

## EFFECT OF SOME OIL EMULSIONS AND WAX TREATMENTS ON PROLONGING STORAGE PERIOD OF WASHINGTON NAVEL ORANGE FRUITS AND THEIR VOLATILE COMPONENTS

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### ABSTRACT

This study was carried out during 2004 and 2005 seasons on the postharvest behavior of Washington Navel orange fruits during cold storage. Fruits were divided into seven groups. The fruits of first group was left without coating (control), while the six other groups were coated separately with wax liquid, jojoba oil, sesame oil, paraffin oil, orange oil and corn oil emulsion, then stored at  $5^{\circ} \pm 1^{\circ}\text{C}$  and 85-90% RH. The fruits were examined every 15 days for 90 days storage period. Coating with jojoba oil and orange oil were the best in reducing fruit decay and weight loss and increasing fruits storage life. As storage days progressed T.S.S. increased , however titratable acidity and ascorbic acid contents decreased.

The headspace volatiles of freshly harvested fruits, coated and uncoated (control) stored fruit samples were collected and subjected to GC and GC-MS. analysis. 35 volatile Components were identified, including 7 esters, 6 terpenic hydrocarbons, 12 alcohols, 5 aldehydes, 3 ketones and others. Volatile composition varied quantitatively during storage rather than qualitatively. Generally, coated fruits showed significant increase in component quantities that consider important to fresh orange flavour such as ethylbutyrate, methylbutyrate, ethylacetate,  $\alpha$ -pinene, octanal, nonanal, neral and linalood. By comparing the volatile profile of coated fruit samples to fresh fruit samples, it was clear that jojoba oil, orange oil and corn oil coating exhibited high quality during storage period.

**Keywords:** Navel orange, cold storage, wax, oil emulsions, volatile components.

### INTRODUCTION

In Egypt citrus is the leading fruit crop produced, navel orange is the principal orange cultivar that represents near 65-70% of the total orange cultivated area. Egypt has a long history in the exportation of citrus to different countries.

Citrus fruits suffer from relatively high loss during the harvesting and handling chain. Such loss is due to unideal postharvest several mechanical damage, operations including storage conditions. Mechanical damage is always associated with pathological infections that increase crop losses after harvest.

*Nagvi and Ting (1990)* showed that jojoba oil is (commonly known as liquid wax) colourless and odourless with unique physical and chemical properties. They found that jojoba oil can easily be hydrogenated into a soft wax that can be used in candle wax, various kinds of polishes and coating material for fruits and pills. *Martínez et al.*, (1991) found that coated Valencia orange fruits with Imazalil solution (IMZ) (800ppm) showed reduction in

weight loss of coated fruits compared with that of uncoated control fruits, after 15 days storage at 20°C, 70% RH. The waxing had no effect on the total soluble solids (T.S.S.), and total soluble solids/acid ratio.

*Rana et al. (1992)* demonstrated that, treating sweet orange fruits with sesame oil emulsion resulted in reducing decay, after 40 days of storage, compared with untreated sample (control). They also found that, percentage of weight loss and total soluble solids of fruits increased with increasing storage time, while juice and ascorbic acid content decreased. *Cuquerella et al. (1993)* reported that water-type waxes reduced shrinkage of Valencia and Washington Navel orange, but had no clear effect on decay. *Subedi et al. (1998)* reported that treating mandarin (*Citrus reticulata*, cv. *Suntala*) fruit, with 6% paraffin wax increased T.S.S content and percentage of fruit weight loss significantly as storage time increased, while significant negative relationship between titratable acidity and length of storage period existed. Fruit rotting percentage was reduced by paraffin wax coating.

*Zhiguo and Eric. (2001)* dipped pears fruits in either corn or soybean oil emulsions (prepared by mixing plant oil, tween 60 and water at 90°C) immediately after harvest. The treated fruits were stored along with the untreated ones (control) at 0°C. They found that the untreated fruits showed high percentage of decay, while both treatments inhibited decay by about 10%. Furthermore, the treated fruits exhibited normal colour change and had higher soluble solids, titratable acidity and volatiles production. *El-Mohamedy et al. (2002)* suggested that alcohols such as nerol,  $\alpha$ -terpineol; citronellol and aldehydes such as citral (constituents of citrus essential oils), can be used for controlling postharvest decay of citrus fruits.

The flavour volatile profile of citrus fruit has been reported to change in response to storage conditions such as temperature (*Nisperos-Carriedo and Shaw, 1990*). It has been reported that waxing of citrus fruit can adversely affect the fruit flavour (*Davis and Hofmann, 1973; Cohen et al. 1990; Hagenmaier and Baker, 1993*), perhaps due to overproduction of volatiles associated with anaerobic conditions, such as ethanol, methanol, and acetaldehyde. Harvested tangerine fruits, especially the wax coated ones are particularly susceptible to off-flavour development (*Cohen et al. 1990*). Another report claimed no adverse flavour effects when "Shamouti" or "Valencia" oranges were coated with various waxes (*Ben-Yehoshua, 1969*).

Of the volatile components important to flavour, esters and aldehydes are the primary contributors to fresh orange flavour (*Bruemmer, 1975*); although other components could also be important (*Shaw, 1977*). Other factors that influence the flavour are correct proportions of the different compounds (*Shaw, 1979*), taste threshold values of volatiles (*Patton and Josephson, 1957*), synergistic effects between volatiles (*Shaw and Wilson, 1980*), and the interaction of nonvolatile with volatile flavour components (*Ahmed et al. 1978a*).

Although various types of coatings have been developed for extending fruit storage life, there are little descriptive informations and quantitative data concerning the effect of films on volatile flavour components. Changes in the volatile components arising from different edible coating during storage in orange have been studied by many authors (*Shaw*

*et al 1990; McCalley and Torres 1992; Moshonas and Shaw 1994, 1997; Peebles et al 1999 and Hagenmaier, 2000).*

Generally, coated fruit showed higher concentrations of many volatile compounds as time of storage increased, most notably ethanol, ethyl butanoate, ethyl acetate, methyl butanoate and  $\alpha$ -pinene. (Baldwin, *et al.* 1995. and Nisperos-Carriedo, *et al.* 1990). The later authors found that use of beeswax emulsion and TAL Pro-long (TAL chemical co.) alone or in combination was the most effective coating in retaining or increasing volatile components.

The present study was designed to determine the influence of coating by five vegetable oil emulsions and a wax on postharvest behaviour and volatile components of navel orange during storage at low temperature, in order to assess the best treatments that maintain the fruit quality. The result of this study may be useful for prolonging the shelf life, marketing and finally to increase the exports of navel orange fruits.

## MATERIALS AND METHODS

The present investigation was carried out for two successive seasons (2004 and 2005), on 12 years - old Washington navel orange trees budded on Volkamer lemon rootstock grown in a private farm at El-Sadat city, Minufiya governorate. Fruits at maturity stage were picked, washed and then dried. The fruits were divided into seven groups. The first group was packed in 3 kilo-one layer carton boxes (control). The second one was sprayed with water containing 2000 ppm Imazalil (IMZ) (Laville 1978). Oil emulsions were prepared by mixing oil with Tween 80 in water at 90°C (Ju *et al.* 2000). The third, fourth, fifth, sixth and seventh groups were treated with jojoba, sesame, paraffin, orange and corn oil emulsions respectively. The fruits were left to dry and then packed in one layer inside carton boxes three kilo each. Three boxes served for each treatment. The fruits were stored at  $5 \pm 1^\circ\text{C}$  and 85-90 RH.

The fruits were periodically examined at 15 days intervals until the end of the storage period (90 days), to determine the following parameters.

### **A-Physical properties:**

**A.1. Decay (%).** The fruit decay percentage was determined at 15 day intervals during the storage period.

**A.2 Weight loss (%).** The fruit weight loss was calculated as the difference between the initial weight of samples at zero time and the weight at different tested periods.

### **B- Chemical properties.**

**B.1. Total soluble solids (T.S.S.).** The total soluble solids of fruit juice were estimated using a hand Refractometer (A.O.A.C., 1990).

**B.2. Titratable acidity.** Acidity was calculated as citric acid in 100g fresh weight (A.O.A.C., 1990).

**B.3. T.S.S. :acid ratio.** T.S.S./ acid ratio was calculated.

**B.4. Ascorbic acid.** Ascorbic acid as mg in 100 ml of juice was estimated using 2,6 dichlorophenol – indophenol as an indicator (A.O.A.C., 1990).

**C.1 Isolation of headspace volatiles.**

The volatiles in the headspace of each sample under investigation were isolated by using a dynamic headspace system. The samples were purged for 1 h. with nitrogen gas (grade of N<sub>2</sub> > 99.99°C) at a flow rate 100 ml/min. The headspace volatiles were swept into cold traps containing diethyl ether and pentane (1:1, v/v) and held at -10 °C. The solvents containing the volatiles were dried over anhydrous sodium sulfate for 1 h. The volatiles were obtained by evaporation of the solvents under reduced pressure.

**C.2 Gas chromatographic (GC) analysis.**

GC analysis was performed by using a Hewlett-Packard model 5890 equipped with flame ionization detector (FID). A fused silica capillary column DB5 (60m x 0.32 mm id) was used. The oven temperature was maintained initially at 50°C for 5 min, then programmed from 50 to 250° °C at a rate of 4 °C/min. Helium was used as the carrier gas, at flow rate 1.1ml/min. The injector and detector temperatures were 220 and 250°C, respectively. The retention indices (Kovats index) of the separated volatile components were calculated with hydrocarbons (C<sub>8</sub>-C<sub>22</sub> Aldrich Chemical CO.) as references.

**C.3 Gas chromatographic-mass spectrometric (GC-MS) analysis.**

The analysis was carried out by using a coupled gas chromatography Hewlett-Packard (5890)/mass spectrometry Hewlett-Packard-MS (5970). The ionization voltage was 70 eV, mass range m/z 39-400amu. The GC condition was carried out as mentioned above. The isolated peaks were identified by matching with data from the library of mass spectra (NIST) and compared with those of authentic compounds and published data (Adams, 1995). The quantitative determination was carried out based on peak area integration.

**D- Statistical analysis.**

The data obtained were statistically analyzed as a complete randomized design according to *Snedecor and Cochran (1972)*. Averages were compared using L.S.D. values at 0.05 level.

## **RESULTS AND DISCUSSION**

### **1. Physical properties:**

#### **1.1. Fruits decay (%).**

The decay percentage of "Washington Navel" orange fruits, developed during cold storage period upon non-traditional treatments is shown in Table (1). Fruits decay percentage increased as the storage period extended under cold storage. Control fruits became unacceptable after 90 days, while treated fruits showed decay (%) ranged between 3.0 and 21.0% after the same period. Up to 75 days of cold storage, fruits treated with jojoba oil and orange oil didn't show any decay and reduced fruit decay under cold storage for 90 days to 6.0%. Whereas wax, sesame oil, paraffin oil and corn oil treatments maintained fruits without decay up to 30 days . Corn oil followed jojoba oil and orange oil, while paraffin oil recorded higher decay percentage at the two seasons, than wax and sesame oil treatments. These results are in agreement with those reported by *Cuquerella et al. (1993)* and *Hamouda (2000)*.

### **1.2. Weight loss (%).**

Table (2) shows the effect of storage periods and oil emulsions on the weight loss percentage of Washington navel orange fruits. Data indicated that, in both seasons prolonging the storage period resulted in increased fruit weight loss. However, untreated fruits (control) lost 11.4% and 11.9% of their weights in the two seasons respectively. Treatment with jojoba oil caused weight loss of 7.0% and 6.9% in the first and second seasons respectively at the end of storage period. Fruits treated with paraffin oil possessed weight loss of 9.0% and 8% in the two seasons respectively after 90 days of cold storage. Weight loss (%) increased as storage period progressed, these results are in agreement with those found by *Rana et al. (1992)* and *Bhadra and Sen (1999)*.

### **2. Chemical properties.**

#### **2.1. Total soluble solids (%).**

Data in Table (3), indicate that total soluble solids percentage of all treated fruits increased as storage prolonged.

Fruits treated with jojoba oil or orange oil in the first season or those treated with sesame oil, jojoba oil or wax in the second season have a constant T.S.S. (%) compared to fruits exposed to other treatments at the end of cold storage. These results are in line with those obtained by *Bhadra and Sen (1999)* and *Zhiguo and Eric (2001)*.

#### **2.2. Titratable acidity (%).**

Fruit juice acidity (%) under different treatments decreased during storage period. (Table 4). The differences among acidity values of oil treatments were not clearly noticed. These findings agreed with those of *Zhiguo and Eric (2001)*.

#### **2.3. T.S.S./ acid ratio.**

The effect of different treatments on T.S.S./acid ratio of Washington navel orange fruits is shown in Table (5). It is clear that T.S.S./acid ratio in fruit juice increased by increasing storage period. Fruits treated with jojoba oil had the lowest values of T.S.S./acid ratio compared with other treatments. These results are in harmony with *Castle and Gmitter (1999)*.

#### **2.4. Ascorbic acid content.**

Table (6) shows that ascorbic acid content of fruit juice upon different treatments decreased with increasing storage period, fruits treated with wax were inferior in ascorbic acid content compared with other treatments. At the end of cold storage (90 days), fruits treated with orange oil surpassed in ascorbic acid content. These results are in accordance with what was obtained by *Bhadra and Sen (1999)* who stated that ascorbic acid content decreased as storage progressed.

### **3- Volatile components in headspace of fresh (zero time) , coated stored and control samples of Navel Orange (cv. Washington).**

The separated volatile components as identified by comparison of their mass spectra with library (NIST) and with those of authentic compounds and published data are cited together with their area percentage in Table (7).

Typical gas chromatograms of the volatiles of fresh (zero time), coated stored samples and control sample (uncoated stored) are shown in Figures (1-4).

Table (1) : Effect of different rind coating on fruit decay (%) of washington navel orange during storage at 5±1C° and 85 – 90 % RH

Days of Storage	First season (2004)							Second season (2005)						
	Rind Coating							Rind Coating						
	Control	Coat 1	Coat 2	Coat 3	Coat 4	Coat 5	Coat 6	Control	Coat 1	Coat 2	Coat 3	Coat 4	Coat 5	Coat 6
Start	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
15	0.0	0.0	0.0	0.0	0.0	0.0	0.0	1.0m	0.0	0.0	0.0	0.0	0.0	0.0
30	9.0g	0.0	0.0	0.0	0.0	0.0	0.0	7.0hi	0.0	0.0	0.0	0.0	0.0	0.0
45	21.0d	3.0i	0.0	6.0h	9.0g	0.0	3.0i	22.0d	2.0kl	0.0	6.0i	9.0h	1.0m	2.0kl
60	30.0c	6.0h	0.0	9.0g	12.0f	0.0	6.0h	33.0c	4.0j	0.0	6.0i	11.0g	2.0kl	3.0k
75	36.0p	6.0h	0.0	9.0g	15.0e	0.0	6.0h	39.0p	6.0i	2.0kl	9.0h	13.0f	2.0kl	6.0i
90	42.0a	12.0f	6.0h	12.0f	21.0d	6.0h	9.0g	43.0a	9.0h	5.0ij	11.0g	15.0e	5.0ij	6.0i

Coat 1:( Wax)- Coat 2 : (jojoba oil) - Coat 3 : (Sesame oil) - Coat 4 : (paraffin oil) - Coat 5: (Orange oil) - Coat 6 : (Corn oil)

Mean Comparison L.S.D. at (0.05)

Table (2) : Effect of different rind coating on fruit weight loss (%) of washington navel orange during storage at 5±1Co and 85 – 90 % RH

Days of Storage	First season (2004)							Second season (2005)						
	Rind Coating							Rind Coating						
	Control	Coat 1	Coat 2	Coat 3	Coat4	Coat5	Coat6	Control	Coat 1	Coat 2	Coat3	Coat4	Coat5	Coat6
Start	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0	0.0
15	1.1i	0.7j	0.6j	0.9ij	1.1i	0.7j	0.9ij	1.5ij	0.8k	0.7k	1.1j	0.9j	0.9j	0.8j
30	3.4g	1.2i	1.2i	1.4i	1.6hi	1.3i	1.3i	3.8h	1.1j	1.1j	1.3j	1.1j	1.3j	1.2j
45	5.7ef	2.3h	2.2h	3.7g	2.8gh	2.4h	2.5gh	4.9f	2.5i	2.6i	3.4h	2.8hi	2.9hi	3.1hi
60	6.2de	3.1g	2.8gh	4.9f	3.8g	3.5g	4.0fg	6.0e	3.4h	3.6h	4.5g	3.6h	3.9gh	4.1fg
75	9.0p	4.8f	4.1fg	5.8ef	6.3e	4.8f	5.0f	8.8b	5.2f	5.1f	6.3e	5.8ef	5.5ef	6.0e
90	11.4a	7.0d	7.0d	8.2c	9.0p	7.4cd	7.9cd	11.9a	7.9c	6.9d	8.0c	8.0c	7.0d	7.9c

Coat 1:( Wax)- Coat 2 : (Jojoba oil) - Coat 3 : (Sesame oil) - Coat 4 : (paraffin oil) - Coat 5: (Orange oil) - Coat 6 : (Corn oil)

Mean Comparison L.S.D. at (0.05)

**Table (3) : Effect of different rind coating on total soluble solids (%) of washing on navel orange during storage at 5±1Co and 85 – 90 % RH**

Days of Storage	First season (2004)							Second season (2005)						
	Rind Coating							Rind Coating						
	Control	Coat 1	Coat 2	Coat 3	Coat4	Coat5	Coat6	Control	Coat 1	Coat 2	Coat 3	Coat4	Coat5	Coat6
Start	10.8e	10.8e	10.8e	10.8e	10.8e	10.8e	10.8e	11.5c	11.5c	11.5c	11.5c	11.5c	11.5c	11.5c
15	10.9e	10.8e	10.9e	10.8e	10.9e	10.8e	11.0d	11.5c	10.8d	11.6c	10.7d	11.8b	11.2cd	11.5c
30	11.3j	11.0d	10.9d	10.9d	11.2d	11.0d	11.3d	12.0b	11.2c	11.6bc	11.3c	11.9bc	11.7bc	11.7bc
45	11.6cd	11.4cd	11.2d	11.5cd	11.7cd	11.4d	11.7cd	12.5b	11.7bc	11.9bc	11.4c	12.1b	12.0b	12.0b
60	12.3b	11.8cd	11.7d	11.5d	12.0bc	11.8bc	12.1b	12.8ab	11.5c	11.9bc	11.6c	12.6ab	12.5ab	12.3ab
75	12.8ab	11.9b	11.9b	11.8b	12.2b	12.0b	12.5ab	13.0ab	11.9bc	12.1b	11.7b	12.7ab	12.7ab	13.0ab
90	13.0a	12.5ab	12.2b	12.7ab	12.6ab	12.2b	12.9c	13.5a	12.3b	12.2b	12.0b	13.2ab	12.9ab	13.1ab

Coat 1:( Wax)- Coat 2 : (Jjoba oil) - Coat 3 : (Sesame oil) - Coat 4 : (paraffin oil) - Coat 5: (Orange oil) - Coat 6 : (Corn oil)  
 Mean Comparison L.S.D. at (0.05)

**Table (4) : Effect of different rind coating on titratable acidity of washington navel orange during storage at 5±1Co and 85 – 90 % RH**

Days of Storage	First season (2004)							Second season (2005)						
	Rind Coating							Rind Coating						
	Control	Coat 1	Coat 2	Coat 3	Coat4	Coat5	Coat6	Control	Coat 1	Coat 2	Coat 3	Coat4	Coat5	Coat6
Start	0.97a	0.97a	0.97a	0.97a	0.97a	0.97a	0.97a	1.01a	1.01a	1.01a	1.01a	1.01a	1.01a	1.01a
15	0.96a	0.93ab	0.94b	0.96a	0.97a	0.94b	0.91c	0.99b	1.00a	0.99b	0.99b	0.99b	0.99b	0.99b
30	0.90c	0.88bc	0.89c	0.90c	0.93ab	0.89c	0.90c	0.96bc	0.90c	0.93bc	0.91c	0.95bc	0.92c	0.95bc
45	0.85de	0.86de	0.83e	0.84de	0.87d	0.84de	0.85de	0.90c	0.89cd	0.90c	0.90c	0.90c	0.91c	0.91c
60	0.84de	0.81f	0.80f	0.80f	0.83ef	0.83ef	0.82ef	0.82e	0.86d	0.85d	0.87de	0.84ed	0.85d	0.81e
75	0.80f	0.79fg	0.78fg	0.79fg	0.80f	0.76g	0.80f	0.77f	0.80e	0.80e	0.81e	0.80e	0.81e	0.79f
90	0.79fg	0.78fg	0.75g	0.78fg	0.79fg	0.75g	0.77fg	0.74g	0.79f	0.74g	0.76fg	0.75g	0.75g	0.75g

Coat 1:( Wax)- Coat 2 : (Jjoba oil) - Coat 3 : (Sesame oil) - Coat 4 : (paraffin oil) - Coat 5: (Orange oil) - Coat 6 : (Corn oil)  
 Mean Comparison L.S.D. at (0.05)

**Table (5) : Effect of different rind coating on T.S.S / acid ratio of washington navel orange during storage at 5±1Co and 85 – 90 % RH**

Days of Storage	First season (2004)							Second season (2005)						
	Rind Coating							Rind Coating						
	Control	Coat 1	Coat 2	Coat 3	Coat4	Coat5	Coat6	Control	Coat 1	Coat 2	Coat 3	Coat4	Coat5	Coat6
Start	11.13f	11.13f	11.13f	11.13f	11.13f	11.13f	11.13f	11.38i	11.38i	11.38i	11.38i	11.38i	11.38i	11.38i
15	11.35f	11.61ef	11.54ef	11.25f	11.98ef	11.49ef	12.09e	11.59h	10.87i	11.69h	10.81i	11.90h	11.29h	11.59h
30	12.37e	12.50e	12.25e	12.17e	12.04e	12.36e	12.56e	12.59g	12.44g	12.55g	12.42g	12.53g	12.72g	12.39g
45	13.65d	13.26d	13.49d	13.69d	13.45d	13.51d	13.77d	13.95f	13.10f	13.21f	12.61g	13.50f	13.23f	13.17f
60	14.64cd	14.57cd	14.63cd	14.38c	14.40c	14.22c	14.76cd	15.73d	13.37f	13.93f	13.33f	15.09d	14.65e	15.19d
75	16.00a	15.06b	15.26b	14.94c	15.25b	15.79b	15.63b	16.88c	14.86e	15.25d	14.53e	15.88ed	15.66d	16.48c
90	16.46a	16.03a	16.27a	15.77b	15.95b	16.27a	16.75a	18.24a	15.57d	15.70d	15.96d	17.51b	16.95c	17.37b

Coat 1:( Wax)- Coat 2 : (Jojoba oil) - Coat 3 : (Sesame oil) - Coat 4 : (paraffin oil) - Coat 5: (Orange oil) - Coat 6 : (Corn oil)  
 Mean Comparison L.S.D. at (0.05)

**Table (6) : Effect of different rind coating on ascorbic acid content of washington navel orange during storage at 5±1Co and 85 – 90 % RH**

Days of Storage	First season (2004)							Second season (2005)						
	Rind Coating							Rind Coating						
	Control	Coat 1	Coat 2	Coat 3	Coat4	Coat5	Coat6	Control	Coat 1	Coat 2	Coat 3	Coat4	Coat5	Coat6
Start	48.36a	48.36a	48.36a	48.36a	48.36a	48.36a	48.36a	48.79a	48.79a	48.79a	48.79a	48.79a	48.79a	48.79a
15	47.50b	48.00a	48.10a	47.90ab	47.90ab	47.50b	46.20c	47.90cd	48.10b	48.20ab	48.00b	48.00b	48.30ab	48.10b
30	44.00de	44.60d	44.70d	44.30de	44.10de	44.30de	44.10de	45.00de	45.80d	46.00d	45.70de	44.90e	45.90d	45.90d
45	41.70h	43.00e	44.10de	43.50ef	42.00e	44.00de	43.70e	42.30g	44.30e	45.20d	44.90e	43.80f	45.00de	45.00de
60	40.20g	42.40f	43.20e	42.80f	41.30gh	43.10e	42.90e	40.10i	42.70g	43.60fg	43.00g	42.90g	44.00e	43.20fg
75	39.20h	41.30fg	42.40g	41.20fg	40.80g	42.60ef	41.80fg	39.40i	41.50h	42.00g	41.60h	41.30h	42.40g	41.90h
90	39.10i	40.30g	41.40h	40.50g	40.50g	41.80h	40.90h	38.80i	39.70i	41.80h	40.20i	39.80i	41.90h	41.60h

Coat 1:( Wax)- Coat 2 : (Jojoba oil) - Coat 3 : (Sesame oil) - Coat 4 : (paraffin oil) - Coat 5: (Orange oil) - Coat 6 : (Corn oil)  
 Mean Comparison L.S.D. at (0.05)



**Table (7): Volatile components isolated by dynamic headspace volatiles of fresh (zero time), coated and uncoated (control) samples of ripe orange stored for 3 months (\*values expressed as relative area percentages to total identified components).**

Peak No.	Kl*	Components	Fresh	Stored samples for 3 months							Methods of identification <sup>b</sup>
			Control (zero time)	Jojoba oil coated	Orange oil coated	Corn oil coated	Sesame oil coated	Paraffin oil coated	Wax treated	Control	
1	602	Methanol	*9.69	7.89	7.75	6.83	2.72	3.95	1.86	5.01	St, Kl
2	617	Ethanol	10.67	9.36	10.67	8.37	5.84	7.25	5.23	6.17	St, Kl
3	628	Acetone	1.90	-	-	-	-	-	-	0.34	St, Kl
4	638	1-Propanol	2.12	0.63	1.01	0.62	-	-	-	0.31	Kl, MS
5	647	Ethyl acetate	2.42	3.17	2.21	3.42	1.42	3.28	2.55	1.23	Kl, MS
6	664	Isobutanol	1.56	0.58	0.57	0.43	0.52	0.43	0.45	0.30	Kl, MS
7	695	1-Butanol	0.09	0.10	0.09	0.15	0.08	0.08	0.17	0.05	Kl, MS
8	731	1-Penten-3-ol	0.08	0.09	0.08	0.10	0.02	0.04	0.18	0.04	Kl, MS
9	740	1-Penten-3-one	0.43	0.38	0.41	0.19	0.10	0.18	0.04	0.15	Kl, MS
10	741	Ethyl propionate	0.42	0.28	0.91	0.29	0.97	2.68	1.14	0.18	Kl, MS
11	753	3-methyl butanol	0.08	0.28	0.28	0.63	-	0.25	0.23	0.21	Kl, MS
12	756	Methyl butyrate	0.71	2.73	2.84	6.58	0.14	2.69	0.42	2.49	Kl, MS
13	770	(E)2-Hexenal	0.73	0.24	0.39	0.18	0.13	0.08	0.15	0.10	Kl, MS
14	797	(Z)3-Hexen-1-ol	0.09	0.04	0.04	0.09	-	0.02	-	0.03	Kl, MS
15	847	Ethyl butyrate	11.49	15.78	25.76	23.59	48.70	66.92	58.44	21.60	Kl, MS
16	939	α-Pinene	0.69	1.04	0.68	0.77	0.73	0.02	0.60	1.23	St, Kl
17	952	Sabinene	0.46	0.06	0.12	0.05	0.20	0.12	0.17	0.06	St, Kl
18	992	Myrcene	1.47	2.18	0.90	1.92	0.34	0.05	0.42	4.05	St, Kl
19	1006	α-Phellandrene	1.01	0.92	1.56	0.58	-	2.56	0.33	0.48	St, Kl

Table (7) Continued

Peak No.	Kl <sup>a</sup>	Components	Fresh	Stored samples for 3 months							Methods of identification <sup>b</sup>
			Control (zero time)	Jjoba oil coated	Orange oil coated	Corn oil coated	Sesame oil coated	Paraffin oil coated	Wax treated	Control	
20	1017	Ethyl hexanoate	0.22	0.31	0.08	0.60	1.90	0.07	1.21	0.58	KI, MS
21	1025	Octanal	1.31	0.93	1.91	0.57	1.50	2.51	1.37	0.52	KI, MS
22	1035	D-limonene	49.52	49.11	36.53	41.24	30.73	5.84	19.90	51.86	St, KI
23	1069	□-Terpinene	0.23	0.17	0.18	0.05	0.11	0.47	0.07	0.10	St, KI
24	1085	Ethyl octanoate	0.41	0.46	0.27	0.21	0.05	0.04	0.11	0.02	KI, MS
25	1097	(Z) linalool oxide	0.10	0.13	0.32	0.27	0.28	-	0.21	0.32	KI, MS
26	1136	Octanol	0.39	0.64	0.58	0.34	0.42	0.37	0.26	0.49	KI, MS
27	1146	(E) linalool oxide	0.13	0.11	0.14	0.29	0.44	-	0.25	0.12	KI, MS
28	1160	Nonanal	0.11	0.30	0.27	0.24	0.51	-	0.50	0.50	KI, MS
29	1164	Linalool	0.16	0.36	0.36	0.28	0.17	0.44	0.44	0.45	KI, MS
30	1211	Ethyl-3- hydroxyhexanoate	0.48	0.35	0.75	0.18	0.85	-	0.29	0.26	KI, MS
31	1216	Terpinen-4-ol	0.15	0.18	0.24	0.10	0.08	-	0.80	0.11	KI, MS
32	1219	Decanal	0.24	0.25	0.24	0.28	0.22	-	0.20	0.29	KI, MS
33	1231	□-Terpineol	0.14	0.16	0.32	0.05	0.19	-	0.20	0.09	KI, MS
34	1256	Carvone	0.22	0.24	0.47	0.37	0.22	-	1.18	0.10	KI, MS
35	1273	Neral	0.15	0.19	0.25	0.12	0.46	-	0.62	0.24	KI, MS

Compounds listed according to their elution on DB5 column.

a: Kovats index.

b: Compound identified by GC-MS (MS) and / or by kovats index on DB5(KI) and / or by comparison of MS and KI of standard compound (St) run under similar GC-MS conditions

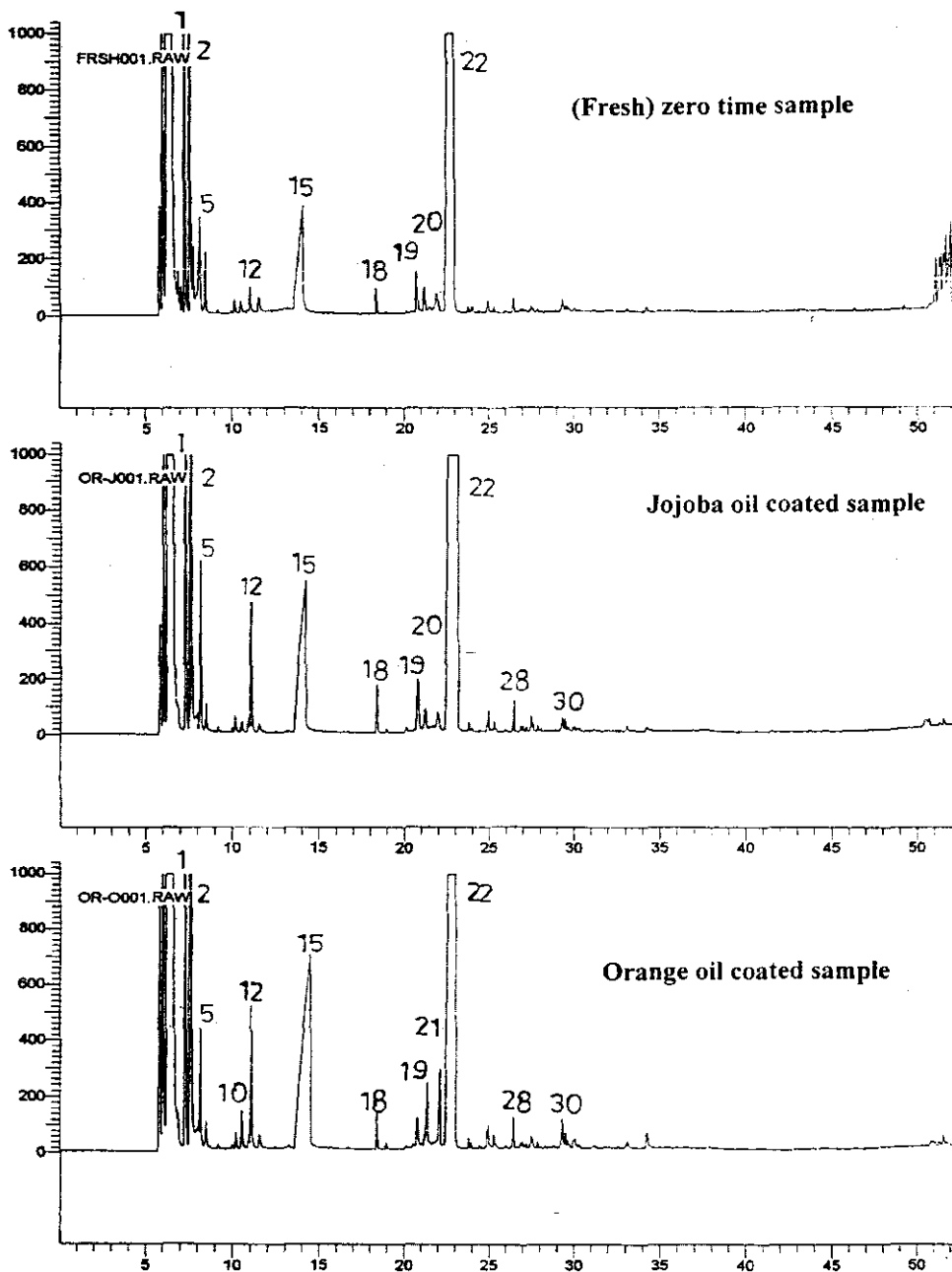


Fig. (1): Gas chromatograph of volatiles in headspace of (Fresh) zero time, jojoba oil coated sample, orange oil coated sample of ripe orange.

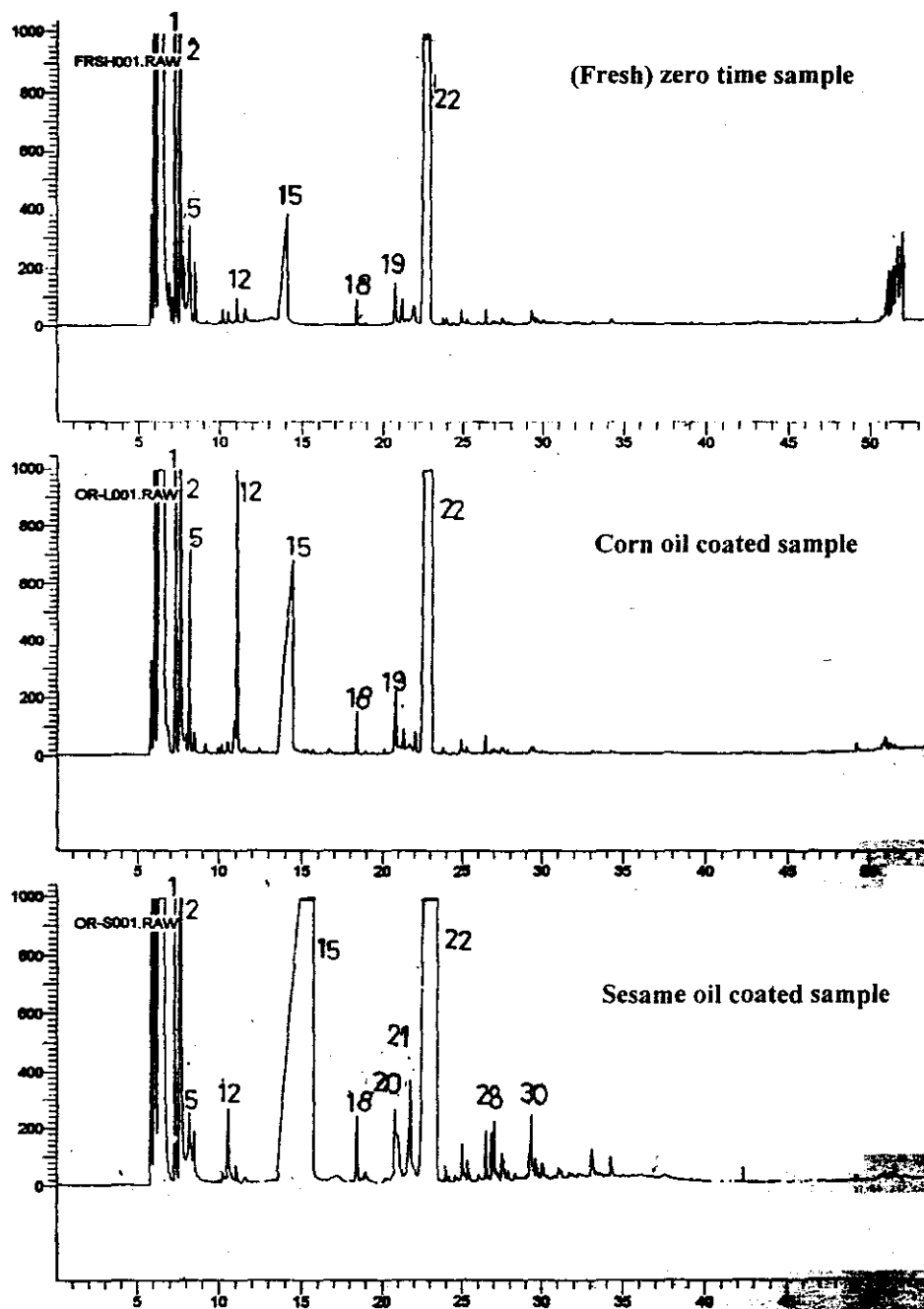


Fig. (2): Gas chromatograph of volatiles in headspace of (Fresh) zero time, corn oil coated sample, sesame oil coated sample of ripe orange.

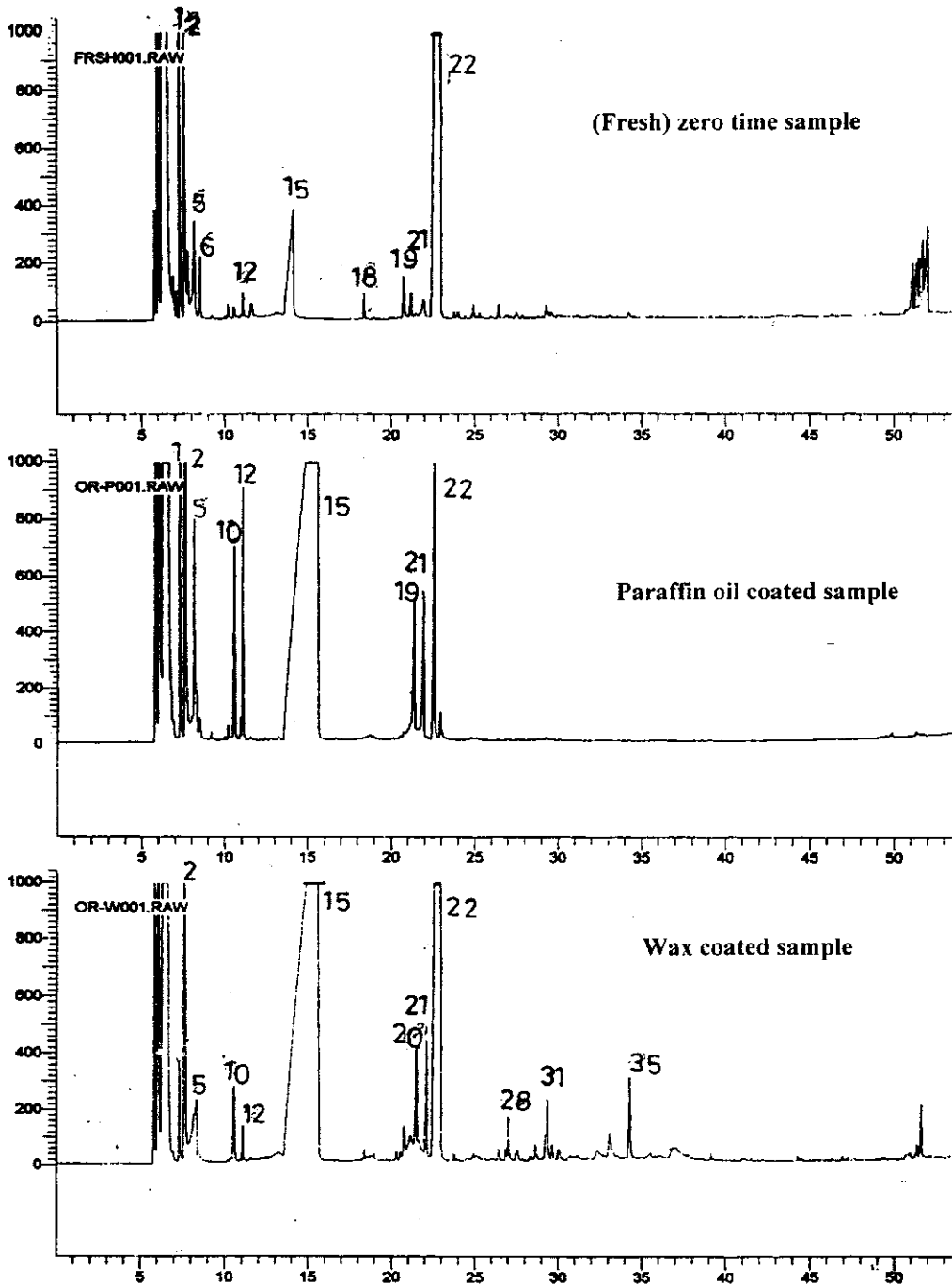


Fig. (3): Gas chromatograph of volatiles in headspace of (Fresh) zero time, paraffin oil coated sample, wax coated sample of ripe orange.

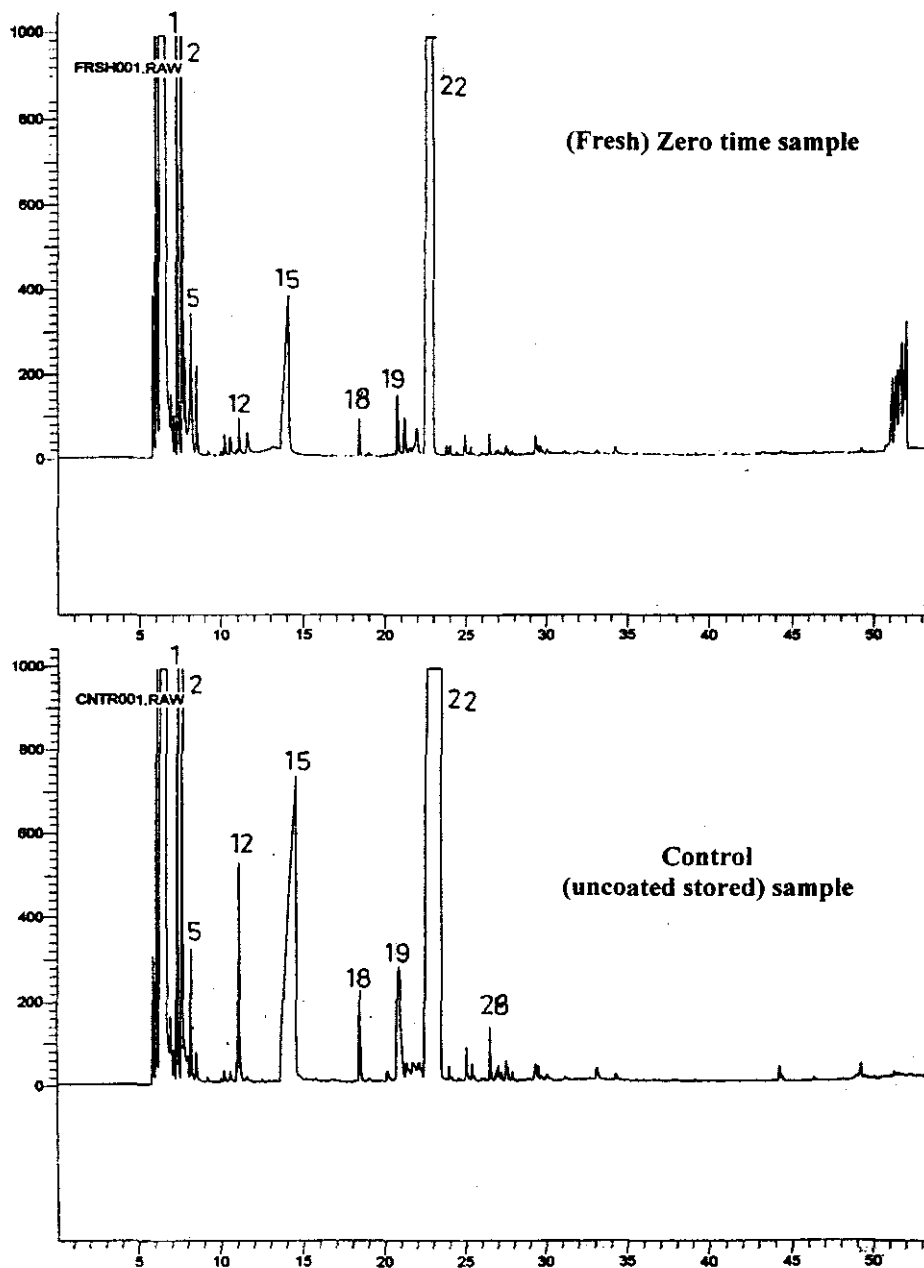
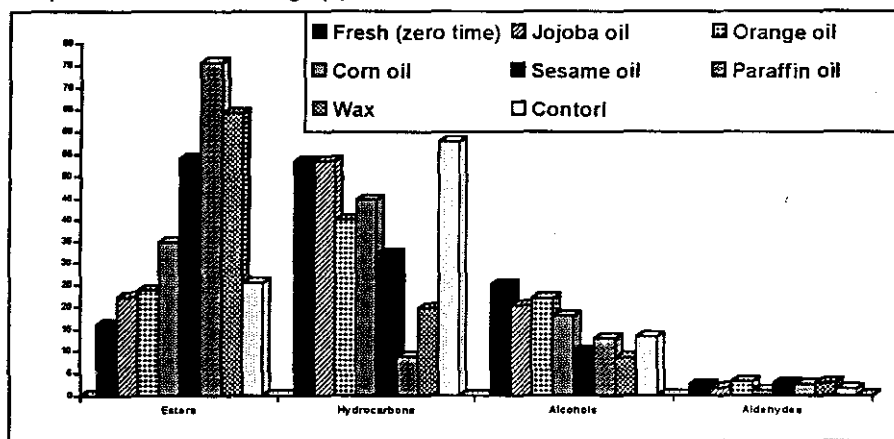


Fig. (4): Gas chromatograph of volatiles in headspace of (Fresh) zero time, control (uncoated stored sample) of ripe orange.

The total area percentages of the main chemical classes of volatile components in the headspace of fresh (zero time), coated stored and control samples are shown in Fig. (5)



**Fig. (5):** The total area percentages of the main chemical classes of volatile components in the headspace of fresh, stored coated and control samples of ripe orange.

Thirty five volatile components were identified in Navel Orange (cv. Washington) grown in Egypt. They included 12 alcohols, 7 esters, 5 aldehydes, 6 terpenic hydrocarbons, 3 ketones and others.

As shown in Table (7), coating treatments resulted mainly in quantitative rather than qualitative differences in the composition of the headspace volatiles of ripe oranges.

**Esters:**

Volatile esters were found to be the major components in volatiles of Washington orange fruits. As shown in Table (7) ethyl butyrate, methyl butyrate and ethyl acetate were the major esters in all samples, these three esters are known to contribute to the "tope-note" of fruit flavours including citrus (*Arctander, 1969*). The total ester content of fresh sample was 16.15% (Fig. 5). In the stored sample coated with jojoba oil, orange oil and corn oil the esters recorded 22.28%, 23.82%, and 34.87% respectively, whereas coating with sesame oil, paraffin oil and wax caused great increase in total ester contents (54.03%, 75.68% and 64.16%, respectively). This notable increase could be attributed to the remarkable increase in ethyl butyrate percentage which recorded in these three samples 48.7%, 66.92% and 58.44%, respectively (Table, 7) . It was also found that ethyl butyrate is the major ester in fresh as well as in all stored coated samples including control one. *Ahmed et al. (1978b)* found that ethyl butyrate is the major volatile ester in orange juices and orange flavour fractions and is an important contributor to desirable flavour in orange products. Also, *Nisperos-Carriedo and Shaw (1990)* reported general decrease in ethyl butyrate, as well as in total esters, which was observed with decreasing quality of the product type. Coated fruit

showed higher concentrations of many volatile compounds as time of storage increased, most notably ethanol, ethyl butanoate, ethyl acetate and  $\alpha$ -pinene (Baldwin *et al.* 1995).

#### **Hydrocarbons:**

As shown in Table (7), it was found that limonene was the major hydrocarbon compound identified in all samples. The total concentration of hydrocarbons in jojoba oil coated sample was the same as in the fresh (zero time) sample, which recorded 53.48% and 53.38%, respectively (Fig 5), with limonene content in both 48.11% and 49.08%, respectively. Also hydrocarbons content was 44.61% in corn oil coated sample with 41.24% limonene. Ahmed *et al.* (1978b) found limonene to be an important contributor to orange flavour. On the other hand, it was found that in Table (7) limonene showed remarkable decrease in four coated samples with orange oil, sesame oil, paraffin oil and wax, which recorded 36.53%, 30.73%, 5.84% and 19.9%, respectively. This result was confirmed by Nisperos-Carriedo (1990) who found general decrease in limonene content in some stored coated samples. Also as shown in Table (7) there is no clear change in limonene content between fresh sample and control stored sample which was also confirmed by Nisperos-Carriedo (1990) who found that levels of limonene, the major volatile hydrocarbon, were fairly constant in the control juice samples during storage.

Other hydrocarbons such as  $\alpha$ -pinene, showed slight increase in most of stored coated samples, while  $\alpha$ -phellandrene showed increase only in orange oil, paraffin oil coated samples. Myrecene showed remarkable increase in stored coated samples with jojoba, corn oil and control samples, while these decreased in the other four coated samples.  $\gamma$ -Terpinene showed variable changes (Table 7).  $\gamma$ -Terpinene has a citruslike aroma, while sabinene has a warm, spicy aroma and flavour (Arctander, 1969).

#### **Alcohols:**

In our study a decrease in concentration of both major alcohols (methanol and ethanol) in all coated stored samples was detected compared with their concentrations in fresh sample (9.69 and 10.67% respectively) Table (7). The values of these alcohols were graduated from 18.42% for the orange oil coated sample to 7.09% for the wax coated sample.

Baldwin, *et al.* (1995) reported that ethanol, methanol and linalool increased roughly 2-fold at both storage temperatures in shellac-coated fruit compared to uncoated control by the end of storage.

Ethanol was the major alcohol present in all samples and in orange oil coated sample, it recorded (10.67%) as in fresh sample. Nisperos-Carriedo and Shaw (1990) confirmed these results. The main function of ethanol in synthetic/flavourings and perfumes is to act as a solvent and to provide a lift to other aromas (Arctander, 1969), and it probably performs a similar function in orange juice products.

The minor alcohols isobutanol, 1-butanol, 1-penten-3-ol, 3-methyl butanol and Z-2-hexen-1-ol showed variable changes in coated stored samples (Table 7). Z-2-hexen-1-ol was an important contributor to the green leafy top-note in fresh orange flavour and in other fruit flavours (Nisperos-Carriedo and Shaw, 1990; Arctander, 1969).



Linalool (other minor alcohol) showed an increase in its concentration in all coated stored samples and control than its concentration in fresh sample (Table 7). Linalool showed significant increase (for at least one storage temperature) for one or both coating treatments by the end of the storage period (Baldwin *et al.* 1995). Linalool was one component of orange peel oil (Ahmed *et al.* 1978b; Shaw, 1979), it was the most odor-active or key compounds of citrus Hyuganatsu aroma (Choi *et al.* 2001). Terpinene-4-ol,  $\alpha$ -Terpineol and octanol also minor alcohols were found in trace amounts in all samples. Terpinene-4-ol and  $\alpha$ -Terpineol were not detected in paraffin oil coated sample. (Table 7).  $\alpha$ -Terpineol is a degradation product of limonene, the major orange oil constituent, and is a known contributor to off-flavour in orange juice at levels of 2ppm or higher (Tatum *et al.* 1975).

#### **Aldehydes:**

Five aldehydes were identified in headspace volatiles of fresh (zero time), coated and uncoated (control) stored samples. These aldehydes are, (E)- $\alpha$ -hexenal, octanal, nonanal, decanal and neral.

From Table (7), (E)-2-hexenal decreased in all coated stored samples and control than fresh sample. 2-Hexenal was reported to be derived from enzymatic oxidation and isomerization of long chain fatty acids (linolenic) during disintegration of fruits (Robertson *et al.* 1990).

Octanal and decanal are generally considered important contributors to orange flavour (Arctander, 1969; Boelens and van Gemert, 1987). In the present study octanal was found to be a major aldehyde and comprised a significant percentage in fresh and orange, sesame, paraffin oils and wax treated stored samples (1.31%, 1.91%, 1.5%, 2.51% and 1.37%, respectively). Nonanal and decanal were absent in paraffin oil coated sample. Nonanal showed remarkable increase in all other samples, it recorded five folds increase in control, wax treated and sesame oil coated samples compared with fresh sample.

From the aforementioned results it can be concluded that, generally coated fruits showed significant increase in components considered important to fresh orange flavour (ethyl butyrate, methyl butyrate and ethyl acetate),  $\alpha$ -pinene, octanal, nonanal, neral and linalool. Jojoba oil, orange oil and corn oil coated samples gave the best results and were the nearest to fresh sample. These three samples showed, besides the previous components, an increase in myrcene and decanal which could add the fruity top-notes of orange flavour.

The very high concentration of esters in paraffin and wax coated samples are not related to good quality. In a previous study (Abd El-Mageed, 2001) the esters comprised the predominant compounds in ripe peach and apricot. Also the control sample had high concentration of terpenic hydrocarbons (57.78%) which according to Moshonas and Shaw (1994) are not related to good quality in orange juice.

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## تأثير بعض مستحلبات الزيوت والشمع على إطالة فترة تخزين البرتقال أبو سررة وأشنجتون ومحتواها من مركبات النكهة

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أجرى البحث على ثمار البرتقال أبو سررة خلال موسمي ٢٠٠٤/٢٠٠٥ المأخوذة من مزرعة خاصة بمدينة السادات وتعرضت الثمار للمعاملات التالية : التغليف بالشمع، التغليف بالمستحلب الزيتي لكل من (زيت الجوجوبا، زيت السمسم، زيت البرافين، زيت البرتقال، زيت الذرة) وثمار أخرى تركت بدون معاملة (استخدمت كعينة قياسية) وتم تخزين الثمار على درجة حرارة ٥°م ونسبة الرطوبة من ٨٥-٩٠%، لمدة ٩٠ يوم. وقد أوضحت النتائج أن التغليف بمستحلب زيت الجوجوبا وزيت البرتقال كانت من أفضل المعاملات حيث قلت من تلف الثمار والفقد في الوزن وزيادة العمر التخزيني. أما السكريات الكلية الذاتية فقد زادت بزيادة فترة التخزين بينما نقصت نسبة الحموضة وحمض الأسكوربيك.

تم جمع المركبات الطيارة لعينة البداية والعينات المعاملة المخزنة وتم تحليلها باستخدام جهاز التحليل الغازي الكروماتوجرافي – طيف الكتلة وقد تم تعريف ٣٥ مركب لنكهة البرتقال منها ٦ مركبات تريپينية و٧ مركبات استرلات للأحماض الأليفاتية و١٢ مركب كحولي و٥ دهيدات و٣ كيتونات والباقي مركبات أخرى. أوضحت نتائج التحليل أن الاختلاف في المركبات الطيارة بين العينة للطازجة (عينة البداية) والعينات المعاملة كان كميًا أكثر منه نوعيًا. الثمار المعطلة عموماً أظهرت زيادة واضحة في المركبات التي تعتبر مسؤولة عن رائحة وطعم البرتقال الطازج ومن هذه المركبات ايثيل بيوتيرات وميثيل بيوتيرات والايثيل استيات والفابيين والأوكتانال والنونال واللينالول .

وبمقارنة نتائج تحليل العينات المعاملة بعينة البداية اتضح أن التغليف بزيت الجوجوبا أو بكل من زيت البرتقال وزيت الذرة قد حافظ على نكهة الثمار طوال فترة التخزين.