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Extraction of Oil from Eucalyptus Camadulensis Using Water Distillation Method

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Abstract

This work was conducted to study the extraction of eucalyptus oil from natural plants (*Eucalyptus camaldulensis* leaves) using water distillation method by Clevenger apparatus. The effects of main operating parameters were studied: time to reach equilibrium, temperature (70 to100°C), solvent to solid ratio (4:1 to 8:1 (v/w)), agitation speed (0 to 900 rpm), and particle size (0.5 to 2.5 cm) of the fresh leaves, to find the best processing conditions for achieving maximum oil yield. The results showed that the agitation speed of 900 rpm, temperature 100° C, with solvent to solid ratio 5:1 (v/w) of particle size 0.5 cm for 160 minute give the highest percentage of oil (46.25 wt.%). The extracted oil was examined by HPLC.

Keyword

Extraction, Water Distillation, Eucalyptus Oil

Introduction

Eucalyptus oil $C_{10}H_{18}O$, is one of the most important essential oil. The oil is extracted from fresh and dried leaves, in addition to branch tips [1]. Eucalyptus oil has biological effects, antibacterial, antiviral and antifungal components and long history of use against the effect of cold, influenza, other respiratory infection, rhinitis and sinusitis [2]. Essential oils chemistry is very complex; in nature essential oils themselves have many chemical ingredients. Some of them play a major part and others a minor part. The ingredients found in essential oils are organic due to their molecular structure which is based on carbon atoms held together by hydrogen atoms. Oxygen atoms and sometimes nitrogen and sulphur atoms are also present [3]. They can be essentially classified into two groups:

A. **Volatile Fraction:** Essential oil constituting of 90–95% of the oil in weight.
B. **Nonvolatile Residue:** This comprises 1–10% of the oil, containing, fatty acids, sterols, carotenoids, waxes, and flavonoids.

The properties of eucalyptus oil is shown below [4]:

Description: Colourless to pale yellow liquid with an aromatic and camphoraceous. odour and pungent, cooling, spicy taste.
Molecular formula: C10H18O.
Molecular weight: 154.
Specific Gravity at 25°C: 0.920 - 0.925.
Refractive Index at 20°C: 1.4550 - 1.4600.
Optial Rotation at 20°C: -0.5° - +0.5°.
Freezing Point: 0°C - +1°C.

Boiling Point at 760 mmHg: 176 - 177°C.
Flash Point: 49°C.
Melting Point at 760 mmHg: 1.5°C.
Solubility: Slightly soluble in water, soluble in: ethanol, ether, chloroform, and hexane.

Extraction

Essential oils can be extracted using a variety of methods, although some are not commonly used today. Currently, the most popular method of extraction is steam extraction, but as technological advances are made more efficient and economical methods are being developed. These include methods such as solvent extraction, supercritical fluid extraction, and microwave extraction. The suitability of extraction method varies from plant to plant and there are significant differences in the capital and operational costs associated [5].

Principles of Distillation

It is imperative to note that a liquid always boils at the temperature at which its vapor pressure equals the atmospheric or surrounding pressure. For any two immiscible liquids, the total number of molecules present in the vapor phase is greater than the number which would be present if either pure liquid were present alone at the same temperature. Hence, the pressure exerted by the vapor mixture will be greater than that exerted by either pure vapor alone [6]. Consequently, the vapor pressure of the whole system increases, that cause to evaporate essential oil at temperature below its boiling point. Thus, any essential oil having high boiling point can be evaporated with steam in a ratio such that their combined vapor pressures equal the atmospheric pressure [3].

Water Distillation

In the manufacture of essential oils using the method of water distillation, the botanic material is completely immersed in water and the still is brought to the boil. The main characteristic of this process is that there is direct contact between boiling water and plant material [3]. Distillation offers better conditions for the osmosis of oil, because the higher temperature and the movement of water, caused by temperature and pressure fluctuations within the still, accelerate the forces of diffusion to such a point that all the volatile oil contained within the plant tissue can be collected. A special case of water distillation it uses the practice of returning the distillate water to the still after the oil has been separated from it so that it can be re-boiled. The principal behind it is to minimize the losses of oxygenated components. Practical advantages of water distillation are that the stills are inexpensive, easy to construct and suitable for field operation. These are still widely used with portable equipment in many countries. This method protects the oils so extracted to a certain degree since the surrounding water acts as a barrier to prevent it from overheating [6].

Experimental Work

Fresh leaves of *Eucalyptus camaldulensis* was collected from the gardens of Baghdad University, the leaves were taken to laboratory and cut out by a pair of scissors to small part. The schematic diagram of the equipment used is shown in figure 1.

The Clevenger consists of a main tube combined with condenser and graduated tube with glass stopcock. A return tube for the aqueous part of the distillate connects the bottom of the measuring tube and the main tube. Eighty grams of fresh and clean

Eucalyptus leaves were placed into the three necked round extraction
n flask and soaked with water. Clevenger apparatus was linked to the flask as shown in figure 2. The flask was heated using heating mantel. Water and leaves, were mixed and allowed to boil. Water and extracted eucalyptus oil evaporate. The vapour mixture condensed using reflux condenser, from condenser distillate water and oil flow in to gradated tube; as the oil is not miscible with water it may be easily separated and started accumulating and distillate water returning to the flask, after the oil has been separated from it, so that can be re-boiled. The oil was allowed to stand for sufficient time, to be clear and then it was collected and stored in dark glass vial in a refrigerator until it has been tested.

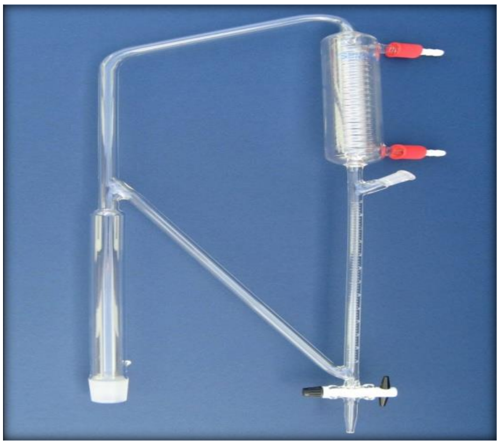

Fig. 1, laboratory glassware Clevenger

Fig. 2, Photographic representation of water distillation unit

Results and Discussion

Effect of Extraction Time

The obtained results are plotted in figure 3. The effect of extraction time on oil extraction was studied until the equilibrium was reached, of particle size 0.5 cm at 100°C with solvent (distilled water) to solid ratio 5:1(ml/g) and agitation speed 300 rpm.

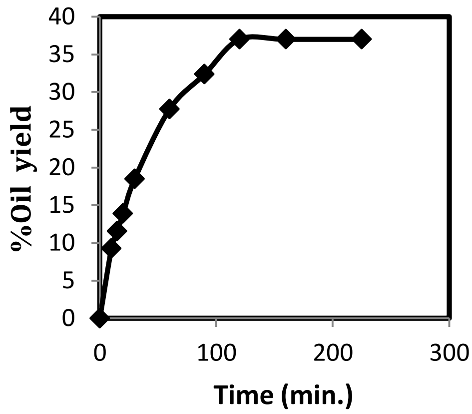

Fig. 3, Effect of extraction time on oil extraction by water distillation *Operating* conditions (P.S.=0.5 cm, S.R. =5/1(ml/g), A.S.=300 rpm, T=100°C)

The results in figure 3 shows, that extraction of oil increased with time. It is also can be observed that the rate of extraction is fast at the beginning of the extraction but gets slow gradually. The reason is that when the meal is exposed to the fresh solvent, the free oil on the surface of particles gets extracted quickly inducing a fast increase in the extraction rate. Furthermore, since the oil concentration is low in the solvent at the beginning of the extraction process, the oil diffuses quickly from the meal to the liquid phase due to the mass transfer effect. As the time passes by, the concentration of oil increases in the solvent resulting in a decrease in the diffusion rate. When the maximum amount of extractable oil is obtained, the oil yield level remains constant even by extending the extraction time; it reaches equilibrium [7]. This results are in agreement with the results which obtained by Cassel and Vargas (2006) [8].

Effect of Temperature

The obtained results are plotted in figure 4. The effect of extraction temperature was examined in the range of 70 to 100°C under condition of particle size 0.5 cm with solvent to solid ratio 5:1(ml/g) and agitation speed 300 rpm until the equilibrium was reached.

At 100°C the liquid boils, as the vapour pressure of liquid equal atmospheric pressure, that cause to ascend the vapour mixture (steam and extractible oil) and yield essential oil .but at temperature below 100°C the vaporized of essential oil with steam cannot occur.

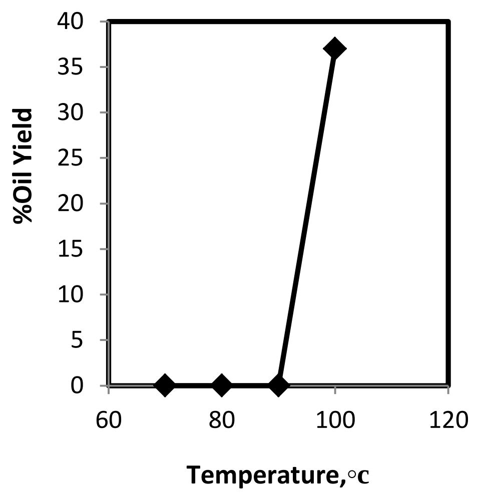

Fig. 4, Effect of temperature on oil extraction by water distillation Operating conditions: (P.S. =0.5 cm, S.R. =5/1(ml/g), A.S. =300 rpm)

Effect of Agitation Speed

The obtained results are plotted in figure 5. The effect of agitation speed was examined in the range of 300 to 900 rpm and compared it with the yield without mixing, under condition of particle size 0.5 cm with solvent to solid ratio 5:1(ml/g) and temperature 100˚C until the equilibrium was reached.

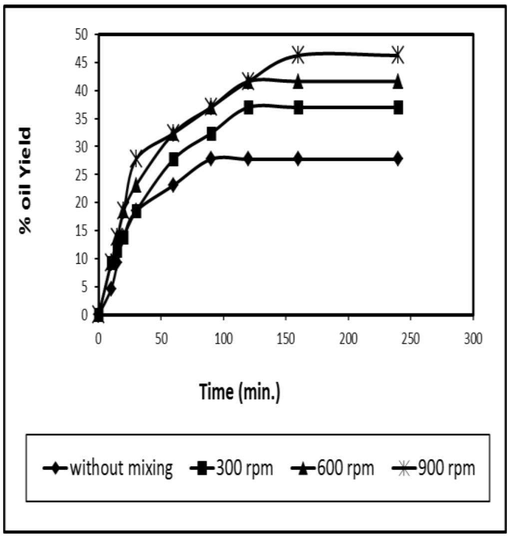

Fig. 5, Effect of agitation speed on oil extraction by water distillation Operating condition: (P.S. =0.5 cm, S.R. =5/1(ml/g), temp. =100ºC)

As seen in figures 5, the oil yield is increased by increasing the stirring speed and there is obvious difference between the two. Figure 3 shows the maximum oil yield without mixing was 27.75 wt.% after 90 minute, but stirring speed of 300 rpm gave yield 37 wt.% after 120 minute and it has reached 46.25 wt.% after 160 minute with stirring speed 900 rpm. From these result the maximum yield of oil by water distillation was 46.25 wt.% after 160 minute with stirring speed 900 rpm. Agitation of the solvent is important because it increases the eddy diffusion and therefore increases the transfer of material from the surface of particle to the bulk of the solution [4] [9].

Otherwise agglomerations of dense material will settle on the bottom and become thermally degraded [3].

Effect of Solvent to Solid Ratio

The obtained results are plotted in figure 6. The effect of solvent to solid ratio was examined in the range of 4:1 to 8:1(ml/g), under condition of particle size 0.5 cm with agitation speed 900 rpm and temperature 100°C until the equilibrium was reached.

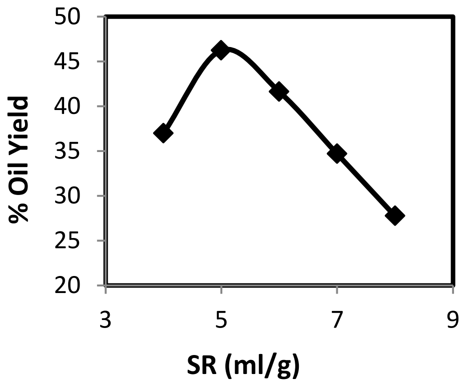

Fig. 6, Effect of solvent to solid ratio on oil extraction by water distillation, Operating conditions (P.S. = 0.5cm, A.S. = 900rpm, temp. = 100°C)

As seen in figure 6, the yield of oil is decreased by increasing the ratio of solvent. The oil yield after 160 minute was 27.75 wt. % at S.R. (8:1), and it has reached 46.25 wt. % at S.R. (5:1). Also observed at S.R. (4:1) the oil yield was 37 wt. % after 120 minute. From these result the maximum yield of oil by water distillation was 46.25 wt. % after 160 minute at S.R. (5:1(ml/g)).

Esters are constituents of essential oils and, in the presence of water, especially at high temperatures, they tend to react with water to form acids and alcohols. If the amount of water is large, the amounts of alcohol and acid will also be large, resulting in a decreased yield of essential oil. And if the amount of water is not enough, as in S.R. (4:1(ml/g)), the plant material may overheat and char [3].

Effect of Particle Size

The obtained results are plotted in figure 7. The effect of particle size was examined in three different size (0.5, 1.5, 2.5cm), under condition of solvent to solid ratio 5:1(ml/g) with agitation speed 900 rpm and temperature 100ºC until the equilibrium was reached.

As seen in figure 7, less oil is extracted from the larger particles compared to the smaller size particles. The reason is that the smaller the size the greater is the interfacial area between the solid and liquid and therefore the higher is the rate of transfer of material; further, the smaller is the distance the solute must diffuse within the solid as already indicated [11]. Therefore, less amount of oil will be transferred from inside the larger particles to the surrounding solution in comparison with the smaller ones [3].

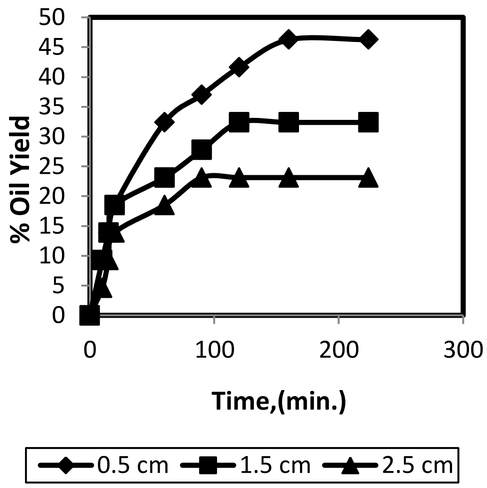

Fig. 7, Effect of particle size on oil extraction by water distillation Operating conditions: (S.R. =5:1, A.S. =900 rpm, temp. =100 ºC)

Conclusions

According to the results obtained from this study, the following conclusions are obtained:

1. The best conditions to yield eucalyptus oil by water distillation method is at 100° C with stirring speed 900 rpm for particle size 0.5 cm and solvent to solid ratio 5:1(v/w) for 160 min.
2. The maximum yield of eucalyptus oil produced by water distillation, under best conditions, was about 46.25 wt. %.

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Abbreviation

Notation	Description
P.S.	Particle Size.
S.R.	Solvent to solid ratio.
A.S.	Agitation speed.
v/w	Volume / Weight.
rpm	Revolution per Minute.
HPLC	High Pressure Liquid Chromatography.