Extraction of essential oils from leaves of the Japanese Lindera umbellata Thunb. by using microwave heating distillation method

Y Yamamoto, T Nawate, and S Mitsudo
Research Center for Development of Far-Infrared Region, University of Fukui, 3-9-1 Bunkyou, Fukui 910-8507, Japan

E-mail: yuuta14@gmail.com

Abstract. In order to investigate effective utilization methods of forest resources and an advanced microwave distillation method, a microwave vacuum distillation system has been developed with commercially available microwave oven. By using this system, essential oil extraction from the Japanese Lindera umbellata Thunb. was carried out under the vacuum condition. The qualitative and quantitative analysis for the components of the essential oil was performed by GC/MS. As the result, the content percentages of nerolidol, selinene and linalool were different from those obtained with the traditional method.

1. Introduction
There are many studies on the essential oils using microwave heating method. The microwave heated distillation method is attracting attention because it enables low cost distillation and it enables different extraction components as compared with the traditional methods [1]. Products market related to aromatherapy is growing 1.2 times over the period from 2011 to 2015 [2]. It is expected that the demand for essential oils will be expected to increase in the forthcoming years. Especially, essential oils from plants, which are inherent in particular countries and regions, are drawing attentions.

On the other hand, in Japan, the efficient use of the forest resources is an important issue in the forest industry. Using forest resources not only as timbers but also as raw materials for essential oils can contribute to solve this problem. Previously, we have studied ceramics sintering by high-power microwave heating [3]. Using these experiences, we have introduced a microwave vacuum distillation system developed by using commercially available microwave oven. The microwave heating has several advantages and interesting features. One of the important features is the direct heating of the core region inside the target. As the high speed heating is possible, the heat treatment time and energy can be reduced. In addition, each component has different absorption rate for the irradiated microwaves. Therefore, it is possible that the characteristics of extracted essential oils can be controlled with the microwave irradiation parameters.

The essential oils of Kuromoji which has a very good smell are extracted as the Japanese aroma, and their components are well studied [4]. So in this paper, we compare the results of the essential oil using this method with one using traditional method.
2. Microwave vacuum distillation system

A microwave vacuum distillation system was constructed by using a commercially available microwave oven. Figure 1 shows a schematic drawing of this system. A still pot made of heat-resistant glass with the capacity of 2.5 little is installed in a microwave oven. The plant which is the raw material of the essential oil is placed in this pot and heated by the microwaves. Microwave power can be set as 200 W, 600 W or 1 kW. The steam which consists of essential oil and water is led to the outside of the microwave oven through the still head and sent into the condenser in which the heated steam is cooled down into the liquid state. Condensed mixture of oil and water is collected into a receiving flask. The essential oil is separated from the water, so as to be a floating on the water surface in the receiving flask. The condenser is the cooled by a water-chiller. An aspirator is used for the system vacuum pumping. A needle valve is used to adjust the pressure. In order to maintain the pressure, the aspirator is cooled with water, and its temperature is controlled during the experiment.

Figure 1. A schematic drawing of microwave vacuum distillation system.
1. Microwave oven, 2. Still pot (Vessel), 3. Condenser, 4. Distillate/receiving flask, 5. Chiller, 6. Aspirator (water vacuum pump), 7. Pressure controller (Needle valve), 8. Manometer, 9. Thermometer (steam temperature), 10. Thermometer (water in chiller), 11. Cooling water inlet, 12. Cooling water outlet.

Figure 2 is a photograph of microwave distillation system.

Figure 2. A photograph of our microwave distillation system.
3. Microwave distillation of Kuromoji

In order to investigate the characteristics of microwave heating distillation, the distillation experiments for Kuromoji have been performed. Water of 1 L and leaves of Kuromoji of 300 g were put into the still pot and distilled with the microwave power of 200 W. Temperature of vaper was kept below 80 °C.

The extracted components was analysed using GC/MS(HP6890 Series GC System). Figure 3 shows histogram of the extracted oil. A mass spectrometry (MS) was also performed simultaneously. From this analysis, the top five extracted components from lindera umbellate were identified (see table 1). The indexes indicated in figure 3 correspond to ones in table 1. Nerolidol and selinene are classified as sesquiterpenes. Sesquiterpenes consist of three isoprene units of C_5H_8, and have the molecular formula of C_{15}H_{24}. Therefore, Sesquiterpenes are large and heavy molecule components of this essential oil. Generally, the main component of the essential oil of Kuromoji obtained by traditional distillation method is about 50% linalool which is classified as monoterpenes (C_{10}H_{16}). This result was different from the fact that the main component of the essential oil of Kuromoji by traditional distillation method was linalool which is classified as monoterpenes. It is considered that monoterpenes, which are lighter molecules than sesquiterpenes, are obtained first in distillation at low temperature. However, in this microwave distillation experiment, sesquiterpenes are mainly extracted. Further studies are needed to clarify whether this is due to special effects such as selective heating by microwave distillation method.

![Figure 3. Chromatogram of extracted oil of Kuromoji by microwave distillation.](image)

**Table 1.** Representative component in essential oil detected by GC/MS.

| Peak | Time(min.) | Area (%) | Name | CAS#         | Qual |
|------|------------|----------|------|--------------|------|
| 1    | 13         | 10.08    | 1,6,10-Dodecatrien-3-ol, 3,7,11-tr ((6E)-Nerolidol) | 040716-66-3 | 91   |
| 2    | 13.87      | 7.68     | delta.-Selinene | 028624-23-9 | 89   |
| 3    | 8.58       | 6.98     | 1,6-Octadien-3-ol, 3,7-dimethyl-(Linalool) | 000078-70-6 | 94   |
| 4    | 10.22      | 6.55     | 2,6-Octadien-1-ol, 3,7-dimethyl-(Nerol),               | 000106-25-2 | 72   |
| 5    | 12.59      | 6.2      | Naphthalene, decahydro-4a-methyl-1(β-Selinene) | 000515-17-3 | 86   |

4. Conclusion

We aimed to develop effective utilization methods of forest resources and advanced research on microwave distillation method. So we’ve developed microwave vacuum distillation system by using...
commercially available microwave oven. Essential oil extraction experiment of Japanese essential oil from Kuromoji was carried out under reduced pressure using this system. The qualitative and quantitative analysis of the components in obtained essential oil was performed by GC/MS. As the result, nerolidol and selinene which are classified as sesquiterpenes are mainly extracted. This result was different from the fact that the main component of the essential oil of Kuromoji by traditional distillation method was linalool which is classified as monoterpenes. Usually, it is considered that monoterpenes, which are lighter molecules than sesquiterpenes, are obtained first in distillation at low temperature. However, in this microwave distillation experiment, sesquiterpenes are mainly extracted. Further studies are needed to clarify whether this is due to special effects such as selective heating by microwave distillation method.

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