



## Extraction and characterization of oleoresin from ginger (*Zingiber officinale* roscoe) rhizomes using blends of monohydric alcohols

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**Abstract** Ginger oleoresin has widespread industrial applications, notably, as a flavoring agent in foods, beverages, pharmaceuticals and as oleochemicals. This study aimed to extract and characterize oleoresin from *Zingiber officinale* Roscoe using different blends of monohydric alcohols. The extraction of oleoresin from ginger powder was achieved with ethanol, isopropanol and butanol in different blend ratios. The oleoresin fractions were characterized by Gas Chromatography-Mass Spectrometry. Extracted oleoresin yield ranges from 9.5-15%, with 50 ml ETOH/25 ml ISP (OR1) resulting in the highest percentage yield and total extracted content. Oleoresin extracts from all alcoholic blends showed the presence of oleic acid, stearic acid, gingerol,  $\alpha$ - zingiberene and palmitic acid. 50 ml ETOH/25 ml ISP (OR2) showed the highest levels of terpenes content characterized by the presence of  $\alpha$ - zingiberene at 13.38%. 50 ml ETOH/25 ml ISP (OR2) and 25 ml ETOH/50 ml BUT (OR4) showed highest and lowest levels of gingerol respectively. The highest content of ester characterized by the presence of 9- Dodecanic acid methyl ester at 22.65% was recorded for OR3. The content of  $\alpha$ - zingiberene, stearic acid, gingerol, palmitic acid and oleic acid recorded ranges from 0.25-13.38, 1.08-25.50 3.52-34.30, 4.77-21.67 and 13.34-46.83% respectively. The results showed that the blends of monohydric alcohols were effective in the extraction of oleoresin from ginger powder. Thus, the four classes of oleoresin present a wide range of application as oleochemicals resin in food, beverage and pharmaceuticals industries.

**Keywords** Alcohols, Esters, Extracts, Oleochemical, Fatty acid, Blend ratio.

### 1. Introduction

Recently, developments in extraction technology have led to the increase use of spice oleoresin as opposed to the spice themselves. The increased prominence of oleoresin over natural spices is due to the merits that oleoresins hold over the use of spice. These advantages include increased economy in use, more uniform flavor and concentration, and lack of microbial contamination [8, 9]. Ginger is often used for the treatment of stomach aches, cardiovascular and motor diseases [9]. It also possesses anti- inflammatory activity and inhibits bacteria growth, as well as provides protection for immune- depressed patient [4].

Extraction of ginger oleoresin, have been extensively studied for several biological activities including antibacterial, anti convulsion, analgesic, antiulcer, gastric antisecretory and antitumor [15]. Ginger oleoresin is characterized by monoterpenes and sesquiterpenic compound while the main pungent compounds in the oleoresin are a series of homologues called gingerols and shogaols. Many active components have been found in ginger;  $\alpha$ - Zingiberene, gingerol and oleic acid [14]. The active component obtained from ginger is a high valued-added product as a result there is a continuous search to improve the yield with better and novel extracts.



The use of ultrasound to extract oleoresin from ginger (*Zingiber officinale* R.) was studied [9]. The extraction was performed by using ethanol as solvent in the presence of ultrasonic irradiations operating at frequency of 42 kHz at extraction temperature of 60 °C. The oleoresin extracted was in the form of dark thick liquid with specific ginger flavor. Based on GC-MS analysis, the use of ultrasound does not give an effect on alteration of main component in ginger oleoresin. There have been reports, on the use of supercritical CO<sub>2</sub> on the extraction of oleoresin from ginger [15] because it's environmentally friendly; the extraction process is safer and serves as a good substitute for organic solvents and volatile organic compound (VOC) emissions.

Also the effects of temperature, pressure and the addition of co-solvent (ethanol (EtOH) and isopropyl alcohol (IsoC3), both at 1.17% (mass)) on the kinetics of extraction of ginger oleoresin were studied [5]. The design used was a 2×2×3 factorial (pressure 200 and 250 bar; temperature: 25 and 35 °C; solvent: CO<sub>2</sub>, CO<sub>2</sub>+EtOH, CO<sub>2</sub>+IsoC3). The identification of the substances present in the oleoresin was performed by GC-MS; GC-FID. The antioxidant activity of the extract fractions was determined using the coupled oxidation of linolenic acid and β-carotene. The results showed that temperature, pressure and solvent significantly affected the total yield. Studies have shown that replacement of single organic solvents such as hexane, ethylacetate and a chlorinated hydrocarbon with a benign binary monohydric alcohol is considered desirable from a technical standpoint [7].

*Zingiber officinale* R. commonly known as ginger is a root but technically a sympodium. The main body is somewhat flattened and cylindrical with several secondary branched refers to as 'fingers' with narrowing tips [7]. It has upright stems and narrow medium green leaves arranged in two on each stem in which plant can grow about 1.2 m tall, the stem is surrounded by the sheathing bases of 2 rank leaves, the leaves are 1.9 cm wide and 17.8 cm long. Reports, on the use of blend of monohydric alcohols (two carbon atoms, three carbon atoms and four carbon atoms) to induce selectivity in the content of extracted oleoresin from ginger rhizomes is scanty. Therefore, this study represents a paradigm shift in the realm of solvent extraction of oleoresin from ginger rhizomes and could provide valuable information regarding the novelty and industrial impact of the final products. Therefore, the main objective of this study is to investigate the effect of monohydric alcohol of the same homologue at different blend ratios on the percentage extracts and characterized the extract fractions physico-chemically.

## 2. Experimental

### 2.1. Chemical

All the chemicals used in this study were analytically grade, purchased from British Drug House (BDH) laboratories and used as supplied without further purification. Double distilled water was used.

### 2.2. Material preparation

Fresh ginger rhizomes were purchased from Mushin market in Lagos- Mainland. Lagos state Nigeria. Stones, sand and leaves that may have accompanied the ginger after harvest, transportation and storage were removed by sorting. The Ginger rhizomes were washed, peeled and cut into uniform size with the help of a locally fabricated ginger slicer developed and fabricated at the Federal Institute of Industrial Research, Oshodi (FIIRO). The sliced ginger was sun dried for 14 days to reach a moisture content level of 8% [2], and then ground using an attrition mill in FIIRO pilot plant. The resulting powder was sieved using a 100 µm mesh to screen out coarse particles/dissected fiber and the fine powder was packaged in a polyethylene bag and kept in laboratory cupboard under dry cool condition.

### 2.3. Characterization

#### Oleoresin extraction

The method [4] with slight modification was adopted. Extraction equipment comprises of a 1000 ml jacketed glass beaker and a 250 ml plastic beaker with a porous base, inserted into a 500 ml glass beaker. Water was maintained insitu in the jacketed beaker. The entire experimental set-up was mounted on a hot plate (Cole Parmer hot plate-solid state, model 4817, USA) and thermo- metrically controlled at 45 °C with the help of a thermometer inserted into the water contained in the 1000 ml glass beaker.



The ginger powder (10 g) was carefully weighed into the 250 ml plastic beaker with a porous base and 75 ml ethanol (OR1) was measured into the 500 ml glass beaker respectively. The former was then placed inside the later, so that the ginger powder entered into contact with the solvent. The system was maintained at 45 oC under constant agitation for 150 min. After, which the 500 ml glass beaker containing the leached phase (oleoresin and excess solvent) was retrieved and allowed to stand for 24 h and filtered (whatmann No. 42) to obtain the filtrate (mixture of oleoresin and solvent). The solvent was recovered from the oleoresin using a rotatory evaporator. The extracted ginger oleoresin was weighed, esterified [4] and dried in a dessicator with anhydrous sodium sulfate. The dried esterified oleoresin was then poured into a sample bottle and stored at room temperature 25 oC prior to GC-MS analysis. This experimental protocol was repeated for, 50 ml Ethanol/25 ml Isopropanol(2:1) OR2, 25 ml Ethanol/50 ml Isopropanol(1:2) OR3 and 25 ml Ethanol/50 ml Butanol (1:2) OR4 respectively.

### Characterization of oleoresin extracts

The extracted ginger oleoresin fractions were characterized for their fatty acids/bioactive contents.

### Gas chromatography/Mass spectrometry (GC/MS) and Identification of Compounds

The GC/MS analyses of the oil were conducted on a Hewlett- Packed HP 5973 mass spectrometer interfaced with an HP 6890 gas chromatograph. The following column and temperature conditions were used; initial temperature 70 oC, equilibration time 3.00 min, ramp 4 oC/min, final temperature 240 oC; inlet: splitless, initial temperature 220 oC, pressure 8.27 psi, purge flow 30 ml/min, purge time 0.20 min, helium gas; column: capillary, 30 m x 0.25 mm i.d; 0.25  $\mu$ m, film thickness 0.7 ml/min, average velocity 32 cm/sec; MS: EI method at 70 eV.

The oils compounds were identified by matching their mass spectra data with those of authentic standards held in the computer library (Wiley 275, New York) and by comparing the calculated retention indices with those in literature. The percentage composition was calculated from summation of the peak areas of the total oil composition [3].

## 3. Results and Discussion

**Table 1:** Percentage Yield of Ginger Oleoresin for various Blend Formulations.

Weight of ginger Powder (g)	Operating Temp (°C)	Solvent Blend	Volume of Solvent (ml)	Yield (%)
10	45	ETOH (OR <sub>1</sub> )	75	12.5
10	45	50 ml ETOH/25 ml ISP (OR <sub>2</sub> )	75	15.0
10	45	25 ml ETOH/50 ml ISP (OR <sub>3</sub> )	75	10.5
10	45	25 ml ETOH/50 ml BUT (OR <sub>4</sub> )	75	9.5

ETOH- Ethanol; ISP- Isopropanol; BUT- Butanol

The percentage yields of oleoresin obtained with varying ratio of monohydric alcohols at 45oC are presented in Table 1. Generally, oleoresin extracted with solvent and solvents blend was thick dark brown colored liquid with a characteristic ginger flavor. The monohydric alcohols blends extracted oleoresin content varied from 9.5-15%. The highest yield (15.0%) and lowest of 9.5% oleoresin was obtained from 50 ml ethanol/25 ml isopropanol (2:1) and 25 ml ethanol/50 ml butanol (1:2) respectively. The oleoresin content as obtained from this study is comparable to 17.4% for Supercritical carbon dioxide [15]. However, the present study shows that the percentage yield is lower than 40.5-50.0% using ultrasound technology [9]. The variations in yield may be ascribed to methods of extraction and environmental factors.

The use of monohydric alcohols with smaller hydrocarbon chain length in greater proportion (75 ml Ethanol and 50 ml Ethanol/25 ml Isopropanol) results in greater oleoresin yield This is may be attributed to more interactions of the preponderant smaller alkyl group in ethanol molecule with larger bioactive molecules, in the substrate were more effective to limits steric hindrance [11], allowing the solvent gain more access to the internal particle structure, thereby, enhancing the removal of the cell contents. Combination of solvents, as evident in 50 ml ETOH/25 ml ISP



gave better result as the synergistic effect shows increased populace of bioactive compound extracted. Therefore, this inquiry presents a new scientific route when increased and specific bioactive compounds are needed in ginger oleoresin for specific use or bioavailability.

**Table 2:** Compositions of oleoresin extracts

Compound	% Composition			
	OR <sub>1</sub>	OR <sub>2</sub>	OR <sub>3</sub>	OR <sub>4</sub>
11-Dodecadiene	1.34	-	-	-
Decenal	1.80	0.80	0.64	-
Curcumen	-	9.24	-	-
$\alpha$ -Zingiberene	0.36	13.38	0.34	0.25
$\beta$ -Farnesene	-	8.99	-	-
$\beta$ -Sesquiphellandrene	-	5.82	5.82	-
Vanillyl acetone	2.63	5.61	1.36	-
1,2,4-Trimethyl 3-nitro- bicyclic (3.3.1) nonan-9-one	-	2.32	-	-
Palmitic acid	21.67	4.77	12.97	12.34
Methyl-tri-decanoate	-	-	-	8.81
9-Dodecanic acid methyl ester	-	-	22.65	-
Palmitoleic	2.55	-	-	-
Oleic acid	40.70	13.34	28.65	46.83
Stearic Acid	2.64	1.08	18.04	25.50
Gingerol	10.08	34.30	6.99	3.52
Olein	11.56	2.35	1.08	-
Eicosanic Acid	6.77	-	-	-
Veridiflurol	-	-	1.46	3.00

Results of GC-MS analysis for each extract are presented in Table 2. All the extracts showed the presence of gingerol, with the highest composition of 35.38% for OR2 (50 ml ethanol/25 ml isopropanol), which suggest that a blend of ethanol and isopropanol may have influenced positively the solubility of this bioactive compound resulting in its large content [7]. The results also present OR2 with the highest percentage extracted content of monoterpenes and sesquiterpenes :  $\alpha$ -Zingiberene (13.38%),  $\beta$ -Farnesene (8.99%),  $\beta$ - Sesquiphellandrene (5.82%) and curcumen (9.24%), compared to other blends of monohydric alcohol. This suggests ethanol molecules easily permeates and solvates the internal cell structure of ginger particle, resulting in higher fractions of these aromatic hydrocarbons [2, 7, 11].

The results as represented indicates that all the solvent blends were effective in the extraction of saturated fatty acids (palmitic and stearic) and monounsaturated fatty acid (oleic), which permits the use of each extract as oleochemical in food mix and personal care products [13]. The total saturated fatty acid (TSFA), i.e. palmitic and stearic acid content for the oleoresins (OR1, OR2, OR3 and OR4) are 24.31%, 5.85%, 31.01%, and 37.84% respectively, presents lower values than their corresponding levels of monounsaturated fatty acid (oleic) [OR1, 40.70%; OR2, 13.34%; OR3 28.65%; OR4 46.83%]. Thus, qualifies the extracts for application in functional foods where low fat and ginger flavor is required [12].

Furthermore, the low saturated fatty content of the oleoresins suggest long time storage without oxidative rancidity and could be suitable for stir-fry cooking owing to its high smoke point [1]. OR4 with the highest oleic acid content may be suitable for pharmaceuticals as the health benefits inherent in this monounsaturated fatty acid could help in addressing cardiovascular disorders [6, 10]. OR2 with high level of gingerol could find wider usage in confectionaries, beverages and pastries where high ginger flavor is required. The low levels of gingerol and high levels of esters in OR3 and OR4 notably; Methyl-tri-decanoate and 9- Dodecanic acid methyl ester distinguishes their potential inclusion in personal care products, dentifrice and nutraceutical.



#### 4. Conclusions

Ginger oleoresin have been extracted from ginger powder using blends of monohydric alcohols and characterized. The oleoresin extracts was characterized by the presence of saturated fatty acids (stearic and palmitic), monounsaturated fatty acid (oleic acid) and terpenes ( $\alpha$ - Zingenberene,  $\beta$ -Farnesene and  $\beta$ -Sesquiphennleindrene). This study shows that the oleoresin fractions contained dietary supplements suitable for human consumption and has also provided detailed information on the quality of extract from each blend of monohydric alcohols. Clearly, this research has shown that oleoresin extract could be tailored to suit specific needs and industrial applications.

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#### References

- [1]. Ajayi LA, Oderinde RA, Kajogbola DO, Uponi JI. (2006). Oil content and fatty acid composition of some underutilized legumes from Nigeria. *Food Chemistry*. 99: 115-120.
- [2]. Alkali, JS, Satimehin, AA. (2004). Drying kinetics of ginger. *Nigerian Food Journal*, 22 105-107.
- [3]. Asekun, OT, Okoh, SO, Familoni, OB, Afolayan, AJ. Chemical profiles and Antioxidant Activity of Essential Oils extracted from the leaf and stem of pakia biglobosa (Jacq) Benth. *Research Journal of Medicinal Plant*. 44: 102-107.
- [4]. Hinneburg, IH, Damien Dorman, J, Hiltunen, R. (2006). Antioxidants activities of extracts from selected culinary herbs and spices. *Food Chemistry* 97: 122-129.
- [5]. Kelly, CZ, Marcia, OM, Marques, R. (2005). Extraction of ginger (*Zingiber officinale* Roscoe) oleoresin with CO<sub>2</sub> and co-solvents: a study of the antioxidant action of the extracts, *The Journal of Supercritical Fluids*. 24 57-76.
- [6]. Manzoor M, Anwar F, Iqbal T, Bhanger MI. (2007). Physicochemical characterization of Moringa concanensis seed and seed oil. *Journal of American Oil Chemical Society*. 84: 413-419.
- [7]. Meadows, AB, Olorunda, AO, Aina TO. (2005). Oleoresin Yield and Gingerol in two varieties of Nigerian Ginger (*Zingiberis officinale* Roscoe) at various maturity stages. *Nigerian Food Journal*, 23 91-105.
- [8]. Miliauskas, G, Venskutonis, PR, Van Beek, TA (2004). Screening of radical scavenging activity of some medicinal and aromatic plant extracts. *Food Chemistry* 85: 231-237.
- [9]. Mohammed, DS, Anwar, F, Pocut, NA, Normalina, A. (2011). Solvent extraction of ginger oleoresin using ultrasound. *Makara Sains* 15 (2): 163-167.
- [10]. Nicolosi RJ, Woolfrey B, Wilson TA, Scollin P, Handelman G, Fisher R. (2004). Decreased aortic early atherosclerosis and associated risk factors in hypercholesterolemic hamsters fed high or mid-oleic acid oil compared to high-linoleic acid oil. *Journal of Nutrition Biochemistry* 15: 540-547.
- [11]. Ramadan MF, Sharanabasappa G, Seetharam YN, Seshagiri M, Moresel JT. (2006). Characterization of fatty acids and bioactive compounds of Kachnar (*Bauhinia purpurea* L.) seed oil. *Food Chemistry*. 98 (2): 359-365.
- [12]. Razip S, Anwar F, Mahmood Z, Shahid SA, Nadeem R. (2012). Characterization of seed oil from different varieties of watermelon [*Citrullus lanatus* (Thunb.)] from Paskistan. *Grasas Y Aceites*, 63: (4) 365-372.
- [13]. Rossell, JB. (1991). Vegetable oil and Fats. In *Analysis of oilseeds, Fats and Fatty Foods*, Rossell, J.B. and Pritchard, J. L.R. Eds; Elsevier Applied Sciences: New York; 261-328.
- [14]. Shanavas B. (2012). India spice oleoresin industry: way forward initiatives. Paper presented at the IFEAT international conference in Singapore, 4-8 November. "Essential Asia" pp 39-49 in the printed conference proceedings.
- [15]. Spiro, M. Kandiah, P. (1990). "Extraction of ginger rhizomes: Kinetic studies with Supercritical carbondioxide" *International Journal of Food Science and Technology*. 25: 328-338.

