior to GC-MS analysis has deficiency in VFC extraction from oily sample, the first part of this research was devoted to the LDPE membrane pouch extraction. This method was developed to extract VFC from butter oil prior to GC-MS analysis. Butter oil represented for oily sample with complex flavor compounds. The butter oil was placed in the pouch and heat-sealed for extraction in diethyl ether. The extraction was taken at 40°C for 1 h and followed by GC-MS analysis. The result showed the wide spectrum of VFC including 12 fatty acids and 7 lactones, which characterized butter oil aroma. The separation of VFC from large molecules of triglycerides was explained by the molecular sieve effect of LDPE membrane. The relatively small and middle molecules such as VFC could diffuse through the amorphous region of the LDPE membrane while the relatively large molecules such as triglycerides in butter oil could not diffuse through the region. The percent recoveries of fatty acids and lactones by this method were 1.00-1.74% and 10.1-11.2%, respectively.

Sensory evaluation of aroma recombine and reference butter oil was assessed to indicate the accuracy of the extraction. The aroma recombine was prepared from 12 odorants in the same concentration as determined in butter oil. The average sensory scores and standard deviations from 10 panelists showed that creamy odor and buttery odor had high scores in both butter oil and aroma recombine. These odors characterized the main aroma of butter oil while sweet odor was undertone of the creamy odor. The sensory scores showed that the components extracted by LDPE membrane pouch method were matched the odor character in butter oil.

The next part is specific lactone release from dehydrated butter oil by non-enzymatic treatment. The effect of moisture, temperature and storage time on lactone release in butter oil was studied. The accumulated lactone concentrations were determined along with moisture content in butter oil samples during storage. The dehydrated samples had moisture content reduced from 948 ppm to 280 ppm and were kept at 5°C or 20 °C for 4 weeks. After storage, the samples were weekly extracted by LDPE membrane pouch method and analyzed by GC-MS for volatile flavor compounds. The lactone concentration highly increased in the first 2 weeks and remained at high level while there was no increase of free fatty acids. Lactones potential were also manipulated to determine the ability of the specific lactone release.

The lactone formation mechanism was proposed with the intermediate structure of 6-membered-ring, which composed of 4- or 5-hydroxy fatty acid glyceride ester and water molecule. Lactone potentials indicated that sample with approximately 750 ppm moisture content and kept at 20°C for 3 weeks gained the optimum condition for lactone release.  $\epsilon$ -Tetradecalactone and  $\delta$ -dodecalactone were predominance in the dehydrated butter oil which gave a pleasant and rich butter flavor after storage without enzyme application.

In conclusion, LDPE membrane is recognized for its permeability and act as molecular sieve. In cooperated with organic solvent, LDPE membrane can separate small molecules such as volatile flavor compounds from large molecules of triglycerides. The method was developed to establish the efficient methodology for volatile flavor compound extraction from butter oil prior to GC-MS analysis without depending on specific apparatus. The generated lactones along with the former fatty acid were expected to characterize the buttery and creamy aroma for butter oil samples. The specific lactone release is the alternative method for lactone generation in flavor industry.