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EXTRACTION OF ESSENTIAL OIL FROM CLOVE'S BUDS USING AN ULTRASONIC-ASSISTED METHOD

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Abstract

Cloves (Syzygium aromaticum L.) are the aromatic dried flower buds of a tree in the family Myrtacae. Clove's essential oil is widely used as aromatherapy and for toothache treatment. Development of extraction technology that could increase oil yield from clove would thus significantly enhance the profitability of the clove's oil and reduce processing costs significantly. It has been shown that ultrasound-assisted extraction method which uses vibrations to extract samples with polar solvents in an ultrasonic bath can enhance extraction of phytochemicals from plant sources while reducing processing time and solvent consumption. In this this research work an essential oils was obtained from clove's buds using an ultrasonic-assisted method with n-hexane as extraction solvent at different ultrasonic machine power rate of 100, 200, 300, 400 and 500 W at constant time of 20 minutes at room temperature, the chemical compounds present in the different essential oil were analyzed using Gas Chromatography-Mass Spectrometer (GC-MS). The GC-MS results show that the maximum content of eugenyl acetate in extracts was 9.24%, and were extracted from clove buds at ultrasonic powers of 300 W.

Key words: Ultrasonic-assisted, GC-MS, clove's buds, n-hexane, Eugenyl acetate

INTRODUCTION

Clove bud oil is derived from clove tree a member of the *myrtaceae family*. This tree is a native of Southeast Asian country like Indonesia (Alma et al. 1990). Three types of essential oil are available from clove species: clove bud oil, clove steam oil and clove leave oil. Each has different chemical composition and flavor. Clove bud oil the most expensive and the best quality product, contains eugenol (80% -90%), eugenyl acetate (15%-17%), and β-caryophyllene (5-12%). It is well known that the amount of secondary compound like essential oil is affected by genetic factor, climate, soil and cultivation techniques (Arslan et al. 2014).

Essential oils are complex mixtures, made up of terpenoid hydrocarbons, oxygenated terpenes and sesquiterpenes. They originate from the plant secondary metabolism and are responsible for their characteristic aroma. Essential oils (also called volatile or ethereal oils, because they evaporate when exposed to heat in contrast to nonessential oils) are odorous and volatile compounds found only in 10% of the plant kingdom and are stored in plants in special brittle secretory structures, such as glands, hairs, ducts, cavities or resin ducts (Ahmadi et al. 2002; Ciccarelli et al. 2008; Liolios et al. 2010). The essential oil of cloves has anesthetic and antimicrobial qualities and is some time use to eliminate bad breath or to ameliorate the pain of bad tooth. Also, clove bud oil has biological activities, such as antibacterial, antifungal, insecticidal and antioxidant properties, and are used traditionally as flavoring agent and antimicrobial material in food (Velluti et al. 2003). Sesquiterpenes found in cloves were investigated as potential anti carcinogenic agent. (Zheng et al. 1992).

Essential oil can be extracted using methods such as hydro-distillation, steam distillation, soxhlet, microwave, and super critical fluids extraction. However, the disadvantages of these processes are that the extracts are constantly heated and this can damage thermolabile compounds and initiate the formation of artifacts (Ogunwale and Udo, 1996). It also caused hydrolysis and water solubilization of some fragrance constituents. Extracts obtained by solvents contain residues that pollute the foods and fragrances to which they are added. These disadvantages can be avoided by using the ultrasonic assisted method. Ultrasonic assisted extraction method of oil extraction from plants has been performed previously by some authors such as (Ying et al. 2011). The conditions explored are ultrasonic powers of 40 to 60 W and temperature ranging from 60 to 90°C. The results showed that the yield of polysaccharides increased significantly with increasing extraction power, and then decreased when the extraction power was over 60 W. The work of Wei Wang et al. showed that the optimal parameters to extract the target compounds from *H.* lyrata were as follows: extraction solvent: 70% aqueous ethanol; solvent to material ratio: 20:1 (v/w, ml/g); extraction time: 20 min under the conditions: ultrasonic frequency: 40 Hz; extraction temperature: 30 ^{0}C. Also, Gutte et al. (2015) carried out an optimization of ultrasonication treatment of extraction of flaxseed oil exploring the following conditions: amplitude (20–80 kHz), temperature (25–40^{0}C), sonication time (20–80 min) and solid to solvent ratio (1:5, 1:10 and 1:15) for extraction of flaxseed oil. The result showed that the ultrasonic treatment at frequency of 40 kHz, temperature of 30^{0}C, extraction time of 40 min and solid to solvent ratio: 1:10 gave best results for extraction. The ultrasonic assisted extraction improves the extraction yield by 11.5% however, none of investigations has examined in detail the extraction of essential oil from the clove buds oil using ultrasonic assisted method at a higher power and room temperature. Since the clove oil has been used widely as pharmaceuticals.

In this study, an essential oil was extracted from clove's buds using an ultrasonic-assisted method by varying its power rate and at room temperature.

MATERIALS AND METHODS

The dried sample of *Eugenia Caryophyllus* (clove) used in this research was purchased from Dutsinma central market, Katsina State Nigeria. The sample were sorted out by hand to remove bad ones and foreign materials and then ground with mortar and pestle into powder using a mesh size of 0.5mm. The n-hexane (BDH) served as an extraction solvent and distilled water was used throughout in the experiment.

Extraction of essential oil from clove buds was carried out using an ultrasonic instrument (UK). 20g of ground powder was weighed using a digital weighing balance, and this was mixed with a 150ml of n-hexane in the 500ml plastic beaker, then the beaker was placed in an ultrasonic bath and the machine was set at power of 100, 200, 300, 400, and 500 Watts for five different samples at 20 minutes each. The crude clove oil was filtered using Whatman No.1 filter paper and the oil were concentrated using rotary evaporator (Model 2215, BUCHI, Switzerland) at 40^{0}C). The oil was collected and stored in opaque, air tight containers (amber bottle) at 4^{0}C for further analysis. The GC/MS analysis was carried out using GC/MS-QP 2010 plus Shimadzu, Japan. After concentration of the oil, the percentage oil yield was determined from equation (1).

$$\text{Percentage oil yield (\%)} = \frac{W_2}{W_1} \times 100 \quad \text{........(1)}$$

Where;

W_1 = weight of powdered sample before extraction of oil (g)

W_2 = weight of powdered sample after extraction of oil (g)

RESULTS AND DISCUSSION

Yield of the oil extracted at different ultrasonic power

Percentage yield of extract in (*Eugenia carryophyllus*) buds at different ultrasonic power is as shown in Table 1. The oil extract was dark brown in color with a characteristics clove odour and the percentage yield of ultrasonic extracts of 100, 200, 300, 400 and 500 Watts were found to be 34.30, 61.70, 71.55, 45.66 and 49.85 respectively. This shows that the percentage yield increase with increase in power up to 300 Watts, as the power reaches 400 Watts, the percentage yield start to decrease this shows that the power of 300 Watts may be the optimum power of essential oil extraction. oil extract at ultrasonic power of less than 300 Watts may be explain in terms of cavitational effects caused by the application of the ultrasonic waves. Cavitation takes place in liquid medium once the media is subjected to rapid, alternating high pressure. The mechanical stress were induced on the cell using ultrasound which causes cellular break down that increases the solubilization of metabolites in the solvent and improves extraction yields. An increase in ultrasonic intensity will contribute to an increase in cavitation effect (Mason et al. 1990). However, as the ultrasonic power increased beyond 300 Watts, the extraction yield decreases. This may be due to the formation of a large amount of cavitation micro bubbles at intensity above 300 Watts and this phenomenon tends to reduce the amount of ultrasonic energy being transmitted to the solvent medium and produce less cavitational effect, reduced formation of microfractures in the biological tissues. This phenomenon explains why further increase in ultrasonic power beyond 300 Watts decreases the extraction yields.

Compositions of the Oil Extracted at Different Ultrasonic Power

Table 2 shows the composition of the eugenol and eugenyl acetate of the clove oil extracted at different ultrasonic powers. It can be seen that clove oil obtained at 500 W contained highest percentage of eugenol (8.092%), followed by that obtained at 200 W (2.78%). Clove oil obtained at 100 W had the lowest percentage of eugenol (0.604%). However, clove oil obtained at 300 W contained the highest percentage of eugenyl acetate (9.242%), which is also the main antioxidant ingredients in clove oil (Lee et al. 2001). The active antioxidant ingredients, eugenol and eugenyl acetate, in the extracted clove oil by ultrasonic assisted method was obtained.

Comparisons of the Clove oil extracted at different Ultrasonic Power

Colour and texture are the prime characteristics and quality factors of essential oil, and extraction yield and extraction time are the important factor for the industrialization (Weiqiang et al. 2007). In table 2, the content of eugenyl acetate was determined by GCMS. It can be seen from table 2 that, the content of the biological ingredients such as eugenyl acetate in the clove oil at 300 W is the highest when compared to the 400 W and 500 W. This may be due to the fact that, at higher ultrasonic power there could be rise in temperature and then the vapor pressure became higher and more bubbles were created, but they collapsed with less intensity due to a smaller pressure difference between inside and outside of bubbles, which reduced cell tissues disruption (Hromadkova et al. 1999). Another reason may be due to the surface tension which decreased with the increase of temperature affecting the bubble formation and collapse. The bubbles may be so easily collapsed at higher power thus reducing the intensity of the mass transfer enhancement.

In the previous studies, the eugenyl acetate content of an essential oil of clove (Syzygium aromaticum) were reported by Alma et al. (2007) and Lee and Shibamoto (2001) as 8.01% and 8.6% respectively. Our results was higher than the values reported.

Table 1: PERCENTAGE YIELD OF CLOVES BUD OIL AT DIFFERENT ULTRASONIC POWER

S/NO	Ultrasonic power (W)	Yield (%)
1	100	34.30
2	200	61.70
3	300	71.55
4	400	45.66
5	500	49.85

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Table 2 COMPOSITIONS OF CLOVES BUD OIL AT DIFFERENT ULTRASONIC POWER

S/NO	Ultrasonic power (W)	Eugenyl acetate (%)
1	**100**	**4.80**
2	**200**	**8.872**
3	**300**	**9.242**
4	**400**	**5.110**
5	**500**	**8.092**

CONCLUSIONS

The yield and composition of clove oil obtained using an ultrasonic assisted method at different ultrasonic powers were compared. Extraction yield and percentage of eugenyl acetate at 300 W were the highest, it can be concluded that extraction at 300 W gave a better result when compared to other four powers (100 W, 200 W, 400 W and 500 W), because it has the highest percentage of active antioxidant ingredients of eugenyl acetate in the extracted clove. Therefore, extraction at 300 W is considered in this experiment as the best for obtaining clove oil with high quality.

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