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Some Physicochemical and Functional Properties of Lemon and Orange Peels

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A B S T R A C T

This study aimed to evaluate some physicochemical and functional properties as well analyze phenolic acids profiles & functional groups of raw and microwave or air-oven dried orange and lemon peels (OP&LP) for potential use as functional ingredients sources for food enrichment. Fresh LP had high %s of moisture, protein, ether extract, fiber and ash than OP. After drying, LP had less ether extract% compared to OP. Ash and fiber contents of LP had more %s compared to OP dried by microwave or air oven. Total dietary fiber content in fresh OP was of less % than LP. Dried LPs using both drying methods were of high total dietary fiber % than that in OPs. Fresh LPs contain more insoluble dietary fiber than OPs. Microwave dried LP and OP had more insoluble dietary fiber than air-oven dried ones. Furthermore fresh and dried OP samples contain more soluble dietary fiber than the LPs. Total flavonoids content of methanolic OP & LP extracts was higher than those of ethanolic extracts. HPLC analysis showed that naringin and hesperidin were the predominant phenolic acids in the tested samples with different concentrations. FTIR spectroscopy analysis was also recorded (400-4000 wave number cm^{-1}). OPs dried by air oven had highest water and oil holding capacities.

Introduction

Recently, untraditional sources of valuable nutrients from food waste by-products have been explored for their utilization in food stuffs. Some previous researches described the application of food waste by-products in food, pharmaceutical development and biotechnology processes.

Citrus by-products are promising economic sources of bioactive ingredients and of valuable technological & nutritional properties; can be utilized as food additives (Marín *et al.*, 2002; Puupponen-Pimia *et al.*, 2002; O'Shea *et al.*, 2012). Citrus waste contains large amount of flavonoids, carotenoids, dietary fiber, polyphenols, ascorbic acid, sugar etc. (Sharma *et al.*, 2017).

OP and LP are the primary citrus byproducts waste generated during processing. They contain dietary fiber and bioactive compounds which are important in food quality evaluation. Total polyphenols and carotenoids in lemons, oranges and grape fruits peels are significantly higher than in peeled fruits (Ramful *et al.*, 2011).

Dietary fiber (DF) as a major constituent of plant foods and has been accepted as an important nutrient in human diet and is considered as a main nutrients in healthy diets due to its ability to reduce cholesterol, diabetes and coronary heart disease and ease constipation (Telrandhe *et al.,* 2012). In addition, DF can impart other uses such functional benefits as gelling, thickening and water binding. The functional properties of DF include the bulk volume, the hydration, hydro-colloidal and rheological properties, which contribute to application in food formula design and food manufacturing (Bodner and Sieg, 2009; Gómez-Ordóñez *et al.,* 2010).

DF mainly consists of soluble (SDF) and insoluble (IDF) fiber fractions. Beneficial effects of SDF include lowering blood lipid and glucose levels (Benavente-Garcia and Castillo, 2008), reducing risks from cardiovascular and colorectal cancer diseases (González-Molina -Molina *et al.,* 2010; Adibelli *et al.,* 2009) and enhanced gastrointestinal immunity (Anderson *et al.,* 2009; Gunness and Gidley, 2010. Meanwhile, cellulose, hemicelluloses and lignin were the main components of the IDF which prevents or relieves the constipation due to the absorption of water from the digestive tract.

Several studies indicated that SDF was more important than IDF in many health aspects (Galisteo *et al.,* 2008; Kethireddipalli *et al.,* 2002). DF of citrus fruit peels contains higher proportions of SDF which exhibit several functional properties, such as glucose retardation index, and WHC & OHC. These properties are more useful for understanding the chemical composition and physiological effects of DF (Jing and Chi, 2013; Kendall, *et al.,* 2010). DF was not only desirable for their nutritional value but also potential use in food formulation with its functional and physicochemical properties (Fabek *et al.,* 2014).

Usually dietary fiber and antioxidants are addressed separately as groups of food constituents. However, little known fact that a considerable proportion of the antioxidant, polyphenols and carotenoids contained in fruit and vegetables are linked to dietary fiber (Saura-Calixto *et al.,* 2007), and some of the postulated benefits of the fiber intake can be attributed to these associated compounds (Martinez *et al.,* 2011). Worthy to note that DF of fruit and vegetables transport a significant amount of polyphenols and carotenoids linked to the fiber matrix through the human gut (Saura-Calixto *et al.,* 2007 and Saura-Calixto and Goni, 2006). It was demon-strated by Chowdhury *et al.,* (2013) that OP (*Citrus sinensis*) contains high amount of vitamin C, phytochemicals, antioxidants, SDF and IDF have been found to be helpful in reduction of the risk for cancers, many chronic diseases e.g. arthritis, obesity and coronary heart diseases. Goñi and Hervert-Hernández, (2011) reported that when several fruit and vegetable by-products are used as high-quality ingredients in functional foods or dietary supplements they can be considered as an excellent source of DF and natural antioxidants. Therefore, the importance of food fibers has led to the development of a large and potential market for fiber-rich products and there is a trend to find new sources of DF that can be used in the food industry (Chau and Huang, 2003). The development of food products with increased dietary benefits from citrus peels have placed not only on the recovery of carbohydrates and pectin (Baker, 1994) but also on the production of potentially important secondary metabolites, such as polyphenols (Manthey and Grohmann, 1996). Consequently, most of the bioactive compounds which extracted from citrus by-products could be used as functional ingredients especially non-digestible carbohydrates (dietary fiber) and bioactive compounds (ascorbic acid and flavonoids) when designing healthy foods

(Marín *et al.,* 2002). Also, the bioactive compounds from citrus by-products such as peel can be evaluated as alternative to synthetic food additives which are associated with negative effects on human health (Ignat *et al.,* 2011). So, the present study intended to evaluate some physicochemical and functional properties of OP and LP focused on their contents of TDF, SDF and IDF, TFC, WHC and OHC, as well to analyze phenolic acids profiles using HPLC in order to use them as functional ingredients for food industry. FTIR spectrum was also recorded for identification of functional group.

Materials and Methods

Materials

Citrus lemon (*Citrus aurantiifolia*) and orange (*Citrus sinensis*) fruits were purchased from an Egyptian local market.

Chemicals

Chemicals, solvents, standards and reagents were purchased from Sigma Chemical Co. (St. Louis, Mo, USA). All other chemicals used were of analytical grade.

Methods

Preparation of Lemon and Orange Peel samples

Lemon and orange fruits were washed by running tap water. The citrus were carefully separated into edible and inedible portions (peel). The obtained fresh citrus peels were cut into small pieces before the drying processes.

Drying Methods

Each of fresh citrus peel pieces was divided separately into two parts and each part was dried using the following two methods:

Air Oven-Drying

The fresh citrus peels pieces were dried in an air oven (Shellab-Model 1350FX.-Made in USA) at 40 ± 2°C for ~ 48 h.

Microwave–Drying

A programmable domestic microwave oven (type Samsung, 77 QH 400148, MF 2015), with a maximum output of 1500W at 2450 MHz) was used for drying the fresh lemon or orange peel pieces samples for 6 min. The dried peel samples were ground to fine powders using a mechanical laboratory grinder and passed through a 24-mesh sieve, then packaged in polyethylene bags and stored at 4±1°C until required for use.

Ethanol and Methanol extraction

Dried powder peels (10g) and (4g) of each lemon or orange sample was extracted with 100 ml of ethanol (70%) and 80 ml of methanol (80%) respectively at room temperature and several agitations with sonication using the ultrasonic device (200 W, 59 kHz, Shanghai Kudos) for 60 min. Both extracts were centrifuged (5000 rpm for 30 min at room temperature). Then the extracts were filtered using filter paper What- man (No.4) according to Jo *et al.,* (2003) and Xu, G. *et al.,* (2008) with some modification.

Analytical Methods

Proximate chemical composition

Moisture, ash, protein, ether extract and crude fiber contents were determined in accordance to the AOAC (2005) methods.

Determination of Dietary fiber

To determine total dietary fiber (TDF), soluble dietary fiber (SDF) and insoluble dietary fiber

(IDF) contents in orange and lemon peel samples, the extraction was carried out following an enzymatic-gravimetric procedure (AOAC 985.29, 2001) with minor modifications. Briefly, peel sample was thoroughly dispersed in 4 times volume of de-ionized water, and the pH of peel dispersion was adjusted to 6.0 with 0.1mol/L NaOH; then % (w/w) heat-stable α-amylase at 95 °C was added, and hydrolyzed with constant stirring at 120 rpm for 30min. After the temperature of the hydrolysate was cooled down to 60°C, neutral protease 0.016% (w/w) was added and further hydrolyzed for 30 min with constant stirring at 120 rpm. At the end, the enzymatic hydrolysis reaction was quenched at 95 °C for 5 min and the hydrolysate was centrifuged at 3800 ×g for 20 min after cooled down to room temperature, the supernatant and sediment were collected. The supernatant was condensed to one-tenth with a vacuum rotary evaporator. Afterward, the concentrated supernatant was mixed with 95% (v/v) ethanol at 4 °C for 12 h and then subjected to centrifugation at 3800 ×g for 15min. The precipitate was dried at 60 °C for 48 hr. The dried flocculate was SDF, which was milled and passed through a 60-mesh sieve and stored at 4 °C. The sediment (IDF) was washed for three times with 70 °C water, dried at 60 °C for 48 hr and milled into powder and passed through a 60-mesh sieve and stored at 4 °C. TDF was the sum of IDF and SDF. With each assay, blanks were run along with samples to measure any contribution from reagents to residue.

Calculations

$$\text{Dietary Fibre (\%)} = \frac{\frac{R_1 + R_2}{2} - p - A - B}{\frac{m_1 + m_2}{2}} \times 100$$

Where:

R_1 = residue weight 1 from m1

R_2 = residue weight 2 from m2;

m_1 = sample weight 1

m_2 = sample weight 2;

A = ash weight from R_1

p = protein weight from R_2; and

B = blank

Blank = $[(BR_1 + BR_2) / 2 - BP - B]$

Determination of total flavonoids content

Colorimetric aluminum chloride method was used for flavonoids determination described by Ebrahimzadeh *et al.,* (2008) with some modifications. Lemon or orange peels powder extracts 0.5 ml solution was separately mixed with 1.5 ml methanol, 0.1 ml of 10% aluminum chloride, 0.1 ml of 1 M potassium acetate and 2.8 ml distilled water then left at room temperature for 30 min. The absorbance of the reaction mixture was measured at 415 nm with spectrophotometer (T80 UV/Visible - PG instrument Ltd - Made in Germany). Total flavonoid contents were calculated as quercetin from a calibration curve, which prepared by preparing quercetin solutions at concentrations 12.5 to 100 mg ml^{-1} in methanol and was calculated by using the following equation:

y = 0.0059

Where:

y = Dependant factor

x = Independant factor (absorbance of sample).

Phenolic acids profiles

HPLC analysis was carried out according to Kim *et al.,* (2006) with slight modifications using an Agilent Technologies 1100 series liquid chromatograph equipped with an auto sampler and a diode-array detector. The analytical column was Agilent Eclipse XDB

C18 (150 x4.6 µm; 5 µm) with a C18 guard column. The mobile phase consisted of acetonitrile (solvent A) and 2% acetic acid in water (v/v) (solvent B). The flow rate was kept at 0.8 mL min^{-1} for a total run time of 70 min and the gradient program was as follows: 100% B to 85% B in 30 min, 85% B to 50% B in 20 min, 50% B to 0% B in 5 min and 0% B to 100% B in 5 min. There was 10 min of post-run for reconditioning. The injection volume was 10 µL and peaks were monitored simultaneously at 280 and 320 nm for the benzoic acid and cinnamic acid derivatives, respectively. All samples were filtered through a 0.45 µm Acrodisc syringe filter (Gelman Laboratory, MI) before injection. Peaks were identified by congruent retention times and UV spectrum and compared with those of the standards.

Functional group analysis--Fourier Transform Infrared Spectroscopy (FTIR)

Methanol extracts of lemon and orange peel were prepared according Xu, G. *et al.,* (2008). For extra-purification 2 ml of both methanolic lemon and orange peel extracts were centrifuged (5000 rpm for 30 min at room temperature) and the extracts were filtered using Whatman No.4 filter paper. The filtrate was then evaporated till dryness under reduced pressure. After that, the obtained powders were collected and each pure lemon or orange peel sample pressed in KBr-disc (spec pure).The Infrared spectra (KBr-disc) were recorded using a Jasco FT/IR-300E spectrometer in range 400-4000 cm^{-1}

Water and oil holding capacities

Water and oil holding capacities (WHC and OHC) of lemon and orange peels powder were determined as described by Chau and Huang, (2003). One gram of powdered sample was weighed, added into 10 mL of distilled water or 10 mL of sun- flower oil and stirred for 1 min. The suspensions were then centrifuged at 2200 ×g for 30 min, and the supernatant volume was measured. WHC or OHC was expressed as gram of water or oil held per gram of sample.

Results and Discussion

Proximate Chemical composition

Chemical composition of fresh OP & LP and their dried samples either by air oven (hot air) or microwave drying methods are shown in Table (1). Fresh LP contained more moisture content (81.23%) than OP sample (74.35%). Nesrine *et al.,* (2012) reported that the fresh citrus peel of Thompson navel, mandarin and lemon are characterized by high moisture.

After drying, using the two mentioned methods, the air dried OP & LP samples still having more moisture than those of microwave dried samples. As regard to microwave dried, LP showed significant higher moisture content when compared to the dried OP by about 10.24%. These findings agreed with Adewole *et al.,* (2014). Also, it is revealed that fresh LP sample contained 11.53% crude protein whereas OP has less crude protein by about 38.51%. The ether extract of dried LP sample showed high amount by ~ 15.90% when compared to OP. Total fiber contents of fresh LP were greater than those of fresh OP, i.e. 16.15 vs 11.48% respectively. After drying, LP sample has more crude protein, fiber and ash contents. With respect to ether extract; OP sample was significantly of more amounts, reached to 2.44, 2.12% compared to 1.42 and 1.35% in LP sample dried by microwave and air oven drying methods, respectively (Table 1). Regarding to ash content, LP had significantly more amounts i.e. reached to 5.92, 5.71% compared to 3.33 and 3.51% in OP sample dried by microwave and air oven methods respectively. These results agreed with Marín

et al., (2007) who found that the OP and LP samples contained ether extract 1.58 and 1.51% on dry weight, as well contain protein 8.82 and 7.00% on dry weight respectively.

Total flavonoids content (TFC)

TFC of the two studied peel samples extracted with methanol was higher than ethanolic extract. TFC of the OP sample extracted with methanol was 506.82 ±0.97 for fresh sample (control). Meanwhile, the microwave and air oven dried OP samples reduced to 309 ± 0.32 and 365.40 ± 0.16 QE / 100g db respectively (Table 2). TFC of dried peels with air oven was higher than microwave-drying samples. The same trend was also noticed in case of ethanolic extract. These findings varied with those of (Hegazy and Ibrahium, 2012) i.e. methanol extracts of orange peel samples either fresh or dried contained more TFC than ethanol extracts. Additionally data in Table (2) indicated that the TFC of LP dried by air oven and extracted with methanol had the highest TFC (469.08 ±0.42 mg quercetin equivalent / 100g DW), followed by microwave drying and control samples with values 442.79 ±0.42 and 430.58 ±0.77mg /100g DW respectively. It was also noticed that TFC content of methanolic extract of LP was higher than ethanolic extract. Kamran *et al.*, (2009); Hayat *et al.*,(2010) and El-Seedi *et al.*, (2012) demonstrated that citrus peels contain a high concentration of phenolic compounds and represent a 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 distinct for flavonols.

Phenolic acids profiles

The standard phenolic acids (e.g. gallic, catachine, rutin, naringeen, hisperdin etc.) in the investigated citrus peels and their corresponding concentrations of each individual phenolic are shown in Table (3) and Fig. (1).

Phenolic acids profile of OP and LP was nearly similar. Among the tested phenolic acids, only pyrogallol, protochatchuic, p-hydroxybenzoic, gentisic and chyrsin were not detected in all samples. Meanwhile, chlorgenic, caffic, syrngic, vanillic, ferulic, rosmarinic and apegnin were not detected in microwave dried OP extracted by ethanol. Also, apegnin was only not detected in microwave dried LP extracted by ethanol under the experimental conditions.

Naringeen and hisperdin were the predominant phenolic acids in all tested samples, with different concentrations. Ethanolic and methanolic extracts of dried microwave OP were (26433.7, 50968.5 and 14751.5, 39969.0 µg/100g sample) respectively. Meanwhile, dried air oven OP were (21127.3, 48405.5 and 12885.6, 30914.2 µg/100g sample) respectively.

Regarding to dried microwave LP naringeen and hisperdin had 7588.1, 16894.0 and 5298.9, 10679.5 µg/100g sample respectively. Dried air oven lemon peels gave 7484.5, 14747.3 and 3140.2, 10767.9 µg/100g sample respectively. All samples were compared to control. Flavanones, hesperidin and narirutin concentrations were calculated by reference to an external standard curve constructed using various concentrations of each standard compounds.

These results are supported by Jiang *et al.*, (2014) who reported that the HPLC analysis showed that the contents of three flavonoid components, narirutin, naringin and neoheperidin, displayed a similar trend as that of total flavonoids in citrus peels. Hesperidin and narirutin are flavanone glycosides

consisting of the aglycones hesperitin and naringenin, respectively, bound to the disaccharide rutinose (ramnosyl-alpha-1,6 glucose (O'Neil *et al.,* (2001) and Tripoli *et al.,* (2007). Wang *et al.,* (2008) found that the flavanone composition of eight citrus peels is present at high levels to naringin, hesperidin and neohesperidin levels, respectively. Moreover, naringin and hesperidin was abundant in C. sinensis peels.

Lemon peel contained a moderate level of hesperidin. Naringin, followed by hesperidin is the main flavonoid glycoside found in orange peel (Wang and Weller, 2006). The naringin, hesperidin and neohesperidin contents were much higher in the peels than in the fruits *(*Wang *et al.*, 2007).

Dietary fiber content

Data from Table (4) show that the total dietary fiber (TDF) content in fresh OP & LP were 65.11 and 65.54% respectively. TDF contents of dried LP samples by the microwave and air oven were higher than that in OP samples. Fresh LP contains more insoluble dietary fiber (IDF) than OP (51.95 compared to 50.82% orange).

Microwave dried LP and OP have more IDF than of air oven dried ones. Regarding to soluble dietary fibers (SDF) content the opposite pattern was cleared i.e. fresh and dried orange peel samples contain more SDF than lemon (14.29 compared 13.59% for orange).

Table.1 Proximate chemical composition of citrus peels (orange & lemon) as affected by air-oven and microwave drying (db)

Component %	Orange peel samples			Lemon peel samples		
	Control (Fresh)	Microwave Drying	Air oven Drying	Control (Fresh)	MicrowaveDrying	Airoven Drying
*Moisture	74.35±0.02	8.51±0.01^{c}	9.96±0.05^{b}	81.23±0.01^{a}	9.48±0.002^{b}	9.58±0.05^{b}
Protein	7.09±0.01^{a}	6.44±0.02^{b}	6.39±0.02^{c}	11.53±0.02^{a}	7.19 ±0.06^{b}	7.06 ±0.05^{b}
Ether-extract	2.75 ±0.01^{a}	2.44 ±0.05^{b}	2.12 ±0.01^{c}	3.27±0.04^{a}	1.42±0.02^{b}	1.35 ±0.005^{c}
Fiber	11.48±0.0^{a}	10.40±0.01^{b}	10.46±0.01^{b}	16.15±0.02^{a}	12.35±0.01^{b}	12.47±0.01^{b}
Ash	4.21±0.08^{a}	3.33 ±0.10^{c}	3.51 ±0.09^{b}	6.58 ±0.01^{a}	5.92 ±0.03^{b}	5.71 ±0.01^{c}

(db) = dry weight basis. *= wet weight basis. Results are presented as means for triplicate analyses ± standard deviation (SD). Means within row with different letters are significantly different ($P \leq 0.05$).

Table.2 Total flavonoids content (mg QE/100g sample) of dried citrus peel extracted by methanol or ethanol

Peel samples	Extract solvents	Control (Fresh)	Microwave-Drying	Air oven-Drying
Orange	Methanol	506.82±0.97^{aA}	309.69±0.32^{cB}	365.40±0.16^{bC}
	Ethanol	376.87 ±1.96^{aC}	241.20 ±0.25^{cC}	273.82 ±0.13^{bD}
Lemon	Methanol	430.58±0.77^{cB}	442.79±0.42^{bA}	469.08±0.42^{aA}
	Ethanol	316.05±0.62^{bD}	317.41±1.22^{bB}	390.75 ±0.68^{aB}

db= dry weight basis. Results are presented as means for triplicate analyses ± standard deviation (SD). Means within row and column with different letters are significantly different ($P \leq 0.05$).

Table.3

Table 3 Phenolic acids profile of orange and lemon peel extracts (µg/100g sample)

Phenolic	1	2	3	4	5	6	7	8	9	10
Gallic acid	950.2	3325.9	1808.9	1056.6	471.8	1510.0	145.2	434.1	ND	ND
Catachine	3301.8	3827.2	39293.9	34098.7	ND	1446.4	17916.4	10065.2	1002.8	2530.1
Chlorgenic acid	ND	ND	572.4	588.5	ND	ND	240.7	261.1	ND	ND
Caffic acid	ND	1332.9	1244.9	1283.5	403.3	597.1	ND	500.9	410.6	505.7
Syrngic acid	ND	ND	1158.5	1150.6	315.1	82.1	445.5	601.7	ND	ND
Vanillic acid	ND	ND	391.0	457.1	ND	309.0	ND	141.5	703.5	ND
Ferulic acid	ND	720.4	1787.8	1621.5	114.3	243.2	837.4	ND	156.5	ND
Sinapic acid	582.0	ND	508.5	523.7	199.9	458.2	240.3	ND	ND	ND
Rutin acid	3401.4	2982.5	4524.0	3717.0	1526.6	1997.7	2212.8	ND	1313.0	464.7
***p*-coumaric acid**	1043.2	ND	1983.6	1009.9	361.8	662.0	401.4	378.5	439.3	83.5
Naringeen	**26433.7**	**21127.3**	**7484.5**	**7588.1**	**12885.6**	**14751.5**	**5298.9**	**3140.2**	**9474.6**	1297.6
Hisperdin	**50968.5**	**48405.5**	**14747.3**	**16894.0**	**30914.2**	**39969.0**	**10679.5**	**10767.9**	**28376.0**	**7101.4**
Rosmarinic acid	ND	ND	ND	9898.0	ND	ND	2261.9	4911.0	ND	535.4
Cinnamic acid	1601.0	899.7	2734.1	2517.4	514.1	1297.4	560.7	464.0	920.5	442.4
Qurcetin	1630.8	1607.9	1989.7	1687.7	797.4	642.3	943.7	2297.9	2883.9	1334.2
Kaempferol	808.0	1390.4	ND	ND	1029.3	ND	ND	ND	395.6	ND

ND = not detected; 1=Ethanolic extract of microwave dried orange peel; 2= Ethanolic extract of air oven dried orange peel; 3= Ethanolic extract of air oven dried lemon peel; 4= Ethanolic extract of microwave dried lemon peel; 5= Methanolic extract of air oven dried orange peel; 6= Methanolic extract of microwave dried orange peel; 7= Methanolic extract of microwave dried lemon peel; 8= Methanolic extract of air oven dried lemon peel; 9= Extract dried microwave orange peel; 10= Extract dried microwave orange peel.

Table.4 Effect of the two different drying methods on dietary fiber of orange and lemon peels

Citrus fruits peels	TDF %	IDF %	SDF %
Fresh orange peels	65.11	50.82	14.29
Dried orange peels by air oven	64.21	50.14	14.07
Dried orange peels by microwave	65.06	50.81	14.25
Fresh lemon peels	65.54	51.95	13.59
Dried lemon peels by air oven	64.9	51.54	13.36
Dried lemon peels by microwave	65.41	51.84	13.57

TDF=Total dietary fiber, IDF= insoluble dietary fiber, SDF= soluble dietary fiber

Table.5 FTIR spectra of the studied samples of orange and lemon peels

	Absorptions peaks cm^{-1}
1M	3366.14, 2929.34, 1633.41, 1413.57, 1263.15, 1058.73, 924.7-774.22, 593.00
2M	3366.14, 2931.27, 1632.45, 1412.60, 1060.66, 592.04
3M	3387.35, 2931.27, 1725.98, 1617.02, 1407.78, 1227.47, 1068.37, 818.63-778.14, 603.61
4M	3385.42, 2933.2, 1967.04, 1725. 98, 1617.02, 1406.82, 1226.50, 1069.33, 819.598-779.10, 603.61
5M	3387.35, 2930.31, 1724.05, 1617.02, 1407.78, 1228.43, 1066.44, 604.57
6M	3366.14, 2928.38, 1729.83, 1636.30, 1415.49, 1262.18, 1060.66, 921.81-775.24, 628.88

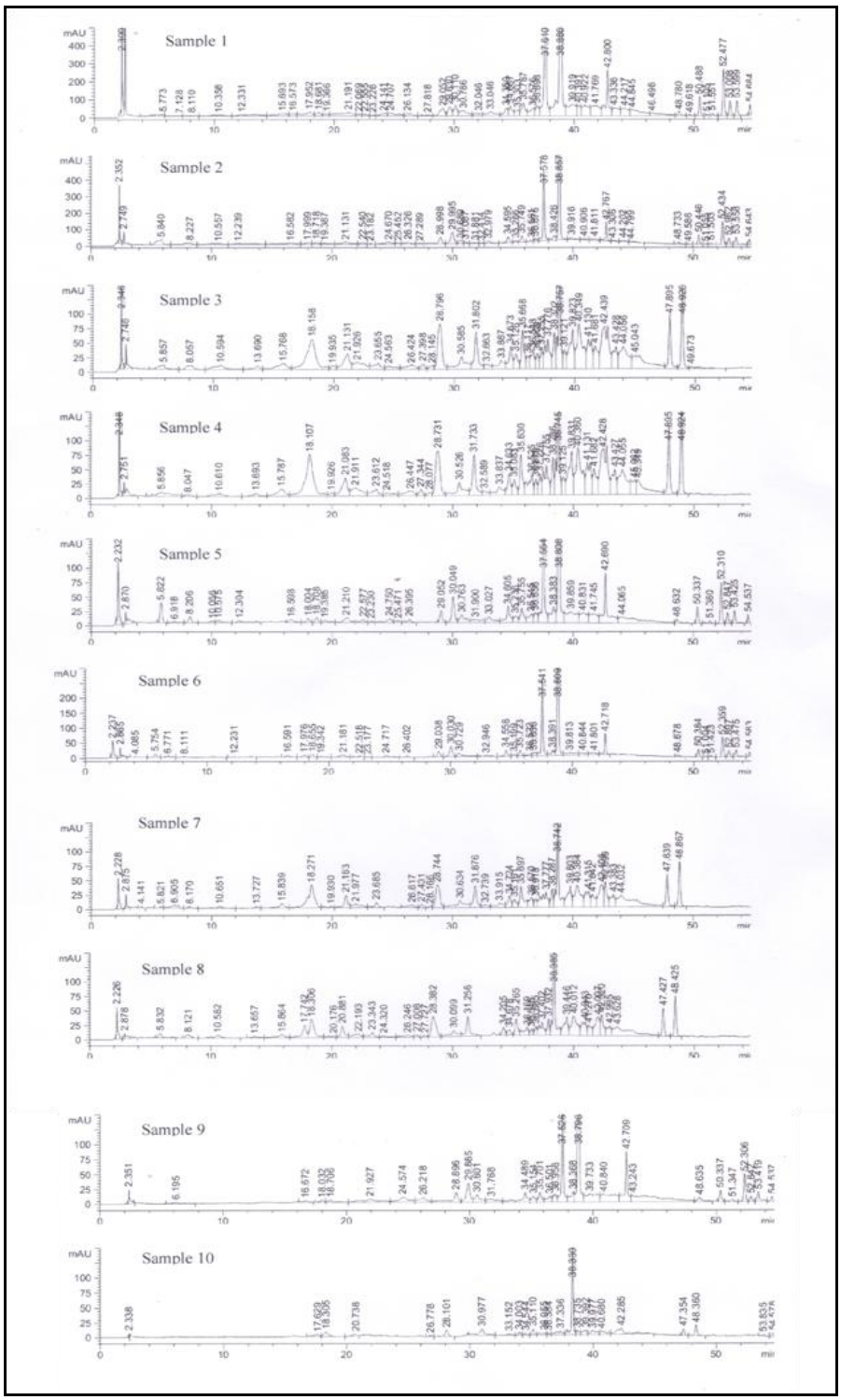
1M=Orange oven drying; 2M=Orange microwave drying; 3M=Lemon Oven Drying; 4M=Lemon microwave drying; 5M=Lemon fresh sample; 6M=Orange fresh sample

Table.6 Water and oil holding capacities (as g of water or oil held/g sample) changes of orange and lemon peels as affected by air oven and microwave drying methods

Peel samples	Parameter	Control (fresh)	Microwave-Drying	Air- oven Drying
Orange	WHC	6.17 ± 0.02^{a}	5.22 ± 0.02^{c}	5.59 ± 0.03^{b}
	OHC	5.28 ± 0.06^{a}	1.32 ± 0.01^{c}	$1.71^{b} \pm 0.01$
Lemon	WHC	5.52 ± 0.03^{a}	4.48 ± 0.04^{c}	5.03 ± 0.03^{b}
	OHC	7.17 ± 0.01^{a}	1.16 ± 0.04^{b}	1.16 ± 0.05^{b}

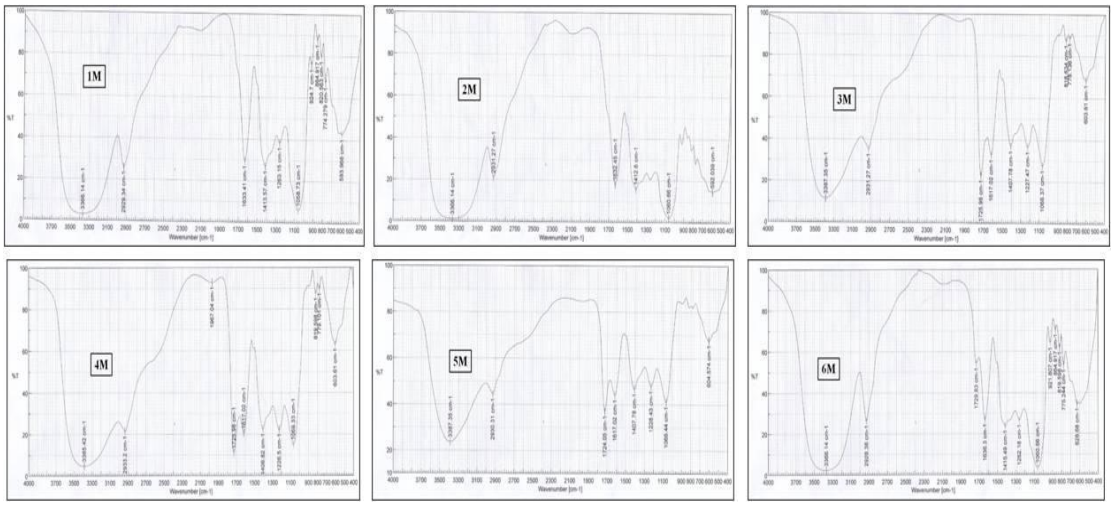
WHC= Water holding capacity; OHC= oil holding capacity. Results are presented as means for triplicate analyses ± standard deviation (SD). Means within row with different letters are significantly different ($P \leq 0.05$).

Fig.1 HPLC profiles of Orange and Lemon citrus Peel Samples (1-10)



Samples: 1=Ethanolic extract of microwave dried orange peel; 2= Ethanolic extract of air oven dried orange peel; 3= Ethanolic extract of air oven dried lemon peel; 4= Ethanolic extract of microwave dried lemon peel; 5= Methanolic extract of air oven dried orange peel; 6= Methanolic extract of microwave dried orange peel; 7= Methanolic extract of microwave dried lemon peel; 8= Methanolic extract of air oven dried lemon peel; 9= Extract dried microwave orange peel; 10= Extract dried microwave orange peel

Fig.2 FTIR spectrum of Orange and Lemon citrus Peel Samples (1-6)



1M=Orange oven drying; 2M=Orange microwave drying; 3M=Lemon Oven Drying; 4M=Lemon microwave drying; 5M=Lemon fresh sample; 6M=Orange fresh sample

Wang *et al.*, (2015) reported the contents of TDF, SDF and IDF in five types of citrus fruit peels had no significant ($P < 0.05$) difference and the binding capacities of SDF in orange; lemon, gonggan and ponkan for sodium cholate and cholesterol were significantly lower than those of grapefruit SDF. Chau and Huang (2003) found that orange peel contained TDF 57% DW and of this; 47.6% & 9.41% DW was the IDF and SDF respectively.

IDF is the dominant fraction that providing health benefits such as intestinal regulation and increased stool volume. Regarding to lemon by-products, Gorinstein *et al.*, (2001) established the levels of DF to be 14 g/100 g (DM) in the lemons peels, compared to 7.34 g/100 g DM in the peeled fruit itself. The TDF contained IDF of 9.04 g/100 g DM and 4.93 g/100 g DM SDF.

Functional group analysis-Fourier transforms infrared spectroscopy (FTIR)

To identify the major functional groups in citrus fruits peel analysis the FTIR spectra was carried out. FTIR spectra of the OP and LP samples are shown in Table (5) and Fig.2 and revealed that:

Intense absorption peaks around 3387-3366 cm^{-1} due to bonded OH groups. The OH stretching vibrations occur within a broad range of frequencies indicating the presence of "free" hydroxyl groups and bonded OH bands of carboxylic acids. In all the peel powder samples broad absorption band in the region 3600–3100cm^{-1} corresponds to the characteristic O–H stretching vibration and hydrogen bond of the hydroxyl groups This band might be due to the inter/intra molecular hydrogen bonding of the fiber back bone (Singthong, *et al.*, 2004).

Absorption peaks around 2933-2928 cm^{-1} can be assigned to stretching asymmetric vibration of CH group. The stretching vibrations of –COOH and $–COOCH_3$ are attributed to the very strong peak around 1729.83-1724.05 cm^{-1} ; 1636-1617 are assigned to carboxylic acid and alkyl carbonate C-OH of carboxyl and 1415-1406 cm^{-1} due to carbonate ion vibrations of lemon peel.

Peaks around 1069.66-1058.73 cm^{-1} are

observed in all studied samples due to the stretching CC, CO and CCO of sugar component Marti, *et al.,* (2009) and John A. Manthey (2006). The wave number between 800 and 1200 in the tested peel samples represents the finger print region of fiber corresponding to CH_3 deformation, C–O–C stretching and O–H bending.

Water- and oil- holding capacities

From Table (6) water holding capacity (WHC) of air oven dried OP samples decreased from 6.17 to 5.59 (as g of water held/g sample); while it is reduced to 5.22 in microwave dried sample. The reducing effect of WHC may be due to the lower temperature used in air drying (40°C). Similar trends were found in case OHC of the fresh and powder samples.

These results agreed with the work of (Abirami *et al.*, 2014) who found that WHC & OHC of Citrus hystrix and Citrus maxima samples were in the range between 5.18 – 8.08 mL water/g samples, 1.26–2.45 mL oil/g sample respectively. Viuda-Martos *et al.,* (2012) mentioned that WHC is related to the SDF content, and high levels of SDF produce a high WHC value. This could be explained by the higher WHC of soluble fibers, such as pectin and gums than cellulosic fibers. So, the higher WHC of LP and OP could be due to the chemical structures, which possess a higher WHC than cellulosic fibers in citrus peel. Sangnark and Noomhorm, (2003) reported that particle size reduction of dietary fibers has been associated with a lower ability to retain water and a lower oil binding capacity. Lario *et al.,* (2004) reported that the high WHC of fiber concentrate could be used as a functional ingredient to avoid syneresis, modification of texture and viscosity and reduce calories of food formulations.

In conclusion, some physicochemical and functional properties of fresh OP & LP and their dried samples either by air-oven (40°C) or microwave for potential use in food industry was the objective of this study. The technological, functionality and nutritional effects of OP & LP samples were determined through their some physicochemical properties. The effect(s) of the used drying methods, on proximate chemical composition, total flavonoids content, phenolic acids profiles, DF content as well WHC & OHC of the tested citrus peel samples, furthermore, analysis of the FTIR spectra was performed to identify major functional groups in the fresh and dried peel samples were discussed in details. HPLC analysis showed that naringin and hesperidin were the predominant phenolic acids in the tested samples with different concentrations. Thus, it can be concluded that orange and lemon peels have important characteristics and can be a promising source of dietary fiber and phenolic compounds which can act important role in evaluation of food quality and potentially can be used as natural sources of functional ingredients or food additives in food enrichment.

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