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Comparison of Essential Oils of Clove Buds Extracted Using Soxhlet and Ultrasonic-Assisted Extraction Methods (SHORT COMMUNICATION)

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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. In this research work an essential oil was obtained from *clove's buds* using Sohxlet and an ultrasonic-assisted extraction methods with n-hexane as extraction solvent. The oil extract was analyzed for its chemical composition using gas chromatography/mass spectrometric (GC-MS). The results of analysis show that the essential oil yield were found to be 54.70% and 71.55% while the eugenyl acetate in extracts obtained were found to be 5.72% and 9.24% for sohxlet and ultrasonic-assisted extraction methods respectively.

Keywords: Sohxlet extraction; Essential oil; Clove bud; Ultrasonic-assisted; 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%). 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).

Different methods can be used to extract essential oil from aromatic plants, 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). Comparison of some essential oils obtained by ultrasonic- assisted with conventional extraction methods and GC analysis of clove oil in ethanol had been studied by some authors (Gutte et al. 2015; Hromadkove et al. 1999; Wenqianq et al. 2007), however, none of investigations has examined in detail the comparison of essential oil composition between clove buds oil obtained by ultrasonic-assisted and the sohxlet extraction techniques. Since

clove oil has been used widely as pharmaceuticals, flavoring and antimicrobial agents in food industry, it is necessary to find the most suitable method for the improvement of the quality of clove oil. The aim of this work is to compare clove oils obtained by the ultrasonic-assisted and the sohxlet extraction. Compositions of clove oil were analyzed by gas chromatography/mass spectrometry (GC–MS).

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.

Determination of Ash Content of Oil sample

The ash content of the clove powder is a measure of metallic constituents in the powder, which indicates the mineral element contained in the powder, which is reflection of nutritional values of the clove powder, when clove powder is completely burnt and turn to ash, high ash content indicate low nutritional value (Almustapha *et al.* 2009).

2g of clove oil sample was weigh; the crucible containing the samples was placed into the lenton furnace thermoset at 600^{o}C and allows to burn for 3 hours until the content became ash. The crucible containing the ash sample was weight using electrical weighing balance, the ash content is determined using the equation (1) as described by (Almustapha *et al.* 2009).

$$\text{Ash content} = \frac{W_3 - W_1}{W_2 - W_1} \times 100 \quad \ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where;

W_1 = weight of empty crucible

W_2= weight of oil sample before ashes + weight of the crucible

W_3= weight of ash sample + weight of crucible after ashes

Determination of Moisture Content of Oil Sample

2g of oil sample of *Eugenia cryophyllus* was weigh, the clean watch containing the sample was placed in an oven at temperature at 105^{o}C for six hours and then weigh. The moisture content of oil sample can be calculated using equation (2) as described by (Udo and Oguwele, 2011).

$$\text{Moisture content} = \frac{W_1 - W_2}{W_2 - W_0} \times 100 \quad \ldots\ldots\ldots (2)$$

Where;

W_0 = weight of the empty crucible (g)

W_1 = weight of fresh sample + crucible (g)

W_2 = weight of dried sample + crucible (g)

Determination of oil sample density

Density of a substance is the relationship between the mass of the substance and how much space it takes up by its volume. Measuring cylinder was placed on weighing balance, clove oil was poured inside the measuring cylinder up to volume of 5cm^3 and the weight of the clove oil was recorded. The density of clove oil can be calculated using equation (3) Almustapha et al. 2009.

$$\text{Density} = \frac{\text{Mass}}{\text{Volume}} \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots (3)$$

Where;

M = Mass of oil sample in gram

V = Volume of oil sample in cm^3

Soxhlet Extraction

60g of the ground clove's buds was weighed and transferred into a filter paper extraction thimble, thimble containing the sample was then place in the soxhlet extractor fitted at the lower portion with a flask containing 300ml of n-hexane used as the extraction solvent. The soxhlet column was then fitted to a reflux condenser and the set up was placed on the heating mantle as temperature increase steadily until n-hexane began to boil and the boiling vapor passes through the condenser and was condensed, vapor now falls back on the porous

thimble containing the powdered seed sample, n-hexane being a solvent dissolved the oil content of the seed sample leading to the formation of homogenous mixture of n-hexane and oil which was collected in the receiver of the soxhlet extractor set- up. The set-up was heated, for about 6 hours. Then the oil extract was concentrated using vacuum evaporator (Model 2215, BUCHI, Switzerland) at 40^{0}C.

Ultrasonic-assisted Extraction

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 between 100 and 500 Watts 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).

$$\text{Percentage oil yield (\%)} = \frac{W_2}{W_1} \times 100 \quad \text{.....(4)}$$

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

Table 1: Physicochemical Properties of an Essential Oil of Clove Bud by Different Extraction Methods

Properties extraction	Sohxlet extraction	Ultrasonic-assisted
Colour	Yellow brown	Clear yellow
Density (g/cm^3)	0.83	0.81
Percentage ash content (%)	0.001	0.0001
Percentage moisture content (%)	18.80	25.70
Boiling point (^{0}C)	134	135

Table 2: Percentage Yield And Eugenyl Acetate Content Of An Essential Oil Of Clove Bud By Different Methods

Method	Percentage yield (%)	Eugenyl acetate (%)	Time of Extraction
Sohxlet extraction	54.70	5.722	6 hr.
Ultrasonic-assisted extraction	71.55	9.242	20 min.

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 (Wenqianq et al. 2007). The oil extract by ultrasonic-assisted and sohxlet methods were clear yellow and yellow brown in colour with a characteristics clove odour respectively. The physicochemical properties by different extraction methods differed significantly for density and it was noted that ultrasonic-assisted method yielded oil with a density of 0.81 g/cm^3. The density of the oil obtained by sohxlet extraction was found to be 0.83 g/cm^3 (Table 1). Comparison of other physicochemical properties of the extracted oil is also shown in Table 1. Comparisons of yield and chemical compositions of the clove oils obtained by

different methods were also listed in Table 2. In Table 2, the content of eugenyl acetate was determined by GC-MS. It can be seen that, both the oil yield and content of the eugenyl acetate in the clove oil by ultrasonic-assisted extraction was higher while that of the sohxlet extraction is lower. Furthermore, the yellow brown colour of extracts by Sohxlet method implies that more undesired impurities and organic solvent residue may be presence. Utrasonic-assisted extraction offers the most important advantages over sohxlet method. Extraction yield of ultrasonic-assisted was about two times as high as that obtained by sohxlet extraction method. The higher content of eugenyl acetate in the extracted oil was obtained at the short extraction time of 20 minutes.

CONCLUSION

The composition and some character of clove oil obtained by sohxlet and ultrasonic-assisted methods were compared. The yield and compositions of the oils obtained differ quantitatively. Extraction yield of ultrasonic assisted was about two times as high as that obtained by sohxlet method. Ultrasonic assisted offers many important advantages over sohxlet method, including higher extraction yield, the highest percentage of active antioxidant ingredients of eugenyl acetate in the extracted clove, shorter extraction time, no thermal degradation of chemical constituents and so on. Therefore, ultrasonic-assisted method is considered as the better method for obtaining clove oil with high quality.

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