

Bioactive Compounds in Some Citrus Peels as Affected by Drying Processes and Quality Evaluation of Cakes Supplemented with Citrus Peels Powder

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ABSTRACT: Citrus peels as orange, mandarin and lemon peels are the most important aspect of this study. Citrus peels are good source of dietary fiber, elements and bioactive compounds. Studies on the effect of drying methods (sun and oven drying at 50°C) on the chemical composition and bioactive compounds of dried citrus peels were conducted. The results showed that oven drying method at 50° C indicated higher levels of crude protein, total dietary fiber, elements (K, Ca, Na, Mg, P, Zn and Fe) and bioactive compounds (total phenolic content, ascorbic acid and flavonoids) than sun-drying of citrus peel samples. On the other hand, this study also evaluated the effects of supplementation of wheat cake with orange and mandarin peels powder at 5%, 10%, 15% and 20% levels (The ratio between orange and mandarin peels was 1:1 by weight). The prepared cakes were analyzed for physicochemical composition and evaluated for sensory properties. The results indicated that cakes prepared with 10% of orange and mandarin (1:1 by weight) peels powder were higher in elements value and were highly scored (significantly ($p < 0.05$)) by panelists as compared to control and other samples. It was concluded that, citrus peels are rich in fiber and bioactive compounds and are recommended to be used as food additives to gain nutritional and healthy benefit.

Keywords: citrus peels, drying methods, cake, chemical composition, bioactive compounds

INTRODUCTION

Citrus is a universal term for plants belonging to family *Rutaceae*, which considered as the most important fruit around the world (Wang *et al.*, 2014). This family has rich phytochemicals sources of many bioactive compounds which are responsible for antioxidant and many other biological activities (Proteggente *et al.*, 2003, Anagnostopoulou *et al.*, 2006, Guimaraes *et al.*, 2009 and Rafiq *et al.*, 2016).

Citrus fruits are highly consumed worldwide as fresh produce, juice, and most often the peel is discarded as waste which contains a wide variety of secondary components with substantial antioxidant activity in comparison with other parts of the fruits (Manthey and Grohmann, 2001, Rana and Blazque, 2012 and Genovese *et al.*, 2014).

Citrus peels are generated as the primary citrus by products represent about 30-50 % of fruit weight during processing. These by products discarded and considered as a huge load to the environment (Wang *et al.*, 2008 and 2014). These by products can be used as ingredients and food additives in food industry for their cheap valuable component (Galanakis, 2012).

In Egypt and many Mediterranean countries, a major quantity of the citrus peels do not use in proper applications. Some efforts were made to use these residues as livestock feed (Farhat *et al.*, 2011).

Natural products present in citrus peels e.g. dietary fibers, flavonoids, carotenoids, folic acid, vitamin C, pectin and essential oils present are very useful for food industry and human health. Also, citrus peels are good source of

phenolic compounds can be extracted and employed as natural antioxidants to prevent oxidation of some foods or may be utilized in designing functional foods (Zaker *et al.*, 2017).

Dietary fiber consists of cellulose, hemicellulose, lignin, pectin, glucans and gums (Figuerola *et al.*, 2005). It could be classified into two major groups: water soluble polymers (SDF), such as pectin and gum, and water insoluble materials (IDF), in which cellulose, hemicellulose and lignin are included (Vergara-Valencia *et al.*, 2007). Dietary fibers show beneficial physiological effects on human health. It has been reported that high dietary fiber consumption is associated with the prevention and treatment of obesity, diabetes, colorectal cancer, atherosclerosis and coronary heart diseases (Borchani *et al.*, 2011). It is well known that the functional properties of dietary fibers have the greatest effect on their functions in foods (El-Refai *et al.*, 2006). The functional properties of plant fiber depend on the IDF/SDF ratio, particle size, extraction condition, structure of the plant polysaccharides and vegetable source. Dietary fiber inserted into bakery products provides benefits on the health of the heart, gastrointestinal pain, reduce the risk of various cancers. Also, dietary fiber have the role of ensuring a good intestinal transit, preventing constipation, reducing fat absorption from the digestive tract, as well as favouring the absorption of toxins (Gallagher and Schneeman, 2001 and Lupton, 2004) .

Drying has become a widely used way of food processing, allowing the extension of the shelf-life of fruits and by-products. However, processing may cause irreversible modifications to the cell wall polysaccharides, affecting their original structure (Femenia *et al.*, 2009). Therefore, the final quality of the dried by-products can be determined by structural and compositional modifications which might occur during the drying processes (Garau *et al.*, 2007 and Manjarres-Pinzon *et al.*, 2013).

The effects of the drying temperature on the quality of dried citrus peels, which are value-added food ingredients, were studied (Chen *et al.*, 2011, Kamal *et al.*, 2011, Al-Juhaimi, 2014, Marey and Shoughy, 2016, Abou Arab *et al.*, 2016 and 2017).

Most of the conventional thermal treatments such as, hot-air drying, vacuum drying and sun-drying are used for food preservation primarily intended to inactivate enzymes, deteriorative microorganisms and reduce water activity. However, high temperatures and long drying periods usually reduce the quality of the final product. It has been reported that many reactions can affect color and minerals during processing of fruits and their derivatives (Bejar *et al.*, 2011).

The citrus peels can serve as source of valuable minerals required for normal functioning as the body system. Iron (Fe) is necessary for the formation of hemoglobin and also plays an important role in oxygen and electron transfer in human body (Mahapatra *et al.*, 2012) and normal functioning of the central nervous system and in the oxidation of carbohydrates, proteins and fats. The observation of anemia in Fe deficiency may probably be related to its role in facilitating iron absorption and in the incorporation of iron into hemoglobin.

Sodium (Na) plays an important role in the transport of metabolites. High sodium intake has been proved to increase high blood pressure (Paul and Shaha, 2004). The ratio of Na /K in any food is an important factor of prevention of hypertension arteriosclerosis, with K depresses and Na enhances blood pressure.

Phosphorus (P) is an essential mineral for all animals too. Deficiency in phosphorus is the most widespread of all the mineral deficiencies effecting livestock. Phosphorus must be balanced with the animal diet of adequate Ca and vitamin D for growth, reproduction, gestation, and lactation (Mahapatra *et al.*, 2012).

The positive impact on zinc (Zn) supplementation on the growth of some stunted children, and on the prevalence of selected childhood diseases such as diarrhea, suggests that zinc deficiency is likely to be a significant public health problem, especially in developing countries (Osendarp *et al.*, 2003).

Baking industry is considered to be one of the major segments of food processing in Egypt. Baked products have popularities in the people because of their availability, ready to eat convenience and reasonably good shelf life (Vijayakumar *et al.*, 2013 and Zaker *et al.*, 2017). Baked foods especially, biscuits and cakes are among these ready to eat foods used at home. However, they contain a very limited amount of therapeutic substances (phytochemicals) and digestion facilitating substances such as bioactive compounds and dietary fibers. These substances can be obtained through partial supplementation of wheat with flour prepared from citrus peels as orange, mandarin and lemon peels (Nassar *et al.*, 2008, Youssef and Mousa, 2012, Akubor and Ishiwu, 2013, Kolo *et al.*, 2016; Zaker *et al.*, 2017).

These peels are rich in nutraceuticals and dietary fibers; to utilize them as food ingredients is in the way of conducting waste management related to new food sources research and development (Mahmoud *et al.*, 2015, Ojha1 and Thapa, 2017)

For this reason, this study was carried out to investigate the effect of two drying methods on the chemical composition and bioactive compounds of dried orange, mandarin and lemon peels and processing them into flour to supplement wheat flour in cake and also evaluated their overall quality characteristics.

MATERIALS AND METHODS

Materials and Chemicals:-

1. Sample collection and Preparation:-

Oranges (Washington Navel orange) (*Citrus sinensis*, L), lemon (*Citrus lemon*, L) and mandarin (*citrus reticulata*, L) were purchased from the local market of Alexandria governorate, Egypt.

Citrus fruits were washed with tap water then peeled. Peels were chopped with a knife in small slices (about 1 cm²).

2. Chemicals: -

All the reagents used in this study were purchased from Sigma-Aldrich, Inc. All other chemicals used were of analytical grade.

Methods

1. Drying methods:-

1.1. Open sun drying method

Five kilogram peels of each citrus variety (orange, lemon and tangerine) were spread on a single layer of white cloth and sun dried until equilibrium moisture content was achieved to 10%. During the sun drying of peels slices, the air temperature and relative humidity were determined by using thermometer and hygrometer. The air temperature and relative humidity was recorded as $30 \pm 2^\circ\text{C}$ and $38 \pm 6\%$, respectively. Open sun drying experiments were done in Jun, 2016 from sunrise to sunset for a period of six days with an average of eight hours per day. Dried peels were ground to a fine powder and passed through a 24-mesh sieve according to the method described by Abou-Arab *et al.*, (2017). The flour was stored at room temperature in dark glass airtight containers until needed.

1.2. Oven drying method

Five kilogram peels of each citrus variety (orange, lemon and tangerine) were oven dried at $50 \pm 2^\circ\text{C}$ for 72 hours until equilibrium moisture content was achieved to 10%. Dried peels were ground to a fine powder and passed through a 24-mesh sieve according to the method described by Abou-Arab *et al.*, (2017). The flour was stored at room temperature in dark glass airtight containers until needed.

2.2. Preparation of ethanolic extract

Fifty grams dried powder of each peel was added to 500 mM ethanol 98%, then blended well using carburetor magnetic and left for 24 h in the dark at 25°C . The obtained extract was nominated using filter paper (Whatman No. 1). The residue was re-extracted (3-4 times) with ethanol. The filtrate was concentrated using rotary vacuum evaporated at 40°C to get rid of the solvent. The filtrate was transferred into dark bottle glass in desiccator in the dark at room temperature for 24 h then placed in opaque bottles and kept in the refrigerator at 4°C until use (Mahmoud *et al.*, 2016).

2.3. Proximate Analysis and dietary fiber

Moisture content was determined after oven drying to a constant weight at 105°C . Proteins, lipids and ash were analyzed according to AOAC methods (AOAC, 2000). Total dietary fiber (TDF), soluble dietary fiber (SDF) and insoluble dietary fiber (IDF) contents of samples were determined with an enzymatic-gravimetric procedure according to AACC, (2011) and Abou Arab *et al.*, (2016).

2.4. Determination of total phenolic

The total phenolic compounds were evaluated using a modified Folin Ciocalteu Reagent (FCR) method (Al-Juhaimi, 2014). One mL of FCR was added to 200 μL of sample or standard solution and mixed thoroughly. After 10 min incubation at room temperature 1mL of 20% Na_2CO_3 solution was added,

followed by thorough mixing. The solutions were incubated further at room temperature for 2 h and absorbance values were recorded using a spectrophotometer (Ultrospecll; Biochrom-LKB, Cambridge, England) using a blank for auto zero at 765 nm. The blank carried all reagents as previously mentioned but was without either sample or standard solution. Total phenolic were expressed as milligrams of gallic acid equivalent per 100 gram (mg GAE/100 g) of dried peel or pulp from individual citrus fruits.

2.5. Quantification of ascorbic acid

0.4mL sample (diluted peel or pulp extracts using methanol) was mixed with 4 mL reagent (mixture of 4 mM NaH_2PO_4 , 2 mM H_2SO_4 , and 0.6 M $(\text{NH}_4)_2\text{MoO}_4$ solutions). 4 mL reagent and 1mL methanol were joined to get blank. The tubes were tightly capped and sealed using paraffin wax and put at 95°C (in a water bath) for 90 min. After this treatment samples were immediately cooled to ambient temperature under running water. The values of absorbance were recorded at 695 nm against a blank. The pure ascorbic acid standard was used to obtain a calibration curve and its comparative contents in the peel and pulp extracts were presented as milligrams of ascorbic acid per 100 (mg/ 100 g) of either of the dried pulp or peel (Al-Juhaimi, 2014).

2.6. Determination of total flavonoids content

Colorimetric aluminum chloride method was used for flavonoids determination according to the method described by Ebrahimzadeh *et al.*, (2008). One gram solution of each extract was mixed separately with 1.5 mL ethanol, 0.1 mL 2% aluminum chloride $\text{AlCl}_3 \cdot 6\text{H}_2\text{O}$, 0.1 mL 1 M potassium acetate and 2.8 mL distilled water and then left at room temperature for 10 min. The absorbance of the mixture was measured at 367 nm on UV/visible spectrophotometer. The quercetin (mg/ ml) was used as a standard for the calibration curve. The total flavonoid content of the samples was calculated by using the following linear equation based on calibration curve according to the following equation:

$$Y = 0.0205X - 1494 r$$

Where, Y is the absorbance and X is the flavonoid content in (mg/ ml) and $r = 0.9992$.

2.7. Determination of elemental contents

The element contents of citrus peels were determined as described by Romelle *et al.*, (2016). An amount of 2 g of fruit peels was dried in air oven at 105 °C for 3 hours. The dried sample was next charred until it ceased to smoke. The charred sample was then a shed in a muffle furnace at 550 °C until a whitish or greyish ash was obtained. The ash was treated with concentrated hydrochloric acid transferred to a volumetric flask and made up to 100 mL before submission to Atomic Absorption Spectrophotometry (AAS). For AAS, a SHIMADZU atomic absorption flame emission spectrophotometer model AA-670 IF with an air-acetylene flame, and wavelength respectively set to 422.7 nm for calcium, 279.5 nm for manganese, 248.3 nm for iron and 213.9 nm for zinc determination was used. Stock solutions (1000 mg/l) of calcium, manganese, iron and zinc were used to prepare working standard solutions with at least 4 concentrations within the analytical range. To eliminate phosphorus interference, lanthanum chloride was added to working standard solutions of calcium and to

the test ash solution destined to calcium determination so that the final solutions contained 1% La. Concentration of each mineral contained in test solutions was calculated from the standard curve prepared. Total phosphorus was assayed by spectrophotometer method (by the sodium phosphomolybdate) according to the Garau *et al.*, (2007).

2.8. Preparation of cakes

Cakes were prepared using the standardized recipe and method given by Sharoba *et al.* (2013) and modified by Zaker *et al.* (2017). The formula used was as follows: 250 g soft wheat flour, 125 g sugar, 53.50 g shortening, 12.50 g of baking powder, 110 g fresh whole egg, 25 g skim dry milk, 2 g vanilla and 70 - 72 ml water. The fat was beaten thoroughly, the sugar was added to butter and mixed until got smooth like cream and then a well blended egg with vanilla were added and mixed together. The blends soft wheat flour (72%) with citrus peel flour as dietary fiber sources (orange, mandarin and lemon peels powder) were replaced with wheat flour at 5, 10, 15 and 20 percent levels), baking powder were stirred together and added alternately to the egg mixture. The mixture was whipped until got smooth. The dough transferred to a greased pan was baked for 25 min. at $200\pm 5^{\circ}\text{C}$ then was cooled at room temperature. Cakes were prepared according to the formula shown in (Table 1).

Table (1). Raw ingredients of processed cakes according to Zaker *et al.* (2017)

| Ingredients | Weight (g) |
|-----------------------------------|------------|
| Soft wheat flour (72% extraction) | 250.0 |
| Sugar | 125.0 |
| Skimmed milk powder | 25.0 |
| Shortening | 53.50 |
| Fresh whole egg | 110.0 |
| Baking powder | 12.50 |
| Vanillia | 2.0 |

2.8.1. Physical characteristics for cakes:-

The weight (g) for cake was determined individually within one hour after baking the average was recorded. The volume (cm^3) of different types of produced cakes was determined by rape seeds displacement method according to (Zaker *et al.*, 2017). Specific volume was calculated using the following equation:

$$\text{Specific volume} = \text{Volume (cm}^3\text{)} \div \text{Weight (g)}$$

2.8.2. Organoleptic evaluation of cakes:-

The sensory evaluation was done on nine point hedonic scale as per the method given by Zaker *et al.*, (2016). The sensory evaluation of prepared cake was carried out by a 25 member trained panel comprising of postgraduate students and academic staff members of faculty who had plenty previous experience in sensory evaluation of bakery products. The panel members were requested to measure the terms identifying sensory characteristics and in use of the score. Judgments were made through rating products on a 9 point Hedonic

scale with corresponding descriptive terms ranging from 9 'like extremely' to 1 'dislike extremely'.

3.2. Statistical analysis:-

Analysis of variance for individual character was done on the basis of the mean values as suggested by Snedecor and Cochran (1980).

RESULTS AND DISCUSSION

1. Proximate composition of fresh and dried citrus peels:-

Proximate composition as described in (Table 2) are provide a general nutritional value of fresh orange, mandarin and lemon peels as well as their samples dried by sun and oven dried at 50 °C methods. Fresh orange peel sample contained more moisture content (72.10 %) than mandarin (71.85 %) and lemon samples (71.10 %). Fresh citrus peel of Valencia orange (*Citrus sinensis*, Rutaceae), mandarin, and lemon are characterized by high moisture contents as reported by Nesrine *et al.*, (2012).

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. Sun and oven drying were stopped at about 10% moisture content in citrus peels. The dried orange peel responses were significantly dependent on drying temperature. The quality of citrus peels samples should be taken into consideration in order to obtain better dried products (Manjarres-Pinzon *et al.*, 2013 and Adewole *et al.*, 2014).

Fresh orange peels contained 1.90 % crude protein whereas lemon peels contained 1.85% and mandarin peel had lowest crude protein 1.80%. Also, ether extract of fresh mandarin peel exhibited the higher percent being 0.95% followed by orange peel as 0.91 % and lemon peel as 0.85%. Ash contents were ranged from 0.80 – 0.98 % in fresh citrus peels. Total dietary fiber content of fresh orange peel (25.10%) was greater than mandarin (24.0 %) and lemon peel (23.9%). Insoluble dietary fiber recorded the highest amount of total dietary fiber. The results of proximate composition in fresh citrus peel were in agreement with Alam *et al.*, (2016) and Abd El-ghfar *et al.*, (2017). The results of dietary fibers were in agreement with Garcia *et al.*, (2010) and Borchani *et al.*, (2012). After drying, orange peels still have more crude protein and total fiber (Table 2). Protein content was 8.21 % in sun drying and 8.72 % in oven drying, respectively comparing to control sample 1.90 %. The trend of the results from this study generally indicates that the oven drying retains higher levels of crude protein than sun-drying. This may be due to the fact that that sun-dried vegetables are prone to dust and dirt contamination, attacks insects, and re-wetting of the drying vegetables by rain (Keding *et al.*, 2007) which might affect the level of protein. Fat content of Valencia orange was 1.57 % and 2.82 % in sun and oven drying, respectively comparing to fresh sample, 0.91 % while fat content of mandarin was higher as 3.94 % and 3.32 % in sun and oven drying, respectively comparing to fresh sample, 0.95%. These differences between treatments reflect the effect of drying methods of proximate composition. Similarly, Siles *et al.* (2010) reported that oven drying lowers lipid

content. Ash content of Valencia orange was in lowest level and varied from 10.03 % in sun -drying and 9.48 % in oven drying, respectively comparing to control samples (fresh) 0.80 %. The variation in ash contents also depends on plant species, geographical origins, their method of mineralization, as well as effect of food processing by drying (Sanchez-Machado *et al.*, 2004).

Total Dietary fiber (TDF) was classified according to its solubility, as soluble dietary fiber (SDF) and insoluble dietary fiber (IDF). As a consequence of such differences in its hydration capabilities, each fiber type produces a different physiological response (Garcia *et al.*, 2010). Table (2) presents the values of IDF, SDF as well as TDF of fresh and dehydrated citrus peels. The high IDF content indicates considerable amounts of celluloses and hemicelluloses present in the citrus peels. A high proportion of IDF could be considered an advantage because IDF can be used by the food industry as an ingredient to increase the content of indigestible insoluble compounds (Ramulu and Rao, 2003, Peerajit *et al.*, 2012).

When comparing dehydrated samples with fresh ones, the results showed that the applied drying temperatures had negative effects on IDF. TDF decreased more after sun drying than after oven drying. Comparable results were reported by Borchani *et al.* (2012) working with dehydrated date fiber concentrate.

It is recommended that the ratio of IDF to SDF should be in the range of 1.0–2.3 for certain food application (Peerajit *et al.*, 2012). The ratio IDF to SDF in fresh Cape gooseberry samples presented a value of 7:1 (Vega-Galvez *et al.*, 2009). Based on the results, drying at 60 °C could be an appropriate working temperature to achieve a final dried product with high quality aspects.

The recommendations regarding the intake of DF are not the same in all countries. The United Kingdom proposes 18 g/d of DF expressed as Nonstarch polysaccharides (NSPs), while an amount of 30 g/d has been proposed by Germany, and in the United States the specified intake should be 38 g/d for men and 26 g/d for women (BeMiller, 2004).

Table (2). Proximate chemical composition of orange, mandarin and lemon peels as affected by drying methods.

| measurments | Orange peel samples | | | Mandarin peel samples | | | Lemon peel samples | | |
|--------------------------------|---------------------|------------|-------------|-----------------------|------------|-------------|--------------------|------------|-------------|
| | Control (Fresh) | Sun Drying | Oven Drying | Control (Fresh) | Sun Drying | Oven Drying | Control (Fresh) | Sun Drying | Oven Drying |
| Moisture | 72.10±0.2 | 10.08±0.11 | 10.02±0.81 | 71.85±0.32 | 10.12±0.21 | 10.15±0.31 | 71.10±0.98 | 11,89±0.22 | 11±0.55 |
| Protein | 1.90±0.16 | 8. 21±0.92 | 8. 72±0.92 | 1.80±0.76 | 8.77±0.62 | 7.92±0.72 | 1.85±0.72 | 7.04±0.35 | 7.89±0.74 |
| Ether-extract | 0.91±0.31 | 1.57±0.65 | 2.82±0.42 | 0.95±0.32 | 3.94±0.91 | 3.32±0.85 | 0.85±0.61 | 2.42±0.93 | 2.55±0.54 |
| Ash | 0.80±0.84 | 10.03±0.91 | 9.48±0.28 | 0.40±0.87 | 7.35±0.19 | 7.22±0.53 | 0.98±0.94 | 7.92±0.63 | 7. 72±0.89 |
| Insoluble dietary fiber | 14.90±0.33 | 42.32±0.14 | 50.22±0.65 | 12.8±0.54 | 44.66±0.45 | 49.74±0.45 | 13.4±0.65 | 44.84±0.95 | 47.98±0.13 |
| Soluble dietary fiber | 10 .20±0.92 | 27.28±0.62 | 19.25±0.21 | 11.2±0.17 | 25.16±0.17 | 21.15±0.20 | 10.5±0.10 | 25.89±0.20 | 23.36±0.10 |
| Total dietary fiber | 25.10±0.46 | 69.60±0.16 | 69.47±0.87 | 24.0±0.74 | 69.82±0.96 | 70.89±0.66 | 23.9±0.37 | 70.73±0.41 | 71.34±0.29 |

Data are expressed as mean ± standard deviation (triplicate).

2. Elements content of citrus peel (mg/100g DM) of fresh and dried citrus peels:-

Table (3) shows the elements content [Calcium (Ca), Potassium (K), Sodium (Na), Manganese (Mg), Phosphors (P), Zinc (Zn) and Iron (Fe)] of citrus by-products (orange valencia, mandarin, and lemon) peels. Ca, K, Na and Mg were the predominant elements in the studied citrus peels. Orange fresh peel presents the highest amount of K (208.5 mg/100g), Ca (159.5 mg/100g), Mg (19.98 mg/100g) and Fe (32.6 mg/100g). Indeed, from many studies, a high proportion of potassium content is related to its assimilation or absorption by tissues. Thus, the great content of the citrus peels elements may have resulted from its abundance in the tissues of the orange (Wastowski *et al.*, 2013). Potassium records an important nutritious role in any organism. Intake of higher potassium content and sodium less could prevent the hypertension, source of the cerebral vascular damages and the heart diseases. It's the main intracellular mineral. It takes part in the muscular activity and to the heart muscular. On the other hand, a dietary with high potassium content of favorable to the bone healthy thanks to its alkaline effect (Cook and Obarzanek, 2009).

The calcium proportion is lower than that potassium. One of the main benefits from calcium related to interactions between cells walls. Therefore, it ensures the cells structure by hard cementing them. Calcium is a cellular component and regulator of the nervous excitability. It's also a factor of ethylene synthesis during the fruits ripening (Morad, 1996). The level of Ca was high in all samples. This is important for humans given its role in bone and tooth development. Furthermore, Ca may be used for the prevention and treatment of hypertension. Mg and K are cofactors for many enzyme reduction (Oscan and Hacisferogccullari, 2007). The citrus peels can serve as source of valuable minerals required for normal functioning as the body system. The utilization of these peels will enhance conversion of waste to wealth. It will also contribute positively to solid waste management and cleaner environments.

Table (3) shows the effect of some different drying methods of the minerals contents of citrus peels. The results showed that the calcium (Ca) contents of the orange valencia, mandarin and lemon peels were increased by drying with the oven-drying more than sun-drying. This results are in agreement with those reported by (Abioy *et al.*, 2014), who reported that the calcium content of baobab leaves were increased significantly by drying with the solar-dried samples had the highest increase in the metal in with incremental values.

In the same trend solar-drying had the highest zinc values. The processing methods had an advantage in improving zinc quality of citrus peels, which showed that it will contribute more to this mineral than other samples.

The results indicated that potassium (K), Na, Mg, P and Fe contents of studied citrus peels were reduced by both drying methods compared with control. The level of components content varies and depends on the type of citrus and method of drying. The reduction in this content may be due to the nature and sensitivity of the components to the level of heat during the drying process (Mahmoud *et al.*, 2015).

Table (3). Mineral composition of orange, mandarin and lemon peels as affected by drying methods

| Components (mg/100g) | Orange peel samples | | | Mandarin peel samples | | | Lemon peel samples | | |
|-------------------------|---------------------|---------------|----------------|-----------------------|---------------|----------------|---------------------|---------------|----------------|
| | Control (Fresh) | Sun Drying | Oven Drying | Control (Fresh) | Sun Drying | Oven Drying | Control (Fresh) | Sun Drying | Oven Drying |
| Calcium (Ca) | 159.5±0.35 | 160.3±0.94 | 163.2±0.39 | 153.4±0.16 | 155.6±0.16 | 154.2±0.12 | 143.3±0.11 | 145.2±0.16 | 144.5±0.13 |
| Potassium (K) | 208.5±0.24 | 201.2±0.21 | 205.4±0.52 | 195.4±0.30 | 193.6±0.12 | 194.7±0.22 | 204.5±0.83 | 200.75±0.20 | 201.3±0.81 |
| Magnesium (Mg) | 19.98±0.29 | 19.32±0.22 | 19.48±0.37 | 12.03±0.38 | 11.8±0.11 | 11.9±0.51 | 12.8±0.42 | 12.7±0.18 | 12.5±0.26 |
| Sodium (Na) | 10.8±0.22 | 9.4 ±0.41 | 10.6±0.12 | 11.4±0.19 | 10.8±0.22 | 11.2±0.32 | 7.8±0.81 | 6.9±0.12 | 7.4±0.21 |
| Phosphorus (P) | 30. 4±0.30 | 27.7±0.37 | 29.5±0.28 | 18.6±0.26 | 15.2±0.19 | 16.7±0.19 | 24.1±0.27 | 20.6±0.15 | 22.3±0.29 |
| Iron (Fe) | 32.6±0.20 | 30.4±0.19 | 32.1±0.24 | 10.0±0.65 | 8.7±0.74 | 9.3±0.43 | 14.9±0.74 | 11.8±0.84 | 13.5±0.63 |
| Zinc (Z) | 9.3±0.12 | 9.2±0.95 | 11.8±0.94 | 7.2±0.53 | 8.2±0.13 | 7.9±0.91 | 5.4±0.81 | 6.8±0.41 | 5.9 ±0.62 |

Data are expressed as mean ± standard deviation (triplicate).

3. Total phenolic content of fresh and dried citrus peels

Polyphenolic compounds (as phenolic acids and flavonoids) are important fruit phytochemicals compounds for their antioxidant activities, their chelation of redox-active metal ions, and inactivation of lipid free radical chains and prevention of hydro peroxide conversion into reactive oxy-radicals (Cabral de Oliveira *et al.*, 2009). Phenolic content can be used as an indicator of antioxidant capacity and as a preliminary screen for any product when planned to utilize as a natural source of antioxidants in functional foods (Viuda-Martos *et al.*, 2011).

Data in (Table 4) illustrated that total phenolic (TPC) amount varied greatly in fresh and dried citrus peels. Fresh orange peel contained highest level of TPC (5032.4 mg Gallic acid/ 100g sample dry weight) than mandarin (4355,8 mg Gallic acid/ 100g dry weight) and lemon (3550.6 mg Gallic acid/ 100g dry weight). TPC of fresh peels are higher than the recovery from dried samples because the water in fresh plant cells can help phenols extraction (Viuda-Martos *et al.*, 2011).

The results shows that the citrus species had highly amount of total phenolic compound which is agreement with Ma *et al.*, (2008) who studied the physical and chemical characteristics of citrus peel and traced high amount of total phenolic compounds. Total phenolic contents decreased in dried citrus peels compared to control samples. In general, TPC in citrus peels decreased more by sun drying than oven drying at 50 °C.

The reduction of phenolic compounds recovered from dried peels may be due to water evaporation and components in the cells (e.g., membranes and organelles) may hold together in the water absence and probably the extraction with solvent become more difficult. Moreover, if the citrus peel is dried before extraction, the recovery is much lower than using the fresh materials (Li *et al.*, 2006). The increase in drying temperature lead to a decrease in total polyphenols content after re-dissolution (Kamran *et al.*, 2009).

Therefore, these reasons may explain present study results (Table 4). Worthy to note, that extraction of polyphenols from plant material is affected by the solubility of the polyphenols in the extraction solvent. Furthermore, solvent polarity plays a key role in increasing the extract contents (Naczka and Shahidi, 2006). Also, Methanol and ethanol were better than the acetone at extracting phenolic compounds owing to their higher polarity and good solubility for phenolic components as indicated by Wieland *et al.* (2006).

4. Ascorbic acid content of fresh and dried citrus peels

Vitamin C (L-ascorbic acid or simply ascorbate) is a water soluble material. It is major in citrus and rich in the flesh and peel of fruits. It can efficiently scavenge diversity of reactive oxygen species (ROS), as a natural free radical scavenger, and give off semi dehydroascorbic acid, clearing O₂ and reducing sulfur radicals (Vega-Galvez *et al.*, 2009). Ascorbic acid contents of the investigated orange, mandarin and lemon peel samples were determined by the 2,6-dichloroindophenol titrimetric method. Their corresponding

concentrations of fresh peels were found 133.8, 151.5 and 161.9 mg/100g dry weight basis (Table 4). Lemon peel contained highest amount of ascorbic acid.

Drying of these citrus peels, either by sun or oven greatly reduced the ascorbic acid concentration to less than half content of their original values (control). These results agreed with Fernandez-Lopez *et al.*, (2004). This could be attributed to the fact that ascorbic acid is not stable at high temperature (Nagi and Roy, 2000). Similar results are reported by Vega-Galvez *et al.*, (2009) that observed 90.20% losses in ascorbic acid content of red pepper dried at 90 °C. Ascorbic acid is one of the substances that contribute to the antioxidant capacity in citrus juices and by products. It contributes to about 56% and about 77% of the antioxidant capacity of citrus juice and peels, respectively (Vinson *et al.*, 2002). Ascorbic acid degradation during drying depends mainly on temperature, time and metal ions traces (Ozkan *et al.*, 2004).

The recommended daily intake of vitamin C varies according to age, sex, risk group cigarette smokers, alcohol users, institutionalized elderly and subjects on certain drugs and criteria applied in individual countries. The recommended dietary allowances for vitamin C in the USA were recently revised upwards, to 90 mg/day for men and 75 mg/day for women based on pharmacokinetic data (EFSA, 2013).

For smokers, these recommended dietary allowances for vitamin C are increased by an additional 35 mg/day. Also for pregnant (85 mg/day) and lactating women (120 mg/day) higher amounts of vitamin C are recommended. The RDAs are in a similar range in other countries. Recent evidence sets the estimate for the needs to maintain optimal health in the region of 100 mg daily (Food and Nutrition Board, 2000).

5. Total Flavonoids content of fresh and dried citrus peels

Citrus peels are rich source of natural flavonoids. Also, phenolic and flavonoid compounds of citrus have high antioxidant activity. Flavonoids possess a broad spectrum of chemical and biological activities including radical scavenging properties. Such properties are especially evident for flavonols (Kamran, *et al.*, 2009, Hayat *et al.*, 2010 and El-Seedi *et al.*, 2012).

Total flavonoids content (TFC) of the investigated citrus peels is revealed in (Table 4). Generally, TFC of the tested peel samples, extracted with ethanol was in highest level in orange peel as 488.5 followed by lemon peel as 468.5 and in lowest level in mandarin peel as 411.5(mg QE/100g sample).

TFC content of citrus peels decreased after drying. These results were agreed with Bengtson *et al.* (2008) with the main reason that loss of flavonoids during heat treatment might be due to dry conditions precisely heat treatment.

TFC content of dried peels with oven was higher than sun dried samples. These results were in agreement with the results of Hegazy and Ibrahim (2012). Data in (Table 4) indicated that the orange peel dried by oven at 50 °C and extracted with ethanol had the highest content (395.9 mg quercetin equivalent / 100g db), followed by sun dried with values of 353.6 mg /100g db.

Considering flavonoids, orange and lemon peels gave the highest concentrations, which is agreed with reports available in literature (Levaj *et al.*, 2009).

The accumulation of the flavonoid compounds in the dried citrus powder could be attributed to the activation of enzymes, which are responsible for the synthesis of compounds, such as phenylalanine ammonia lyase (PAL) and chalcone synthase (CSH), by UV-C irradiation (Solovchenko *et al.*, 2008). Previous studies have shown that enzymes are still active in dried plant tissues.

Total flavonoids content in this study were in accordance with previously published data (Levaj *et al.*, 2005 and Levaj *et al.*, 2009). At the same time, total flavonoids content of mandarin peels investigated in this work are approximately three fold higher than it was determined by Levaj *et al.*, (2005) and it is higher than values of Abeysinghe *et al.*, (2007).

Table (4). Effect of drying methods on total phenolic content (mg Gallic acid/100g sample), ascorbic acid content (mg/100g db) and total flavonoids content (mg QE/100g sample) of dried orange, mandarin and lemon peels

| Peel Samples | Control (Fresh) | Drying methods/ Ascorbic acid | |
|-----------------|-----------------|-------------------------------|---------------|
| | | Sun drying | Oven drying |
| Orange | | | |
| Total phenolic | 5032.4 ± 0.42 | 2453.7 ± 0.97 | 3026.3 ± 0.62 |
| Ascorbic acid | 133.8 ± 0.68 | 63.8 ± 0.42 | 65.7 ± 0.44 |
| Flavonoids | 488.5 ± 0.11 | 353.6 ± 0.65 | 395.9 ± 0.11 |
| Mandarin | | | |
| Total phenolic | 4355.8 ± 0.26 | 3280.5 ± 0.66 | 3645.8 ± 0.71 |
| Ascorbic acid | 151.5 ± 0.66 | 70.6 ± 0.74 | 72.8 ± 0.63 |
| Flavonoids | 411.5 ± 0.69 | 301.8 ± 0.85 | 321.5 ± 0.65 |
| Lemon | | | |
| Total phenolic | 3550.6 ± 0.36 | 2504.40 ± 0.72 | 2632.8 ± 0.73 |
| Ascorbic acid | 161.9 ± 0.77 | 82.5 ± 0.31 | 84.8 ± 0.38 |
| Flavonoids | 468.5 ± 0.87 | 322.6 ± 0.75 | 361.5 ± 0.74 |

Data are expressed as mean ± standard deviation (triplicate).

6. Physical properties of produced cake:-

Data presented in (Table 5) show that, the addition of orange peel powder increased volume of cake under investigation; these results indicated the important of adding dietary fiber sources on the volume of cake. Also, addition of dietary fiber sources increased specific volume. The trend of increasing in the specific volume related with high amount of dietary fiber sources. These results are in agreement with that of Saeed, (2010).

7. Chemical composition of orange and mandarin peels powder substituted cake:-

The values (Table 6) show that protein and fat contents decreased with increasing orange peel powder concentration, this is due to replacing the refined wheat flour and vegetable fat which are major source of the protein and fat. On

the other side, for cake carbohydrate, total insoluble and soluble dietary fiber contents increased by increasing the level of orange peel powder and reached to 13.41, 7.17 and 6.24 percent at level 20 percent for orange and mandarin peels powder, respectively, as from the proximate composition of the orange peel powder it is clear that peel powder is a major source of the dietary fibers. The obtained results for the proximate composition and dietary fibers were similar to the results reported by Nassar *et al.*, (2008) and Bandyopadhyay *et al.*, (2014). Nassar *et al.*, (2008) revealed increase in fiber and ash content and decrease in protein with increase in mandarin peel percentage in biscuit. Youssef and Mousa, (2012) also reported increase in ash content and fiber content in biscuit with addition of different citrus peel powder. Romero-Lopez *et al.*, (2011) also reported increase in fiber and ash in muffin when prepared with citrus peel. Increasing of substitution levels of orange and mandarin caused increases in total phenolic compounds (TPC) so that cake with 20% orange and mandarin peels powder showed the highest one. These results are in agreement with Ojha and Thapa, (2017) who published that the levels of TPC increased in prepared biscuits with increasing mandarin peel powder.

8. Elemental composition of cakes:

The mean values of minerals composition of wheat cakes, and fortified wheat cakes with citrus peel powders are outlined (Table 7). The data revealed that 10% Navel orange Abo-Sora and mandarin peels powder supplemented wheat cakes had the highest Ca, Na and K contents, respectively. Meanwhile, the latter 10% Baladi lemon peel recorded in addition the highest P content among all studied biscuits on dry weight basis. Such data coincide with Cohen *et al.* (1984), Sulamyn *et al.* (1990), and Youssef (2007).

Table (5). Physical properties of prepared cake

| Substitute level (%) Orange + mandarin Peels (1:1 by weight) | Specific volume (cm ³ /g) | Volume (cm ³) | Weight (g) |
|--|--------------------------------------|---------------------------|------------|
| 0 | 2.378 | 1083.97 | 453.98 |
| 5 | 2.585 | 1178.52 | 455.89 |
| 10 | 2.765 | 1256.32 | 456.32 |
| 15 | 2.987 | 1378.85 | 458.99 |
| 20 | 3.214 | 1487.20 | 462.41 |

Table (6). Chemical composition of orange and mandarin peels powder substituted cake

| Sample (%) | Protein | Fat | Ash | Total Dietary fiber | Insoluble fiber | Soluble fiber | Total phenolic contents |
|-----------------|-------------|-------------|-------------|---------------------|-----------------|---------------|-------------------------|
| Control | 9.78 ± 0.40 | 9.79 ± 0.12 | 0.9 ± 0.04 | 3.14 ± 0.92 | 2.10 ± 0.46 | 1.04 ± 0.54 | 66.2 ± 0.82 |
| 5 (O+M) | 8.14 ± 0.33 | 8.45 ± 0.10 | 1.2 ± 0.06 | 8.21 ± 0.27 | 5.41 ± 0.16 | 2.8 ± 0.11 | 233.8 ± 0.13 |
| 10 (O+M) | 7.52 ± 0.30 | 8.10 ± 0.10 | 1.34 ± 0.07 | 10.01 ± 0.05 | 6.15 ± 0.19 | 3.86 ± 0.14 | 498.6 ± 0.68 |
| 15 (O+M) | 6.20 ± 0.25 | 7.38 ± 0.09 | 1.91 ± 0.10 | 11.85 ± 0.35 | 7.8 ± 0.24 | 4.05 ± 0.17 | 780.2 ± 0.42 |
| 20 (O+M) | 6.01 ± 0.24 | 8.20 ± 0.10 | 2.42 ± 0.13 | 13.41 ± 0.47 | 8.17 ± 0.25 | 5.24 ± 0.22 | 1022.8 ± 0.93 |

Data are expressed as mean ± standard deviation (triplicate). Orange peel (O) and Mandarin peel (M)

9. Sensory evaluation of prepared cake

Sensory evaluation of cake containing different levels of orange and mandarin (1:1 by weight) peels powder as compared to the control cake is shown in (Table 8). The data revealed that incorporation of orange and mandarin peels powder had marked improvement in colour, appearance and textural profile of prepared cake up to concentration of 10% while further increase in concentration showed reduction in appearance, colour, flavour and texture as well as taste characteristics. The overall acceptability of cake was determined by taking average of all the values pertaining to appearance, colour, flavour, texture and taste. It was found that sample containing 10 % of mixed peels powder found to secure maximum score (8.30) (significantly ($p < 0.05$)) followed by 5% (7.36) and control (7.16) while least overall acceptability was observed in sample containing 20% of mixed peels powder.

On the basis of overall acceptability of cake, it could be concluded that incorporation of orange and mandarin peels powder in preparing cake up to the level of 10 per cent was superior to all other treatments and control sample and hence, 10% mixed peels powder incorporation in preparation of cake could considered optimum with respect to sensorial quality characteristics. The results were in conformity with Ojha and Thapa, (2017) who published that the biscuit made from 6 % mandarin peel powder incorporation was comparable to control in-terms of overall acceptability.

Cake prepared with 10 % orange and mandarin peels powder was comparable to control biscuit made from wheat flour based on sensory evaluation with increased bioactive component. The result shows that there is an ample opportunity for use of mandarin peel in various other food products also.

Table (7). Elemental composition of wheat cakes and fortified wheat cakes with citrus peels powders

| Type of cakes | Elemental content (mg/100g) | | | | | |
|-----------------|-----------------------------|-------------|------------|-------------|------------|------------|
| | Fe | Ca | Mg | Na | K | P |
| Control | 30.33a±0.32 | 0.02a±0.058 | 0.02a±0.03 | 0.24a±0.17 | 0.11a±0.40 | 0.18±0.18 |
| 5%(O+M) | 10.33 _b ±0.02 | 3.52b±0.028 | 4.02b±0.17 | 2.24b±0.02 | 6.11b±0.05 | 0.68a±0.14 |
| 10%(O+M) | 18.48b±0.07 | 5.08b±0.004 | 7.01c±0.19 | 3.24bc±0.05 | 9.14b±0.30 | 1.04b±0.26 |
| 15%(O+M) | 19.23b±0.03 | 6.05b±0.008 | 8.02c±0.24 | 4.26cd±0.08 | 8.17b±0.20 | 1.17b±0.22 |
| 20%(O+M) | 21.43a±0.12 | 7.04 b±0.04 | 7.02c±0.40 | 4.41cd±0.12 | 7.23b±0.05 | 1.22b±0.05 |

Data are expressed as mean ± standard deviation (triplicate).

Orange peel (O) and Mandarin peel (M)

Table (8). Sensory evaluation of cake

| Sample Code | Sensory attributes | | | | | |
|-----------------|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|-------------------------|
| | Colour | Appearance | Texture | Taste | Flavor | Overall acceptability |
| Control | 7.20 ^a ±0.23 | 7.17 ^a ±0.18 | 7.19 ^a ±0.31 | 7.13 ^a ±0.32 | 7.12 ^a ±0.05 | 7.16 ^a ±0.14 |
| 5%(O+M) | 7.37 ^a ±0.27 | 7.28 ^a ±0.22 | 7.25 ^a ±0.35 | 7.48 ^a ±0.20 | 7.42 ^a ±0.22 | 7.36 ^a ±0.40 |
| 10%(O+M) | 8.2 ^b ±0.33 | 8.53 ^b ±0.26 | 8.31 ^b ±0.31 | 8.25 ^b ±0.26 | 8.23 ^b ±0.33 | 8.30 ^b ±0.20 |
| 15%(O+M) | 6.39 ^c ±0.53 | 6.98 ^c ±0.43 | 6.12 ^c ±0.26 | 6.52 ^c ±0.05 | 6.86 ^c ±0.20 | 6.57 ^c ±0.30 |
| 20%(O+M) | 4.97 ^d ±0.33 | 4.71 ^d ±0.03 | 4.99 ^d ±0.22 | 5.82 ^d ±0.14 | 5.08 ^d ±0.18 | 5.11 ^d ±0.26 |

Each value represents the average of ten determinations. Orange peel (O) and Mandarin peel (M)
Means with different letters in the same row are significantly different by Duncan's Multiple Range Test at (P < 0.05
± (SD) standard deviation.

CONCLUSION

Citrus peels as Orange, mandarin and lemon peels are the most important aspect of this study. Citrus peels are good source of dietary fiber, elemental and bioactive compounds. The level of components content varies and depends on the type of citrus peels and drying methods. The results showed that the oven drying method at 50 °C recorded highest levels of crude protein, total dietary fiber, minerals (K, Ca, Na, Mg, P, Zn and Fe) and bioactive compounds (total phenolic contents, ascorbic acid and flavonoids) than sun-drying in citrus peel samples. The results indicated that cakes prepared with 10% of orange and mandarin peels powders (1:1 by weight) contained high total dietary fiber and bioactive compounds. It can be concluded that incorporation of orange and mandarin peels up to the level of 10 per cent in formulating cake preparations enhanced the nutritional value particularly with respect to dietary fiber, physicochemical quality and overall acceptability of cakes. Moreover, citrus peels could be recommended as useful value added functional ingredients for food industry.

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الملخص العربي

المركبات الحيوية في قشور بعض الموالح وتأثيرها بطرق التجفيف وتقدير جودة الكيك المدعم بمسحوق القشور

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تتركز الدراسة في هذا البحث على قشور بعض الموالح مثل البرتقال واليوسفي والليمون. تعتبر قشور الموالح مصدراً غنياً بالألياف والمعادن والعديد من المركبات الحيوية. ويتوقف تركيز هذه المركبات على قشور نوع الموالح وأيضاً على طرق التجفيف المختلفة لها. ويرجع إنخفاض تركيزات تلك المركبات خلال التجفيف إلى مدى حساسية تلك المركبات إلى درجة الحرارة والفترة الزمنية. أوضحت هذه الدراسة أن التجفيف بالفرن على درجة ٥٠ م° قد سجل أعلى محتويات لكل من البروتين الخام والألياف والعناصر (البوتاسيوم - الكالسيوم - الصوديوم - المنجنيز - الفوسفور - الزنك - الحديد) والمركبات الفعالة المختلفة (المركبات الفينولية - حمض الإسكروبيك - الفلافونات) وكان له تأثير أقل على محتويات تلك المركبات مقارنة بالتجفيف الشمسي. ومن ناحية أخرى فإن تلك الدراسة هدفت إلى تأثير إضافة وتدعيم كيك دقيق القمح بمسحوق القشور الجافة لكل من البرتقال واليوسفي (١:١ بالوزن) بنسب ٥% ، ١٠% ، ١٥% ، ٢٠%. وقد تم إجراء التحاليل الطبيعية والكيميائية والحسية على الكيك المحضر. وأوضحت النتائج الكيميائية أن الكيك المحضر بنسبة ١٠% مسحوق البرتقال واليوسفي كان محتويًا على نسب مرتفعة من الألياف والمركبات الفعالة. ويمكن إستنتاج أن خلط أو إضافة مسحوق البرتقال واليوسفي في حدود ١٠% بمكونات الكيك يؤدي إلى إرتفاع القيمة الغذائية وأيضاً الخواص الحسية للكيك المحضر بالمقارنة بالكنترول والعينات الأخرى.

وأيضاً يمكن التوصية بأن إضافة قشور الموالح ذات القيمة الغذائية المرتفعة من الألياف والمركبات الفعالة حيويًا يمكن إضافتها إلى الأغذية الوظيفية في الصناعات الغذائية لإكتساب فوائد تغذوية وصحية.