



## EVALUATION OF ANTIOXIDANT ACTIVITY OF SOME SPICES AND THEIR APPLICATION IN CROISSANT AND FILLING CREAM

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### ABSTRACT

Natural antioxidants have gained considerable interest in recent years for their role in preventing the auto oxidation of fats, oils and fat containing food products. In this study, six spices containing natural antioxidants were evaluated for their retarding fat oxidation compared to butylated hydroxyl toluene (BHT). The yield obtained from water extract of the tested spices ranged between 20% and 50 %. Carob gave the highest yield followed by anise and cinnamon, then caraway and fennel, while ginger gave the lowest yield. The free radical scavenging activities measured by 2-diphenyl-1-picryl-hydrazyl (DPPH) were 88.08%, 81.69%, 79.62%, 78.93%, 73.06%, and 71.50% for cinnamon, anise, carob, caraway ginger, and fennel, respectively at concentration of 2250 µg/ml. Cinnamon showed higher antioxidant activity on linoleic acid oxidation than BHT followed by anise, carob and ginger. While, fennel and caraway recorded closely antioxidant activity to that of BHT at low concentration of 100 µg/ml. Total phenolic content of the tested spices varied ranging from 11.19 to 22.95 mg as gallic acid / gm dry extract in carob and caraway, respectively. Depending upon the sensory evaluation of croissant containing different levels of tested spices, a concentration of 1.5% of anise, caraway fennel and cinnamon was chosen, while the chosen concentration of ginger was 2.5% and carob was 1% for testing their effectiveness against oxidation of lipid in croissant. However, a concentration of 2% of anise, fennel, ginger and carob was chosen while, the chosen concentration of caraway and cinnamon was 1% for testing their effectiveness against oxidation of lipid in filling cream. Addi-

tion of tested spices gave an excellent antioxidant effect on croissant and filling cream compared with the effect of BHT. The increase in both peroxide and acid values after 14 and 28 days respectively were lower than of control and BHT. Carob, ginger, caraway and cinnamon were more effective in controlling lipid oxidation during storage. In conclusion addition of tested spices as sources of effective natural antioxidants retarded lipid oxidation and maintain the quality of croissant and filling cream during storage.

### INTRODUCTION

Antioxidant compounds in food play important roles as health-protecting factor. Antioxidants are also widely used as additives in fats and oils and in food processing to prevent or delay spoilage of foods. Spices and some herbs have received increased attention as sources of many effective antioxidants, (Suhaj, 2006).

Lipid oxidation and bacterial contamination are the main factor that determine food quality loss and shelf-life reduction. Therefore, delaying lipid oxidation and preventing bacterial cross-contamination are highly relevant to food processors, (Decker, *et al* 1995).

The use of synthetic antioxidants in food products is being questioned, consumers have also become more cautious about the nutritional quality and safety of food additives. In food industry, antioxidants are used to retard the oxidative degradation of fats by inhibiting the formation of free radicals. Synthetic antioxidants, such as butylated hydroxyl toluene (BHT), butylated hydroxyl anisol (BHA) and propylgallate (PG) are widely used. (Shahidi, 2000 and Kosar, *et al* 2007).

There are some serious problems concerning the safety and toxicity of such synthetic antioxidants related to their metabolism and possible ab-

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sorption and accumulation in body organ and tissues. (Linderschmidt, *et al* 1986).

Many antioxidant compounds, naturally occurring in plant sources have been identified as free radicals or active oxygen scavengers. Recently, interest has considerably increased in finding naturally occurring antioxidant for use in foods or medicinal materials to replace synthetic antioxidants, which are being restricted due to their side effects such as carcinogenicity (Zheng and Wang, 2001). There is a growing interest in the use of natural antioxidants for increasing the shelf life of foods. This is mainly due to an obvious consumer preference for natural ingredients combined with concerns about toxic effects of synthetic antioxidant activities. (Puertas-Meji *et al* 2002).

A great number of spices and aromatic herbs contain chemical compounds exhibiting antioxidant properties. These properties are attributed to a variety of active phytochemicals including vitamins, carotenoids, terpenoids, alkaloids, flavonoids, lignin, simple phenols and phenolic acids, etc. (Liu and Ng, 2000). Spices can be add to foods in several forms as whole spices, as ground spices, or as isolates from their extracts. spices are aromatic and pungent food ingredient (Suhaj, 2006).

Anise (*Pimpinella anisum* L.) is an annual herb and a grassy plant with white flowers and small green to yellow seeds. Anise seed, one of its various extracts, or the soluble from of spice is used in seasoning for dry sausage (Farrell, 1985 and Gulcin, *et al* 2003).

Carob has long been eaten as food. A part from being chewed as a sweetmeat, carob pods are processed to a cocoa -like flour which is added to cold or heated milk for drinking. It has been combined with wheat flour in making bread or pancakes and used in ice cream, salad dressings, sauces, canned meats and fish, jelly, mustard, and other food products. (Morton, 1987 and Grealley *et al* 1992).

Ginger is the common name for the monocotyledonous perennial plant *Zingiber officinale*, the edible part of the plant is commonly used as aspic in some parts of the Middle East its powder is used as a spice for coffee, (Taghizadeh, 2007). Cinnamon (*Cinnamomum zeylanicum*) is reddish-brown and has a warm, spicy, woody aromatic and used commercially in confections, ice cream, chewing gums and sauces. Cinnamon are often used as flavoring cereals (Farrell, 1985 and Archer, 1988).

Fennel (*Foeniculum vulgare*) flavor has been described as being warm, spicy, anise like, green herbaceous. Fennel seed is used in soups, sausage, tomato dishes and meat. Caraway seed (*carum carvi* L.) have a characteristic agreeable odor, and aromatic, pleasant, warm, sharp test. Caraway seed is used in many of baked goods-breads, roast goose, seafoods and cabbage, potato soups (Farrell, 1985).

Reddy, *et al* (2005) evaluated the antioxidant activity of some plant extracts and their application in biscuits and found that the addition of extracts of amla (*Embllica officianalis*), drumstick leaves (*Moringa oleifera* ) and raisins (*Vitis vinifera*) gave an excellent antioxidant effect on the biscuits compared with the effect of BHA.

Cake manufactures face a major problem of lipid oxidation and mould growth which limits the shelf-life of their products. The use of antioxidants and preservatives can reduce this problem (Lean and Mohamed, 1999).

The objective of this work was to evaluate the effect of six spices ( anise ,caraway ,fennel , cinnamon, ginger and carob ) as antioxidant compared with ( BHT) and to investigate the possibility of their application at the best selected concentration as natural antioxidant in croissant and filling cream.

## MATERIALS AND METHODS

### 1. Materials

#### 1.1. Spices

Anise, caraway, fennel, cinnamon, ginger and carob were purchased from local market at Cairo.

#### 1.2. Croissant and filling cream ingredients

Wheat flour was obtained from Cairo South Company of Milling (EL-Haram Milling). Corn oil, fine sucrose (commercial grade), fresh yeast, salt, shortening and margarine were purchased from local market, Cairo. Egypt. Non fat milk was obtained from Dena farm.

#### 1.3. Chemicals

Potassium sorbet was obtained from Gomhoria, Company, Egypt. Folin-Ciocalte Reagent, butylated hydroxyl toluene (BHT), 2, 2-diphenyl-1-picryl-hydrazyl (DPPH), gallic acid, linoleic fatty acid were obtained from Sigma-Aldrich Chime, Germany.

## 2. Methods

### 2.1. Preparation of water extracts and its yield of tested spices

For water extraction, 1g sample of spice in a fine powder form was extracted with 20 ml boiling water using magnetic stirrer for 15 min then, the extract was filtered over whatman No. 1 paper (Gulcin, *et al* 2003). The filtrate was then dried at 70°C in air oven and the yield was calculated in percent of spice.

### 2.2. Evaluation of antioxidant activity of tested spices

Antioxidant activity of tested spices were tested individually in comparison with a synthetic antioxidant (BHT) as follows:-

#### 2.2.1. Free radical scavenging activity (DPPH test)

The free radical scavenging activity of tested spices was measured by 2,2 diphenyl-1-picrylhydrazil (DPPH) method according to **Tepe *et al* (2005)** One ml of 0.1mM methanolic solution of DPPH was added to 3ml of various concentrations of each spices (250 -2250 µg spice/ml). After 30 min incubation period at ambient temperature, the absorbance of the reaction was measured against a blank at 517 nm using spectrophotometer Shimadzu UIV-1201 (Shimadzu Co., Ltd., Kyoto, Japan). Lower absorbance of the reaction mixture indicates higher free radical scavenging activity. Inhibition of free radical scavenging in percent was calculated using the following equation:-

$$\text{Inhibition \%} = (A_0 - A_s / A_0) \times 100$$

Where:

**A<sub>0</sub>** : the absorbance of control

**A<sub>s</sub>** : the absorbance of the test sample

#### 2.2.2. Linoleic acid emulsion test

Antioxidant activity of tested spices was determined using a diene conjugated formation (linoleic acid emulsion) method according to **Lingnert, *et al* (1979)**. A substrate consisted of 2.86g of linoleic acid emulsified with an equal amount of Tween 20 in phosphate buffer, pH 7.0 (0.1mol/ l). at high speed for 1 min. Different concentrations of each spice (100, 200, 400 and 600 µg spice/ml) were mixed with 5 ml emulsions and incubated at 50°C for 20 h . Absorbance was then measured at

234 nm using spectrophotometer Shimadzu UIV-1201 (Shimadzu Co., Ltd., Kyoto, Japan).

#### 2.2.3. Determination of total phenolic compounds

Total phenolic compounds in water extracts of tested spices were determined with Folin- Cioclteu reagent according to the method of **Slinkard and Singleton (1977)**. One ml of water extract of each spice was mixed thoroughly in test tube with 1ml of Folin-Cioclteu reagent. Three minutes later, 3ml of Na<sub>2</sub> CO<sub>3</sub> was added and the mixture was allowed to stand for 2 h with intermittent shaking. The absorbance was measured at 760 nm using spectrophotometer Shimadzu UIV-1201 (Shimadzu Co., Ltd., Kyoto, Japan). The concentration of total phenolic compounds in water extract of spice was determined as milligrams of gallic acid equivalent per gram of dry extract weigh according to **Gulcin, *et al* (2003)**.

## 3. Technological methods

### 3.1. Croissant manufacture

Croissant was prepared using the suggested formula according to **Sternbagen and Hosoney (1994)**.

Wheat flour, corn oil, salt, non fat milk, fine sucrose, fresh yeast and different concentrations of tested spices were mixed with a sufficient amount of water to form dough with constant consistency. The dough was then rested for 15 min, before incorporating 0.4 kg of layering fat (margarine) by envelope method. The paste was rested for another 15 min more and gauged to 10 mm thickness by using sheeter, the sheet was gauged to 8 mm, and then the ends were folded first and then folded again like book. After another 15 min of rest period, the paste was gauged to 7 mm thickness and given two fourfold turns before final gauging to 3.0 mm. The paste was cut into triangular pieces. The pastries were placed into a proofing cabinet and proofed at 85 % humidity and 38°C to 60 min, then the pastries were baked at 85%humidity and 38°C for 60 min, then the pastries were baked at 225°C for 25 min. Thereafter pastries were allowed to cool at room temperature before organoleptic evaluation.

### 3.2. Filling cream manufacture

Filling cream was prepared using the modified formula according to **Jeffery, (1993)**.

### Croissant

Flour	Corn oil	Salt	Fresh yeast	Fine sucrose	Non fat milk	Potassium sorbet	Margarine
1.0 kg	100g	15 g	15g	100g	40g	1.0g	400g

### Filling cream

Fine sucrose	Shortening	Lecithin	Salt	Non fat milk	Potassium sorbet
62.55g	34.78g	0.05g	0.04g	2.48g	0.10

The shortening was creamed for 3 min then fine sucrose was added to the creamed shortening and beaten for 5 min. The other ingredient and different concentrations of tested spices were added gradually on the (sugar-fat mixture) and beaten for 3 min. Thereafter, evaluate the sensory properties of filling cream.

#### 4. Chemical analysis

Moisture content, Peroxide and acid values of extracted lipids from croissant and filling cream containing tested spices were determined according to **A.O.A.C., (2000)**.

#### 5. Sensory evaluation

A10 panelists from the staff members of the Food Science Department. Faculty of Agriculture, Ain Shams. University were asked to evaluate appearance, color, odor, taste and overall acceptability of the processed filling cream. They were also asked to evaluate texture, crust color, crumb color, taste and overall acceptability of the processed croissant according to **Bennion and Bamford, (1983)**.

#### 6. Statistical analysis

The experimental data were analyzed using analysis of variance and Duncan's multiple range test at ( $P < 0.05$ ). The data were analyzed according to User's Guide of Statistical Analysis System at computing center of Faculty of Agriculture, Ain Shams University (**SAS, 1996**).

## RESULTS AND DISCUSSION

### 1. Water extraction Yield of tested spices

The percentage yield obtained from water extract of tested spices is represented in **Fig. (1)**. It

was clear that carob gave the highest yield, followed by anise and cinnamon then caraway and fennel. being 50, 40, 40, 30, 30 and gm / 100gm dry spices respectively while ginger gave the lowest yield, it was 20%. Different findings were reported by **Yen and Chuang, (2002)**, they found the yield obtained from water extract of (*Cassia tora* L.) was 2.58 %. **Kosar, et al (2007)** found that the yield obtained with methanolic extract of sumac was 29.77 %.

### 2. Antioxidant activity of tested spices

Antioxidant activity of tested spices was carried out by two tests, namely DPPH test and linoleic acid emulsion test :-

#### 2.1. DPPH radical scavenging activity

Relatively stable organic radical DPPH has been widely used in determining of antioxidant activity of single compounds as well as different plant extracts. The method is based on the reduction of alcoholic DPPH solution in the presence of a hydrogen donating antioxidant. The reduction capability of DPPH radicals was determined by the decrease in its absorbance at 517 nm, which is induced by antioxidants. (**Yamaguchi et al 1998**).

Free radical- scavenging capacity of different concentrations of some spices measured by DPPH are given in **Fig. (2)**. DPPH radical scavenging activities (%) were increased with increasing concentration of tested spices from (250 to 2250 µg/gm). The water extract of anise at high concentration (2250 µg/gm) showed similar antioxidant activity to that of BHT. Cinnamon at concentrations of 750 and 2250 µg/gm notably reduced significantly DPPH free radical, with an efficacy much greater than the reference BHT being 83.07%, 88.08% and 81.35% respectively. On the other hand, the least DPPH inhibition was recorded by carob 20.50% at concentration of

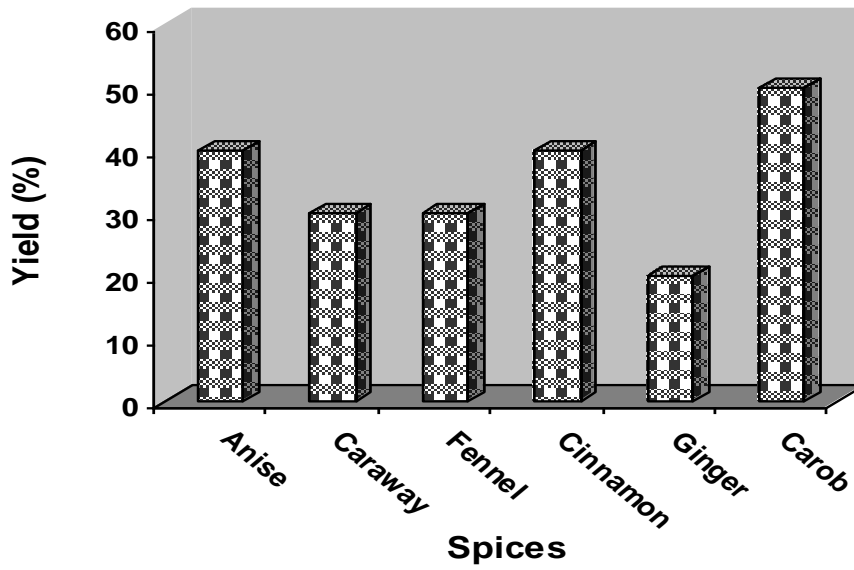


Figure 1. Yield (%) of the tested spices

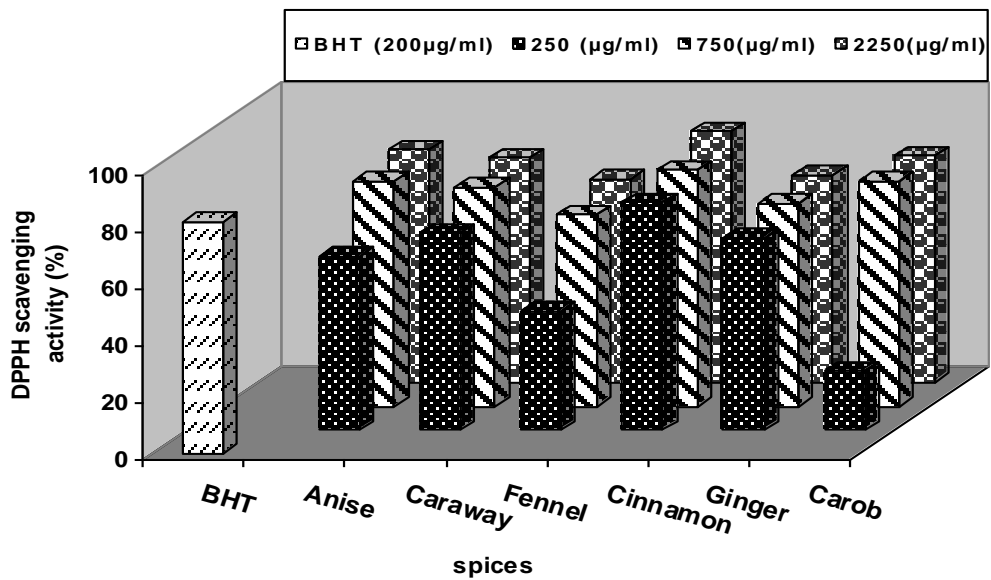


Figure 2. DPPH scavenging activity (%) of different concentrations of tested spices

250 µg/gm. These results are in harmony with those of **Gulcin, et al 2003**) who found that the scavenging effect of water extracts of anise on the DPPH radical decreased which 64.07%. **Su, et al (2007)** mentioned that cinnamon had the greatest capacity to quench DPPH, while black peppercorn had the least ability to quench DPPH.

## 2.2. Linolic acid emulsion test

The antioxidant activity of different concentrations of tested spices in the linoleic acid emulsion is shown in **Fig. (3)**. Antioxidant activity was determined by the decrease in its absorbance at 234 nm, which induced by antioxidant. Increasing the concentration of spices tested up to 100µg/gm emulsion resulted an increase in antioxidant in all samples.

Generally all the concentrations of tested spices recorded higher antioxidant activity compared with BHT. Cinnamon showed the highest antioxidant activity at all concentration followed by anise, carob and ginger. While fennel and caraway recorded closely antioxidant activity which was at the least position. These results are in agreement with those obtained by **Gulcin et al (2003)**. Who found that the antioxidant activity of anise extract increased with increasing concentration.

## 3. Total phenolics content of tested spices

Phenol are very important plant constituents because of their radical scavenging ability due to their hydroxyl group. (**Gulcin, et al 2003**). Typical phenolics that possess antioxidant activity are known to be mainly phenolic acid and flavonoids. Phenolic acid are a major class of phenolic compounds, widely occurring in the fruits, vegetable and spices (**Wojdylo, et al 2007**

The amount of total phenolic in tested spices are given in **Fig. (4)**. The amount of total phenolics, measured by Folin-Cio-Calteu method varied in some spices under this study and ranged from 11.19 to 22.95 mg gallic acid/gm dry extract. Caraway had the highest levels of total phenolics followed by fennel then cinnamon and anise being 22.95, 21.27 19.83 and 19.7 mg as gallic acid/gm dry extract, while carob had the lowest one of total phenolics (11.19 mg gallic acid/gm dry extract). Different results were reported in this aspect whereas some authors found correlation between the polyphenol content and the antioxidant activity, others found no such relationship. (**Moure et al 2001**).

## 4. Application of tested spices in food products

### 4.1. Croissant

#### 4.1.1. Selection of best concentration of tested spices

Statistical analysis of panelist scores for sensory properties of croissant containing different concentrations of tested spices were evaluated to choice their best concentration. The received scores of croissant containing different concentrations of tested spices were statistically analyzed using Duncan's multiple range test and the results are given in **Table (1)**.

No significant difference was observed in all sensory properties of croissant containing 1.0%, 1.5% anise, caraway, fennel or cinnamon as well as 1.5, 2.5% ginger and 1.0% carob compared to control. It was noticed that the addition of more than 1.5% of each of anise, caraway, fennel or cinnamon and more than 1.0 %carob reduced all the sensory properties of croissant. Meanwhile, the increasing the concentration of ginger up to 2.5 %, the increasing the acceptability and mean score values of croissant. Therefore, a concentration of 1.5 % of anise, caraway fennel and cinnamon was chosen to test their effectiveness against oxidation of lipid in croissant while the chosen concentration of ginger was 2.5 % and carob was 1%.

#### 4.1.2. Moisture content of croissant during storage

The moisture content of croissant containing tested spices during storage at room temperature are given in **Table (2)**. Moisture content of croissant ranged between 19.67% ± 0.39 in fennel croissant to 23.30 % ± 0.51 for ginger croissant at zero time. During storage, the moisture content of all samples gradually decreased, it reached a values ranging 13.72 % ± 0.25 for control to 18.14 % ± 0.15in cinnamon croissant after 14 days. The highest losses in moisture content of croissant were observed for control samples and BHT.

#### 4.1.3. Measurements of lipid oxidation

##### 4.1.3.1. Peroxide value (PV)

The primary products of lipid peroxidation are hydroperoxidations. Therefore, determining the concentration of peroxides is one clear index of lipid peroxidation. Changes occurring in PV values in extracted lipids from croissant containing

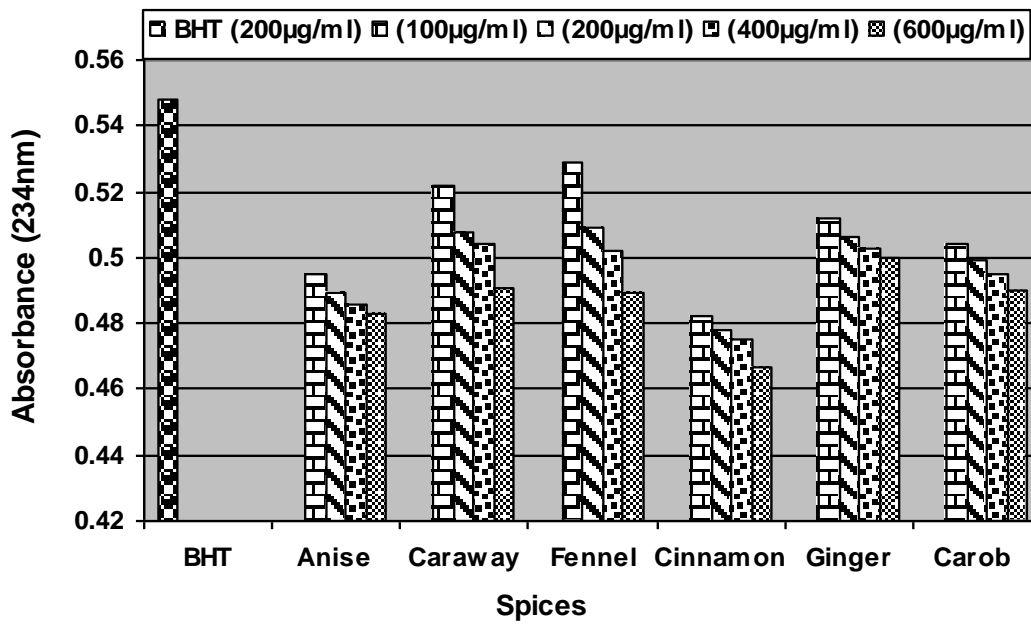


Figure 3. Antioxidant activity of different concentrations of tested spices in the linoleic acid emulsion

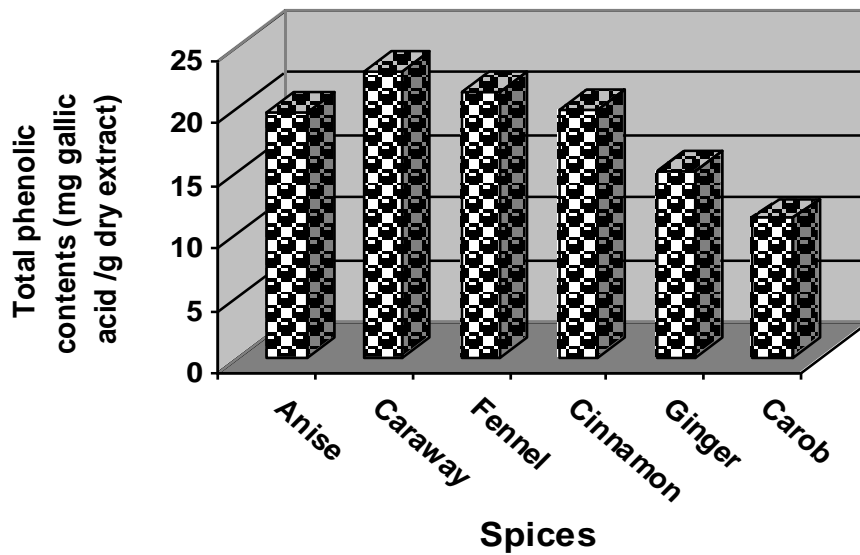


Figure 4. Total phenolic contents of the tested spices

Table 1. Statistical analysis of scores of sensory properties of croissant containing different concentration of some spices

Conc. (%)	Means* of scores of sensory properties					
	Texture	Crust color	Crumb color	Odor	Taste	Overall acceptability
Anise						
Zero	8.6 <sup>A</sup>	8.5 <sup>A</sup>	7.9 <sup>A</sup>	7.8 <sup>A</sup>	8.0 <sup>A</sup>	7.8 <sup>A</sup>
1.0	8.4 <sup>A</sup>	8.3 <sup>A</sup>	8.0 <sup>A</sup>	7.7 <sup>A</sup>	7.8 <sup>A</sup>	7.7 <sup>A</sup>
1.5	7.9 <sup>A</sup>	8.2 <sup>A</sup>	8.2 <sup>A</sup>	8.2 <sup>A</sup>	8.3 <sup>A</sup>	8.1 <sup>A</sup>
2.5	6.9 <sup>B</sup>	6.7 <sup>B</sup>	6.4 <sup>B</sup>	6.2 <sup>B</sup>	6.4 <sup>B</sup>	5.9 <sup>B</sup>
Caraway						
Zero	8.6 <sup>A</sup>	8.5 <sup>A</sup>	7.9 <sup>A</sup>	7.8 <sup>A</sup>	8.0 <sup>A</sup>	7.8 <sup>A</sup>
1.0	7.5 <sup>B</sup>	8.1 <sup>A</sup>	7.8 <sup>AB</sup>	8.0 <sup>A</sup>	7.9 <sup>A</sup>	8.2 <sup>A</sup>
1.5	7.9 <sup>AB</sup>	7.9 <sup>AB</sup>	7.8 <sup>AB</sup>	7.6 <sup>AB</sup>	8.0 <sup>A</sup>	8.2 <sup>A</sup>
2.5	7.3 <sup>B</sup>	7.1 <sup>B</sup>	6.9 <sup>B</sup>	6.7 <sup>B</sup>	6.6 <sup>B</sup>	6.7 <sup>B</sup>
Fennel						
Zero	8.6 <sup>A</sup>	8.5 <sup>A</sup>	7.9 <sup>B</sup>	7.8 <sup>A</sup>	8.0 <sup>BC</sup>	7.8 <sup>BC</sup>
1.0	8.3 <sup>A</sup>	8.4 <sup>A</sup>	8.3 <sup>AB</sup>	8.5 <sup>A</sup>	8.2 <sup>AB</sup>	8.4 <sup>AB</sup>
1.5	8.6 <sup>A</sup>	8.6 <sup>A</sup>	8.6 <sup>A</sup>	8.5 <sup>A</sup>	8.8 <sup>A</sup>	8.7 <sup>A</sup>
2.5	7.7 <sup>B</sup>	7.5 <sup>B</sup>	7.7 <sup>B</sup>	7.9 <sup>A</sup>	7.4 <sup>C</sup>	7.3 <sup>C</sup>
Cinnamon						
Zero	8.6 <sup>A</sup>	8.5 <sup>A</sup>	7.9 <sup>A</sup>	7.8 <sup>B</sup>	8.0 <sup>A</sup>	7.8 <sup>B</sup>
1.0	8.5 <sup>A</sup>	8.4 <sup>A</sup>	8.4 <sup>A</sup>	8.4 <sup>AB</sup>	8.3 <sup>A</sup>	8.4 <sup>A</sup>
1.5	8.1 <sup>A</sup>	8.3 <sup>A</sup>	8.0 <sup>A</sup>	8.5 <sup>A</sup>	8.6 <sup>A</sup>	8.6 <sup>A</sup>
2.5	7.5 <sup>B</sup>	7.2 <sup>B</sup>	6.2 <sup>B</sup>	6.8 <sup>C</sup>	6.9 <sup>B</sup>	6.9 <sup>C</sup>
Ginger						
Zero	8.6 <sup>A</sup>	8.5 <sup>A</sup>	7.9 <sup>B</sup>	7.8 <sup>AB</sup>	8.0 <sup>AB</sup>	7.8 <sup>AB</sup>
1.0	7.9 <sup>B</sup>	7.8 <sup>B</sup>	7.7 <sup>B</sup>	7.4 <sup>B</sup>	7.4 <sup>B</sup>	7.2 <sup>B</sup>
1.5	8.2 <sup>AB</sup>	8.3 <sup>AB</sup>	8.0 <sup>AB</sup>	8.1 <sup>AB</sup>	7.8 <sup>AB</sup>	8.3 <sup>A</sup>
2.5	8.5 <sup>A</sup>	8.4 <sup>AB</sup>	8.6 <sup>A</sup>	8.3 <sup>A</sup>	8.4 <sup>A</sup>	8.4 <sup>A</sup>
Carob						
Zero	8.6 <sup>A</sup>	8.5 <sup>A</sup>	7.9 <sup>A</sup>	7.8 <sup>AB</sup>	8.0 <sup>A</sup>	7.8 <sup>AB</sup>
1.0	7.8 <sup>AB</sup>	8.0 <sup>AB</sup>	7.7 <sup>A</sup>	8.2 <sup>A</sup>	8.2 <sup>A</sup>	8.3 <sup>A</sup>
1.5	7.5 <sup>BC</sup>	7.5 <sup>BC</sup>	7.0 <sup>B</sup>	7.4 <sup>BC</sup>	7.0 <sup>B</sup>	7.1 <sup>B</sup>
2.5	6.7 <sup>C</sup>	7.0 <sup>C</sup>	7.0 <sup>B</sup>	6.9 <sup>C</sup>	6.2 <sup>C</sup>	6.2 <sup>C</sup>

\*: Means in a columns showing the same letter are not significantly different (  $p > 0.05$  )



Table 2. Moisture content (%) of croissant containing tested spices during storage at room temperature

Spices	Moisture content ( % ) during storage periods ( days)				
	Zero	2	6	10	14
Control	20.27 ± 0.34	18.97 ± 0.22	18.37 ± 0.03	16.19 ± 0.13	13.72 ± 0.25
BHT	20.18 ± 0.14	18.57 ± 0.19	17.8 ± 0.17	15.61 ± 0.22	15.96 ± 0.03
Anise	20.82 ± 0.51	17.84 ± 0.39	18.35 ± 0.14	17.28 ± 0.28	17.01 ± 0.10
Caraway	20.49 ± 0.55	19.35 ± 0.12	16.87 ± 0.07	16.18 ± 0.10	16.08 ± 0.09
Fennel	19.67 ± 0.39	18.77 ± 0.32	16.44 ± 0.02	16.06 ± 0.02	15.97 ± 0.08
Cinnamon	21.52 ± 0.56	20.19 ± 0.29	18.22 ± 0.41	18.18 ± 0.25	18.14 ± 0.15
Ginger	22.30 ± 0.51	17.88 ± 0.10	17.62 ± 0.19	16.80 ± 0.13	16.04 ± 0.19
Carob	21.79 ± 0.62	19.70 ± 0.05	18.80 ± 0.06	16.29 ± 0.35	16.12 ± 0.20

Data expressed as mean of 3 replicates ± standard deviation

the chosen concentrations of tested spices during storage at room temperature are given in **Fig. (5)**. All The six natural antioxidant used and BHT were slowed down the rate of peroxide formation. The PV of all samples which contained synthetic antioxidant (BHT) or natural antioxidants were lower than that of control sample during storage for 14 days of storage at room temperature. Croissant which contained the tested spices showed lower PV ranged from 2.3 to 3.5 meq /kg oil, compared to 5 meq /kg oil of control after 14 days of storage. These results are in agreement with those of **Lean and Mohamed, (1999)**.

#### 4.1.3.2. Acid value (AV)

The acid value of croissant containing the best chosen concentrations of tested spices during storage at room temperature is summarized in **Fig. (6)**. An increase in AV was observed in all croissant samples during storage. The increase was considerably higher in control sample compared to croissant containing the tested spices. After 14 days of storage the values were 3.13, 1.63 and 1.13 for control sample, BHT and carob respectively, while the AV of other samples ranged between 1.25 to 2.53. These result are accordance with those of **Reddy, et al (2005)**.

#### 4.1.4. Sensory evaluation of croissant during storage

The sensory evaluation of croissant appears to be necessary since the addition of natural antioxi-

dants, and BHT increased shelf life of product by. For these reasons, a questionnaire was conducted to detect the acceptability of croissant samples. The sensory properties of croissant containing the best concentration of the tested spices during storage at room temperature are given in **Table (3)**. Croissant containing cinnamon, caraway and fennel showed significantly superior overall acceptability than control and other samples after 14 days. The minimum reduction in overall acceptability was observed after 6 days in croissant containing BHT and control samples. It can be noticed that croissant containing cinnamon, caraway, fennel and carob were comparable to those sample containing BHT and control sample being able to maintain the quality of croissant during storage for 14 days.

## 4.2. Filling cream

### 4.2.1. Selection of best concentration of tested spices

Statistical analysis of panelist scores for sensory properties of filling cream containing different concentration of tested spices were statistically analyzed to choice the best concentration of spices and the results are given in **Table (4)**.

It was observes that with increasing the addition level of anise, fennel, ginger and carob up to 2%, the sensory properties improved and received the highest score values. Concerning caraway and cinnamon, the panelist prefer the filling cream

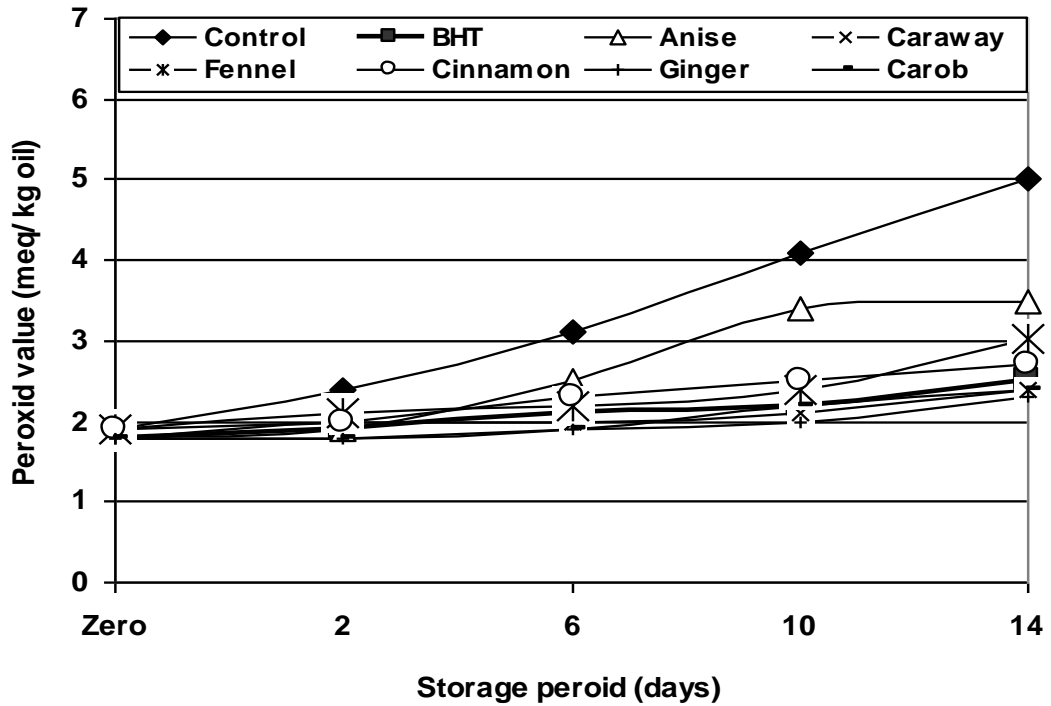


Figure 5. Peroxide value of croissant containing tested spices during storage at room temperature

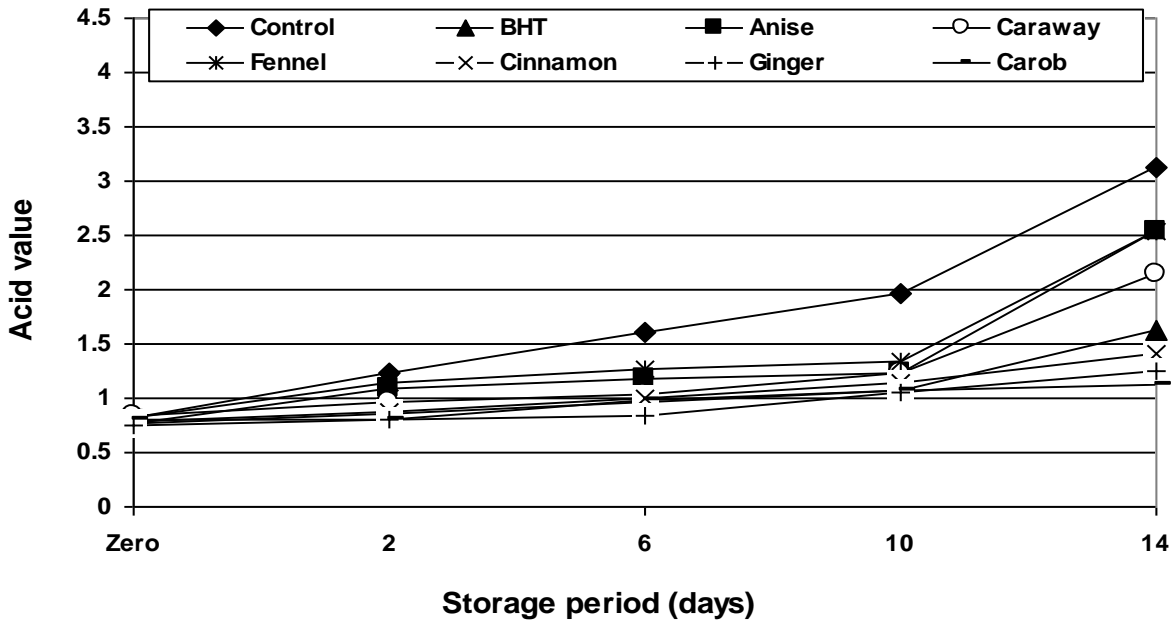


Figure 6. Acid value of croissant containing different spices during storage at room temperature

Table 3. Statistical analysis of scores of sensory properties of croissant containing tested spices during storage at room temperature\*

Storage periods (days)	Croissant containing							
	Control (zero)	BHT (0.02%)	Anise (1.5%)	Caraway (1.5%)	Fennel (1.5%)	Cinnamon (1.5%)	Ginger (2.5%)	Carob (1.0%)
Texture								
Zero	8.6 <sup>Aa</sup>	8.5 <sup>ABa</sup>	7.9 <sup>BCa</sup>	7.9 <sup>BCa</sup>	8.6 <sup>Aa</sup>	8.1 <sup>ABCa</sup>	8.6 <sup>Aa</sup>	7.8 <sup>Ca</sup>
2	8.4 <sup>ABa</sup>	8.6 <sup>Aa</sup>	7.7 <sup>CDab</sup>	8.0 <sup>BCDa</sup>	8.6 <sup>Aa</sup>	8.2 <sup>ABCa</sup>	8.6 <sup>ABa</sup>	7.6 <sup>Dab</sup>
6	6.2 <sup>Eb</sup>	6.7 <sup>Deb</sup>	7.1 <sup>CDbc</sup>	6.8 <sup>Db</sup>	7.5 <sup>BCb</sup>	7.4 <sup>BCb</sup>	8.4 <sup>Aa</sup>	7.7 <sup>Ba</sup>
10	5.5 <sup>Dc</sup>	5.5 <sup>Dc</sup>	6.5 <sup>Ccd</sup>	6.8 <sup>BCb</sup>	6.5 <sup>Cc</sup>	7.4 <sup>Ab</sup>	7.4 <sup>Ab</sup>	7.1 <sup>ABbc</sup>
14	4.4 <sup>Ed</sup>	5.5 <sup>Dc</sup>	5.9 <sup>CDd</sup>	6.6 <sup>Bb</sup>	6.3 <sup>BCc</sup>	6.7 <sup>Bc</sup>	7.2 <sup>Ab</sup>	6.6 <sup>Bc</sup>
Crust color								
Zero	8.4 <sup>ABa</sup>	8.5 <sup>ABa</sup>	8.2 <sup>ABa</sup>	7.9 <sup>Ba</sup>	8.6 <sup>Aa</sup>	8.3 <sup>AB a</sup>	8.4 <sup>ABa</sup>	8.0 <sup>ABa</sup>
2	8.4 <sup>ABa</sup>	8.5 <sup>Aa</sup>	8.3 <sup>ABa</sup>	8.0 <sup>ABa</sup>	8.3 <sup>ABa</sup>	8.0 <sup>ABa</sup>	8.4 <sup>ABa</sup>	7.9 <sup>Ba</sup>
6	7.5 <sup>Ab</sup>	6.7 <sup>CDb</sup>	7.4 <sup>ABb</sup>	6.9 <sup>BCDb</sup>	7.6 <sup>Ab</sup>	6.6 <sup>Db</sup>	7.3 <sup>ABCb</sup>	7.3 <sup>ABb</sup>
10	7.2 <sup>Ab</sup>	6.5 <sup>Bb</sup>	6.6 <sup>Bbc</sup>	7.2 <sup>Ab</sup>	7.3 <sup>Abc</sup>	6.9 <sup>ABb</sup>	7.3 <sup>Ab</sup>	7.3 <sup>Ab</sup>
14	6.4 <sup>Cc</sup>	6.4 <sup>Cb</sup>	7.0 <sup>Ac</sup>	6.8 <sup>ABb</sup>	7.1 <sup>Ac</sup>	6.6 <sup>BCb</sup>	7.0 <sup>Ab</sup>	7.0 <sup>Ab</sup>
Crumb color								
Zero	7.9 <sup>BCa</sup>	8.8 <sup>Aa</sup>	8.3 <sup>ABa</sup>	7.8 <sup>BCab</sup>	8.6 <sup>Aa</sup>	8.0 <sup>BCa</sup>	8.6 <sup>Aa</sup>	7.7 <sup>Ca</sup>
2	8.0 <sup>Ca</sup>	8.7 <sup>Aa</sup>	8.0 <sup>Ca</sup>	7.9 <sup>Ca</sup>	8.6 <sup>ABa</sup>	8.2 <sup>ABCa</sup>	8.1 <sup>BCa</sup>	7.8 <sup>Ca</sup>
6	7.0 <sup>Bbc</sup>	7.1 <sup>ABb</sup>	7.4 <sup>ABb</sup>	7.3 <sup>ABbc</sup>	7.5 <sup>Ab</sup>	6.4 <sup>Cb</sup>	7.3 <sup>ABb</sup>	7.3 <sup>ABab</sup>
10	7.2 <sup>Ab</sup>	6.4 <sup>Bc</sup>	6.7 <sup>ABc</sup>	7.0 <sup>Adc</sup>	7.2 <sup>Ab</sup>	6.8 <sup>ABb</sup>	6.9 <sup>ABb</sup>	6.9 <sup>ABb</sup>
14	6.6 <sup>BCc</sup>	6.3 <sup>Cc</sup>	6.7 <sup>BCc</sup>	6.6 <sup>BCd</sup>	7.3 <sup>Ab</sup>	6.6 <sup>BCb</sup>	7.3 <sup>Ab</sup>	7.0 <sup>ABb</sup>
Odor								
Zero	7.9 <sup>ABa</sup>	7.7 <sup>Ba</sup>	8.2 <sup>ABa</sup>	7.6 <sup>Ba</sup>	8.5 <sup>Aa</sup>	8.5 <sup>Aa</sup>	8.3 <sup>ABa</sup>	8.2 <sup>ABa</sup>
2	7.8 <sup>BCa</sup>	7.9 <sup>BCa</sup>	7.9 <sup>BCab</sup>	7.6 <sup>Ca</sup>	8.3 <sup>ABa</sup>	8.5 <sup>Aa</sup>	7.9 <sup>BCab</sup>	7.9 <sup>BCab</sup>
6	6.3 <sup>Db</sup>	6.8 <sup>Cb</sup>	7.6 <sup>Bb</sup>	7.5 <sup>Ba</sup>	7.3 <sup>Bb</sup>	8.4 <sup>Aa</sup>	7.7 <sup>Bab</sup>	7.4 <sup>Bbc</sup>
10	5.6 <sup>Ec</sup>	6.6 <sup>Db</sup>	6.6 <sup>Dc</sup>	7.5 <sup>ABa</sup>	7.1 <sup>BCDb</sup>	7.7 <sup>Ab</sup>	7.3 <sup>ABCb</sup>	6.9 <sup>CDcd</sup>
14	5.0 <sup>Dd</sup>	5.5 <sup>Cc</sup>	6.7 <sup>Bc</sup>	7.4 <sup>Aa</sup>	7.2 <sup>Ab</sup>	7.5 <sup>Ab</sup>	6.4 <sup>Bc</sup>	6.6 <sup>Bd</sup>
Taste								
Zero	8.0 <sup>BCa</sup>	7.6 <sup>Ca</sup>	8.3 <sup>Aa</sup>	8.0 <sup>BCab</sup>	8.8 <sup>Aa</sup>	8.5 <sup>ABa</sup>	8.4 <sup>ABa</sup>	8.2 <sup>ABCab</sup>
2	8.0 <sup>ABa</sup>	7.7 <sup>Ca</sup>	8.1 <sup>ABa</sup>	8.1 <sup>ABa</sup>	8.4 <sup>Aa</sup>	8.6 <sup>Aa</sup>	8.1 <sup>ABab</sup>	8.4 <sup>Aa</sup>
6	6.1 <sup>Cb</sup>	6.4 <sup>Cb</sup>	7.4 <sup>Bb</sup>	7.4 <sup>Bb</sup>	7.4 <sup>Bb</sup>	8.4 <sup>Aa</sup>	7.7 <sup>Bbc</sup>	7.6 <sup>Bbc</sup>
10	5.6 <sup>Db</sup>	6.6 <sup>Cb</sup>	6.7 <sup>Cc</sup>	7.5 <sup>ABab</sup>	7.3 <sup>ABb</sup>	7.6 <sup>Ab</sup>	7.3 <sup>ABc</sup>	7.0 <sup>BCcd</sup>
14	6.0 <sup>CDb</sup>	5.6 <sup>Dc</sup>	6.7 <sup>Bc</sup>	7.4 <sup>Ab</sup>	7.2 <sup>Ab</sup>	7.4 <sup>Ab</sup>	6.4 <sup>BCd</sup>	6.6 <sup>Bd</sup>
Overall acceptability								
Zero	7.8 <sup>Ba</sup>	8.3 <sup>ABa</sup>	8.1 <sup>ABa</sup>	8.2 <sup>ABa</sup>	8.7 <sup>Aa</sup>	8.6 <sup>Aa</sup>	8.4 <sup>ABa</sup>	8.4 <sup>ABa</sup>
2	8.0 <sup>A a</sup>	8.2 <sup>Aa</sup>	8.0 <sup>Aa</sup>	8.3 <sup>Aa</sup>	8.6 <sup>Aa</sup>	8.5 <sup>Aa</sup>	8.2 <sup>Aa</sup>	8.1 <sup>Aa</sup>
6	6.3 <sup>Cb</sup>	6.7 <sup>Cb</sup>	7.3 <sup>Bb</sup>	7.5 <sup>Bb</sup>	7.6 <sup>Bb</sup>	8.2 <sup>Aab</sup>	7.4 <sup>Bb</sup>	7.6 <sup>Bb</sup>
10	6.1 <sup>Dbc</sup>	6.4 <sup>Db</sup>	7.2 <sup>BCb</sup>	7.4 <sup>BCb</sup>	7.1 <sup>Cc</sup>	8.0 <sup>Ab</sup>	7.5 <sup>Bb</sup>	7.2 <sup>BCbc</sup>
14	5.6 <sup>Cc</sup>	5.8 <sup>Cc</sup>	6.7 <sup>Bb</sup>	7.4 <sup>Ab</sup>	7.2 <sup>Abc</sup>	7.3 <sup>Ac</sup>	6.7 <sup>Bc</sup>	7.0 <sup>ABc</sup>

\*Means in a row showing the same capital letters are not significantly different ( $p > 0.05$ )\*Means in a column showing the same small letters are not significantly different ( $p > 0.05$ )

Table 4. Statistical analysis of scores of sensory properties of filling cream containing different concentration of some spices

Conc. (%)	Means* of scores of sensory properties				
	Appearance	Color	Odor	Taste	Overall acceptability
Anise					
Zero	8.7 <sup>A</sup>	8.7 <sup>A</sup>	8.4 <sup>A</sup>	8.3 <sup>B</sup>	8.6 <sup>AB</sup>
0.5	8.3 <sup>A</sup>	8.2 <sup>A</sup>	7.7 <sup>B</sup>	8.0 <sup>B</sup>	8.1 <sup>B</sup>
1	8.9 <sup>A</sup>	8.6 <sup>A</sup>	8.8 <sup>A</sup>	8.5 <sup>AB</sup>	8.7 <sup>AB</sup>
2	8.4 <sup>A</sup>	8.5 <sup>A</sup>	8.6 <sup>A</sup>	9.0 <sup>A</sup>	8.9 <sup>A</sup>
Caraway					
Zero	8.7 <sup>A</sup>	8.7 <sup>A</sup>	8.4 <sup>A</sup>	8.3 <sup>AB</sup>	8.6 <sup>A</sup>
0.5	8.4 <sup>A</sup>	8.6 <sup>A</sup>	7.6 <sup>B</sup>	7.7 <sup>B</sup>	7.7 <sup>B</sup>
1	8.4 <sup>A</sup>	8.7 <sup>A</sup>	8.7 <sup>A</sup>	8.7 <sup>A</sup>	8.8 <sup>A</sup>
2	7.5 <sup>B</sup>	7.3 <sup>B</sup>	7.0 <sup>B</sup>	6.8 <sup>C</sup>	6.6 <sup>C</sup>
Fennel					
Zero	8.7 <sup>A</sup>	8.7 <sup>A</sup>	8.4 <sup>A</sup>	8.3 <sup>AB</sup>	8.6 <sup>A</sup>
0.5	8.2 <sup>A</sup>	8.3 <sup>A</sup>	7.9 <sup>A</sup>	7.4 <sup>C</sup>	7.2 <sup>B</sup>
1	8.1 <sup>A</sup>	8.3 <sup>A</sup>	8.1 <sup>A</sup>	7.6 <sup>BC</sup>	7.5 <sup>B</sup>
2	8.3 <sup>A</sup>	8.3 <sup>A</sup>	8.6 <sup>A</sup>	8.6 <sup>A</sup>	8.7 <sup>A</sup>
Cinnamon					
Zero	8.7 <sup>A</sup>	8.7 <sup>A</sup>	8.4 <sup>A</sup>	8.3 <sup>A</sup>	8.6 <sup>A</sup>
0.5	8.3 <sup>A</sup>	8.1 <sup>A</sup>	8.4 <sup>A</sup>	8.4 <sup>A</sup>	8.4 <sup>A</sup>
1	8.7 <sup>A</sup>	7.9 <sup>A</sup>	8.5 <sup>A</sup>	8.5 <sup>A</sup>	8.6 <sup>A</sup>
2	7.1 <sup>B</sup>	6.3 <sup>B</sup>	6.6 <sup>B</sup>	6.9 <sup>B</sup>	6.9 <sup>B</sup>
Ginger					
Zero	8.7 <sup>A</sup>	8.7 <sup>A</sup>	8.4 <sup>A</sup>	8.3 <sup>A</sup>	8.6 <sup>A</sup>
0.5	7.6 <sup>B</sup>	7.2 <sup>C</sup>	5.9 <sup>B</sup>	6.1 <sup>B</sup>	6.1 <sup>C</sup>
1	7.8 <sup>B</sup>	7.6 <sup>BC</sup>	7.8 <sup>A</sup>	7.6 <sup>A</sup>	7.7 <sup>B</sup>
2	8.2 <sup>AB</sup>	8.3 <sup>AB</sup>	7.9 <sup>A</sup>	8.3 <sup>A</sup>	8.2 <sup>AB</sup>
Carob					
Zero	8.7 <sup>A</sup>	8.7 <sup>A</sup>	8.4 <sup>A</sup>	8.3 <sup>A</sup>	8.6 <sup>A</sup>
0.5	7.7 <sup>B</sup>	7.9 <sup>B</sup>	6.0 <sup>C</sup>	6.0 <sup>C</sup>	5.7 <sup>C</sup>
1	7.4 <sup>B</sup>	7.7 <sup>B</sup>	7.2 <sup>B</sup>	7.5 <sup>B</sup>	7.3 <sup>B</sup>
2	8.1 <sup>AB</sup>	8.2 <sup>AB</sup>	8.3 <sup>AB</sup>	8.2 <sup>AB</sup>	8.3 <sup>A</sup>

\*: Means in a columns showing the same letter are not significantly different ( $p > 0.05$ )

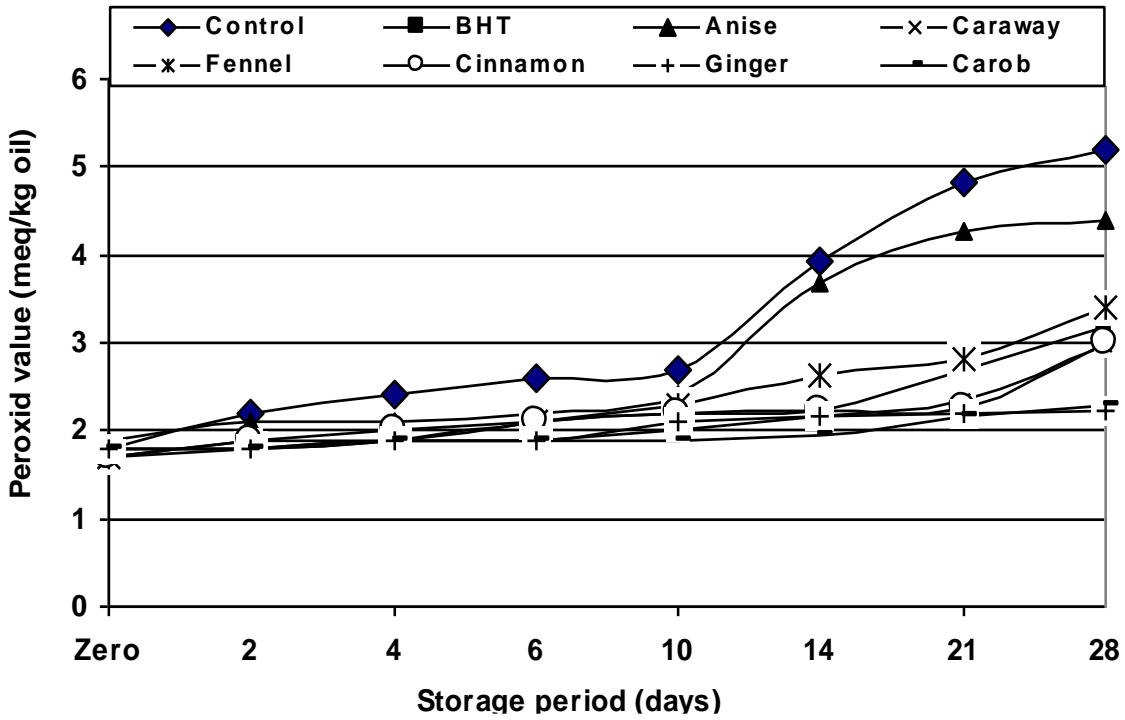


Figure 7. Peroxid values of filling cream containing tested spices during storage at room temperature

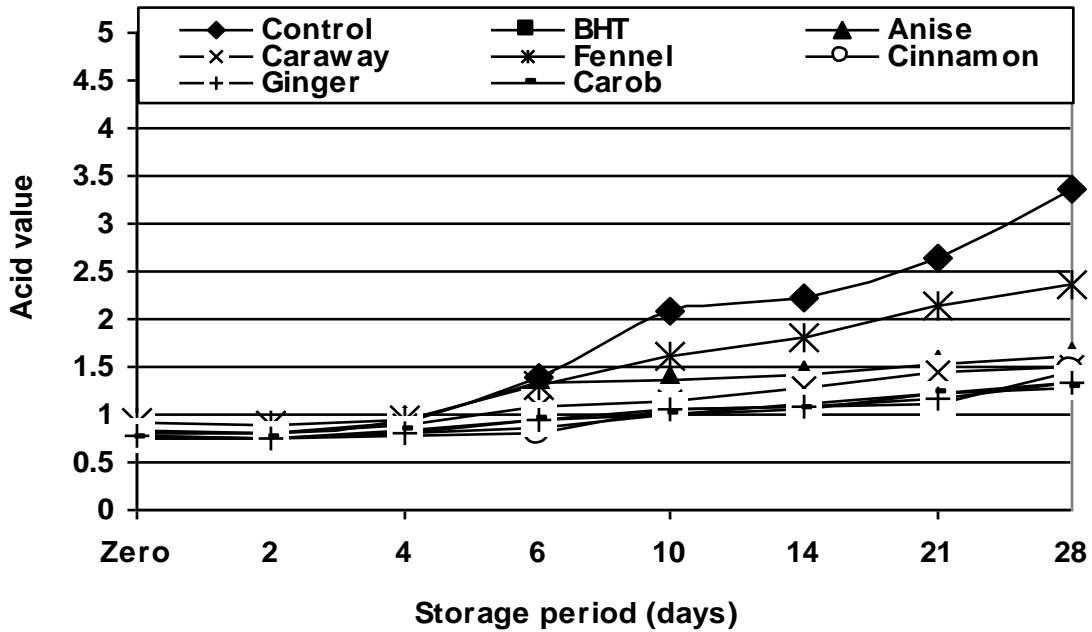


Figure 8. Acid value of filling cream containing some spices during storage at room temperature





containing 1% of each, increasing the added level more than 1%, it impaired the filling cream acceptability. Therefore, the concentration of 2 % of anise, fennel, ginger and carob was chosen to test their effectiveness against oxidation of lipid in filling cream, while the chosen concentration of caraway and cinnamon was 1%.

#### 4.2.2. Moisture content of filling cream during storage

Moisture content of filling cream containing some spices showed a stable or slightly increase during storage for 28 days. It ranged between (0.60 to 0.82 %) from zero to 28 day respectively.

#### 4.2.3. Measurements of lipid oxidation

##### 4.2.3.1. Peroxide value (PV)

Changes occurring in PV values of the filling cream containing the best chosen concentrations of tested spices during storage at room temperature are given in **Fig. (7)**. All samples showed an increase in PV with increasing storage period. PV of lipid extracted from control sample reached 5.2 meq/kg oil after 28 days. The lower PV was observed in the filling cream containing ginger, carob and caraway being 2.22, 2.30 and 2.96 meq/kg oil respectively compared with synthetic antioxidant BHT (3.18 meq/kg oil), after 28 days of storage. These results are in accordance with those of **Reddy, et al (2005)** who mentioned that the peroxide value of biscuits containing plant extracts increase with increased time of storage compared with control sample.

##### 4.2.3.2. Acid value (AV)

The acid value of filling cream containing the best chosen concentrations of tested spices during storage at room temperature is summarized in **Fig. (8)**. The AV of all samples increased with increasing the storage period. The highest AV was observed in control sample and filling cream containing anise and fennel being 3.36, 1.61 and 2.35, respectively after 28 days. Filling cream containing carob and ginger recorded the same effect of BHT. These results are similar to that of **Karpinska et al (2001)**.

##### 4.2.4. Sensory evaluation of filling cream during storage

The sensory properties of filling cream containing the chosen concentrations of the tested

spices during storage at room temperature are given in **Table (5)**. Filling cream containing cinnamon, caraway and carob showed significantly superior overall acceptability than control and other samples after 28 days. The minimum reduction in overall acceptability was observed after 6 days in filling cream containing BHT and control sample. It can be noticed that filling cream containing cinnamon, caraway, ginger and carob were comparable to those sample containing BHT and control sample being able to maintain the quality of filling cream during storage for 28 day.

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