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CHEMICAL AND TECHNOLOGICAL STUDIES ON KHALAS DATE SEEDS POWDER

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ABSTRACT: This study was aimed to estimate the nutritional value of khalas date seed powder and pita bread, prepared by replacing 5, 10, 15 and 20% of wheat flour by khalas date seed powder. Results indicated that the content of carbohydrate (%), protein (%), crude fibre (%), crude oil (%), ash (%), of khalas date seeds were 64.3, 4.69, 17.12, 4.62, and 1.09%, respectively. The total phenolic content (TPC), total flavonoids and radical scavenging activity (%RSA) were 430.43±1.2 mg GAE/100 g, 346. 13 mg RE/100 g and 79.12±1.9%, respectively. The seed oils contained 51.42% saturated fatty acids and 48.58% unsaturated fatty acids. Fiber content of whole wheat pita bread was higher compared to regular pita bread. It remained lower in 5% date seed powder bread, was quite similar in 10% date seed powder bread, and increased to 9.08% in 20% date seed powder bread compared to whole wheat pita bread. The total phenols, total flavonoids and radical scavenging activity content of pita bread fortified with date seeds increased as the amount of date seeds increased. The pita bread with20% date seeds had the highest percent for each of total phenols, total flavonoids and radical scavenging activity which valued 187.11 mg/100g, 235.45 mg RE/100 g and 43.38%, respectively. While control regular pita contained 72.31 mg/100g, 41.42 mg RE/100 g and 3.53%, respectively. Pita bread supplemented with 15% date seeds, showed high score in overall acceptability when compared to control pita bread and other various concentrations of pits. Generally, changes in dough properties were greater using different concentrations of date seeds with differences for overall acceptability and had high acceptability compared to control pita bread**.**

Key words: Date seeds powder, pita bread, sensory quality, radical scavenging activity.

INTRODUCTION

In the context of the alarming worldwide public health problems, including increasing prevalence of nutrition-related diseases **(Malik and Razig, 2008**), functional ingredients/foods are emerging as a new mode of thinking about their relationship between food and health in daily life. Hence, the attempts to develop functional foods with elevated nutritional quality, capable of preventing nutrition-related diseases, are becoming a major focusing the human nutrition area (**Magrone *et al.*, 2013).** Saudi Arabia is one of the most important countries and is considered to be the genetic center for the origin of date palm. Historically this tree has been crucial for the survival of nomadic tribes in Saudi Arabia (**Hoop, 2003**). Saudi Arabia is considered to be one of the major dates producing and exporting countries (**Al-Showiman and Baosman, 1992**). There are more than 400 cultivars of fruiting date palm of economi value (**Date-Palm Symposium, 1982; Fayadh and Al-Showiman, 1990**). Date palm fruit is considered a popular and widely consumed food in the Middle East and North Africa regions (**Ghnimi *et al.*, 2015**). Date fruit is composed of a fleshy pericarp (85-90%) and a seed (also named kernel) (10-15%) (**Hussein *et al.*, 1998**). Date seed do not have a smell or odor and has a slightly bitter taste bland. In general, it has a light and dark brown. Today, a various studies regarding date seed have been published in order to determine the functional properties of

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date seed is used for food and non-food items such as thermal properties (**Rahman *et al.*, 2007**), treatment and diet (**Hussein *et al.*, 1998**) (**Aldhaheri *et al.*, 2004**), the composition of macro and micronutrients (**Habib and Ibrahim, 2009**), the composition of phenolic acids (**Al-Farsi and Lee, 2008**), as an ingredient of bread (**Almana and Mahmoud, 1994**), and protein solubility (**Hamada *et al.*, 2002**). Date seed are highly recommended for use in foods and dietary supplements. Because, it is a very good source of dietary fibers (**Al-Farsi and Lee, 2008**). Total mineral content found in single seed is comparable to the mineral content of barley. The minerals contained in date seed include sodium, potassium, calcium, iron, copper, magnesium, manganese, zinc, phosphorus, lead, cadmium. This suggests that date seeds are a good source of minerals, and also can be used to replace barley in food products (**Ali-Mohamed and Khamis, 2004)**. The amount of dietary fiber found in date seed valued about 58% of which 53% .soluble dietary fibers as hemicellulose, cellulose and lignin (**Aldhaheri *et al.*, 2004; Al-Farsi and Lee, 2008**). In comparison, the higher dietary fiber was detected in other studies conducted in three different varieties of date seed, the number of fibers between 65 to 69 percentage. This shows the high content of lignin and resistant starch (**Hamada *et al.*, 2002**). On the other hand, protein is also found in date seed with a sizeable amount. Albumin, globulin, prolamin and glutelin are soluble protein which found in seeds of the current date (**Habib *et al.*, 2014**) revealed a total amount of polyphenols of 50.2 mg/g, with the primary compounds being epicatechin and catechin, whose antioxidant properties are well established (**Fraga and Oteiza, 2011**). Phenolic acids contained in date seed are the gallic acid, acid protocatechuic acid p-hydroxybenzoic acid vanilla, caffeic acid, p-coumaric acid, ferulic acid, acid m-coumaric and o-coumaric acid (**Al-Farsi and Lee, 2008**). Based on boundries, **Satuhu (2010)**, indicated that the highest antioxidant content is in that seed, included phenolic. The date kernel is typically thrown away or used as feed for animals (**Al-Farsi and Lee, 2008**) representing a great loss of its functional ingredients (**Almana and Mahmoud, 1994)** such as antioxidants, polyphenols and dietary fibers (**Habib *et al.*, 2014; Srisena *et al.*, 2017**) which have several functional activities including prebiotic and antioxidant activities. So, there is growing attention for uncovering the nutritional role and functional capacity of date seed. Although date seed is still limited for developing value added products, several products fortified with date seeds such as bread (**Almana and Mahmoud, 1994**), yoghurt (**Tammam *et al.*, 2013; El-Kholy, 2018**), were investigated. Furthermore, coffee-like beverages based on roasted date powder preparations are commercialized in Saudi Arabia and The United Arab of Emirates (**Ghnimi *et al.*, 2017**). In view of the above, date seed being an inexpensive rich source of dietary fibers could be exploited as prebiotic (**Al-Thubiani and Khan, 2017**) which can interact with the intestinal microflora especially probiotic bacteria. The date fruit seeds are also listed in folk remedies for the management of diabetes, liver diseases and gastrointestinal disorders in traditional Egyptian medicine (**Duke, 1992**). It has been reported that the extracts of date seeds ameliorate gastric ulceration in rats (**Al- Qarawi *et al.*, 2005**) and possess an anti- inflammatory activity in the rat adjuvant arthritis model (**Doha and Al-Okbi, 2004**). **Salah and Al-Maiman (2005)** have reported that feeding the defatted date seed flour to rats reduced the plasma triglycerides, total cholesterol and low- density lipoprotein

Since pita bread is the major staple food in Saudi Arabia and the region and identified as the major contributor to daily amino acids (AA) intake, the addition of date seed powder to the recipe of pita bread might enable the development of a new functional bread, containing relatively higher amounts of fiber and antiox-idants in comparison to the regular pita bread and whole wheat pita bread. The aim of this study was to characterize the chemical properties of date seed powder as well as determination chemical properties and sensory evaluation of pita bread containing different levels of date seed powder

Materials

Date palm seeds of the Koalas variety were used in this study. 4 kg samples were collected randomly from tamer (fully ripe dates) batches at the end of the season, with no preference to size, color, appearance or firmness. Bread-making ingredients such as wheat flour (extracting rate 72%, instant yeast, sugar and salt, *etc.*), and oil were purchased from local market at Najran.

Methods

Date palm seeds of the Khalsa variety, was washed and dried in an oven at 30°C for 48 hours. Pits were crushed using pestle and mortar followed by high speed laboratory blender and then sieved to obtain finely divided powder. Seeds powder was stored under refrigeration until processing and analyses.

Preparation of Date Seed Powder and Fortified Pita Bread (DSPB)

The formula of pita breads are shown in Table 1 including the dough contained flour (100 parts) 1500g, sugar (2.4 parts) 36 g, salt (1 part) 15 g, yeast (0.8 parts) 12g and distilled/deionized water (50 parts) 750 ml. Date seed powder replaced a part of the flour. Different replacement levels were considered, according to the amount of fibers found in whole wheat bread (around 7.4%, according to the USDA National Nutrient Database for Standard Reference, 2012) and to not exceed the current recommendations of 25 g/day for adult woman and 38 g/day for adult man: 5, 10, 15 and 20% date seed powder. The regular pita bread and the whole wheat pita bread were used as controls. The ingredients were mixed in a dough mixer at low speed to obtain smooth continuous dough. Dough was fermented at 40°C for 25 min. The dough was then divided into small balls (75 ± 3 g) and proofed at 40°C for 25 min. The balls were flattened into sheets 1.7 mm thick, proofed again at 40°C for 15 min and baked at 500°C for 1 min. The bread loaves were cooled to room temperature, placed in polyethylene bags and stored at −20°C until used.

Bread Crushing

Each sample of bread was first crushed in a mixer (20 min) till they formed fine bread crumbs and was transferred into new packs with labels.

Chemical Composition

Moisture, crude protein, crude fat, crude fiber and carbohydrate (by difference) of khalas date seeds powder (KDSP) and pita bread formulas were done according to (**AOAC, 2000**). Total phenols were estimated by the Folin-Ciocalteu method reported by **Elfalleh *et al.* (2009**). DPPH scavenging activity was determined using a modified method of **Ohnishi *et al.* (1994**). The free radical scavenging activity of food extracts were tested, indicated as bleaching of the stable 1,1 -diphenyl-2-picrylhydrazyl radical (DPPH). Fatty acids were extracted and separated by the method described by **Stroescu *et al.* (2013)**. Mineral contents *i.e.*, (calcium, phosphorus, potassium, magnesium, sodium, iron, Cupper, manganese and zinc of wheat, khalas date seeds powder and pita bread were determined according to the methods described in **AOAC (2000**). The samples were wet acid digested using a nitric acid and perchloric acid mixture (HNO3, HCLO4, 2:1 *V/V*). The amounts of iron, zinc, and manganese in the digested sample were determined using a GBC Atomic Absorption 906. Sodium and potassium were determined by flame photometer 410. Calcium and magnesium were determined using Double Beam Atomic Absorption. Phosphorus was determined according to the method described in **AOAC (2000)**

Sensory Evaluation of Pan Bread

Samples of pita bread were evaluated by 10 panelists (staff in College of Education, Najran University) for taste (20), odor (20), crumb distribution (15), crust color (15), crumb color (15) and general appearance (15). The total value of these sensory properties were evaluated as overall acceptability and descriptive category as follows: 90-100: very good 80-89: good, 70-79: satisfactory: less than 70: questionable **(Khorshid *et al.*, 2011)**.

Sugar Analyses

Date seed powder, diluted with distilled water in 1/100 ratio, was kept for 1 hr., in a shaking water bath (Nu¨ve) at 508°C and then filtered. One millilitre of extract was transferred to two tubes and 1 ml of phenol solvent (80%) was added to each tube. In the mixture, 5 ml HCl (95.5%) was added quickly and then the tubes were left to cool for 30 min. The absorbance of the mixtures in the tubes was measured at 490 nm wavelength using a UV-vis spectropho-tometer (Schimadzu, Japan). Calibration graphics of saccharose, raffinose, stachyose, galactose, fructose and glucose were prepared and by the use of the graphics the amounts of these sugars in the seed extracts were calculated **(Dubois *et al.*, 1956)**.

Table 1. Formula composition of date seed powder pita bread

Ingredient (g)	Control		Pita bread DSP formula (g)			
	Regular pita bread	Whole wheat pita bread	5%	10%	15%	20%
Wheat flour	1500.0	1500.0	1425	1350.0	1275	1200
date seed powder	-		75	150	225	300
Instant yeast	12	12	12	12	12	12
Sugar	36	36	36	36	36	36
Salt	15	15	15	15	15	15
Water	750	750	750	750	750	750
Total	2313	2525	2525	2525	2525	2525

Statistical Analysis

Statistical analysis was carried out according to **Fisher (1970)**. LSD (Least significant difference) test was used to compare the significant differences between means of treatments (**Waller and Duncan, 1969**).

RESULTS

The findings present in Table 2 show the chemical composition of khalas date seeds (KDS). The results indicated that the content of carbohydrate (%), protein (%), crude fibre (%), crude oil (%), ash (%), of khalas date seeds were 64.3, 4.69, 17.12, 4.62 and 1.09%, respectively. The obtained results indicated that total phenolic content (TPC), total flavonoids and radical scavenging activity (%RSA) were 430.43±1.2 mg GAE/100 g, 346.13 mg RE/100 g and 79.12±1.9%, respectively. Date seeds were shown to be rich in antioxidants, for which antihyperlipidemic, anticancer and antimutagenic properties were identified. The determination of their polyphenolic profile by UPLC-DAD-ESI-MS, (**Habib *et al.*, 2014**) revealed a total amount of polyphenols of 50.2 mg/g, with the primary compounds being epicatechin and catechin, whose antioxidant properties are well established (**Fraga and Oteiza 2011**).While its content of some minerals (ca, P, K, Mg, NaFe, Cu, Mn and Zn mg/100g) were 26.4, 306.3, 368.2, 89.3, 16.8, 2.7, 0.39, 0.68 and 0.24, respectively. The date seeds from (Khalas) were evaluated with respect to sugar compositions. The fructose, stachyose, sucrose, galactose, raffinose and glucose (g/kg) contents of date seeds were 3.60, 3.79, 3.23, 2.21, 2.91 and 2.98, respectively. The results obtained in the present study regarding minerals and sugar compositions are in a general agreement with those of **Al-Juhaimi and Ozcan (2012)**.

The fatty acid compositions of date seeds are given in Table 3. In all of them, 13 fatty acids were present, five of which were unsaturated, whereas the others were saturated. The most abundant fatty acids of the date seed oils were oleic (41.14%), lauric (22.90%), myristic (13.68%), palmitic (9.83%), Linoleic (6.89%) and stearic (3.45%) acids. The seed oils contained 51.42% Saturated fatty acids, 48.58% unsaturated fatty acids, these results were on the same line with those recorded by **Habib *et al.* (2013)**.

Table 4 shows the moisture, protein, fat and fiber contents of the different bread samples. The moisture content ranged from 26.36 to 33.81%, with the whole wheat pita bread having the lowest value (26.36±3.45) and the 5% date seed powder pita bread having the highest one (33.81±2.65). However, no differences were detected among the samples. Similarly, the protein content did not differ among the samples, but ranged from 12.41% in 20% date seed powder pita bread to 14.51% in regular pita bread. In terms of fat content, it was quite similar in regular and whole wheat pita breads, and increased in a linear manner in date seed

Table 2. Chemical composition of khalas date seeds

Content of date									
Moisture (%)	**Carbohydrate (%)**	**Crude protein (%)**	**Crude fiber (%)**	**Crude oil (%)**	**Ash (%)**	**Energy kcal/ 100g**	**Radical scavenging activity (%RSA)**	**Total phenol (mg GAE/ 100 g)**	**Total flavonoids (mg RE/ 100g)**
8.18±1.24	64.30±2.12	4.69±1.31	17.12±1.0	4.62±1.50	1.09±0.42	317.54±3.43	79.12±1.91	430.43±1.27	346. 13±1.41
Minerals mg/100g									
Ca	**P**	**K**	**Mg**	**Na**	**Fe**	**Cu**	**Mn**	**Zn**	
26.4 ± 2.1**1**	306.3 ± 4.**52**	368.2 ± 1.2**2**	89.3 ±2 .8**0**	16.8±**0.80**	2.7 ± 1.4**1**	0.39 ± 0.1**0**	0.68 ± 0.21	0.24±0.06	
Sugar profile of date seeds									
Fructose (g/kg)	**Stachyose (g/kg)**	**Sucrose (g/kg)**	**Galactose (g/kg)**	**Raffinose (g/kg)**	**Glucose (g/kg)**	**Fructose (g/kg)**			
3.60± 0.22	3.79 ± 0.43	3.23 ± 0.11	2.21 ± 0.51	2.91 ± 0.42	2.98 ± 0.13	3.60± 0.22			

Table 3. Fatty acids compositions of khalas date seed oils (%)

	(%)
Caproic (C 6:0)	0.40±0.21
Caprylic (C 8:0)	0.89±0.08
Lauric (C 12:0)	22.90±2.43
Myristic (C14:0)	13.68±1.68
Palmitic (C 61:0)	9.83±1.54
Palmitoleic (C 61:1)	0.12±0.09
Stearic (C 18:0)	3.45±0.20
Oleic (C 18:1)	41.14±3.43
Linoleic (C 18:2)	6.89±1.12
Linolenic (C 18:3)	0.20±0.06
Arachidic (C 20:0)	0.19±0.02
Gadoleic (C 20:1)	0.23±0.04
Behenic (C 22:0)	0.08±0.01
Saturated fatty acid	51.42±2.51
Monounsaturated fatty acid	41.49±3.43
Polyunsaturated fatty acid	7.09±1.32
Total	100

Table 4. Chemical composition of date seed powder fortified pita bread

Product	Moisture (%)	Protein (%)	Fat (%)	Fibers (%)	Total phenolics mg GAE/100 g bread	Total flavonoids (mg RE/ 100 g bread)	Radical scavenging activity (%RSA)
Whole wheat pita bread	26.36±3.45	13.84±2.51	0.08±0.04	5.97±1.21	99.14±3.23	129 ,13±2.7	7.22±2.40
Regular pita	31.23±3.23	14.51±1.05	0.07±0.05	1.31±0.13	72.31±4.56	41.42±1.4	3.53±0.90
5% date seed powder pita	33.81±2.65	13.36±1.11	0.19±0.15	2.65±0.09	75.07±5.00	122.21±2.5	9.98±1.45
10% date seed powder pita	32.67±1.34	13.25±0.21	0.39±0.17	5.89±1.15	89.51±3.30	160.32±1.6	14.65±3.32
15% date seed powder pita	31.12±2.78	12.97±0.54	0.44±0.45	8.09±0.16	136.32±1.54	190.34±2.7	27.57±0.21
20% date seed powder pita	31.56±1.38	12.41±0.31	0.71±0.32	9.08±1.1	187.11±5.20	235.45±3.2	43.38±0.36

powder bread from 5 to 20% of date seed powder, reaching a maximum of 0.71%. Not surprisingly, fiber content of whole wheat pita bread was higher compared to regular pita bread. It remained lower in 5% date seed powder bread of 2.65%, was quite similar in 10% date seed powder bread of 5.89%, and increased to 9.08% in 20% date seed powder bread compared to whole wheat pita bread.

The total phenols content of the pita bread fortified with date seeds increased as the amount of date seeds increased. The pita bread with20% date seeds had the highest percent of total phenols and total flavonoids (187.11 mg/100g and 235.45 mg RE/100 g bread, respectively) compared with control regular pita (72.31 mg/ 100 g and 41.42 mg RE/100 g bread, respectively.). Radical scavenging activity (%RSA) of pita bread increased by the addition of date seeds. The addition of 20% date seeds caused an increase in radical scavenging activity (%RSA) to 43.38% compared with 3.53% for control regular pita bread. This may be due to date seeds has powerful antioxidants activity as reported by **El-Mergawi *et al.* (2016)**.

Sensory evaluation of pita bread fortified with date seeds powder at various levels are shown in Table 5. Most of panelists preferred the odor and taste of Pita breads made from 10% and 15% of the date seeds powder, they commented that the odor and taste of 5% of date seeds with wheat flour was nearly like the control bread. However, pita bread made with 10% and 15% of date pits powder received the highest overall score than pita bread containing wheat flour without any supplementation. These results are in agreement with **Besbes *et al.* (2009)** and **Bouaziz *et al.* (2010)** they published that by-product of date-processing industries could be regarded as an excellent source of food ingredients with interesting technological functionality that could also be used in food as an important source of dietary fiber. However, **Najafi (2011**) showed that bread containing 10% date seed had higher dietary fiber content and similar sensory properties to the wheat bran control, but lower color, flavor, odor, and overall acceptability sensory scores.

The crust and crumb colors were different in pita bread containing 5% and 10% compared with the control bread, while, in pita bread containing 15% date seeds there was increase than that of control (**El-Porai *et al.*, 2013**). The results of crust, crumb colors and general appearance showed that there were changes between control pita bread (100% SWF) and pita bread containing date pits powder, confirmed by **Mirghani *et al.* (2012**) who evaluated taste, texture, aroma, appearance and overall acceptability for pan breads fortified with date pits which can be a source of dietary fibers, without any negative impact on sensory quality of end-products. Pita bread supplemented with 15% date seeds, showed high score in overall acceptability when compared to control pita bread and other various concentrations of pits. Generally, changes in dough properties were greater using different concentrations of date seeds with differences for overall acceptability and had good acceptance to most members compared to control pita bread.

Table 5. Sensory evaluation of pita bread fortified with date seeds powder at various levels

Type of bread	Taste 20	Odor 20	Crumb distribution (15)	Crust color 15	Crumb color 15	General appearance 15	Overall acceptability
Regular pita	18.10± 1.3	18.2±1.4	13.78±	13.87±1.5	12.79±0.9	14.1±0.8	90.84±1.2
Whole wheat pita	18.1±1.2	17.34±0.8	12.32±	12.67±0.7	11.98±1.3	12.23±1.2	87.32±0.8
5% date seed powder pita	18.50±0.5	18.67±2.1	13.08±	14.75±1.5	13.52±2.2	14.32±0.8	92.84±3.1
10% date seed powder pita	18.95±2.1	19.70±1.8	12.92±	14.81±0.9	13.95±0.5	14.67±0.3	95±2.4
15% date seed powder pita	19.10±1.5	19.75±1.6	12.88±	14.85±2.3	14.22±0.3	14.71±1.1	95.51±3.2
20% date seed powder pita	18.80±0.9	19.40±0.8	12.56±	14.81±0.8	13.50±0.6	14.31±2.3	93.82±2.5

The average of total score was converted to a descriptive category as follows: V.good: 90-100, Good: 80-89, Acceptable: 70-79, Poor: less than.

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دراسات كيميائية وتكنولوجية على مسحوق بذور تمر الخلاص

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هدفت هذه الدراسة إلى تقدير القيمة الغذائية لمسحوق بذور تمر الخلاص والخبز العربى المحضرين باستبدال ٥ ، ١٠، ١٥ و ٢٠% من دقيق القمح بمسحوق بذور تمر الخلاص وتحديد أفضل نسب الاستبدال التي تؤدي إلى الحصول على خصائص حسية مقبولة و أشارت النتائج إلى أن محتوى الكربوهيدرات والبروتين والألياف الخام والزيت الخام والرماد لبذورتمر الخلاص كانت (٦٤.٣ ، ٤.٦٩ ، ١٧.١٢ ، ٤.٦٢ ، ١.٠٩%) على التوالي، كما كان محتوى الفينولات الكلية والفلافونويد والنشاط الكاسح للشقوق الحرة في بذور تمر الخلاص ٤٣٠.٤٣و٣٤٦.١٣ ملليجرام لكل ١٠٠ جرام و٧٩,١٢% على التوالي، كما احتوى الزيت المستخلص من البذور على ٥١,٤٢% احماض دهنية مشبعة و٤٨,٥٨% احماض دهنية غير مشبعة، وكان محتوى الألياف اعلى في الخبز المعد من القمح الكامل عن الخبز المعد من دقيق القمح العادى. وظل محتوى الألياف منخفض في الخبز المعد باستخدام ٥% من مسحوق بذور التمروالذى كان متشابه لحد ما مع الخبز المعد مع ١٠% من مسحوق بذور التمرولكن بزيادة محتوى الخبز من مسحوق بذور التمر الى ٢٠% زاد محتوى الألياف ليصل الى ٩,٠٨% وزاد محتوى الخبز العربى من الفينولات والفلافونيويد والنشاط الكاسح للشقوق الحرة بزيادة محتوى الخبز من مسحوق بذور التمر وكان اعلى محتوى مع وجود نسبة ٢٠% من مسحوق بذور التمر في الخبز حيث بلغت نسبة الفينولات الكلية والفلافونيد والنشاط الكاسح للشقوق الحرة ١٨٧,١١و٢٣٥,٤٥ ملليجرام/١٠٠ جرام خبز و٤٣,٣٨% على التوالي مقارنة بالكونترول ٧٢,٣١ و٤١,٤٢ ملليجرام لكل ١٠٠ جرام و٣,٥٣% على التوالي كما أظهرت النتائج ان الخبز العربى المحتوى على ١٥% من مسحوق بذور التمر حصل على اعلى درجة من ناحية التقبل العام بشكل عام حصل تغير في خصائص الخبز باستخدام تركيزات مختلفة من مسحوق بذور التمر ورغم وجود اختلاف في التقبل العام إلا أن كل العينات المحتوية على مسحوق بذور التمر كان لها قبول جيد مقارنة بالكنترول

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