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Study of some chemical composition of orange peel and its extracts

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Abstract
The yield ranged from 12.9 percent to 62.7 percent for ethanol and ethyl acetate extracts for four types of citrus peels (Mandarin, Aurantim, blood orange, and navel orange), with ethanol extracts yielding the highest yield of ethyl acetate. Naval peels yielded the maximum amount of extract, according to the results (62.7 percent). Moisture, ash, carotenoids, chlorophylls, proline, and sugar were discovered to be characteristics of orange peel. Different proportions were found in all cultivars of the peel, according to the findings. A single noticeable peak at 320nm was discovered in an ultraviolet-visible spectrophotometric scan of the extract.
Keywords: *Citrus peels, extraction, chemical composition.*

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I. Introduction

Citrus is a generic word for plants belonging to the Rutaceae family, which produces the world's most significant fruit (Wang et al., 2014). This family has a lot of phytochemicals that contain many bioactive compounds that are responsible for antioxidants and other biological functions (Proteggente et al., 2003, Anagnostopoulou et al., 2006, and Rafiq et al., 2016). Citrus fruits are widely consumed as fresh products and juice, and the peel is frequently discarded as waste, even though it contains a wide range of secondary components with significant antioxidant activity when compared to other sections of the fruit (Manthey and Grohmann, 2001, Rana and Blazque, 2012 and Genovese et al., 2014).

There are 140 different types of citrus fruit. Orange, tangerine, lemon, grapefruit, and other citrus fruits are among the many species (Kamal et al., 2011). Citrus fruits are high in ascorbic acid (vitamin C) and other organic acids, and they are mostly utilized fresh. Peels, pulp, and seeds of citrus fruits are high in dietary fiber, phenolic compounds, carotenoids, flavonoids, aromatic oils, and other chemicals found in concentrated juice (Hue, 1999). Citrus fruit production peaked at around 82 million tons in 2009-2010. A total of 50 million tons of oranges wereproduced (US Department of Agriculture, 2010). The percentage of fruit crusts utilized in juice production is around 34%, and the percentage of fruit crusts used as secondary products is around 44% (Li, 2006). Oranges are the most productive citrus, with artificial processing accounting for 33% of overall production. This results in the production of 15 million tons of secondary residues, which are a good source for the extraction of pigments such as carotenoids, pectin, and other pigments, and industrial treatments were very important to find new ways to profit from waste and dispose of pollution, and they are also useful as food additives and in medicine and pharmaceuticals (Verontica et al., 2008).

Because the residues of citrus manufacturing make up 45–50% of the weight of the original fruit and the peels make up around 30% of the total weight, they constitute a significant difficulty for food manufacturers and generate several environmental issues (Shukla et al., 2015). Researchers discovered that citrus peels have higher biologic activity than other parts of the citrus fruit. They are used as a natural addition and antioxidant in food products such as jelly. Researchers have become more interested in natural sources of biologically active compounds and functional foods in recent years. (Shukla et al., 2015; Shukla and Moon, 2009).

Epoicarp (internal), flavedo (External Colored Terminal), and albedo are the three types of peels. Phenols, amino acids, essential oils, carotenoids, pectin, flavonoids, sugars, vitamins, and minerals are found in the peels, making them protective against a variety of ailments (Wang et al., 2014). Citrus peels have the most complex carotenoids when compared to other fruits, as well as a high quantity of epoxides and flavonoids, accounting for 70% of total carotenoids (Bailey and Curl, 1961). Vitamin C, which is an organic acid dissolved in water and an antioxidant, is abundant in citrus peel (Cioroi, 2007, Hacizevki, 2009). Diabetes, cardiovascular illness, stroke, ophthalmology, dermatitis, and lung cancer are all treated using peel extract. Reduced total cholesterol, triglycerides, high cholesterol, and low-density lipoprotein minimize clinical risk (LDL). Because carotenoids and polyphenols are abundant in many vegetable wastes, most fruit and vegetable wastes can be used as potential source materials for extracting these vital components. The usage of these materials helps the environment and saves money.However, a sensory evaluation of these items is required (Rodriguez, et al., 2006).

II. Experimental Part

Material and methods

Preparation of citrus peel:

Four types of citrus peels were collected, which are orange peels (Mandarin, Aurantim, blood orange, and navel orange). The four orange fruits were obtained from the local markets in Al-Khums city/Libya.

Preparation was done in the inorganic chemistry laboratory/faculty of science / Elmergib University where four kinds of ripe orange fruits were taken and cleaned of dust and impurities and peeled and left the peels to dry at room temperature in a place far from direct sunlight and then the samples were ground with an electric grinder until they became a fine powder and then store them in the refrigerator until use.All chemicals in this study were purchased from BDH (ENGLAND) and Fisher chemical.

Preparation of Alcoholic extract:

Alcoholic extracts were made using the soaking method, in which 10 g of powder was weighed for each sample, and 100 ml of 70% ethanol and 80% ethyl acetate were added separately, then mixed well with a shaker and left for 3 days at room temperature, then filtered the extract using filter paper (Whatman 1.N0), then concentrated the filtrate with a (Rotary Vacuum evaporator) at 40-50°C; shake off (Boukhary et al., 2016).

Determination of the chemical compositionof citrus peels:

The content of moisture and ash were determined according to (A.O.A.C,2000).

Determination of sugars:

The free sugar was determined using a colorimetric method after the sugars in orange peel were extracted (Dalaly& Hakim, 1987).

The following steps are used to estimate sugars using the phenol method:

Take 100 mg of the plant material and soak it in 3 ml of 80% ethanol in a dark spot for 48 hours. After that time has passed, the alcohol is evaporated from the sample by placing it in an 80°C water bath. To each sample, add 20 mL distilled water, 2 mL extract, 5 mL phenol (5%), and 5 mL concentrated sulfuric acid (96%) while avoiding contact with the tube walls, resulting in a golden brown loofah.To make the color uniform, shake the samples with the shaker for 10 minutes before placing them in a water bath at 30°C for 15 minutes.

At a wavelength of 490 nm, the optical density is measured, and the concentration of sugars is calculated using the formula below.

Sugars concentration = 1.24 + 97.44 * (reading at 490).

Determination of proline by Drier &Gorning method 1974:

For extraction takes 100 mg of crushed orange peels, and add 2 ml of methanol 40%.Heat the whole in a water bath for 60 minutes at 85°C, with the tubes closed to prevent evaporation, and cool the tubes.

Coloring process:

Take 1 ml of the previous extract, 300 ml acetic acid, 2 ml acetic acid, and 10 ml Ninhydrine 1 mL of the resulting combination (120 ml of distilled water with 80ml Orthophosphoric acid).Boil the mixture for 30 minutes to create a yellow-orange to a red gradual color solution based on the proline concentration, and then add 5 ml of (Toluene) to each tube, shake to obtain two layers, and take the upper class. Then, using a spectrophotometer at a wavelength of 528 nm, add a quantity of Na_2SO_4 to each sample to dry the remaining water.

Calculate the amount of proline according to the following equation.

Proline quantity:Y (µmol/mg(ps)) = 0.62*DO/M.S

Y: Amount of Proline, DO: optical density, MS: dry matter. (Drier &Gorning 1974).

Determination of total Chlorophylls and Carotenoids:

Approximately 600 mg of orange peel extract was completely dissolved in 2 mL of cyclohexane. The calorimetric determination of chlorophyll and carotenoid was performed using the Minguezmethod(Minguezet al., 1991). The chlorophyll fraction has the highest absorption at 670 nm, while the carotenoid fraction has the highest absorption at 470 nm. E_0 = 613 for pheophytin as a major component in the chlorophyll fraction and E_0 = 2,000 for lutein as a key component in the carotenoid fraction were the coefficients of specific extinction used. As a result, the pigment content was determined as follows:

Chlorophyll (mg/kg) = ($A_{670} \times 10^6$) / (613 × 100 × d).
Carotenoid (mg/kg) = ($A_{470} \times 10^6$) / (2, 000 × 100 × d).
Where A is the absorbance and d is the spectrophotometer cell thickness (1 cm).

Ultraviolet-visible scanning:

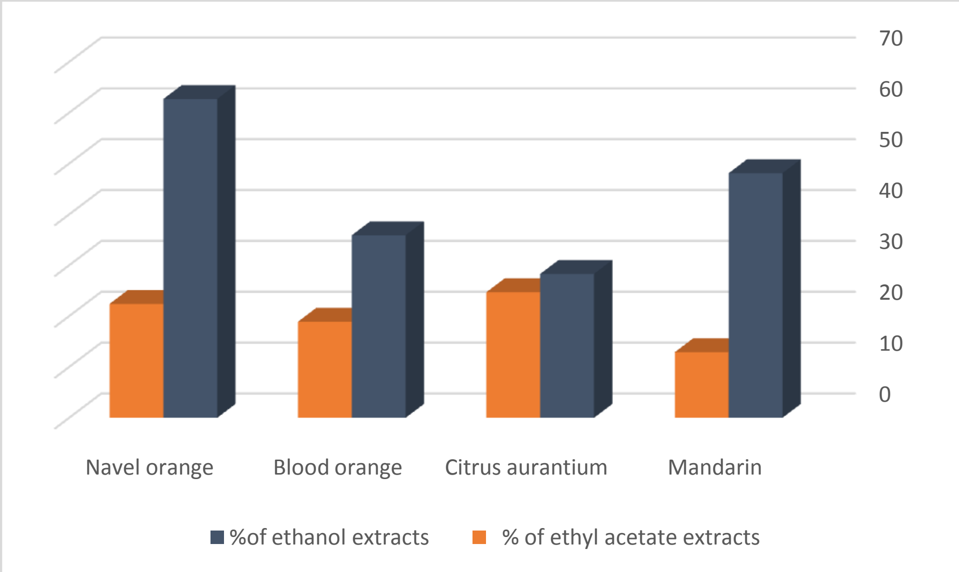
This was accomplished utilizing the Jenway model 6305 ultraviolet-visible spectroscopy method**.** The instrument was turned on and left running for about 3 minutes to stabilize it; 1 ml of the extract was measured out and made up to 10 ml with n-hexane (1 in 10 dilution); n-hexane was used as the blank. The extract and a blank solution were put into the apparatus, which was then scanned for the wavelength of maximum absorption.

III. Results and discussion

Table (1). The percentage of extracts ethyl alcohol and ethyl acetate.

Sample	The percentage of extracts	
	Ethanol extracts (%)	Ethyl acetate extracts(%)
Mandarin	48.1	12.9
Citrus aurantium	28.3	24.7
Blood orange	35.9	18.9
Navel orange	62.7	22.4

Figure (1). The percentage of extracts ethyl alcohol and ethyl acetate.



The percentages of ethyl alcohol and ethyl acetate extracts are shown in Table 1. The extraction of orange peels was done with the help of ethanol and ethyl acetate. The extracted oil was yellow to orange in color, had an orange scent, was pungent in scent, and had an oily viscosity. The yields achieved varied from 12.9 % to 62.7 %. Ethanol produced the highest output of navel orange peel extracts and ethyl acetate produced the lowest yield of mandarin peel extracts. In fact, the proportion varies depending on the harvesting season, plant portions, plant variety, sampling, and growing conditions. As a result, all of these variables can influence the yield. These characteristics can also affect the properties and composition of essential oils (Merve&Oguz, 2018).

Table (2). Measurement of the best absorbance of orange peel extract at a wavelength 320nm.

Sample	Abs
Mandarin	0.737
Citrus aurantium	0.729
Blood orange	0.701

Navel orange	0.735

Figure (2). Measurement of the best absorbance of orange peel extract at a wavelength 320nm.

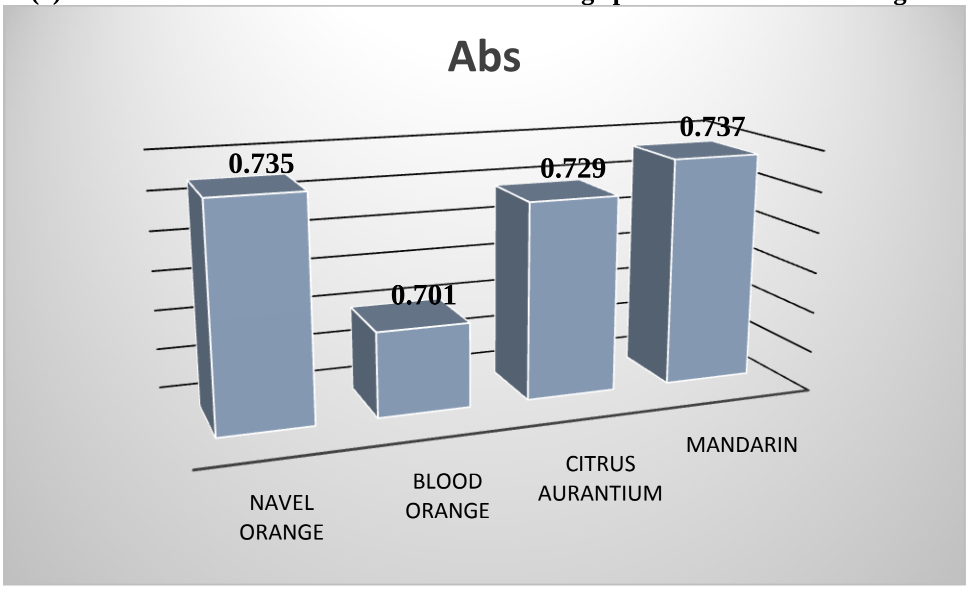
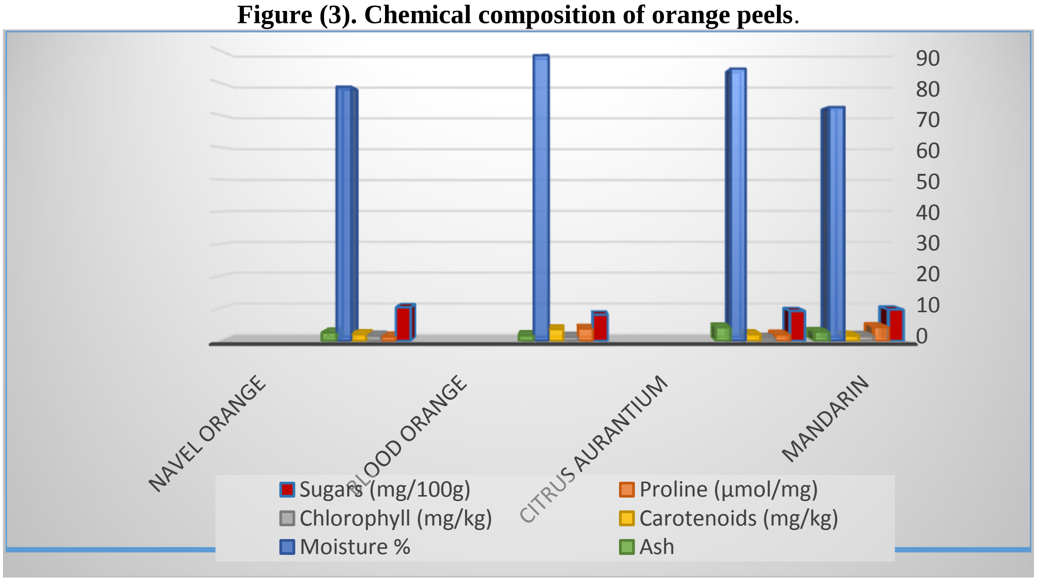


Table (2) shows the results of UV-visible (UV-visible) scanning of orange peel extract, with a maximum absorption wavelength of 330 nm. As a result, the comparatively conspicuous presence of a certain oil component within the composition of the oil is shown. Vekiari et al.,stated in 2002 that the major component of orange essential oil had a percentage composition of 90 to 95%.

Table (3). Chemical composition of orange peels.

Sample	Ash (%)	Moisture (%)	Carotenoids(mg/kg)	Chlorophyll (mg/kg)	Proline (μmol/mg)	Sugars (mg/100g)
Mandarin	2.86	72.38	1.71	1.61	4.368	**9.74**
Citrus aurantium	4.27	84.22	2.155	1.02	2.031	**9.289**
Blood orange	1.85	88.39	3.71	1.305	3.881	**8.119**
Navel orange	2.68	78.65	2.135	1.729	1.416	**10.465**

Figure (3). Chemical composition of orange peels.



The chemical composition of various citrus peels is shown in Table 3. The percentage of ash in the citrus aurantium peel was greater (4.27%). The obtained results of orange peels by Nassar et al. (2008) were incomparable, ranging from 0.5 to 4.7%. In addition, the ash value has been estimated at 3.50 % (Rivas et al., 2008).

In terms of moisture content, blood orange peels had the highest value (88.39%), with no significant difference between other peels with high moisture content. These findings are backed up by (Hayam& Ahmed 2018), who found that the moisture content of orange peel samples was high (74.35%). Similarly, Nesrine et al. achieved high moisture content results for examined orange peels in 2012.

Additionally, the blood orange peels had the highest concentration of carotenoids (3.71 mg/kg), which is why blood orange has a dark red color due to the high pigment level. The presence of various water-soluble pigments and the presence of carotenoids (-carotene) contribute to its hue. Oranges with a more reddish tint in their peel could be found (Laura et al., 2019).

Furthermore, citrus fruits, particularly oranges, are well recognized as one of the most complex natural sources of carotenoids (Dugo et al., 2009). As seen in table 3, the sugar content was highest in the navel peels (10.465mg/100g). These findings were similar to those of a recent study (Assa et al., 2013) that measured the concentration of reducing sugars in orange peels and found that the value ranged from 0.39 to 0.52 mg/100g, which was considerably different. When the sugars in orange peel were measured in 2009, the result was (23%) (Narjiset al., 2009). When compared to other peels, it has a higher concentration of total chlorophyll (1.729 mg/kg). Pigments are pigments that contain chlorophyll, such as carotenoid. As a result, the natural color of citrus fruit is composed of two phases: chlorophyll degradation and carotenoid production. Citrus color variation is caused by chlorophyll degradation during degreening and the appearance of carotenoid pigment (Albert C. and Charles R. (1981), Sumiasih I., et al., 2017). Mandarin peels, on the other hand, have a higher proline level (4.368 mol/mg). Due to its osmoprotectant action, proline is a non-toxic, highly soluble amino acid with a low molecular weight. It plays an important role in the stability of cell structures like membranes and proteins (Ashraf and Foolad 2007 Verbruggen N, and Hermans C. 2008). Finally, changes in the level of chemicals between the cultivars investigated are attributed to growth conditions, geographical location, and climate variability.

IV. Conclusion

The aim of this work is to contribute to the study of some of the chemical contents of mandarin, citrus aurantium, blood orange, and navel orange peels. Where the level of the content of the components varies on the type of citrus peel and the environment in which it grows. In this paper, two extractive solvents were used, and the absorption spectrum extracted obtained was characterized and determined to contain carotenoids and chlorophyll by a spectrophotometer UV-Visible. Waste orange peels should be prepared as a natural alternative material in industries, which stimulates economic growth and preserves the environment from the danger of pollution.

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