



## Research Article

# Efficiency of Micro and Nano Encapsulated Orange Peel Essential Oils on Quality of Sponge Cake

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### Abstract

**Background and Objective:** Orange peel essential oil is commonly used as flavoring agents in food products and it contains bioactive compounds such as anti-oxidants and others. This study was aimed to recover bioactive compounds extracted from orange peel, where it features as a cheaper waste in the form of micro and nano-encapsulated to prevent rancidity of fat during storage of bakery products and improving their physical properties. **Materials and Methods:** Bioactive compounds (Essential oil) were extracted from orange peel and prepared in micro and nano-encapsulation form; it was added to a sponge cake and the chemical-physical properties of this sponge cake were evaluated. **Results:** Results showed that chemical composition of mixed cakes with non-encapsulate or encapsulated orange peel EO was not significantly affected other than moisture. The color parameters were significantly affected compared to control sample. Weight and volume of sponge cakes mixed with orange peel EO were increased. While, the values of hardness and cohesiveness were lower due to the softened of texture compared to control. Results indicated that the nano-encapsulated orange peel EO can be used in food applications successfully without any change in its characteristics with any loss of its anti-oxidant activity. Storage for 15 days did not affect the anti-oxidant activities for both micro and nano-encapsulate EO. **Conclusion:** The outcome of this study indicated that the use of orange peel EO micro and nano-encapsulation forms as natural anti-oxidants enhance their quality and stability and improve their properties.

**Key words:** Micro-encapsulation, nano-encapsulation, flavoring agents, orange peel essential oil, sponge cake, bakery products, bioactive compounds, cheaper waste

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**Data Availability:** All relevant data are within the paper and its supporting information files.

## INTRODUCTION

Fat and oils are the main ingredients of many foods, especially bakery products, which contain a large amount of fat and are often needed to develop the flavor, texture and color attributes of those products. These fats and oils contain unsaturated fatty acids and are therefore highly susceptible to oxidation causing an oxidative degradation of food products. Oxidative degradation of fats and oils reduces the shelf life of fat-containing food safety and nutritional quality. These problems are met with thus during storage hence needed it to maintain the oxidation stability of fats and oils, increase the shelf life of foods<sup>1</sup>.

Orange peel essential oil is commonly used as a flavoring agent in food products (e.g., pickles, pastries, beverages, cheese and bread) and as a component in pharmaceutical products<sup>2</sup>.

Orange has been recognized as an environmental scourge of the community. Orange peels are the source of natural antioxidants. It is valuable to use these wastes at a cheap and efficient source and with minimal costs. Polyphenols are often present in a reasonable concentration in the nonedible outer part of the fruit compared to the edible inner part<sup>3</sup>. The use of orange peel as a source of antioxidants may be of great economic benefit to food manufacturers.

Synthetic antioxidants have been used to delay or reduce oxidative degradation of foods. Some synthetic antioxidants have been found to be carcinogenic and therefore discourage their use in the food additives. This triggers interest in seeking safer ways of natural antioxidants such as using plant origin that would serve the same purpose of preventing oil rancidity<sup>4</sup>. The replacement of synthetic antioxidants by natural substances may be beneficial because of health effects and functions, potential nutritional benefits, therapeutic effects and melting of both oil and water, which are important as emulsifiers in the diets<sup>5</sup>.

Increased awareness of consumers regarding natural foods and safety of food additives has forced the food industry to create a need to identify natural and possibly safer sources of food antioxidants. Natural antioxidants include various products to delay oxidative degradation of fat, improve the quality and nutritional value of food. Therefore, the investigation of natural antioxidants has been a major research interest such as the use of orange peel on other plant material for possible antioxidant potential<sup>6</sup>. Residual wastes of orange after processing represent approximately 50%. They

contain bioactive compounds capable of improving health by preventing diseases. They serve as antioxidants, reducing platelet aggregation and stimulate the immune system. It posses antibacterial, antiviral and modulate the detoxification enzymes<sup>7-9</sup>.

Orange fruit seeds are good sources of vegetable oils rich in phenolic compounds, carotenoids, tocopherols and phytosterols<sup>10</sup>.

Micro-encapsulation technology is widely used in several industries, especially food industries, since it can increase solubility, enhance stability and improve the controlled release properties of flavor compounds, color and texture properties. Also to extend the shelf-life of products such as antioxidants, essential oils and enzymes etc<sup>11</sup>.

The aim of this study was to use orange peel as a by-product of the factories to extract bioactive compounds that have the ability to prevent oxidation of fat, when used in bakery products. Nano and micro-encapsulation orange peel essential oil was made to maintain the efficiency of these active compounds. Physico-chemical properties and sensory evaluation of sponge cake products was studies.

## MATERIALS AND METHODS

**Materials:** 2,2-diphenyl-1-picryl hydrazyl (DPPH), sodium alginate, 2-thiobarbituric acid, n-hexane, Folin-Ciocalteu's reagent, isopropanol, potassium hydroxide, chloroform-glacial acetic acid, sodium thiosulphate, potassium ferricyanide and trichloroacetic acid were obtained from Sigma Aldrich, Germany.

### Methods

**Isolation of essential oil from orange peel:** One hundred gram of crushed dried plant materials (orange peel) were subjected to 3 h of hydrodistillation using Clevenger-type apparatus to yield the essential oil (HedaS, Model 2000, 220 V, 50 Hz, 500 W), National Research Centre (NRC).

### Preparation of micro and nano-encapsulation of orange peel Essential Oil (EO)

**Micro-encapsulation:** Micro-encapsulation of oil was conducted using emulsion extrusion technique described by Voncina *et al.*<sup>12</sup>. Sodium alginate was dissolved in distilled water to produce polymer solutions with a concentration of 2% w/v; the solution was left standing for 3 h to disengage bubble before use. Afterwards, polymer solution (100 mL)

and EO (1 mL) were homogenized into a 200 mL beaker with stirring at a speed of 300 rpm for 10 h using magnetic stirrer. The oil was gradually added to the polymer solution during mixing until the desired oil loading was obtained. Fifty milliliters of alginate-oil emulsion were then sprayed into a collecting water bath containing calcium chloride solution (2 w/v %) using an Inotech Encapsulator with a 450 m nozzle. The resulting micro-capsules were allowed to harden in cross-linking solutions for 3 h. The orange peel EO loaded polymer beads were collected from the cross-linking solutions using a sieve. Finally, the micro-beads were rinsed twice with distilled water; tissue paper was used to absorb the surface excessive water and oil onto the wet micro-capsules.

**Nano-encapsulation:** In the current investigation, nano-encapsulation of orange peel EO was carried out according to the modified method of Terjung *et al.*<sup>13</sup> using homogenization (Homogenizer PRO 400 PC, SN: 04-01198, USA) model in a matrix comprising sodium alginate and Tween 20 ( $T_{20}$ ). Sodium alginate was dissolved in deionized water (3 g/100 mL), to which sodium alginate and an emulsifier Tween 20 were added to a final concentration of 100 mL. Optimization of batch size and process parameters were set through several experimental trials. The proportion of orange peel EO: Sodium alginate:  $T_{20}$  was varied during preliminary trials and it was found that the ratio of 3:10:1 of the same gave optimum yield of powder with desirable attributes.

One gram of orange peel EO was added to 100 mL solution containing 3 g sodium alginate gel and 1%  $T_{20}$ . The emulsion was created by mixing the solution in a high-pressure homogenizer at 18,000 rpm for 30 min. The temperature was kept at 35°C and storage at 4°C until used.

**Sponge cake preparation:** Sponge cakes were prepared according to Bennion *et al.*<sup>14</sup> with some modifications. The forms of sponge cakes at four different types were prepared by the addition of orange peel essential oils [non-encapsulate (0.5 g), micro-encapsulate (1 g) and nano-encapsulate (1 g)] and the control without orange peel essential oil (0 g). One hundred of cake were poured in baking pans, then placed in a pre-heated oven and baked at 160°C for 35 min. The cooled cakes were packed in polypropylene bags at room temperature before physico-chemical and sensory evaluation analyses.

**Transmission Electron Microscopy (TEM):** Twenty microliters of diluted samples (orange peel EO extract encapsulated) was placed on a film-coated 200 mesh copper specimen grid for 10 min and the fluid excess was eliminated using filter paper. The grid was then stained with one drop of 3% phosphotungstic acid and allowed to dry for 3 min. The coated grid was dried and examined under the TEM microscope. The samples were observed by operating<sup>15</sup> at 160 kV.

**Differential Scanning Calorimetry (DSC):** The thermal stability of orange peel essential oil forms (non-encapsulated, nano-encapsulated and micro-encapsulated) was determined using a Differential Scanning Calorimeter (DSC), model 823E from Mettler Toledo. Ten milligram samples were placed in aluminium crucibles. The samples were analyzed under a flow of nitrogen gas (40 mL min<sup>-1</sup>). A dynamic scan was performed at a heating rate of 10°C min<sup>-1</sup> over a temperature ranged from 0-300°C. Evaporation enthalpies were calculated by peak area integration of DSC profiles<sup>16</sup>.

#### **Physico-chemical characteristics of sponge cake forms**

**Chemical composition of sponge cake samples:** The physico-chemical characteristics including moisture, fat and total ash of sponge cake control and sponge cakes with addition of orange peel essential oil and its micro or nano-encapsulated were measured according to AOAC<sup>17</sup>. While, total carbohydrates were calculated by difference according to the methods of AOAC<sup>18</sup>. The protein was determined by Kjeldahl method of AACCC<sup>19</sup>. Results were expressed on a wet basis. The volume and specific volume of the sponge cake was determined according to methods of AACCC<sup>20</sup>.

**Color measurement:** Color parameters ( $L^*$ ,  $a^*$  and  $b^*$ ) of sponge cake samples (control, with non-encapsulated, micro and nano-encapsulated orange peel oil) were determined using a spectro-colorimeter (Tristimulus Color Machine) with the CIE lab color scale (Hunter, Lab Scan XE-Reston VA, USA) in the reflection mode. The instrument was standardized with white tile of Hunter Lab Color Standard (LX No.16379).  $L^*$  (lightness/darkness that ranges from 0-100),  $a^*$  (redness/greenness that ranges from -120 to 120) and  $b^*$  (yellowness/blueness that ranges from -120 to 120) were measured<sup>21</sup>.

**Textural properties of cakes:** The Texture Profile Analysis (TPA) of sponge cake samples (2×2×2 cm) from

the midsection of the sponge cake samples (contain non-encapsulated, micro and nano-encapsulated orange peel essential oil) compared with control were performed using a texture analyzer (Texture Pro-CTV1.6 Build, Brookfield, Engineering Labs, Inc.) with a 36 mm diameter cylindrical probe, 50% compressing and a test speed of 1.0 mm sec<sup>-1</sup>. The crust of cake samples was removed in cake texture determination<sup>22</sup>. The texture parameters recorded were consistency, hardness, cohesiveness, adhesiveness, springiness, resilience, gumminess and chewiness and the texture parameter of cake was averaged from three replications<sup>23,24</sup>.

**Determination of TBA and AWRC:** The amount of lipid oxidation in different substituted sponge cake was determined by the 2 thiobarbituric acid (TBA) method according to AOCS<sup>25</sup>. However, the staling of different sponge cake samples containing different forms of orange peel essential oil (non, micro and nano-encapsulate) were measured by Alkaline Water Retention Capacity (AWRC) according to AACC<sup>19</sup>.

**Sensory evaluation:** Sensory evaluation was performed with the help of 20 specialists in baking technology in Food Technology Department, National Research Centre; Egypt. Sensory evaluation was performed in first day and after 5th, 10th and 15th days of baking to evaluate cells, grain, texture, crumb color and flavor of the sponge cake samples. Panelists evaluated the samples in a testing area<sup>26</sup>. The objective sensory quality of sponge cake is described by its sensory profile which is constituted by sensory attributes according to Lawless and Heymann<sup>27</sup>.

**Extraction of oil from sponge cake:** Hexane was used to extract orange peel essential oils by cold percolation method<sup>28,29</sup> from sponge cake forms. About 3 g of sponge cake was taken in 25 mL of hexane in a conical flask and then kept on a rotary shaker at 120 rpm for 2 h. After 2 h, the extract was filtered and evaporated using a rotary vacuum evaporator to obtain the oil and was stored in air tight bottles at 4°C.

#### **Rancidity analysis**

**Free Fatty Acid value (FFA %):** The FFA value was determined according to PORIM<sup>30</sup> test methods. About 0.5 g of orange peel EO extracted from sponge cake was weighed into an Erlenmeyer flask. About 50 mL isopropanol and 0.5 mL of

phenolphthalein was added and neutralized by dropwise addition of 0.1 N potassium hydroxide till a faint but permanent pink color was obtained. Expression of result in Eq. 1 as oleic acid:

$$\text{FFA (\%)} = 28.2 \times N \times V/W \quad (1)$$

where, N is the normality of NaOH solution, V is the volume of NaOH solution used in mL and W is the weight of oil sample.

**Peroxide Value (PV):** The PV was performed according to AOAC<sup>31</sup>, with slight modifications. Orange peel EO extracted from sponge cake (2 mg) was dissolved in a blended solution of 30 mL chloroform-glacial acetic acid (3:2, v/v). A saturated solution of KI (1 mL) was added. After the addition of 75 mL distilled water, the mixture was titrated against sodium thiosulphate (0.1 M) until the yellow color almost disappeared. PV (meq kg<sup>-1</sup>) was calculated as follows in Eq. 2:

$$\text{PV (meq kg}^{-1}\text{)} = C \times (V - V_0) \times 12.69 \times 78.8/m \quad (2)$$

where, C is the sodium thiosulphate concentration (mol L<sup>-1</sup>), V and V<sub>0</sub> represent the volumes of sodium thiosulphate exhausted by the samples and the blank, respectively (mL) and m is the mass of orange peel EO extract (mg).

**Thiobarbituric acid (TBA):** Thiobarbituric acid values (TBA) of the sponge cake samples were measured after 0, 5, 10 and 15 days of storage at room temperature according to the AOCS<sup>32</sup> method.

#### **Quantitative phytochemical analysis**

**DPPH radical scavenging assay:** The free radical scavenging activity of orange cake EO extracts from sponge cake forms during storage periods were measured by using 2, 2-diphenyl-1-picrylhydrazyl (DPPH) by the modified method of McCune and Johns<sup>33</sup>. The reaction mixture (3.0 mL) consisted of 1.0 mL DPPH in methanol (0.3 mM), 1.0 mL methanol and 1.0 mL of concentration (10 µg mL<sup>-1</sup>) orange peel EO extract diluted in methanol was incubated for 10 min, in dark, after which the absorbance was measured at 517 nm using a UV-VIS Spectrophotometer against a blank sample<sup>29,34</sup>.

**Reducing power:** The reducing power of orange peel extracts from sponge cake samples during storage periods were quantified by the method described by Siddhuraju and

Becker<sup>35</sup> with minor modification. Orange peel EO extract ( $10 \mu\text{g mL}^{-1}$ ) in 1 mL methanol (80%) were mixed with 5 mL phosphate buffer (2 M, pH 6.6) and 5 mL potassium ferricyanide (1%). These mixtures incubated at  $50^\circ\text{C}$  for 20 min. About 5 mL trichloroacetic acid (10%) was added and the mixture was centrifuged at 3000 rpm for 10 min. The absorbance of the pink color mixture was measured spectrophotometrically at 700 nm.

**Statistical analysis:** Experiments and analysis was conducted triplicate and represented as Mean  $\pm$  SD. Data were evaluated using the analysis of variance one-way ANOVA and differences between means of parameters were compared using the Duncan's test at the 5% significance level. Statistical analysis was performed using SAS program (Statistical Analytical Systems, Cary, NC).

## RESULTS AND DISCUSSION

**Transmission Electron Microscopy (TEM):** The particles shape of encapsulated orange peel essential oil was measured and

illustrated in Fig. 1. Transmission Electron Microscopy (TEM) of micro-encapsulated orange peel essential oil particles in sodium alginate beads showed spherical shape of about 150-360 nm diameter (microscale). The external surface of each particle was almost regular and smooth, showing that sodium alginate polymer forms a continuous film surrounding the orange peel essential oil droplets.

The results in Fig. 2 illustrated encapsulated orange peel EO in sodium alginate gel. It appeared to be made up of single spherical units, its diameter ranged between 24.03-65.88 nm (nano-scale), due to the use of homogenizer to make nano-particles. The external surface of each unit was almost regular and smooth, showing that the polymer of sodium alginate gel forms a continuous film surrounding the orange peel essential oil droplets.

### Differential Scanning Calorimetry (DSC)

**DSC of orange peel EO (non-encapsulated):** The apparent melting enthalpy values calculated from the DSC of extracted orange peel EO (non-encapsulated) and its nano and micro-encapsulated in sodium alginate gel and beads,

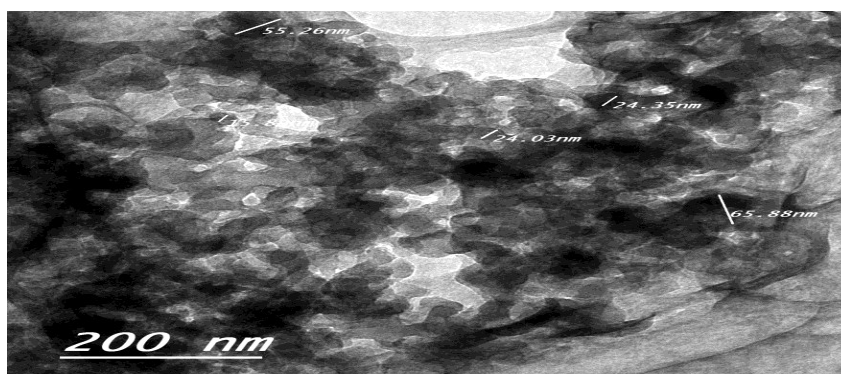


Fig. 1: Transmission electron microscopy morphology of micro-encapsulated orange peel EO in sodium alginate beads

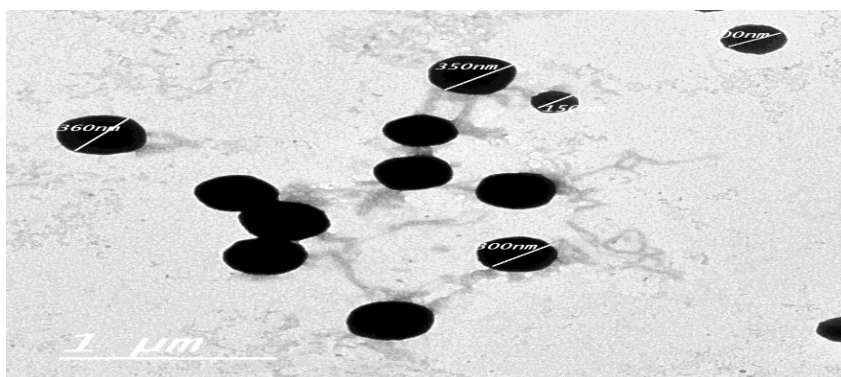


Fig. 2: Transmission electron microscopy morphology of nano-encapsulated orange peel EO in sodium alginate gel

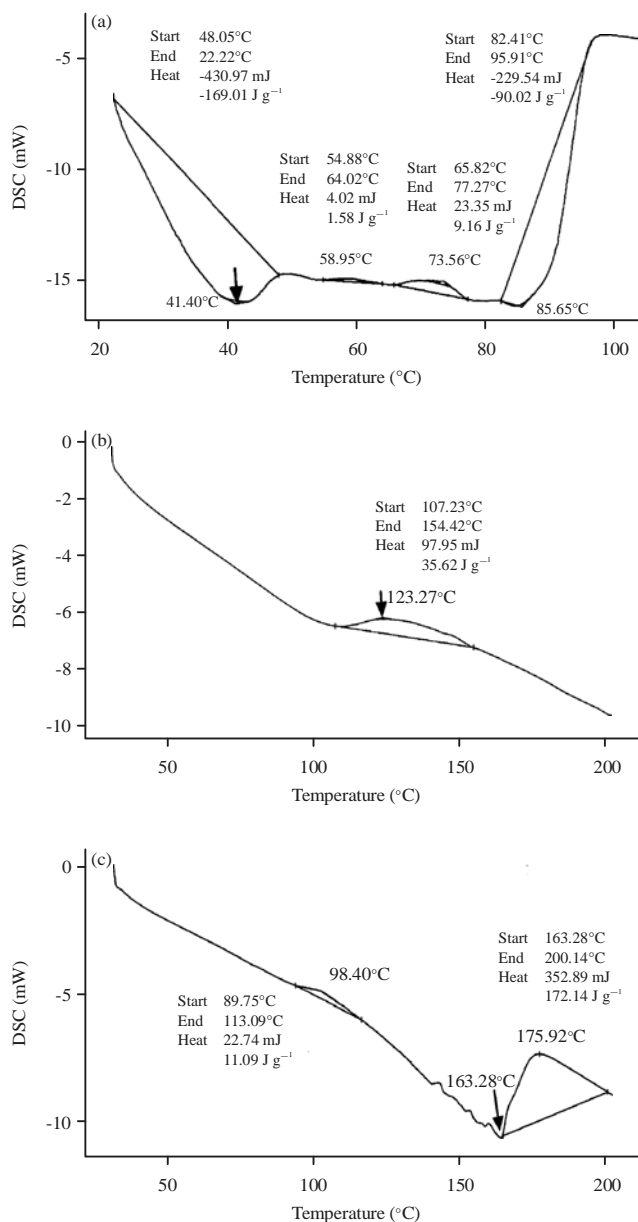


Fig. 3(a-c): (a) DSC of Orange peel EO (non-encapsulated), (b) DSC of orange peel EO microencapsulated in sodium alginate beads and (c) DSC of Orange peel EO that nano-encapsulated in sodium alginate gel

respectively. The thermal stability was measured using Differential Scanning Calorimetry (DSC). Result in Fig. 3a showed that the melting point of orange peels EO (non-encapsulated) being 41.40°C. This indicated the inability of the orange peel EO to withstand the high temperatures during the baking process (sponge cake) and therefore lose its activity.

**DSC of orange peel EO (micro-encapsulated):** The DSC of micro-encapsulated orange peel EO was shown in Fig. 3b. The melting point of micro-encapsulated orange peel EO was 123.27°C; the active compounds degrade and lose their activity at this temperature. The results showed that the process of encapsulation essential oils increase the thermal stability compared with orange peel EO (non-encapsulated), which helps the potential to be used in industrial applications in the food that is exposed to high thermal coefficients maintaining its antioxidant activity.

**DSC of orange peel EO (nano-encapsulated):** The thermal stability of nano-encapsulated orange peel EO was measured by DSC and results were shown in Fig. 3c. The melting point being 163.28°C of nano-encapsulated orange peel EO, which can be used in food applications successfully without any change in its characteristics and no loss of its antioxidant activity. This can be due to the ability of nanomaterial packaging to protect those compounds.

**Chemical composition:** The effect of micro and nano-encapsulated orange peel EO on chemical composition of sponge cake were determined and presented in Table 1. The obtained results showed that moisture content of sponge cake (control sample) reached 26.0%, while that of sponge cake containing non-encapsulate, micro-encapsulate and nano-encapsulate orange peel EO declined to 24.0, 25.5 and 24.5%. On the other hand, there were no significant differences observed in chemical composition of the

Table 1: Effect of micro and nano-encapsulated orange peel EO on chemical composition of sponge cake forms

Samples	Moisture (%)	Protein (%)	Fat (%)	Ash (%)	Carbohydrate (%)
Wheat flour (72%)	9.52±0.11	8.95±0.23	1.62±0.01	0.41±0.05	79.77±1.65
Cake (Control)	26.00±0.15	9.2±0.11	20.0±0.05	1.15±0.01	43.65±0.32
<b>Cake mixed with orange peel EO</b>					
Non-encapsulated	24.0±0.19	8.85±0.13	20.06±0.02	1.059±0.01	46.03±0.12
Micro-encapsulated	25.5±0.14	9.0±0.05	20.12±0.01	1.062±0.02	44.32±0.34
Nano-encapsulated	24.5±0.12	9.05±0.08	20.15±0.02	1.093±0.01	45.21±0.09

Table 2: Effect of micro and nano-encapsulated orange peel EO on cake color parameter

Samples	Crust			Crumb		
	L*	a*	b*	L*	a*	b*
Cake (Control)	58.24±0.22	12.28±0.19	19.16±0.09	72.83±1.32	3.10±0.03	22.99±0.22
<b>Cake mixed with orange peel EO</b>						
Non-encapsulate	57.85±0.29	12.42±0.11	19.19±0.16	70.77±1.11	3.64±0.09	22.76±0.18
Micro-encapsulated	55.65±0.36	12.54±0.09	19.60±0.12	70.50±0.65	3.39±0.07	22.47±0.15
Nano-encapsulated	59.38±0.42	12.10±0.12	19.54±0.19	73.32±0.72	3.16±0.05	20.66±0.11

L\*: Lightness, a\*: Redness, b\*: Yellowness

Table 3: Effect the addition of micro and nano-encapsulated orange EO on TPA parameters of sponge cake

Samples	Hardness (N)	Cohesiveness (-)	Springiness (mm)	Gumminess (N)	Chewiness (Nm)
Cake (Control)	130±2.11	320±2.34	250±2.03	380±3.04	300±1.50
<b>Cake mixed with orange peel EO</b>					
Non-encapsulate	129±2.31	318±3.11	255±2.52	390±2.06	310±2.54
Micro-encapsulated	127±2.23	315±2.25	260±3.03	400±2.95	315±1.55
Nano-encapsulated	121±2.04	312±3.16	262±2.25	410±3.54	320±1.87

Table 4: Effect the addition of micro and nano-encapsulated orange EO on staling and baking quality of sponge cakes

Parameters	Sponge cake (Control)	Cake mixed with orange peel EO		
		Non-encapsulated	Micro-encapsulated	Nano-encapsulated
<b>Baking quality</b>				
Weight (g)	76±0.95	78±1.15	80.5±1.19	82.0±1.06
Volume (cc)	160±1.08	170±1.25	170±1.15	165±1.05
Specific volume	2.11±0.32	2.18±0.09	2.11±0.15	2.0±0.13
<b>Alkaline water retention capacity (AWRC) (Staling)</b>				
Zero	380±1.02	370±2.09	350±1.11	340±1.03
5 days	330±1.22	350±1.35	340±1.20	335±1.10
10 days	290±1.16	310±1.22	320±1.35	325±1.40
15 days	250±1.0	290±1.04	300±1.21	310±1.15

EO: Essential oil

sponge cakes that was mixed with non-encapsulated or encapsulated orange peel EO compared to control sample.

**Color quality:** Color of sponge cake affect directly the consumer attention and is evaluated using Hunter laboratory colorimeter. Results in Table 2 showed significant differences in the color parameters of the cakes with different essential oil types of orange peel (non-encapsulate, micro-encapsulated and nano-encapsulated) compared to control sponge cake sample. The sample of sponge cake containing nano-encapsulate orange peel EO was more lightness (L) than the other sponge cake sample containing micro-encapsulate and non-encapsulate orange peel EO in crust 59.38; it was high in crumb 73.32. Therefore, crumb color reflected of used raw materials colors and their interactions. The sample of sponge cake with added nano-encapsulate was closer to control sample. These results are in agreement with Kordonowy and Youngs<sup>36</sup>, Kim *et al.*<sup>37</sup> and Ramy *et al.*<sup>38</sup>.

It could be noticed that sponge cake sample containing micro-encapsulate orange peel EO in crust resulted in significant increase in redness (a) and yellowness (b) being 12.54 and 19.60, respectively compared with other samples. While, decreased (a) significantly in crumb of cake

sample containing nano-encapsulate orange peel EO being 3.16 compared with the other samples.

**Textural profile analysis:** Physical properties of sponge cake were tested by Texture Profile Analysis (TPA). The TPA analysis of mixed orange peel EO non and encapsulate forms with sponge cake were compared with control sponge cake sample (Table 3). Results showed that sponge cakes hardness values decreased with the addition of orange peel EO and the least hardness sample was contain nano-encapsulate orange peel EO. This results in an improvement in the properties of sponge cake produce with an increase in its shelf life. The cohesiveness of orange peel cake EO was affected by the decline compared to control one.

Results also showed that springiness, gumminess and chewiness values of orange peel cake EO were increased compared to control one. These results proved that the increase in cake volume and softness of texture could be due to addition of orange peel EO.

**Baking quality and staling:** The effect of orange peel EO addition on baking quality of sponge cake was shown in Table 4. The addition of nano-encapsulated EO on sponge

cake resulted in increase of weight (g) compared to control and other samples. Otherwise, non and micro-encapsulated orange peel EO addition on sponge cake increment of volume (cc) than control sample. These results showed that addition of orange peel EO to sponge cake was capable at trapping higher amount of air in it especially that contain micro-encapsulate orange peel EO.

While, the specific volume of orange peel EO sponge cake with (non, micro and nano-encapsulate) after baking at 160°C for 35 min not affected.

The results of the effect of storage period on staling of orange peel EO sponge cake shown in Table 4. Results obtained showed that orange peel EO sponge cake contained higher amount of Alkaline Water Retention Capacity (AWRC) being 380, 330, 290 and 250% for 0, 5, 10 and 15 days storage, respectively. While, micro-encapsulate and nano-encapsulate orange peels sponge cakes contained decreased amount of AWRC in zero time but an increase in AWRC was observed during different storage periods compared to the control sample. This was due to the ability of sodium alginate (beads or gel) to imbibe with water, swelling and retention of water for a long time and delay staling.

**Sensory evaluation of sponge cake samples:** Effect of the addition of micro and nano-encapsulated orange EO on the sensory properties of sponge cake was evaluated during storage at room temperature ( $29 \pm 3.0^\circ\text{C}$ ) for 15 days. Figure 4 indicated that the flavor values of sponge cake mixed with non-encapsulate orange peel EO was higher than other tested samples at zero time and after 5 days being 9.1 and 8.3, respectively but the values subsequently deteriorated to 7.6 and 6.5 after 10 and 15 days, respectively.

There was no significant difference between cake crumb color and texture values of all sponge cake and control sample during storage period. While grain values were similar in both control sample and cake mixed with non-encapsulate orange peel EO at all different storage periods. But this value decreased in sponge cake mixed with micro and nano-encapsulate orange peel EO.

So, Fig. 4a-d depicted that there was no significant difference between cake flavor of control sample and cakes of those mixed with non-encapsulate, micro-encapsulation or nano-encapsulation EO after storage for 10 or 15 days. The same trend was also observed in crumb color, texture, grain and cells.

**Lipid oxidation (rancidity):** The primary products of lipid peroxidation are hydroperoxidations. Therefore, determining the concentration of peroxidase is one clear index of lipid

peroxidation. Changes occurring in the Acid Value (AV), Peroxide Value (PV) and thiobarbituric acid (TBA) of sponge cake samples with addition of orange peel EO forms were evaluated during storage and presented in Table 5. Acid value in the 1st day for sponge cake (control) and non-encapsulate, micro-encapsulate and nano-encapsulate orange peel EO were 1.12, 1.00, 0.90 and 0.88 mL equiv  $\text{O}_2/\text{kg}$ , respectively. After 15 days of storage at room temperature they increased to 1.95, 1.31, 1.25 and 1.20 mL equiv.  $\text{O}_2/\text{kg}$ , respectively. This result was better than the mentioned by Wagdy and Taha<sup>39</sup> after 15 days storage, where they stated that Acid Value (AV) of fortified butter sponge cake with jojoba hull at zero time was 0.71%, while after three weeks of storage changed to 3.88%. This increase at zero time is due to the hydrolysis of oil to fatty acids and formation of aldehydes and ketones<sup>40</sup>.

The effect of orange peel EO sponge cake on Peroxide Value (PV) during storage period (15 days) at room temperatures was studied in Table 5. The PV of sponge cake (control) during 1-15 days ranged between 2.30-3.52 mEq  $\text{kg}^{-1}$ , while non-encapsulated, micro-encapsulated or nano-encapsulated orange peel EO sponge cakes for the same storage period little increased to 2.23-2.62, 2.20-2.5 and 2.17-2.46, respectively. Thiobarbituric acid value (TBA) in the same table showed the higher value of TBA in control sample than other tested sample.

While, non-encapsulated and nano-encapsulated orange peel EO reduced TBA values compared to the control sample during the storage periods. All values were less than  $0.15 \text{ mg kg}^{-1}$  are acceptable because the values higher than  $0.15 \text{ mg kg}^{-1}$  are unacceptable and rancid<sup>41</sup>. At the end of storage (15 days), all cake samples were still acceptable.

All orange peel sponge cake EO or encapsulated samples had lower TBA values compared to control one, indicating that orange peel EO incorporated into cakes exhibited antioxidant properties and preventing lipid oxidation in cakes.

The obtained results suggested that orange peel EO and their encapsulated EO forms were effective in suppressing the oxidation of oils or fats in the sponge cakes. The oxidation stability of sponge cake is due to the addition of natural antioxidants. So, the addition of orange peels to all forms of cakes either capsulated or non-capsulated prevents lipid peroxides formation and delayed oxidation during storage.

#### **Antioxidant activity of orange peel EO forms in sponge cake samples**

**DPPH radical scavenging activity:** The effect of orange peels EO addition to all cakes forms on antioxidant activity was evaluated and the results are presented in Fig. 5.



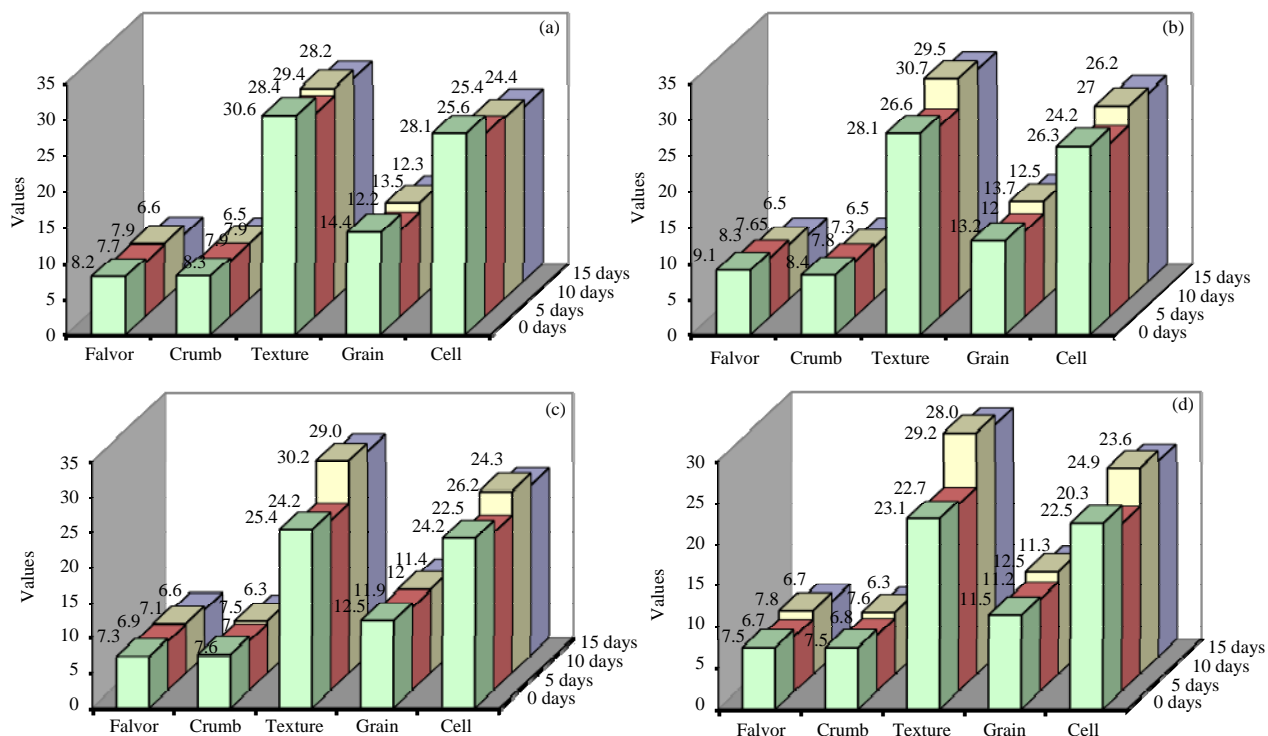


Fig. 4(a-d): Effect of mixing micro and nano-encapsulated orange EO on the sensory properties of sponge cake forms during storage for 15 days at room temperature, (a) Sponge cake (Control), (b) Sponge cake with non-encapsulate orange peel EO, (c) Sponge cake with micro and (d) Sponge cake with nano-encapsulate orange peel EO

Table 5: Effect of non-encapsulate, micro-encapsulated and nano-encapsulated of orange peel EO forms addition on the chemical quality of sponge cake during storage

Quality parameter	Cake (Control)	Sponge cake mixed with orange peel EO forms			LSD
		Non-encapsulated	Micro-encapsulated	Nano-encapsulated	
<b>First day</b>					
Acid value	1.12±0.11 <sup>a</sup>	1.00±0.13 <sup>b</sup>	0.90±0.02 <sup>c</sup>	0.88±0.01 <sup>c</sup>	0.06
Peroxide value	2.30±0.12 <sup>a</sup>	2.23±0.15 <sup>b</sup>	2.20±0.12 <sup>c</sup>	2.17±0.13 <sup>d</sup>	0.03
Thiobarbituric acid	0.10±0.002 <sup>a</sup>	0.08±0.001 <sup>b</sup>	0.07±0.006 <sup>c</sup>	0.06±0.002 <sup>d</sup>	0.01
<b>After 5 days</b>					
Acid value	1.35±0.09 <sup>a</sup>	1.16±0.11 <sup>b</sup>	1.00±0.09 <sup>c</sup>	0.96±0.05 <sup>d</sup>	0.04
Peroxide value	2.82±0.11 <sup>a</sup>	2.50±0.09 <sup>b</sup>	2.41±0.11 <sup>d</sup>	2.32±0.15 <sup>c</sup>	0.11
Thiobarbituric acid	0.15±0.01 <sup>a</sup>	0.08±0.03 <sup>b</sup>	0.08±0.05 <sup>b</sup>	0.07±0.01 <sup>c</sup>	0.01
<b>After 10 days</b>					
Acid value	1.62±0.11 <sup>a</sup>	1.22±0.13 <sup>b</sup>	1.12±0.10 <sup>c</sup>	1.08±0.09 <sup>d</sup>	0.10
Peroxide value	3.12±0.13 <sup>a</sup>	2.60±0.33 <sup>b</sup>	2.50±0.33 <sup>f</sup>	2.36±0.33 <sup>a</sup>	0.15
Thiobarbituric acid	0.20±0.03 <sup>a</sup>	0.12±0.03 <sup>b</sup>	0.10±0.01 <sup>c</sup>	0.10±0.02 <sup>c</sup>	0.02
<b>After 15 days</b>					
Acid value	1.95±0.13 <sup>a</sup>	1.31±0.15 <sup>b</sup>	1.25±0.17 <sup>c</sup>	1.20±0.11 <sup>d</sup>	0.05
Peroxide value	3.52±0.10 <sup>a</sup>	2.62±0.11 <sup>b</sup>	2.50±0.15 <sup>d</sup>	2.46±0.17 <sup>e</sup>	0.03
Thiobarbituric acid	0.25±0.03 <sup>a</sup>	0.15±0.01 <sup>b</sup>	0.12±0.02 <sup>c</sup>	0.12±0.03 <sup>c</sup>	0.02

Results showed that both non-encapsulated EO and nano-encapsulated EO extracted from cake displayed good antioxidant activities during storage periods compared to untreated sponge cake (control). The obtained

data showed that antioxidant activity of orange EO was maximized at zero time compared to untreated sponge cake, where DPPH of non, micro and nano-encapsulated orange peel EO in sponge cake samples reached 24.55,

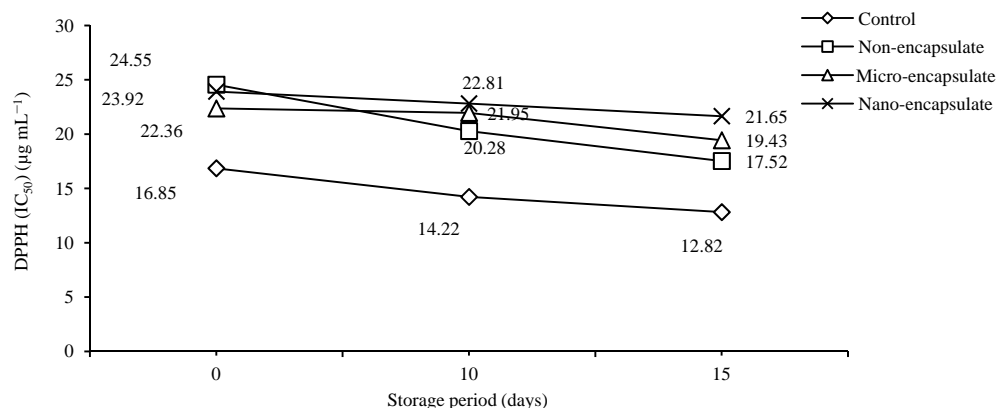


Fig. 5: Effect of non, micro and nano-encapsulation orange peel EO forms addition on DPPH radical scavenging activity of sponge cake samples during storage period

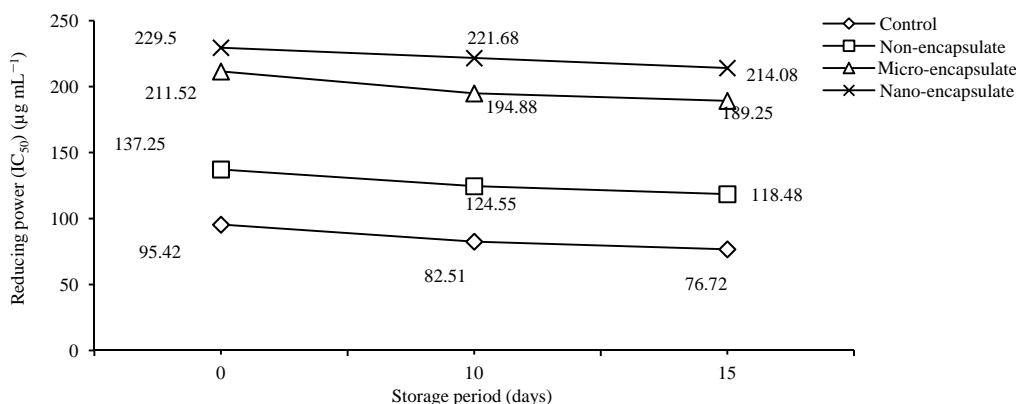


Fig. 6: Effect of non, micro and nano-encapsulated orange peel EO on reducing power in sponge cake samples on storage period

22.36 and 23.92  $\mu\text{g mL}^{-1}$ , respectively. It was 16.85  $\mu\text{g mL}^{-1}$  in untreated cake (control). Furthermore, DPPH of non, micro and nano-encapsulated EO declined slightly and was lower than non-encapsulate EO during sponge cakes storage. The DPPH of micro and nano-encapsulated orange peel EO declined to 19.43 and 21.65  $\mu\text{g mL}^{-1}$  after 15 days, respectively, while it was decreased to 12.82 and 17.52  $\mu\text{g mL}^{-1}$  in control and non-encapsulated orange peel EO sponge cakes. These results indicated that nano-encapsulation of orange peel EO has been able to protect the bioactive compounds from loss of antioxidant activity compared to other samples and increase the shelf-life of food products.

**Reducing power:** The reducing power of nano and micro-encapsulated orange EO in sponge cake was evaluated and presented in Fig. 6. These results indicated the reducing power of EO represented by DPPH radical scavenging activities. The

micro and nano-encapsulated orange peel EO extracted from cake had higher reducing power than untreated (control) or those treated with non-encapsulate orange peel EO during storage period at room temperature. Also, reducing power of micro and nano-encapsulated EO was slightly decreased in cracker during storage. This reducing power was decreased from 211.52 and 229.50 at zero time then decreased to 189.25 and 214.08  $\mu\text{g mL}^{-1}$  after 15 days, respectively.

The present study showed higher antioxidant activity at a concentration (10  $\mu\text{g mL}^{-1}$ ) of orange peel EO extract than those obtained by Mahmoud *et al.*<sup>42</sup>, who used maltodextrin and Arabic gum in orange peel nano-encapsulated. This is due to the different method of extracting from the previous work, which leads to different nature of the antioxidants in the resulting extracts. In present study, the method used in encapsulation process is completely different, which use sodium alginate as a wall material in the tow types (beads and gel) for produced micro and nano-encapsulate of orange

peel EO, respectively. So, the results of antioxidants quality and the ability to extend shelf life of sponge cake more efficient than the previous results. This study will help the researcher to uncover the importance of encapsulation technique for bioactive compounds to protect them by sodium alginate that many researchers were not able to apply in food industries.

On the other hand, orange peel oil can be used in edible-coating of whole or slices fruit such as Radi *et al.*<sup>43</sup> who studied nano-emulsion and micro-emulsion techniques of pectin-based edible coating containing orange peel oil quality parameters for orange slices stored.

### CONCLUSION

This study concluded that orange peel essential oils can be used as antioxidant in baking product (sponge cakes) without the effect of baking temperature, rancidity or change in physical and chemical properties of the final product during storage due to encapsulation techniques.

### SIGNIFICANCE STATEMENT

This study discovered that the used of orange peel EO extract and its encapsulated forms (micro-encapsulated and nano-encapsulated) in bakery products can be beneficial to increase storage stability at room temperature and delay staling in sponge cake added to orange peel EO extract encapsulated specially nano-encapsulated form, which had the ability to prevent lipid peroxides and consequently reduce the rancidity of staining during storage period, increasing the storage capacity compared to control sample. The apparent volume in cakes and the springiness, gumminess and chewiness values of sponge cakes increased with addition of orange peel EO micro or nano-encapsulated. The results showed that addition micro-encapsulate orange peel EO is capable at trapping higher amount of air in it.

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