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UTILIZATION OF CAROB PODS IN THE PRODUCTION OF ALTERNATIVE TO COCOA POWDER

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ABSTRACT

Roasting process of carob pods at various temperatures for different times was carried out to study the effect of its process on the chemical constituents, minerals content, phenolic compounds and antioxidant activity. Volatile compounds were also determined for roasted carob powders. The obtained results revealed that the protein content was 5.42% (on dry weight basis), which decreased with increasing the processing temperatures. Whereas, ash increased gradually by increasing the roasted temperatures but lipids was decreased. The predominant mineral in carob powders was calcium followed by sodium, potassium and magnesium, respectively whereas; the raw carob pods contain 21.07mg/g of total phenolic compounds. Roasting treatment caused to the degradation of phenolic compounds which increased with increasing the roasting temperatures. The antioxidant activity of the carob powders was determined by 2,2-diphenyl-1-picryl-hydrazyl (DPPH) as free radical scavenging. However, the activity of antioxidant was decreased owing to the roasting process. On the other hand, sensory characteristics for both hot drinks and prepared cakes showed greater preference, especially for samples contained roasted carob powders at 160˚C for 30 min. and/or 40min. compared with that in tested samples and control. Seven aroma compounds were positively identified by using GC–mass spectrometry. The major aroma compounds of carob powders were 3-methyl butanal and methyl propanal which was found in chocolate and it's responsible for chocolate flavor. Moreover, other volatile compounds were identified in roasted carob powders and which also found in dark chocolate such as 2-Heptanone, Pentan-2-ol and Linalool. Also, Nonane-2-one and Hexanal were found and identified in roasted carob powders.

INTRODUCTION

The carob tree (*Ceratonia siliqua L.*) has been widely cultivated in Mediterranean countries for at least four thousand years. This tree was distributed by Arabs in Mediterranean area. The two principal components of the fruit of carob tree are pods and seeds. The seeds, which comprise about 10% of weight of the fruit, are composed principally of galactomannans **(Calixto & Canellas, 1982).**

Carob pods is rich in sugars 40-60 %, low protein 2.7-3.5%, lipids 0.4-0.8% and high in phenolic compounds (mostly highly condensed tannins) that account for nearly 20 mg/g on dry weight basis **(Davies *et al* 1970; Alumot *et al* 1980; and Lambraki *et al* 1996).** Many compounds extracted from carob pods such as fructose and ethanol **(Marakis & Marakis, 1996)**, C-glycosyl flavones **(Batista & Gomes, 1993)** α-galactosidase and endo β-mannase **(Kontos &Spyropoulos, 1995; Hashem *et al* 2001),** sugar syrup **(Petit & Pinilla, 1995)**, citric acid **(Roukas, 1999)** and other compounds.

The possibility of processing roasted carob powder using various temperatures and different periods has been studied by **(Yousif & Alghzawi, 2000),** they studied the physical and chemical characteristics of the carob powders roasted at 150°C for 60 min and they found that the roasted carob powder contained 9.03, 5.82, 2.84 and 0.74% moisture, protein, ash and fat, respectively. Whereas, carob powder contained 38.7% total sugar, 7.24% fiber and 3.75% tannins.

The majority of the carob pulp is milled for the production of carob powder, which is roasted to enhance its cocoa-like flavor for its utilization as a cocoa and/or chocolate substitute and also as extender in the food industry.

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Recently, **Afoakwa *et al* (2009)** found that flavour released from dark chocolate volatiles and quantified by static headspace gas chromatography using GC–MS. Sixty-eight (68) flavour compounds were identified, comprising alcohols, aldehydes, esters, ketones, furans, pyrans, pyrazines, pyridines, pyroles, phenols, pyrones and thiozoles. From GC– olfactometry, 2-methylpropanal, 2-methylbutanal and 3-methylbutanal had chocolate notes. With cocoa/roasted/nutty notes were trimethyl-, tetramethyl-, 2,3-dimethyl-, 2,5-dimethyl-, 3(or 2),5-dimethyl-2(or 3)-ethyl- and 3,5(or 6)-diethyl-2-methylpyrazine and furfuralpyrrole. Compounds with fruity/floral notes included 3,7-dimethyl-1,6-octadien-3-ol and5-ethenyltetrahydro-R,R,5-trimethylcis- 2-furanmethanol.

The absence of caffeine and theobromine give carob powder great potential as a component in the coloring, flavoring and sweetening of wide range of confectionery products, beverages, biscuits and cakes sold in health food stores worldwide **(Arrighi *et al* 1997).** On the other side, carob is useful for treating infant diarrhea, **Leob *et al* (1989).** In addition, carob tannins do bind with toxins and inhibit growth of bacteria. Dietary fiber and sugars from carob pod may make food more viscous in the stomach and thus interfere with reflux and acid into the esophagus **Greally *et al* (1992).** Moreover, it contains radical scavenges which have attracted special interest because they can protect the human body from the attacks of active oxygen species and free radicals that may cause cancer and other disease **(Murakami *et al* 2003).** Also, these mechanisms are believed to be related to the antioxidant activity of polyphenolic compounds.

The pod of the carob fruit has long been used as a feed for livestock and in human nutrition, including sweets, biscuits and processed drinks, because of its high sugar content **(Davies *et al* 1970 and Khair *et al* 2001).**

The aim of this investigation is to study the effect of roasting carob at different temperatures on the chemical constituents, minerals, phenolic compounds, antioxidant activity and volatile compounds as well as sensory evaluation to produce alternative products of cocoa powder.

MATERIALS AND METHODS

Materials

Carob pods *(ceratonia siliqua)* were obtained from Borg EL-Arab region, Alexandria Governorate. Raw cocoa powder was purchased from local market to be used as a comparison sample.

Technological methods

Seeds were separated from carob pods and roasted at 150, 160 and 170˚C for 30 and 40 minutes in enforced air oven drier then milled to obtain fine particle which pass from 100-mesh sieves. The powder carob samples were kept in tri-layer bags and stored at –18˚C until used.

Preparation of carob powder products

- Hot carob drinks

Hot carob drinks were prepared by using 15 g of carob powders with 0.5% vanillin, 5% skim milk and 5% sucrose with 200ml hot water, while hot cocoa drink was prepared as mentioned previously by substitution of cocoa powder instead of carob powder and used as control.

- Preparation of cakes

The cake formulation and production was performed mentioned by **Soliman, (1996)** as follows: The cakes formula consists of wheat flour (90 g.), roasted carob powder (10g.), fresh whole eggs (about 120-150 g.), baking powder (2g.), vanillin (1 g.) and sugar (100g.). Then, all ingredients were mixed by using blender for 10 min. The prepared cake was placed in a baking pan (7.8 Cm inner diameter and 3.5 heights) then baked at 180˚C for 35 min. in air oven and compared with control by using 10g of cocoa powders instead of carob powders and mixed with the same ingredients. After baking, cakes were allowed to cool at room temperature.

Analysis of samples

Moisture content, total sugars, ash, lipids, crude protein (N×6.25), crude fiber and phenolic compounds were determined according to **AOAC (1995).**

Calories of tested samples were calculated on the base of: 1g of total sugars or protein equals 4 kilo calorie per 100 g. and 1g of fat equals 9 kilo calorie per 100 g. as mentioned by **Lawrence (1965)**.

Minerals Mg, Na, Zn, Mn, Fe, Ca and K were estimated according to **Kasai *et al* (1997).** Total phenolics were estimated by using photometric method with Folin's reagent as reported by **(AOAC 1970)** and total antioxidant activity was determined according to the method of **Brand-Williams *et al* (1995).**

Isolation of volatile substances: Carob powder was extracted by steam distillation for 2hr. The200µl of collected extractions were concentrated under vacuum then deacidified with extract water, extracted with distillation by diethyl ether. The organic phase was dried over anhydrous sodium sulphate and concentrated under vacuum at 40˚C. **(MacLeod and Forcen, 1992).**

GC-MS analysis: GC-MS analysis was performed by using Perken Elmer (CT 06859 USA) equipped with flame ionization detector (FID). A fused silica capillary column DB5 (60 m × 0.32mm id) was used. The oven temperature was started with 60˚C then programmed from 60 to 225˚C a rate 2˚C min. Helium was used as carrier gas at flow rate 2 ml/min. The injector and detector temperature were 250˚C.

Sensory evaluation: The sensory evaluation of prepared cakes and hot drinks were carried out with three replicates by 10 trained panelists. The investigated attributes of cakes and hot drinks were flavor (odor and taste), texture, colour and overall acceptability. All investigated attributes were scored of 10 degree.

Statistical analysis: Experimental data was carried out using SPSS software, version 12.0. Differences among groups were evaluated using one way analysis of variance. Significant differences were indicated by analysis of variance, group means were compared using the Duncan test. A probability of $P < 0.05$ was considered as significant using the statistical analysis system **SAS Institute Inc., 1999**).

RESULTS AND DISCUSSION

Proximate chemical composition of raw carob pods before and after roasting, at different temperatures is represented in **Table (1)**. Results showed that, the moisture content in raw carob pods was 7.53%, while it was decreased to 2.12, 1.94 and 1.50 % after roasting at 150, 160 and 170˚C for 30 min., respectively. Meanwhile, the corresponding values for decremental of moisture were 2.03, 2.00 and 1.42% after roasting for 40 minutes. Protein content was 5.42% in carob pods and then decreased with increasing the processing temperatures. Ash content was increased gradually by increasing the roasted temperatures. On contrast lipids decreased from 1.94 in raw carob powder to 1.82, 1.76 and 1.59 % after 30 min. and 1.85, 1.80 and 1.73% after 40 min. of roasting at 150, 160 and 170˚C, respectively.

Also, carob powder contained higher levels of crude fibers and lipids, compared with cocoa powder. This might be considered as an advantage from nutritional point of view to consider it as a good replacer of cocoa powder in food processing.

Mineral contents in raw and roasted carob powders at different temperatures for 30 and 40 min. were determined and results obtained are presented in **Table (2)**. These results revealed that, calcium had the highest minerals content of carob powders obtained by roasting at various temperatures (150, 160 and 170˚C) for 30 and/ or 40 min., but less than in cocoa, followed by Na, K and Mg, respectively. Whereas, it contained little amounts of Zn, Mn and Fe compared with that in cocoa powder.

Total polyphenolic compounds was extracted and determined in raw and processed carob powders and the obtained results are shown in **Table (3)**. Total polyphenolic compounds were 21.07 mg/g in raw carob pods. Thermal treatment caused to decrease of polyphenolic compounds which decreased with increasing roasting temperatures. Decremental percentages in processed carob were 18.60, 38.63 and 45.47% after being roasted at 150, 160, and 170˚C for 30 min. respectively. While, the extending roasting time to 40 min. at the same temperatures caused to decremental values of total polyphenolic compounds to 25.39, 42.52 and 51.40%, respectively. These decrements directly affected by the activity of natural antioxidants which determined by 2,2-diphenyl-1-picryl-hydrazyl DPPH as free radical scavenging. Results in the same table showed that, the antioxidant activity percentage in raw carob was 90.09%, and then decreased to 70.28, 69.93 and 67.05% after roasted for 30 min., while the activity of antioxidant was decreased to 65.50, 64.53 and 63.82% after being roasted at 40 min. for 150, 160, and 170˚C, respectively.

Organoleptic evaluation is considered to be of utmost importance since it reflects the consumer preference. Results of sensory attributes were recorded by well trained panelists for hot drinks prepared by using cocoa as control and roasted carob powders are represented in **Table (4)**. These results revealed that the carob powder roasted at 160˚C for 30 and 40 min. were recorded the highest scores compared with other samples including control, while, roasted samples at 150˚C for 30 min. recorded the same scores compared with the untreated carob powders used as control followed by roasted at 150˚C. Meanwhile, the other tested samples prepared by roasted carob powders

Table 1. Proximate chemical composition* of raw and carob powders roasted at different temperatures for various times

Constituents	Raw	Roasted for 30 min.			Roasted for 40 min.			Cocoa powder
		150˚C	160˚C	170˚C	150˚C	150˚C	170˚C	
Moisture	7.53	2.12	1.94	1.50	2.03	2.00	1.42	2.51
Protein	5.42	5.13	4.79	4.58	4.71	4.59	4.52	22.90
Total sugars	54.36	59.13	57.45	56.72	61.24	58.39	55.41	2.16
Ash	2.72	2.81	2.87	2.99	2.92	2.95	3.10	6.40
Lipids	1.94	1.82	1.79	1.59	1.85	1.80	1.73	22.88
Crude fiber	6.81	5.58	5.14	4.97	5.32	5.00	4.65	4.93
Energy(Kcal/100g)	256.58	273.30	264.80	259.51	280.45	268.12	255.29	306.16

* % on dry weight basis

Table 2. Minerals content (mg/100g) of raw and carob powders roasted at different temperatures for various times (on dry weight basis)

Minerals	Raw	Roasted for 30 min.			Roasted for 40 min.			Cocoa powder
		150˚C	160˚C	170˚C	150˚C	160˚C	170˚C	
Mg	15.88	15.91	15.95	15.99	15.92	15.98	16.12	538.30
Na	152.00	152.32	152.85	152.97	152.66	152.79	152.96	983.44
Zn	0.94	0.97	1.05	1.12	0.97	1.10	1.17	7.14
Mn	1.66	1.64	1.60	1.52	1.63	1.60	1.49	2.23
Fe	2.38	2.30	2.25	2.20	2.33	2.10	1.97	10.87
Ca	488.6	491.4	494.5	499.7	491.2	494.6	499.8	134.58
K	135.4	136.21	138.83	142.21	137.35	139.52	145.79	1552.79

Table 3. Total phenolic compounds and total antioxidant activity in raw and carob powders* roasted at different temperatures for various times

Items	Carob powder	Roasted for 30 min.			Roasted for 40 min.		
		150˚C	160˚C	170˚C	150˚C	160˚C	170˚C
*Total phenolic compounds (mg/g)	21.07	17.15	12.93	11.49	15.72	12.11	10.24
T.A.A (%) **	90.09	70.28	69.93	67.05	65.50	64.53	63.82

* on dry weight basis **T.A.A. Total antioxidant activity.

Table 4. Sensory evaluation of hot drinks prepared by using cocoa powder as control and roasted carob powders

	Color	Taste	Odor	Texture	Overall acceptability
Control (Cocoa drink**)**	7.72 $^{a b}$	7.78 $^{a b}$	7.67 ^{a}	9.11 ^{a}	7.89 ^{a}
Roasted for 30 min.					
150˚C	7.44 $^{a b}$	7.33 $^{b c}$	7.39 $^{a b}$	8.39 $^{b c}$	7.00 $^{b c}$
160˚C	7.94 ^{a}	8.39 ^{a}	7.89 ^{a}	8.83 $^{a b}$	7.89 ^{a}
170˚C	6.44 $^{c d}$	6.22 ^{d}	6.33 $^{c d}$	6.50 ^{f}	6.06 ^{d}
Roasted for 40 min.					
150˚C	7.22 $^{a b}$	7.06 ^{c}	6.83 $^{b c}$	8.00 $^{c d}$	6.78 $^{a b c}$
160˚C	7.17 $^{b c}$	7.72 $^{a b c}$	7.28 $^{a b}$	7.39 $^{d e}$	7.17 $^{a b}$
170˚C	6.33 ^{d}	6.17 ^{d}	6.06^{d}	7.17 $^{e f}$	6.25 $^{c d}$
LSD	0.616	0.532	0.594	0.612	0.581

Values with different superscript letters (a) – (f) in the same column are significantly different according to the Duncan ($p<0.05$)

Table 5. Sensory evaluation of prepared cakes with raw cocoa as control and roasted carob powders

	Color	Taste	Odor	Texture	Overall acceptability
Control	7.80 ^{a}	7.90 $^{a b}$	8.00 $^{a b}$	8.20 $^{a b}$	8.30 $^{a b}$
Roasted for 30 min.					
150˚C	7.65 ^{a}	7.80 $^{a b}$	7.80 $^{a b}$	8.20 $^{a b}$	8.30 $^{a b}$
160˚C	8.10 ^{a}	8.20 ^{a}	8.20 ^{a}	8.40 ^{a}	8.60 ^{a}
170˚C	7.90 ^{a}	7.90 $^{a b}$	7.50 $^{b c}$	8.20 $^{a b}$	8.05 $^{a b c}$
Roasted for 40 min.					
150˚C	7.90 ^{a}	7.80 $^{a b}$	7.70 $^{a b c}$	8.30 $^{a b}$	8.05 $^{a b c}$
160˚C	7.60 ^{a}	7.50 $^{b c}$	7.70 $^{a b c}$	7.80 $^{b c}$	7.90 $^{b c}$
170˚C	6.90 ^{b}	7.00 ^{c}	7.20 ^{c}	7.40 ^{c}	7.45 ^{c}
LSD	0.5974	0.5741	0.5968	0.5658	0.6509

Values with different superscript roman letters (a) – (c) in the same column are significantly different according to the Duncan test ($p<0.05$)

Table 6. Identification of some volatile components in raw and carob powder roasted at 160˚C for 30 and 40 min.

Volatile components	tR	Raw carob powder (%)	Carob powder roasted at 160˚C for 30 min. (%)	Carob powder roasted at 160˚C for 40 min. (%)
Methyl propanal (chocolate flavor).	5.07	2.72	1.78	0.13
3-methyl butanal (chocolate flavor).	7.32	4.29	7.40	36.95
Pentan-2-ol	8.76	0.81	6.85	13.17
Hexanal	13.77	2.01	0.29	0.27
2-Heptanone	19.45	2.61	2.54	2.11
Nonane-2-one	25.50	4.33	3.71	2.55
Linalool	36.62	1.02	0.04	0.03

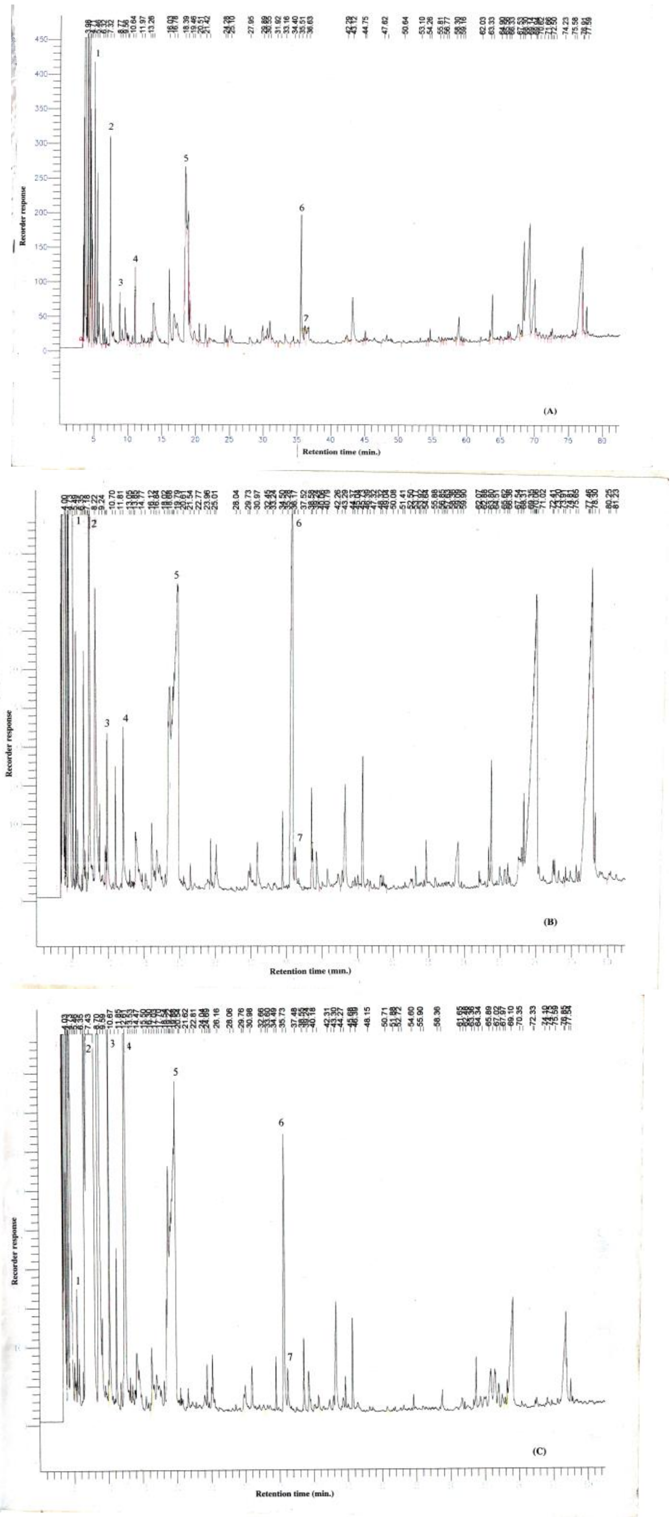

Fig. 1. Gas chromatogram of the volatile compounds in raw and roasted carob powders (A) Raw carob powder, (B) Roasted carob powders at 160˚C for 30min. and (C) Roasted carob powders at 160˚C for 40min. (1) Methyl propanal, (2) 3-Methyl butanal, (3) Pentan-2-ol (4) Hexanal (5) Heptan-2-one (6) Nonane-2-one (7) Linalool

at 170˚C for 30 and 40 min., had the lowest significantly scores compared with control for all tested attributes. Therefore, the best carob drinks recorded the highest scores was prepared by roasting carob powder at 160˚C for 30 min.

The characterization of sensory evaluation for cakes prepared by adding roasted carob powders processed at various temperatures compared with control cake prepared by using cocoa powder are represented in **Table (5)**. Results revealed that significant increase in color, taste, odor, texture and over all acceptability for prepared cakes with roasted carob powder at 160˚C for 30 min. which recorded the highest scores; followed by samples roasted at 150˚C for 40 min. These treatments recorded the highest acceptable attributes comparing with those obtained by control (cocoa powder) and other treatments. Meanwhile, the lowest score was recorded after being treated of sample at170˚C for 40min. **Yousif and Alghzawi, (2000)** noticed that, the best time and temperature for roasting of kibbled carob was at150˚C for 60 min. according to sensory results.

Volatile compounds of carob powders were analyzed by using GC-MS for raw and best processed samples which recorded the highest scores with organoleptic evaluation which was processed at 160˚C for 30 and 40 min. are represented in **Table (6) and Fig. (1)**.Results shows that, seven compounds were identified. The major indicated compounds were 3-methyl butanal and Methyl propanal, these two compounds are responsible to chocolate flavor **Afoakwa *et al* (2009)**. So, release 3-methyl butanal and/or methyl propanal caused to chocolate flavor. Roasted carob caused to increase of 3-methyl butanal from 4.29 in raw carob to 7.40 and 36.95 after roasted at160˚C for 30 and 40 min. respectively. Some compounds were identified in roasted carob and also found in dark chocolate such as, Pentan-2-ol which increased from 0.81 to 6.85 and 13.17% for raw and roasted carob for 30 and 40 min., respectively while, 2-Heptanone decreased from 2.61 to 2.54 and 2.11%, methyl propanal from 2.72 to 1.78 and 0.13 and Linalool decreased from 1.02 to 0.04 and 0.03%, respectively. Other compounds were identified with roasted carob such as Nonane-2-one which decreased from 4.33 to 3.71 and 2.55% and Hexanal from 2.01 to 0.29 and 0.27. Overall aroma and flavor profile could be affected with roasting of carob to produce carob powders which may be due to the thermal degradation of the high sugar content and its related reaction pathways rather than to maillard reaction as explained by **Arrighi *et al* (1997).**

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