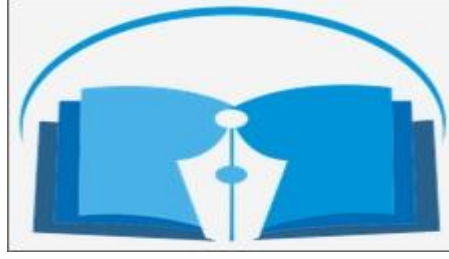




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- المجلة ترحب بما يرد عليها من أبحاث وعلى استعداد لنشرها بعد التحكيم .
- المجلة تحترم كل الاحترام آراء المحكمين وتعمل بمقتضاها .
- كافة الآراء والأفكار المنشورة تعبر عن آراء أصحابها ولا تتحمل المجلة تبعاتها .
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Determination of Chemical and Physical Properties of Essential Oil Extracted from Mixture of Orange and Limon Peels Collected from Al-khoms–Libya

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ABSTRACT: The peel of citrus is known to be rich in essential oil. Essential oil compositions and properties vary among the citrus species and the climate factors play an important role. Essential oil to be suitable for the specific commercial uses should have basic properties. In this study, extracted oil of six citrus species [citrus sinensis, citrus aurantium, citrus nobilis, citrus reticulata and two kinds of lemon] were characterized. The moisture content ranged between 33.33% and 78.85%. and the oil yield was up to 38.85%. The other parameters were analysed as acid value, saponification number, ester number, refractive index, density and specific gravity and their highest values in this study are (7.51 mg/KOH/g), (43.24 mgKOH/g), (149.38 mgKOH/g), (1.4542), (0.973 g/cm³) and (1.22) respectively. These parameters showed variations with the species. The characterization of the citrus extracted oil gave its physical and chemical properties. Values that indicated it could be used for production of different valuable products.

Key words: Essential oil, citrus, physical and chemical properties.

INTRODUCTION

Citrus fruits belong to six genera (Fortunella, Eremocitrus, Clymendis, Poncirus, Microcitrus, and Citrus), which are mainly grown in tropical and sub-tropical areas of Asia, but the major commercial fruits belong to genus *Citrus*. The genus citrus includes about sixteen kind of Citrus trees such as oranges, mandarins, lime, lemons and grapefruits. The essential oil is present in the fruit's peel in great quantities [1, 2].

The essential oils can be obtained from plant material, it may be extracted from leaves, seeds, peels, or stalks that depending on the kind of the plant [1]. Essential oils are the most important by-product usually obtained from peel of Citrus and make up the largest sector of the world production of essential oils [3]. Several techniques can be used to extract essential Oils from different parts of the aromatic plant, including water or steam distillation, solvent extraction, expression under pressure, supercritical fluid and subcritical water extractions [4].

Citrus essential oils are mixtures of many chemical compounds that can be approximated into three fractions: terpene hydrocarbons, oxygenated compounds and nonvolatile compounds. The terpene fraction can constitute from 50 to more than 95% of the oil [1]. Essential oil from citrus is a large type of natural flavors and fragrances which is popularly used in food products such as sweets, beverages, and cakes, daily chemical products such as soap, perfumes. Citrus oil is also utilized in health care field whereas is a registered active ingredient in 15 pesticide products used



as insecticides, insect repellents, and dog and cat repellents and broadly used as pharmaceutical components[5,6].

Citrus oil can be extracted from the peel by different conventional methods like steam distillation, solvent extraction, maceration, cold pressing and modern techniques such as effleurage, super critical CO₂ extraction and turbo distillation [5].The purpose of this study was to determine the physical and chemical properties of oils that extracted from various kinds of orange fruit and lemons including Saponification number, PH number, yield, refractive number, density, and specific gravity.

I.MATERIAL AND METHOD

Six different kinds of citrus, citrus sinensis, citrus aurantium, citrusnobilis, citrus reticulate and two kinds of lemon were taken for our study.Citrus rinds were peeled off carefully with the help of a sharp razor blade. The samples were checked to ensure that none of the white flesh under the rind was included in the sample, then each sample was dried using oven at temperature of 40° and grinded into powder.

Solvent Extraction Procedure

10 g of the sample was used for each run, and it was executed using Soxhlet apparatus that is set up. In the Soxhlet apparatus, the solvent in the round bottom flask was heated from the heating mantle to become evaporated and got condensed down through the sample where it was able to extract the oil along, thereby, giving a mixture of oil and solvent, which was later separated.

Chemical and Physical Properties

Chemical Properties

The moisture and PH of the samples were determined from the powder of citrus that means before extraction of oil. In addition, the properties of citrus oil extractedthatdetermined of each samples included Saponification number, yield, acid value and ester value.

Determination of moisture content

One gram of the peels of citrus fruits samples was placed into the uncoveredcrucibles and dried in the oven and then kept at 105°C forthree hours. The dried sample is next weighed to a constantweight. This process is repeated three times to reach theconstant weight [8].

$$\text{Moisture\%} = \frac{\text{lossed wieght of orange peel}}{\text{initial wieght of sample taken}} \times 100(1)$$

Estimate the PH in the samples

A mass of 1 gram of dry citrus peel powder is weighed and placed in a 100 ml baker and add 50 ml of distilled water to a magnetic stone. Light heating is carried out for 45 minutes and the samples are then filtered and the PH is measured by a PH meter[7].

Calculation of oil yield

The yield of the oil extracted using each of thethree methods of extraction was calculated using Equation (2).

$$\text{Yield\%} = \frac{\text{Weightof oil extracted}}{\text{Weightof sampleused}} \times 100\% \quad (2)$$



Determination of saponification value of the essential oil

Saponification value, being the weight of potassium hydroxide expressed in milligrams that is required to saponify 1 g of oil was also determined in this work. To carry out this, 2 g of the oil was weighed into a 200-ml conical flask into to which 50ml of 0.5 M of KOH was added. The resulting mixture was refluxed for 30 minutes, followed by addition of 3 drops of phenolphthalein indicator, and it was titrated against 0.5 M of HCl until coloration disappeared. This procedure was repeated without the oil and the titer value was determined from the blank value [9]. The saponification value was calculated using Equation (3).

$$\text{Saponification value} = \frac{(t_2 - t_1) \times 28.1}{W} \quad (3)$$

Determination of acid value of the oil

To determine the acid value of the extracted oil, 2g of the oil sample was weighed into a conical flask containing 50 ml of isopropyl alcohol. 3 drops of phenolphthalein indicator were added to the mixture. The resulting mixture was titrated against 0.1 M NaOH [9], and Equation (4) was applied to calculate the acid value of the oil.

$$\text{Acid value} = \frac{5.61 \times \text{titre value}}{\text{Weight of sample}} \quad (4)$$

Determination of ester value of the oil

Ester value, which is defined as the number of milligrams of potassium hydroxide required to saponify the fatty acid esters in one gram of the oil, was also determined for the oil extracted in this work. It was obtained as the difference between the saponification value and the acid value of the oil [9] as given in Equation (5).

$$\text{Ester value} = \text{Saponification value} - \text{Acid value} \quad (5)$$

Physical Properties

Physical properties of citrus oil extracted determined on each samples included refractive index, density and specific gravity. The refractive index of citrus oil determined by refractometer device. A drop of oil was poured around the prism of refractometer and refractive index was noted down. The density of the extracted oil was determined by weighing an empty beaker and recording its value. Thereafter, citrus extracted oil was poured into the beaker and the weight was taken. The density of the oil was calculated using equation (6) and specific gravity was calculated using equation (7).

$$\text{Density} = \frac{\text{Weight of oil sample}}{\text{Volume of oil in the beaker}} \quad (6)$$

$$\text{Specific gravity} = \frac{\text{Weight of oil sample}}{\text{Weight of water dripping}} \quad (7)$$

II. RESULTS AND DISCUSSION

Chemical characterization of extracted essential oil

The results obtained for the chemical properties of citrus oil samples extracted from six types of peels of citrus orange, bitter orange, mandarin, young lemon and lime, are given in table (1).



Table (1): chemical characteristics of extracted citrus oils sample.

Samples	PH (20 C ⁰)	Moisture%	Yield%	Acid Value mgKOH/g	Saponification Value mgKOH/g	Ester Value mgKOH/g
Blood orange (citrus sinensis)	4.90	58.39	38.85	6.73	156.11	149.38
Bitter orange (citrus aurantium)	4.64	58.33	16.35	7.08	100.98	93.90
Tangerine (Citrus nobilis)	4.89	72.38	32.9	6.18	75.94	69.76
Mandarine (citrus reticulata)	4.69	78.85	19.3	6.57	66.90	60.33
Small lemon	4.30	72.34	17.5	7.51	46.83	39.32
Lemon	3.94	33.33	22.1	6.29	24.43	18.14

Before starting to discuss the results, climatic factors play a major role in the difference in content and properties of citrus essential oil between species [10]. Therefore, the properties of citrus oil change from one study to another and from one type to another. For this reason, there are no standard values for the physical and chemical properties of citrus oil. However, the results obtained from this study can be compared with some studies conducted in different countries and cities.

Table 1 shows the chemical properties of citrus oil samples extracted from six types of peels of citrus blood orange, bitter orange, tangerine, mandarin, young lemon and lime. The moisture content ranged between 33.33% and 78.85%, and the highest moisture content was for tangerine orange peel, and this result is somewhat consistent with a study conducted in the state of Goiás, central western Brazil, where the moisture content of Lima oranges, beer oranges, Tahitian lemons, sweet lemons and tangerines was 70.3, 66.6, 72.6, 79.3 and 77.1, respectively [13].

Orange peel oil showed a pH value between 3.94-4.90. These values are within the range according to previous studies, in Goiás state, midwestern Brazil, the PH value of Lima, Beira and Puncan mandarin oranges were 4.78, 3.54 and 3.75, respectively [13], and in Oyo state, Nigeria, the PH value of oranges was 5.2 [11]. The result of the percentage yield was between 16.35 %-38.85 %, which is very similar to a previous study in Bauchi State, Nigeria, where the percentage yield of orange peel oil was about 30% [11].

Finding the acid value content in this study it was between 6.18-7.51mg/KOHg, and the result obtained is comparable with the previous result in Sikkim Himalaya, where the acidic value of mandarin peel oil at different altitudes for the ripe and immature stages ranged from 5.27 to 8.30 and from 5.74 to 8.17 respectively and for the raw lemon It ranged from 6.77 to 8.97 and from 6.70 to 8.43 for the mature and immature stages, respectively [10].

The saponification number value in this study ranged between 43.24 - 11.156 mgkOH/g which is close to several studies where In Sikkim Himalaya , the saponification value were 66.38, 83, and 86 mgkOH/g in mandarinePomelo, and rough lemon respectively in immature stage [10], in Oyo state, Nigeria the saponification value of orange was 41.25 mgkOH/g [11] , in Imo state, Nigeria the saponification value was 177.03 mg KOH/g for sweet orange [12] and in Bauchi state , Nigeria the saponification value of orange was 43.71 mgkOH/mg [1].

The value of the ester number in this study ranged between 18.14– 149.38 mgKOH/g and these values are similar to many studies, where the ester value of oranges in Bauchi State, Nigeria was



39.83 [1], and in Oyo State, Nigeria, the ester value of oranges was 28.96 [11], and in Sikkim Himalayas, the ester value of coarse lemon for the ripe and unripe stages ranged from 127.50 to 156.16 and from 28.30 to 67.40 respectively and for mandarin for the ripe and unripe stages at different heights ranged from 109.07 to 157.54 and from 21.70 to 57.93 [10].

Physical characterization of extracted essential oil

Moreover, the extracted citrus oils samples were analyzed to determine its physical properties, and given in table (2)

Table (2): physical characteristics of extracted citrus oils samples

Samples	Refractive index	Density (g/cm ³)	Specific gravity	Electric conductivity	TDS
Blood orange (citrus sinensis)	1.3961	0.973	1.08	3.90 ms	2.35 g/l
Bitter orange (citrus aurantium)	1.4395	0.878	1.10	6.57 ms	3.92 g/l
Tangerine (Citrus nobilis)	1.3767	0.889	1.006	3.45 ms	1.95 g/l
Mandarine (citrus reticulata)	1.4340	0.90	1.13	4.04 ms	2.40 g/l
Small lemon	1.4542	0.973	1.22	8.62ms	5.15g/l
Lemon	1.3712	0.895	1.01	6.15ms	3.68g/l

As reported in the table (2), the measured refractive index at a temperature of 17 C° was ranged from 1.3712 to 1.4542 of the essential oils samples. The highest refractive index value was for the essential oil of young lemon peel of 1.4542 and the lowest value for the essential oil of blood orange peel of 1.3712 and this result is somewhat consistent with a study conducted in Sikkim Himalayas, the refractive index of coarse lemon for the ripe and unripe stages ranged from 1.46 to 1.48 and for mandarin for the ripe and unripe stages at different heights ranged from 1.46 to 1.48 and from 1.46 to 1.49 respectively [10].

From the obtained results in table (2), it could be observed that the density at 17 C° of the essential oils samples were found ranged between 0.858 g/cm³ and 0.973 g/cm³. The present results are in agreement almost with those obtained in Minch town in Ethiopia [14] and in Baunchi state, Nigeria [1].

It can be observed from the table (2), that specific gravity at 17 C° of the extracted essential oils samples were found ranged from 1.01 to 1.22. Most of the extracted essential oils have specific gravity ranging from 0.696-1.88, and this result is somewhat consistent with a study conducted in Minch town in Ethiopia [14].

From the experimental results obtained from the extraction and the characterization of the essential oil from orange and lemon peels have shown that the maximum refractive index, density and specific gravity of essential oil extracted from young lemon peels. In fact, the differences of some present results were due to the agronomic and technological conditions influences the varietal characteristics appreciated by the chemical composition analysis.



CONCLUSION

Physical and chemical properties of extracted citrus oil were comparable with some previous studies and values reported in the literature. However, differences were probably due to some factors as species, locality, climate and the method that used in oil extraction.

The chemical and physical properties values obtained from the characterization of the oil indicated that, it could be used in different process industries of other valuable products.

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