

Novel Closed System Extraction of Essential Oil: Impact on Yield and Physical Characterization

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Abstract. We have developed an extraction technique derived from the purification technique of bulb to bulb distillation, a novel and green approach for the extraction of essential oil from fresh plant materials. This solvent and water free approach is based on a simple principle involving the application of vacuum system in a closed system under a reduced pressure and temperature to extract essential oils. The extraction has been compared with a conventional technique, hydro distillation. Essential oil isolated by solvent free extraction were quantitatively (yield) and qualitatively (aromatic profile) better to those obtained by hydro distillation. The present apparatus permits fast and efficient extraction, avoids water and solvent consumption, less contamination, lesser processes and allows substantial energy savings. The results showed that by using this solvent free extraction technique, the essential oil produced is lighter in color, higher yield, contains cleaner and distinguishable peaks in GC representing better purity and produced a stronger aroma compared to the essential oil produced from hydro distillation.

Keywords: solvent free, essential oil extraction

1. Introduction

Essential oils are concentrated complex mixtures of many natural components that contain volatile aromatic compounds extracts from fragrant plants. It can be extracted from the seeds, flowers, leaves, wood bark, stems, fruits, rhizomes and roots, from a wide variety of plant shrub and tree species. Essential oils are an important source of raw material to the pharmaceutical, cosmetic and food industries. The constituent of essential oil are synthesized in plant by the secondary metabolites and stored in grandular trichomes, oil cells or ducts in plant tissue [1]. Essential oil compositions are commonly mixture of terpenes, mainly monoterpenes and sesquiterpenes, aliphatic compound and aromatic compound [2].

Currently, the conventional methods for extraction of essential oil that are widely used by the industry are steam distillation, hydro-distillation and solvent extraction. These methods have some weaknesses which is highly affecting the yield and quality of the essential oil. For steam distillation and hydro-distillation, the high temperature used can cause chemical modification of the essential oil component especially to the heat sensitive volatile compound [3]. Furthermore, the use of solvent extraction is not an effective way due to the present of solvent residue in the product and also resulting in the loss of the highly volatile component of the essential oil [4]. The disadvantages related to these techniques have led to the searching for new alternative extraction processes. Supercritical fluid extraction is one of the latest techniques used for extraction of essential oil. In contrast to the conventional method, the extraction by supercritical fluid extraction is better because the essential oil produce is high in quality [4]. However, referring to [5] this technique is not cost effective due to the use of super critical fluid for extraction and other compound is also extracted such as resins and the vegetable waxes.

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Considering the fact that the pure form of essential oil with best quality is hard to get, this new method of extraction is invented and tested to discover its potential. Our extraction method works on the principle that boiling point of a substance is lowered when the pressure is reduced. This technique can be applied to any plant that has volatile compounds. The extraction process is done in a vacuum closed system. Vacuum condition is needed to distill and release the oil from the plants oil cells or duct under reduced pressure. The pressure in the system is reduce, making it possible to boil the liquid at lower temperature. The essential oil produced from this technique will be pure as there are no solvent used for extraction. This technique was developed because most essential oils cannot be distilled at atmospheric pressure because the boiling point is too high and distilling at normal boiling point will cause decomposition of compound. So by using this technique, extraction of essential oil can be done at lower temperature to preserve the phytochemical constituents. This solvent free extraction technique can produce high quality essential oil with higher yield due to the closed system.

2. Materials and Methods

2.1. Closed system extraction and solvent free extraction technique

Fresh leaves of aromatic plants were grinded and to break them into smaller pieces and increasing the area of contact. Then, the grind leaves were put in a flask which was connected to another flask as a receiving flask. Firstly, the raw material was cooled down to a very low temperature to prevent decomposition and to avoid premature oil evaporation. Vacuum and nitrogen gas was applied on and off to remove air and replacing it with nitrogen. Then vacuum was applied lastly before extraction begun. Then the receiving flask was cooled to a very low temperature where the essential oil would be condensed. Heat was placed below the raw material flask at certain temperature according to the targeted compound range temperature and pressure. Vapors containing water and essential oil were collected at the receiving flask at the end of the extraction process. Water and oil was separated and anhydrous sodium sulfate was used to dry the excess water.

2.2. Gas chromatography (GC) analysis

The essential oil extracted was analyzed by using Gas Chromatography to study the composition of the oil. The gas phase chromatography analysis was carried out using Gas Chromatography equipped with a flame ionization detector and BPX70 column (25 m x 0.25 mm, df: 0.25 μ m) with a split flow rate of 60 mL /min, nitrogen gas as carrier gas and temperature programming 5 min at 50 $^{\circ}$ C and 6 $^{\circ}$ C /min up to 200 $^{\circ}$ C, injector temperature was 220 $^{\circ}$ C and detector temperature was 235 $^{\circ}$ C.

3. Results and Discussion

3.1. Yield analysis

Table 1:Yield of the closed system extraction, solvent free extraction of various leaves

Types of leaves	Weight of leaves (g)	Weight of essential oil (g)
<i>Chromalaena odorata</i>	50	0.05
Citronella	50	0.45
<i>Baeckea frutescens</i>	50	3.31
Orange peel	50	2.88

Table 2:Yield from steam distillation of various leaves

Types of leaves	Weight of leaves (g)	Weight of essential oil (g)
<i>Chromalaena odorata</i>	50	0.025
Citronella	50	0.255
<i>Baeckea frutescens</i>	50	1.720
Orange peel	50	1.050

3.2. Physical characterization

Table 3: Colour comparison of the essential oil extracted from solvent free extraction and steam distillation of various leaves

Types of leaves	Solvent free extraction	Steam Distillation
<i>Chromalaena odorata</i>	Yellowish	Greenish
Citronella	Pale yellow	Pale green
<i>Baeckea frutescens</i>	Colorless	Light yellow
Orange peel	Colorless	Light Yellow

Table 4: Odor comparison of the essential oil extracted from solvent free extraction and steam distillation of various leaves

Types of leaves	Solvent free extraction	Steam Distillation
<i>Chromalaena odorata</i>	Pungent	Burnt
Citronella	Heavy minty	Minty
<i>Baeckea frutescens</i>	Fresh piney	Burnt piney
Orange peel	Sharp citrusy	Citrusy

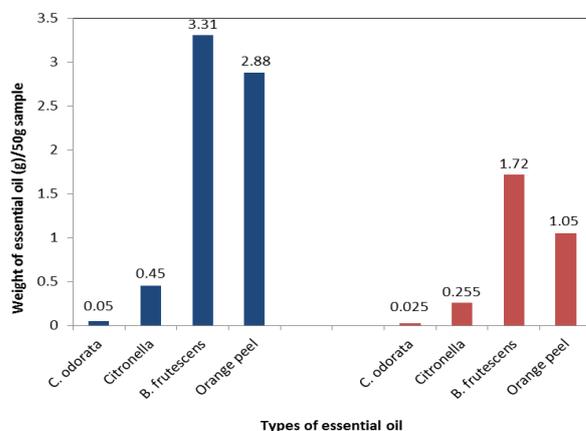


Fig. 1: Comparison chart between solvent free extraction and steam distillation

3.3. Scanning electron microscope (SEM)

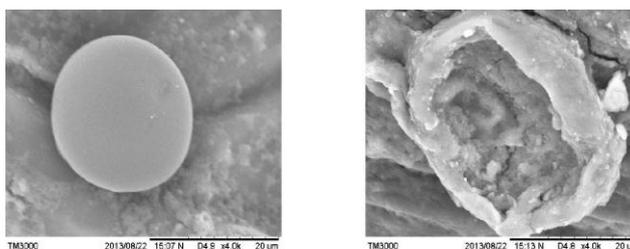


Fig. 2: Essential oil gland (oil cell) before extraction and ruptured essential oil gland after extraction with solvent free extraction

3.4. Analysis by gas chromatography (GC)

From the superimposed graph between solvent free extraction technique and steam distillation in Figure 5, it can be observed that same compound is available in essential oil obtained from the two different techniques. For solvent free extraction technique [Fig. 3] the compound detected at retention time of 6

minute to 20 minute showed higher amount of the compound as the entire peak height is higher compared to the peak for hydro-distillation. The compounds detected at retention time longer than 20 minutes were lesser but distinguishable, on the other hand for the steam distillation technique [Fig. 4], more peaks showed up after 20 minutes and the compound peaks are messier. This implies that new compounds were generated during the extraction which have reduced the intensity peaks for the lower retention time compounds.

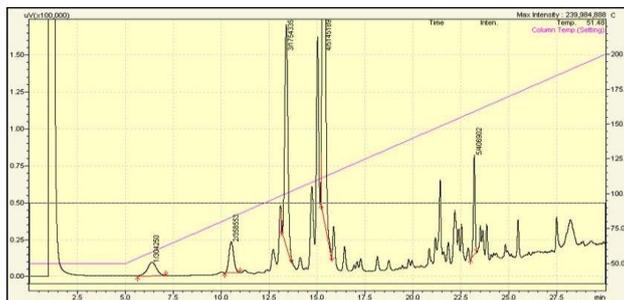


Fig. 3: Graph for GC analysis of extraction by solvent free extraction technique

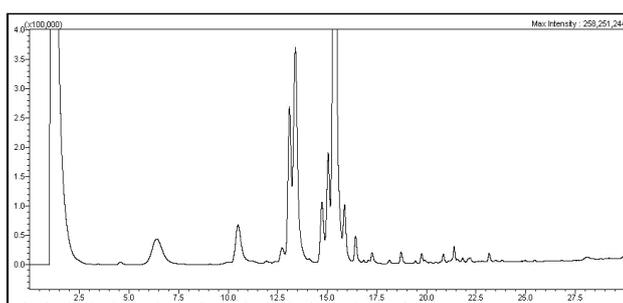


Fig. 4: Graph for GC analysis of extraction by steam distillation technique

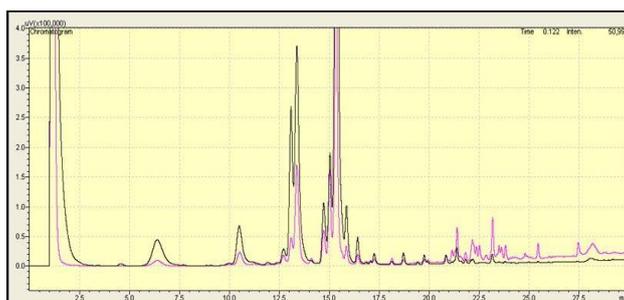


Fig. 5: The superimposed graph between the solvent free extraction techniques indicated by black line and the extraction by steam distillation by pink line.

4. Conclusion

Closed system extraction technique produced superior quality essential oil and higher yields due to zero loss to surrounding or vacuum. This essential oil extraction technique can be the future of essential oil extraction in the industry. This technique can revolutionize, increase and hasten the amount of study on essential oil in the future. This technique saves time and cost efficient since the extraction has fewer processes, no purification needed, contains less impurities and saves energy. The solventless extraction technique is applicable in extraction of essential oil for any plant species that contain volatile and sublimation compound property. This technique is better than the current conventional technique because it can be used to extract essential oil from heat sensitive botanical material since the extraction can be done at room temperature or temperatures below 100 °C.

5. Acknowledgment

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6. References

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