[ED04] Process design of hydro-distillation in ginger oil production

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Introduction

Currently, there are a few conventional and modern methods of extracting essential oils such as by hydro-distillation (Purseglove *et al*., 1981), solvent extraction (Chrissie, 1996), supercritical fluid extraction (Kelly *et al*., 2002) and microwave extraction (Soud *et al*, 2002). Hydro-distillation is the oldest and most common method of extracting essential oil since it is economically viable and safe. Although distilling equipment has gradually improved through the years, the method for extracting essential oil from the plant has changed very little especially in this region. Thus, the aim of this study was to study the operational conditions involve in the extraction of ginger oil based on the yield of ginger oil using the hydro- distillation process so that recommendations on the improvements on the present design used in Malaysia can be made.

Materials and Methods

The sample

The ginger used in this research was purchased at the Selayang Market, Selangor. As in most spices, the ginger has to undergo a series of pre-treatment during sample preparation to ensure maximum yield and quality of the oleoresin and essential oil (Noor Azian *et al*., 2004). Two types of sample were used; sliced and ground ginger.

Extraction of ginger oil by hydro-distillation

Ginger oil is extracted by hydro-distillation; water distillation and steam distillation. The ginger oil extraction was done at atmospheric pressure and excess pressure. Steam distillation in this study was done by packing 1 cm of the 400 grams of fresh ground ginger on a wire mesh above 7 litre of distilled water and steamed in a closed still proper. However, steam was vented as to ensure atmospheric pressure throughout. Whereas, steam distillation at gauge pressures (0.3 bar, 0.6 bar, 0.9 bar and 1.2 bar) was carried out by not allowing steam out of the vessel over the specified duration.

The water distillation was carried out at atmospheric pressure. The procedures were quite the same as for the steam distillation. The only difference was that the sample was submerged in water rather than put on a tray. The effects of steaming the dried ginger samples prior to hydro-distillation at varied time and pressure were observed. The calculation for the yield of the ginger oil is as follows:

$$\text{Yield (\%)} = \frac{\text{Weight of ginger oil collected (g)}}{\text{Initial weight of sample (g)}} \times 100\%$$

Vapour Pressure Measurement

The design of distillation equipment is based on vapour liquid equilibrium compositions. In this study, the VLE data was collected from experimental works. The VLE experiments were done under vacuum to determine the vapour pressure of the ginger oil and mixture of the ginger oil and water. 60 ml of distil water is filled into a glass-sampling flask (figure 1). The rotary pump and the diffusion pump are switched ON for about 45 minutes before the experiments can be carried out and the vacuum pump is to be switched ON throughout the experiment. Heating will start immediately after a steady pressure recorded by the gauge was achieved. The sample will be heated slowly for 1^0C/min and later 0.05^0C/min when approaching the supposedly boiling point. The boiling temperature is determined when the liquid temperature is the same as the vapour temperature at a constant pressure. The same experimental procedure and amount of sample was carried out for the determination of the boiling point of ginger oil. For the vapour pressure measurement of the mixture of water and ginger oil, the 60 ml of the mixture contain 1 percent of ginger oil and 99 percent water.

The same experimental procedure was also applied.

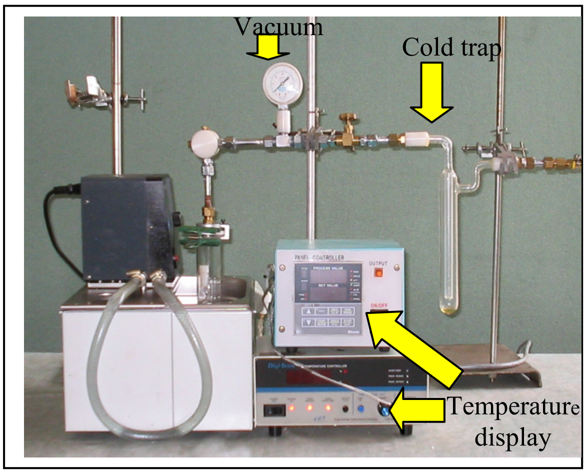

FIGURE 1 Vapour pressure measurement set up.

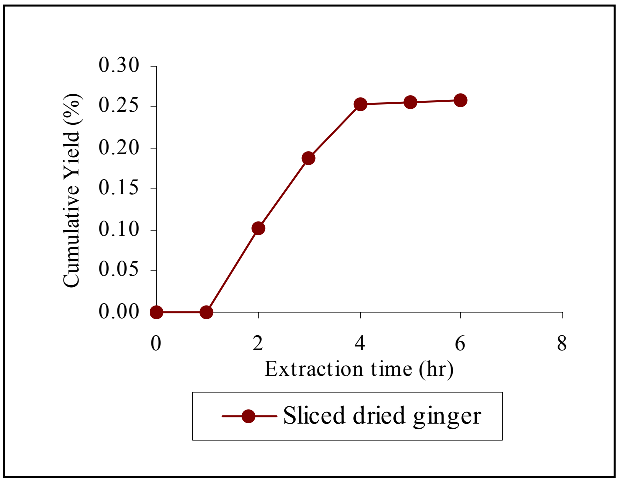

FIGURE 2 The cumulative ginger oil recovery over the extraction time at atmospheric pressure.

Quality analysis

The Refractometer is the fastest and reliable technique in quality control assessments and conformation in this study. The literature value for refractive index (RI) of ginger oil is in the range of 1.4880 to 1.4950. In this study, a Seiko heat flux DSC was also used as a method of determining the degradation temperature of the ginger oil.

Results and Discussions

Effects of extraction time on yield

Figure 2 shows the ginger oil collected during the 6 hours of water distillation. The graph shows that there was no ginger oil collected during the first hour of extraction. This occurs since the energy supplied into the system, was used to heat the water and sample inside the boiler. Ginger oil and water can only be seen condensed after the first hour of extraction time. It was also observed that most of the ginger oil distilled out during the next 2 hours. Prolonging the extraction time will achieve a constant value of the ginger oil collected. Therefore, in order to have minimum energy consumption, 4 hours of extraction is enough to do the extraction.

Effects of pressure on yield

The effects of varied absolute pressure on the yield of the ginger oil was carried out using sliced dried ginger (Figure 3). From the figure, it can be concluded that higher pressure resulted in higher yield. In this case, extraction done at 2.2 bar gave an average oil yield of 0.34 percent. Nevertheless, the yield achieved through this extraction is considered to be low since it is lower than the literature value. Results show that it is not practical to extract ginger oil at excess pressure.

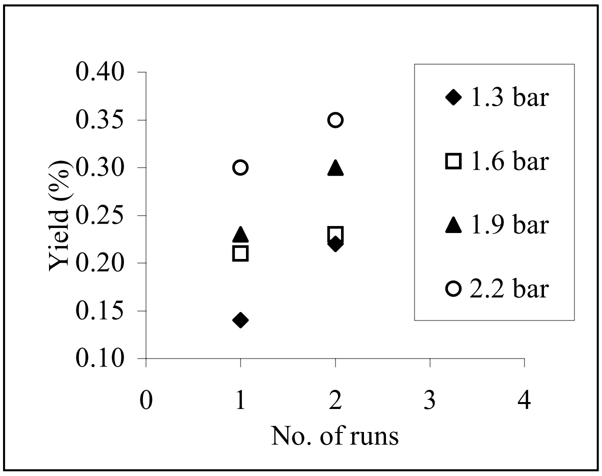

FIGURE 3 The yield of the ginger oil collected by steam distillation at excess pressure for sliced ginger.

Figure 4 showed the results for yield of ginger oil extracted by water distillation and steam distillation of ground dried ginger at atmospheric pressure. From this study, it seemed that water distillation of ground dried ginger gave the highest recovery of the ginger oil (1.53%) whilst the steam distillation gave only 1.42% of ginger oil. Even though a higher ginger oil recovery was achieved by water distillation, this was not the only factor that determined the ideal process. The RI of the ginger oil for both processes was also taken into consideration. The RI of the ginger oil for both processes (Figure 5) showed that ginger oil extraction at excess pressure resulted in a very low values RI in the range of 1.4772 to 1.4775, which was far from the literature value of 1.4880 to 1.4950. This was possibly due to the overheating of the material, while waiting for the vapours to be distilled out.

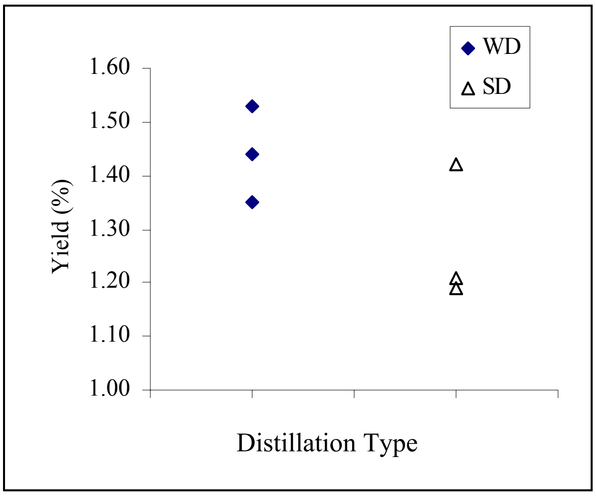

FIGURE 4 Water distillation (WD) and steam distillation (SD) of ground dried ginger at atmospheric pressure.

Vapour Pressure Measurement of Pure Water, Ginger Oil and Mixture of Ginger Oil and Water Under Vacuum

From the DSC study, it was illustrated that ginger oil decomposed at 85.8^{0}C at atmospheric pressure. This justified the need for the VLE experiments to be done under vacuum. From figure 6 it was observed that the boiling point of ginger oil is 141.0^{0}C. The boiling point of the mixture, the temperature at which the total vapour pressure equals 760 mmHg is 97.5^{0}C. From the same graph, the vapour pressure of water at this temperature can be estimated as 710 mmHg. Thus the vapour pressure of ginger oil is (760-710 mmHg) 50 mmHg. Results suggested that ginger oil can be extracted under vacuum at a much reduced pressure.

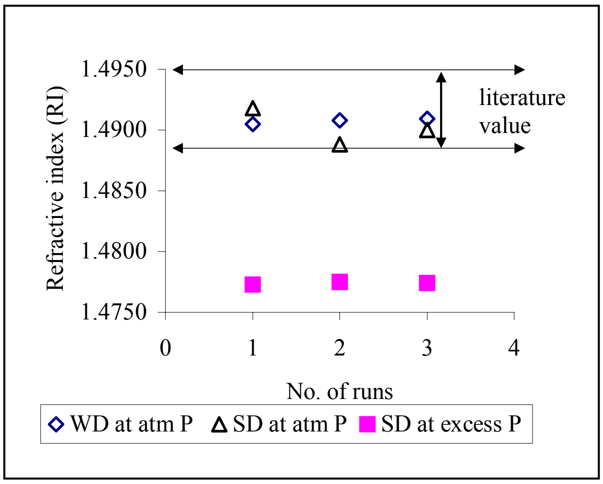

FIGURE 5 The RI for the ground ginger that underwent water distillation (WD) and steam distillation (SD) at atmospheric pressure and excess pressure.

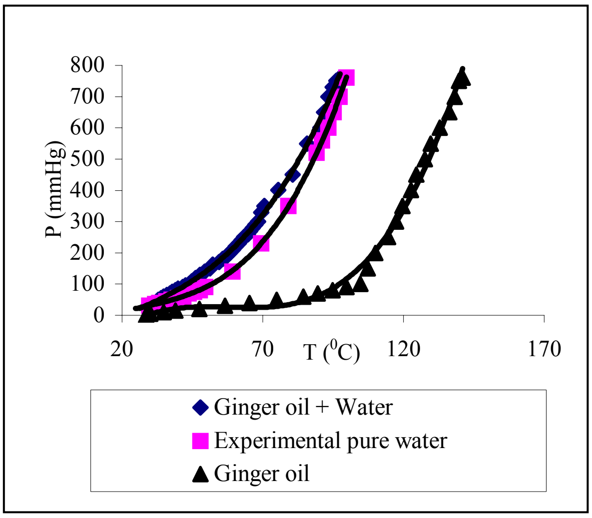

FIGURE 6 Vapour pressure of pure water, ginger oil and mixture of water and ginger oil.

Improve Design of the Steam Distillation Equipment

The equipment required for carrying on distillation of plant materials depends upon the size of the operation and the type of distillation to be carried out. There are there main parts, which are (i) the retort or still proper, (ii) the condenser and (iii) the receiver for the condensate or oil separator. Improvements on the present design used in Malaysia can be made based on the experimental data achieved in this study.

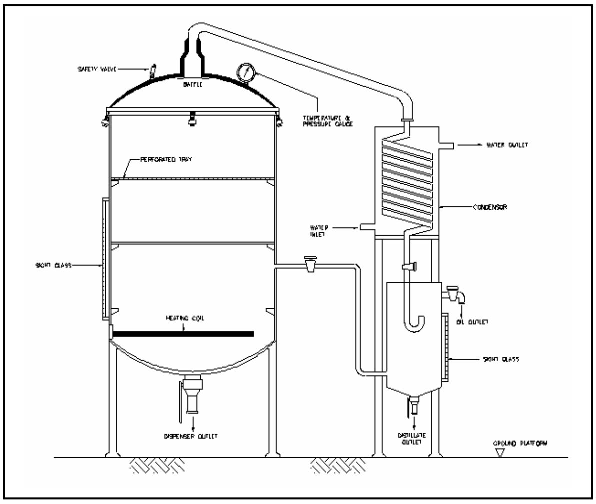

FIGURE 7 The improved steam distillation equipment set up.

The results from this study suggested that ginger oil be extracted by steam distillation. The equipment must be made of stainless steel since ginger oil consists of phenols, which will react with copper and any other metal (Figure 7). For larger and fixed installations, steam distillation is unquestionably offers the most advantages. Among other advantages of extracting ginger oil by steam distillation are as follows:

(1) Without heat damage at ambient temperature throughout.
(2) Without exposure to vacuum stripping.
(3) Without loss of quality or fragile components.
(4) Without being exposed to acidic gases or oxygen.
(5) Without the need for alcohol refinement.
(6) Simple to operate and easy to clean.
(7) Non-hazardous, robust and simple to maintain.
(8) Low energy and labour cost.
(9) Less costly than vacuum distillation.

Ginger oil is a type of essential oil that is not suitable to be extracted at excess pressure since ginger oil decomposed at high temperature as illustrated by the quality analysis of ginger oil in the previous discussion. Therefore, it is much more practical to be extracted at atmospheric pressure. In this case, wet steam is used to distil out the ginger oil.

Minor improvements were done on the design of the equipment currently used in Malaysia. In this design as shown in figure 6, a heating coil was used to boil the water inside the vessel. This will allow sufficient heat supplied into the system without burning the sample inside it. The height of the still depends on the porosity of the plant material. Plant material can be put on two trays at the same time as to allow more ginger oil recovered. A basket can also replace the trays. Another different feature in this design is that a sight glass was designed on the left side of the vessel as shown in figure 7 to make sure there was sufficient amount of water for distillation to happen. A gooseneck leads from the center of the spherical top cover to the condenser. The gooseneck is designed not to be high, slightly curved and gradually descending as to avoid it from acting as a reflux condenser. A baffle is put at the opening to the gooseneck as to filter out particles that might also be evaporated in the process. As a precaution, a safety valve was also put into the design. The still is also well insulated to conserve heat. This is done to avoid excess condensation of steam within the still as a result of heat losses from its surface. The bottom of the retort is also provided by a drain valve sufficiently wide enough to dispense water inside the vessel. This drain valve also serves as an outlet for the wash water, when the still is cleaned.

The condenser used in this design is a vertical coil condenser as shown in figure 7 that in which coils are inserted into a tank supplied with running cold water. The vertical condenser was chosen since it will not take up a lot of space. Long coils were used so that less cooling water is needed to make sure that the contact with the vapours and with the flowing condensate lasts longer. This will also permit the absorption of more heat, so that at the end the temperature of the condensate will approach that of the inflowing cooling water.

The third essential part of the equipment is the oil separator. Figure 7 shows the design of the oil separator. The oil separator permits the removal of water whether the oil being distilled is heavier or lighter than water. Since the total volume of water condensed is always much more than the quantity of oil, the water will be removed continuously. The separator was also designed so that water can be returned into the still and redistilled during distillation as shown in figure 7.

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References

Chrissie, W. (1996). "The Encyclopedia of Aromatherapy." Vermont: Healing Arts Press.

Kelly, C. Z., Marcia, O. M. M., Ademir J. P and et. al. (2002). "Extraction of Ginger (*Zingiber officinale* Roscoe) Oleoresin with CO2 and Co-solvent: A Study of the Antioxidant Action of the Extracts." *Supercritical Fluids* 10: 11.

Noor Azian Morad, Mustafa Kamal Abd Aziz and Nurul Azlina Mohamed. (2004). "Changes of Cell Structure in Ginger During Processing." *Journal of Food Engineering* 62: 359-364.

Purseglove, J. W., Brown, E. G., Green C. L. and Robbins, S. R. J. (1981). "Spices." London: Longman.

Soud, A. A., Yunus, R. M., Aziz, R. A. (2002). "Malaysia.Exploration of Essential Oil Extraction by using Microwave Technique." The proceedings of RSCE and 16th SOMCHE: Malaysia. 1369-1375.