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Bioactive Compounds Content of Citrus Peel as Affected by Drying Processes

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***Abstract*—**The present investigation studied the content of bioactive compounds as ascorbic acid, β-carotene, and flavonoids, and the effect of drying methods (microwave, solar, and air oven drying) on its level in citrus peel. These levels were decreased significantly ($p < 0.05$) due to the dried methods. The percentage of ascorbic acid content loss of orange *C. Valencia* were 46.64, 52.95 and 68.83% with microwave, solar and air oven methods, respectively comparing to fresh samples. Also, the percentages of β-carotene loss of orange *C. Valencia* were 38.89, 52.42 and 87.14% with microwave, solar and air oven methods, respectively. Total flavonoid content recorded 453.33, 396.67 and 327.50 mg QE/100g dw, with dried by microwave, solar and oven methods, respectively compared with control in orange, *C. valencia*. These results revealed that microwave drying procedure was the most effective method which maintained citrus bioactive compounds content (ascorbic acid, β-carotene and flavonoid) followed by solar. On the other hand, air oven drying came in the last order due to direct heat treatment.

***Keywords*—**Ascorbic acid, β-carotene, flavonoids, microwave, solar, air oven drying.

I. Introduction

CITRUS fruits and juices are important sources of essential minerals and bioactive compounds as ascorbic acid, carotenoids, flavonoids, phenolic compounds and pectin's [1]. Epidemiological studies on dietary citrus flavonoids show that it is capable of reducing the risk of coronary heart disease and degenerative diseases, besides their effect as anti-carcinogenic and anti-inflammatory [2] and is attracting more and more attention not only due to their antioxidant properties but also because of their lipid anti-peroxidation effects [3]. Due to their high flavonoid content, citrus peels could be exploited by both pharmaceutical and food industries for functional foods production.

In fact, phenolic compounds, particularly flavonoids, have antioxidant activity towards free radicals, it is principally based on their structural which like conjugation, number, and place of phenolic hydroxyl and other groups [4]. Therefore, phenolic compounds, vitamin C, and carotenoids of the peels and fruit by-products is used as active ingredients in the production of functional food products when designing healthy foods or as synthetic preservatives substitutes [5].

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The peel of citrus fruit which represents mostly one-half of the fruit has the highest concentrations of flavonoids [6] and it has more vitamin C (ascorbic acid) than its juice according to the USDA National Nutrient data base. Therefore, extracts from peels were found to have a good total radical antioxidative potential (TRAP) [7].

Every year, there are large quantities of citrus fruits being processed into juice, which disposal represents a growing problem of having millions of tons of by-products with the risk of prone to microbial spoilage, thus, drying of peel is the first step of the production of citrus bioactive compounds by-products. Science drying and methods of extraction are the most important factors affecting the quantities and biological activities of phenolic compounds, the most of the conventional thermal treatments like, hot-air drying, vacuum drying, and sun-drying, which are used for food preservation primarily intended to inactivate enzymes, deteriorating microorganisms and reduce water activity. However, high temperatures and long drying periods usually reduce the product quality [8]. For that, the aim of the present study is to investigate the effect of drying on the bioactive compounds concentrations of citrus by-products as well as methods of extraction.

II. Materials and Methods

A. Plant Materials

Ripened and fresh fruit [orange (*C. Valencia, C. Balady*) and tangerine (*C. Reticulate*)] peels were washed and peeled off manually and their edible portions were carefully separated. Fresh citrus peels cut into cubes of 1 cm^3 before processing.

B. Chemicals and Reagents

All chemicals and reagents were (Sigma, Aldrich & Fluka) purchased from Sigma-Aldrich Chemical Co. (St. Louis, Mo, 63103USA). All other chemicals and reagents used were of analytical grade.

C. Technological treatments

All of the tested samples of citrus peels prepared were dried at 40 ^oC on perforated trays by using the different drying methods: Microwave drying, solar drying and air oven drying. Finally, it was milled to pass through 100 mesh screen sieve, as well as to fresh sample (control) according to [9].

D. Extraction Procedure

Methanolic extracts were made as follows: Citrus peel powder (4 g) was extracted with 80 ml of 80% methanol by holding in an ultrasonic device (200 W, 59 kHz,) for 60 min at room temperature. The mixture was then filtered through Whatman No. 4 filter paper [10].

E. Methods

1. Determination of Vitamin C (Total Ascorbic Acid)

2,6-dichloroindophenol titrimetric method [11] is used to determine the vitamin C content sample extracts. Results are expressed in mg ascorbic acid/g dry weight.

2. Determination of β-carotene

Determination of β-carotene contents was applied according to the method described by [12]. The absorbance of the filtrate was measured at (453, 505, 645 and 663). The results were expressed as µg of carotenoid per g of extracts. A content of β-carotene was calculated according to:

$$\beta\text{- Carotene (mg/100 ml)} = 0.216 \times A663 - 1.220 \times A645 - 0.304 \times A505 + 0.452 \times A453$$

3. Determination of Total Flavonoids Content

Colorimetric aluminum chloride method is used for flavonoids determination according to the methods described by [13], [14]. Total flavonoid contents are calculated as quercetin from a calibration curve, which is prepared by preparing quercetin solutions to concentrations 12.5 to 100 mg ml^{-1} in methanol.

F. Statistical Analysis

The data obtained from study was statistically subjected to analysis of variance (ANOVA) and means separation was calculated by [15]. The least significant difference (LSD) values are used to determine significant differences between means and to separate means at $p< 0.05$ using SPSS package version 15.0.

III. Results and Discussion

Drying of peel is the most important step for the utilization of citrus bioactive compounds from by-products, which provides a safe limit of microbial and chemical corruption in addition to reducing the amount of peel which facilitates the handling and storage of the peel. The present investigation proved the effect of various drying methods (microwave, solar and air oven drying) on the different types of citrus by-products (Orange *C. Valencia*, Orange *C. Balady* and Tangerine *C. Reticulate*) with minimum loss of bioactive compounds.

A. Drying Methods and Their Affected on Ascorbic Acid (Vitamin C) Content of Citrus Peel

Ascorbic acid is one of the sensitive vitamins that is affected by heat treatments. In our study, different drying methods were objected and data presented in Table I. There is a significant difference ($P<0.05$) between samples whereas, ascorbic acid concentrations of citrus samples varied between types. The highest value of ascorbic acid was detected in tangerine, *C. reticulate* (139.81 mg/100 g dw), followed by *C. valencia* (128.13 mg/100 g dw) and *C. balady* (89.79 mg/100 g dw). These levels decreased influencing by drying methods. For example, in orange *C. valencia*, ascorbic acid contents are recorded 68.38, 60.29 and 39.94 mg/100 g dw dried by microwave, solar and air oven methods, respectively. These results revealed that microwave drying procedure was the most effective method which maintained ascorbic acid contents of different citrus types followed by solar then air oven drying methods. The percentages of ascorbic acid content loss of orange *C. valencia* were 46.64, 52.95 and 68.83% with microwave, solar and air oven methods, respectively comparing to fresh samples (Fig. 1). This could be attributed to the fact that ascorbic acid is not stable at high temperature [16]. Similar results are reported by [17] that observed 98.20% losses in ascorbic acid content of red pepper dried at 90 ˚C.

B. Drying Methods and Their Affected on β-Carotene Content of Citrus Peel

Data in Table II showed that the contents of β-carotene in the collected samples are quite variable. Different types of citrus peels (Orange *C. valencia*, Orange *C. balady* and Tangerine *C. reticulate*) are affected significantly ($P < 0.05$) by different drying methods (microwave, solar and air oven drying). Results indicated that drying of the plants by applying the three techniques resulted in a degradation and decrease to β-carotene when compared to control.

The highest value of β- carotene (Table II) was detected in *C. valencia* (72.32 µg/100g dw), followed by *C. balady* (54.88 µg/100g dw) and tangerine *C. reticulate* (49.84 µg/100g dw). These levels were decreased by dried methods by the microwave, solar and air oven, which are recorded as 44.19, 34.42 and 23.76 µg/100g dw in Orange *C. valencia*, respectively. These results revealed that microwave drying procedure was the most effective method which maintained β-carotene contents of different citrus peel types followed by solar and air oven methods. β-carotene contents of orange *C. valencia* decreased to 38.89, 52.42 and 87.14% with microwave, solar and air oven methods, respectively (Fig. 2). Data proved that three drying procedures had significant effect ($P<0.05$) on β-carotene levels in which orange *C. valencia* was the most affected one followed by orange *C. balady*, then tangerine *C. reticulate*.

Heat treatment of drying methods of citrus plays an important role in decreasing β-carotene, while, solar-drying causes marked losses in β-carotene due to exposure to greater solar radiation particularly ultra violet (UV) rays, which catalysed β-carotene oxidation leading to loss of vitamin activity [18].

The carotenoids are greatly sensitive to light, heat, oxygen, acids. Whatever the processing method, carotenoid retention decreases with longer processing time, higher processing temperature, and cutting or puréeing of the food [19].

Citrus peels showed the presence of good amount of β-carotene which made them a primary attraction for further studies for the use of nutraceutical and pharmaceutical industries.

TABLE I
EFFECT OF DRYING METHODS ON ASCORBIC ACID CONTENT (MG/100 G DW) OF CITRUS PEEL

Sample types	Control		Microwave- drying		Solar-drying		Air oven-drying		L.S.D at 5 %
	Concentrations (mg/100 g dw)	Loss %	Concentrations (mg/100 g dw)	Loss %	Concentrations (mg/100 g dw)	Loss %	Concentrations (mg/100 g dw)	Loss %	
Orange *C. valencia*	128.13ᵃ±0.28	-	68.38ᵇ±1.36	46.64	60.29ᶜ±0.20	52.95	39.94ᵈ±0.99	68.83	1.62
Orange *C. balady*	89.79ᵃ±2.13	-	46.19ᵇ±2.13	48.56	42.18ᶜ±0.58	53.03	27.76ᵈ±0.75	69.09	2.38
Tangerine *C. reticulate*	139.81ᵃ±0.35	-	70.58ᵇ±1.21	49.51	62.79ᶜ±0.49	55.09	30.10ᵈ±1.38	78.47	2.13

- All values are means of triplicate determinations ± standard deviation (SD). - Means within rows with different letters are significantly different (P < 0.05).

TABLE II
EFFECT OF DRYING METHODS ON β-CAROTENE CONTENT (μG/100G DW) OF CITRUS PEEL

Sample types	Control		Microwave- drying		Solar-drying		Air oven-drying		L.S.D at 5 %
	Concentrations (mg/100 g dw)	Loss %	Concentrations (mg/100 g dw)	Loss %	Concentrations (mg/100 g dw)	Loss %	Concentrations (mg/100 g dw)	Loss %	
Orange *C. valencia*	72.32ᵃ ±0.79	-	44.19ᵇ±0.48	38.89	34.42ᶜ±1.17	52.42	23.76ᵈ±1.21	87.14	1.81
Orange *C. balady*	54.88ᵃ±1.46	-	35.13ᵇ±1.27	35.99	26.71ᶜ±1.54	51.34	17.51ᵈ±0.69	68.09	2.42
Tangerine *C. reticulate*	49.84ᵃ±1.82	-	31.70ᵇ±1.49	36.40	25.63ᵈ±1.60	48.57	17.68ᵈ±0.78	64.52	2.47

- All values are means of triplicate determinations ± standard deviation (SD). - Means within rows with different letters are significantly different (P < 0.05).

TABLE III
EFFECT OF DRYING METHODS ON FLAVONOIDS CONTENT (MG QE/100G DW) OF CITRUS PEEL EXTRACTED BY METHANOL

Sample Types	Concentrations (mg QE/100g dw)				L.S.D at 5%
	Control	Microwave- drying	Solar-drying	Air oven-drying	
Orange *C. valencia*	469.17ᵃ±44.46	453.33 ᵇ±8.04	396.67 ᶜ±12.33	327.50ᵈ±2.50	44.16
Orange *C. balady*	486.67 ᵃ±12.83	453.33 ᵇ±8.04	435.00 ᶜ±2.50	150.83 ᵈ±3.82	14.86
Tangerine *C. reticulate*	455.83 ᵃ±3.82	437.50 ᵇ±5.00	418.33 ᶜ±16.65	251.67 ᵈ±6.29	17.77

-All values are means of triplicate determinations ± standard deviation (SD) - Means within rows with different letters are significantly different (P < 0.05).

C. Effect of Drying Methods on Total Flavonoids Content of Citrus Peel

Flavonoids represent the main bulk of the phenolic compounds of citrus [20]. The flavonoids perform an essential role in the health benefit due to its strong antioxidant activities [21].

Data in Table III and Fig. 3 showed the contents of total flavonoids in different citrus peel extracted by methanol. The level of flavonoid of different citrus peel is quite variable among the different type of the sample sources. Flavonoids of citrus peel (Orange *C. valencia*, Orange *C. balady*, and Tangerine *C. reticulate*) were affected significantly (P <0.05) by drying methods (microwave, solar and air oven drying). Results indicated that the highest concentration of flavonoids content was found in methanolic extract from *C. balady*, followed by *C. valencia* and *C. tangerine*, which recorded 486.67, 469.17 and 455.83 mg QE/100g dw, respectively. These levels decreased significantly (p < 0.05) due to the dried methods. On the other hand, in Orange *C. valencia,* total flavonoid content recorded 453.33, 396.67 and 327.50 mg QE/100g dw, dried by microwave, solar and oven methods, respectively compared to control.

The results revealed that the microwave drying procedure was the most perfect method of retention flavonoid content of different citrus types followed by solar and oven methods. The law amounts of flavonoids of the oven dried citrus peel may be caused by the high-temperature treatment that was used in the oven drying. These results were agreed with [22] with the main reason that loss of flavonoids during heat treatment might be due to dry conditions precisely heat treatment.

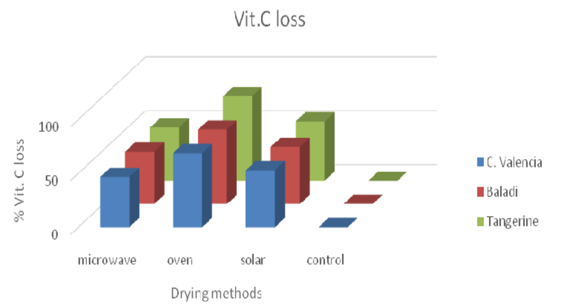

Fig. 1 Drying methods and its affected on ascorbic acid loss (%) of citrus peel

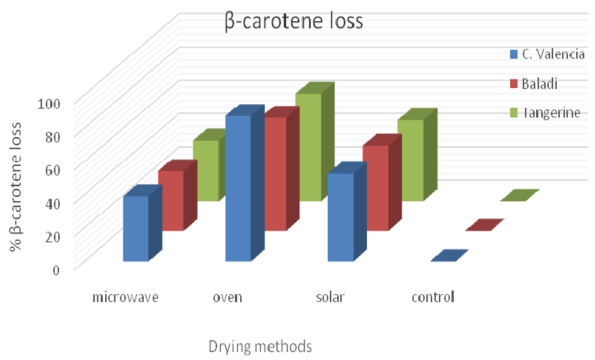

Fig. 2 Drying methods and its affected on β- carotene loss (%) of citrus peel

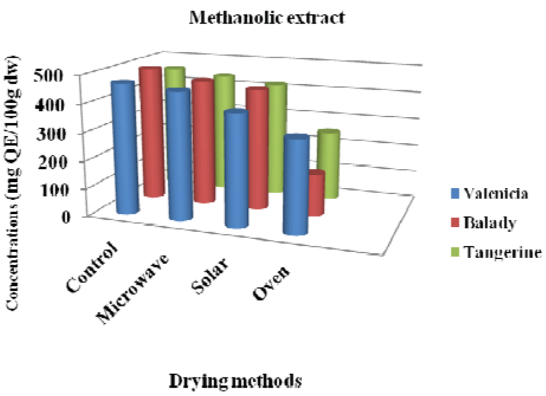

Fig. 3 Drying methods and its affected on flavonoids content (mg QE/100g dw) of citrus peel extracted by methanol

Data proved that total flavonoid concentrations of orange *C. balady* were the most affected by the three drying procedure, followed by Tangerine *C. reticulate* and Orange *C. valencia.* Results of the present study showed that methanolic extracts were better for flavonoid extraction. Similar results reported by [23].

IV. Conclusion

From the available data, it could be concluded that ascorbic acid (vitamin C), β-carotene (vitamin A) and total flavonoids content of citrus peel are relatively high. So, it could be recommended to utilizing as bioactive ingredients sources in order to produce functional food with choose the suitable methods of drying as microwave drying method.

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