

## Evaluation of a Polyethylene-Candelilla Coating for Tangerine Fruit cv. Sai Num Pung

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### ABSTRACT

*Tangerine fruit cv. Sai Num Pung were coated with four microemulsion wax formulations. The main ingredients were polyethylene and candelilla in ratios : 100:0, 75:25, 60:40 and 0:100 (100% PE, 75% PE, 60% PE and 0% PE). Coated and washed non-coated fruit (control) were stored in plastic baskets at 25±2°C and 87±4% RH. The accumulation of ethanol content increased with storage time. Fruit coated with 0% PE had ethanol content over 1,500 ppm after 16 days which imparted off-flavor but in other coatings, after 21 days storage while non-coated fruit never reached this value. Non-coated fruit and fruit coated with 100% PE had the highest percentage of weight loss and internal O<sub>2</sub> but the lowest ethanol content and internal CO<sub>2</sub>. Addition of candelilla wax to polyethylene decreased the percentage of weight loss and internal O<sub>2</sub> but increased internal CO<sub>2</sub> and ethanol content. Coating treatments did not influence total soluble solids, titratable acidity, pH values or vitamin C content of tangerine juice. However, there were significant changes during storage. Titratable acidity and vitamin C content of all coated fruit decreased and total soluble solids and pH values increased during storage.*

**Key words:** Tangerine fruit; Microemulsion wax coating; Ethanol; Physico-chemical evaluation

### INTRODUCTION

Tangerine fruit cv. Sai Num Pung is a hybrid cultivar grown commercially in northern Thailand. The fruit is flat (oblate) with an orange peel and firm texture. The flesh is tender, juicy with a sweet/sour taste, and has a rich flavor. Harvested fruit, especially those which are waxed, develop off-flavor after a few days at room temperature (30-35°C) (Ke and Kader, 1990). Cohen et al., (1990)

emphasized the necessity of wax coating to impart a high gloss to the skin and to prevent water loss and shrinkage, thus prolonging the marketing life of fresh fruit. However, Davis and Hoffman (1973) showed that coating various citrus fruit cultivars affected respiration and composition of the internal atmosphere, leading to the development of distinct off-flavor. High ethanol content is an indication of off-flavor (Cohen et al., 1990; Ke and Kader, 1990). Coatings formed an additional barrier on the fruit through which gases must be passed and modified peel gas permeance and ability to block openings in the peel. Therefore, they had different effects on gas exchange (Hagenmaier and Baker, 1993).

This study aims to evaluate the effect of experimental wax coatings on internal gases, ethanol content, gloss, weight loss and chemical changes in order to provide information for selection of the best coating for the tangerine fruit cv. 'Sai Num Pung'.

## MATERIALS AND METHODS

### Fruit

Tangerine fruit cv. Sai Num Pung were harvested in December 2006 from Chiang Mai Thanathon Co., Ltd. in Chiang Mai Province. Fruit were selected for the mean diameter about 6.0-6.5 cm with no defects. Coatings were applied to fruit by hand with latex gloves. The average amount applied was about 0.20 g per fruit. After air-drying, coated and washed non-coated fruit (control) were stored in plastic baskets at  $25\pm 2^\circ\text{C}$  and  $87\pm 4\%$  RH. All analyses were determined on day 1 and every 5 days for 21 days.

### Coatings

Wax microemulsion coatings were prepared in our laboratory as previously reported by Hagenmaier and Baker (1995); Hagenmaier (2000); Hagenmaier and Goodner (2002). The compositions of all coatings are shown in Table 1. Polyethylene wax (grade A-C 673-P, Honeywell Specialty Chemicals, New Jersey, USA), candelilla wax (grade S.P.75, Strahl & Pitsch Inc., W. Babylon, NY, USA), oleic acid (Panreac Quaimica SA, Barcelona, Spain), myristic acid (Scharlau Chemie S.A., Barcelona, Spain),  $\text{NH}_3$  (Merck KGaA, Darmstadt, Germany), morpholine (Panreac Quaimica SA, Barcelona, Spain) and water were used for preparing polyethylene, candelilla and polyethylene-candelilla wax microemulsion coatings.

**Table 1.** Compositions of the coatings used in the experiment.

| Coating name                              | Abbreviation | Components   |
|---|--------------|--|
| Polyethylene wax microemulsion            | 100% PE      | 18.3% polyethylene wax, 3.7% oleic acid, 2.6% morpholine and 75.4% water   |
| Candelilla wax microemulsion              | 0% PE        | 18% candelilla wax, 3.7% oleic acid, 1.1% NH <sub>3</sub> , and 77.2% water  |
| Polyethylene-candelilla wax microemulsion | 75% PE       | 16% polyethylene wax, 4% candelilla wax, 3.8% oleic acid, 1.1% myristic acid, 1.1% NH <sub>3</sub> and 78% water   |
| Polyethylene-candelilla wax microemulsion | 60% PE       | 12% polyethylene wax, 8% candelilla wax, 3.8% oleic acid, 1.1% myristic acid, 1.1% NH <sub>3</sub> and 74.0% water |

### Internal gas

Gas samples (ten fruit per treatment of each experiment) were withdrawn with a syringe (previously flushed with N<sub>2</sub> to remove traces of oxygen) from the columella of fruit held under water. For determination of internal O<sub>2</sub> and CO<sub>2</sub> concentrations, a loop (167 µl) was used for on-column injection into a gas chromatograph (Shimadzu 8A, Japan) fitted with a CTR-1 column (2 m, 6 mm o.d., 6 ft long, Alltech, Deerfield, IL, USA). Column flow rate was 100 ml/min. Temperatures were 65 and 200°C, respectively, for the column and thermal conductivity detector. Peak areas obtained from standard gas mixtures were determined before and after correction for 0.93% Ar in the atmosphere.

### Ethanol content

Juice samples for ethanol were pooled from nine tangerine fruit per treatment of each experiment. The juice was extracted with a hand-squeezed kitchen juicer. Ethanol content in juice was determined using an ethanol test kit (Ethanol 229-29 from Diagnostic Chemicals Limited, Charlottetown, PE, Canada).

### Flavor

Juice flavor was evaluated after smelling and tasting fruit segments from three fruit for each treatment, by 8 panelists, using rating on a 15 cm line, labeled 'very fresh or very good' at the high end, and 'fermented or over-ripe' at the low end.

### Weight loss

Weight loss during storage was determined from ten fruit set aside for each treatment, by subtracting sample weights from day 1 and expressed as % weight loss every 5 days for 21 days.

### Moisture content of the peel

The moisture content of the peel was determined by the hot air oven (hot air oven, MEMMERT, UM 500, Germany) at 105°C for 72 hours (Hall, 1980). Moisture content was reported on wet weight basis.

## Gloss

Gloss was measured at 60° from a line normal to the fruit surface with the model micro-TRI-gloss reflectance meter (BYK Gardner Inc., Silver Spring, MD, USA) fitted with a shield having a 19 mm diameter hole (Hagenmaier and Baker, 1994). The values of gloss, reported in Gloss Units (G.U.), are means of 10 readings per fruit. Gloss was also evaluated using ranking method by 20 panelists. After storage for 1 day and every 5 days, peel gloss was also scored on trays with three fruit by using a subjective scale of 1 to 5 where 5 = excellent shine, 4 = good shine, 3 = fair shine, 2 = slight shine and 1 = no shine by 8 panelists.

## Chemical compositions

### *Total soluble solids, titratable acidity and pH value*

A digital refractometer (ATAGO N1, Japan) was used to determine the total soluble solids (TSS) of juices. Titratable acidity (TA) was measured by direct titration to pH 8.1 with 0.1 N NaOH standardized solution and the results were expressed as g citric acid per 100 ml juice (AOAC, 2000). The pH value of juice was determined with pH meter (Consort P 407, SCHOTT GERATE, Belgium).

### *Vitamin C content*

Tangerine juice was filtered through 0.45 µm membrane filter. The reduction reactions were done as follow: 15 ml of the filtrate or standard stock solution L-ascorbic acid (Merck KGaA, Darmstadt, Germany) were mixed with 35 ml of 4% oxalic acid (Ajax Fine Chem, Wellington, New Zealand) (AOAC, 2000) The slightly modified HPLC method (Furusawa, 2001) was used to determine the ascorbic acid (mg/100 g juice) of tangerine juice. The HPLC was constituted of a LC-10A pump and SPD-10A UV variable wavelength detector (Shimadzu, Osaka, Japan) set up at 245 nm. The column was a Shimpack (Shimadzu) NH<sub>2</sub> bonded-phase 250 mm x 4.6 mm i.d., 5 µm particle size, the mobile phase was a mixture of 0.5% (v/v) acetic acid (Merck KGaA, Darmstadt, Germany) solution (in water) and methanol (Fisher Scientific UK Limited, Loughborough, UK) (90:10, v/v) at a flow rate of 1 ml/min. The volume injected was 20 µl. The pure (99.5%) L- ascorbic acid (Merck KGaA, Darmstadt, Germany) was used as standard.

## Statistical analysis

Statistix 8 (Analytical Software, Tallahassee, USA) was used for computation of statistical parameters. Data were treated for multiple comparison by analysis of variance with LSD between means determined at  $P=0.05$ . Linear regression and correlation was also done in some analyses.

## RESULTS AND DISCUSSION

### Internal gases

Internal gas concentrations of stored tangerine fruit were markedly affected by coating (Table 2 and Figures 1a and 1b). During 21 days storage at 25±2°C and 87±4% RH, coated fruit decreased internal O<sub>2</sub> and increased internal CO<sub>2</sub> in

comparison with non-coated fruit. Fruit coated with 100% PE, 75% PE and 60% PE coatings had higher internal O<sub>2</sub> and lower internal CO<sub>2</sub> than 0% PE. There was no difference between fruit coated with 75% PE and 60% PE coatings. Adding candelilla wax into the formulation tended to decrease the gas permeability of coating. Fruit coated with 75% PE and 60% PE coatings had lower internal O<sub>2</sub> and higher internal CO<sub>2</sub> than 100% PE. Mandarin fruit coated with polyethylene wax and polyethylene-candelilla wax coatings and stored 7 days at 21°C had internal O<sub>2</sub> about 5.7% and 6.0% and internal CO<sub>2</sub> about 7.0% and 10.4%, respectively (Hagenmaier, 2002).

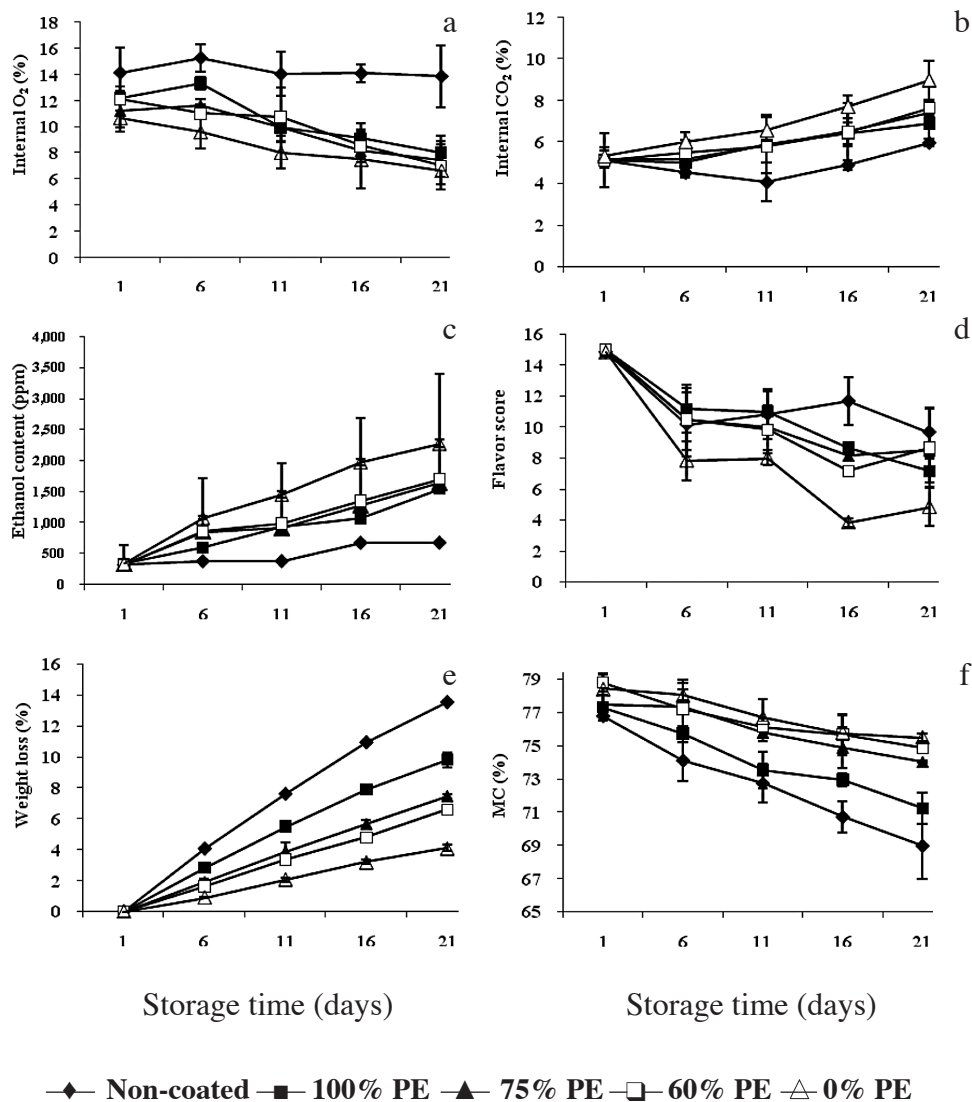
**Table 2.** Mean internal gas concentrations (O<sub>2</sub> and CO<sub>2</sub>), ethanol content and flavor score of tangerine fruit cv. 'Sai Num Pung' after 1 and 21 days storage.

| Treatment  | O <sub>2</sub> (%) |         | CO <sub>2</sub> (%) |         | Ethanol (ppm) |         | Flavor score |         |
|------------|--------------------|---------|---------------------|---------|---------------|---------|--------------|---------|
|            | 1 day              | 21 days | 1 day               | 21 days | 1 day         | 21 days | 1 day        | 21 days |
| Non-coated | 14.14a             | 13.87a  | 5.11                | 5.97c   | 318c          | 679 e   | 14.83        | 9.67a   |
| 100% PE    | 12.20ab            | 8.04b   | 5.13                | 6.87bc  | 343a          | 1,545d  | 15.00        | 7.17ab  |
| 75% PE     | 11.23b             | 7.47bc  | 5.18                | 7.40b   | 334b          | 1,649c  | 15.00        | 7.50a   |
| 60% PE     | 12.10ab            | 7.03bc  | 5.11                | 7.62b   | 319c          | 1,694c  | 15.00        | 7.67a   |
| 0% PE      | 10.69b             | 6.68bc  | 5.31                | 8.97a   | 338ab         | 2,272a  | 14.83        | 4.83bc  |
|            |                    |         | ns                  |         |               |         | ns           |         |

Mean values in the same column that are followed by different letters are significantly different ( $p < 0.05$ , LSD).

### Ethanol content

During storage for 21 days, non-coated fruit had the lowest ethanol content (Table 2 and Figure 1c). Fruit coated with 100% PE, 75% PE and 60% PE coatings showed relatively low ethanol content. In contrast, ethanol content of fruit coated with 0% PE coating rapidly increased. Ethanol content of the juice from coated tangerine fruit varied considerably for each coating treatment. Coatings tended to overly restrict the exchange of O<sub>2</sub> and CO<sub>2</sub> in the atmosphere. Fruit those internal O<sub>2</sub> concentration became too low to sufficiently support anaerobic respiration resulted in high values of internal ethanol, acetaldehyde and internal CO<sub>2</sub> (Ke and Kader, 1990; Hagenmaier, 2002). High ethanol content is an indication of off-flavor (Ahman and Khan, 1987; Cohen et al., 1990; Ke and Kader, 1990; Hagenmaier, 2000). Off-flavor has been reported for citrus fruit with an ethanol content higher than 1,500 ppm (Cohen et al., 1990, Ke and Kader, 1990; Hagenmaier 2002). In the present study, tangerine fruit stored for 16 days showed ethanol content of 1,976 ppm when coated with 0% PE coating, indicating off-flavor. Ethanol content of fruit coated with 100% PE, 75% PE and 60% PE coatings were higher than 1,500 ppm (1,545, 1,649 and 1,694 ppm, respectively) only after 21 days storage, while non-coated fruit never reached this value. Thus, tangerine fruit cv. 'Sai Num Pung' coated with 100% PE, 75% PE and 60% PE coatings can be expected to maintain acceptable flavor for about 3



**Figure 1.** Changes in internal O<sub>2</sub> (a), internal CO<sub>2</sub> (b), ethanol content (c), flavor score (d), weight loss (e) and moisture content (MC) of the peel (f) of tangerine fruit cv. Sai Num Pung coated with various coatings during storage at 25±2°C and 87±4% RH for 21 days.

weeks at room temperature.

The relationship between ethanol content and internal  $O_2$  of tangerine fruit coated with 60% PE coating was linear with negative correlation ( $R^2=0.97$ ) and positive correlation was found between ethanol content and internal  $CO_2$  ( $R^2=0.95$ ). This indicated that low internal  $O_2$  and high internal  $CO_2$  increased the accumulation of ethanol. A positive correlation between ethanol content and internal  $CO_2$  had also been reported for 'Valencia' oranges and 'Marsh' grapefruit (Hagenmaier and Baker, 1994; Hagenmaier, 2000).

### Flavor score

The flavor scores of stored tangerine fruit were markedly different for coatings (Table 2 and Figure 1d). During 21 days storage, non-coated tangerine fruit had the highest flavor scores. Fruit coated with 100% PE, 75% PE and 60% PE coatings had higher flavor scores than fruit coated with 0% PE coating. Flavor score decreased linearly with increasing ethanol content ( $R^2=0.92$ ) from tangerine fruit coated with 0% PE coating. A high negative correlation between flavor and ethanol content has also been reported for 'Valencia' orange (Ke and Kader, 1990; Hagenmaier, 2000).

### Weight loss

Waxing the fruit is practised to help reduce water loss (Cohen et al., 1990). In this study, the percentage of weight loss in non-coated tangerine fruit was higher than coated fruit during storage for 21 days (Figure 1e). The average rate of weight loss in non-coated fruit was 0.56% per day while in fruit coated with 100% PE, 75% PE, 60% PE and 0% PE coatings were 0.42, 0.34, 0.30 and 0.19% per day, respectively. Candelilla wax was incorporated into the formulation in order to reduce the melting point, hardness of the polyethylene wax and also to reduce weight loss of coated fruit (Hagenmaier, 1998). Thus, 60% PE coating which had more candelilla wax in the formulation than 75% PE coating tended to have less weight loss.

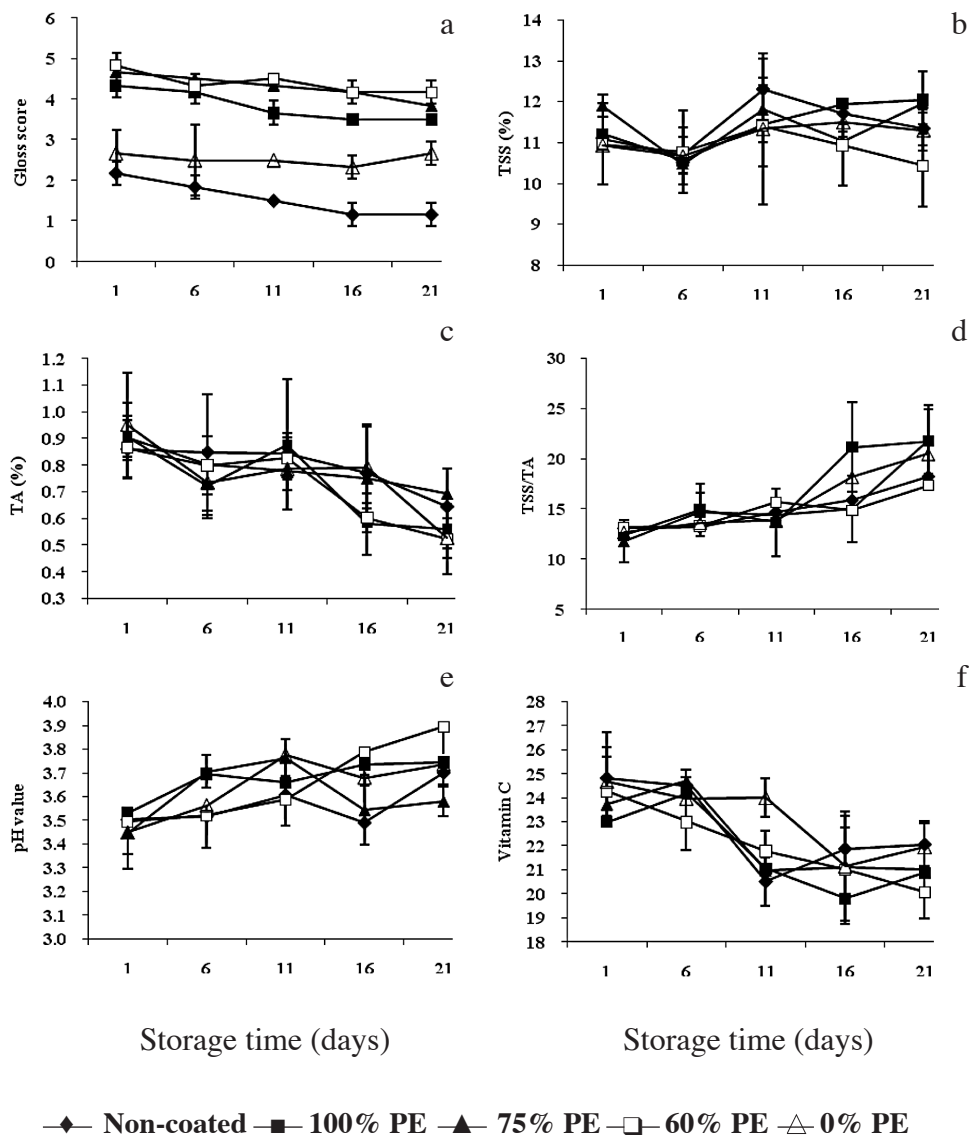
### Moisture contents of the peel

Peel dehydration is the major reason for commercial decline of the orange (Ben-Yehoshua, 1969). Drying of peel is also related with fruit weight loss (Hall, 1980). During storage tangerine fruit for 21 days, the moisture contents of the peel gradually decrease (Figure 1f). The mean rate of moisture loss from the peel was highest in non-coated tangerine fruit, about 0.34% per day and fruit coated with 100% PE, 75% PE, 60% PE and 0% PE coatings were 0.22, 0.17, 0.19 and 0.12% per day, respectively.

### Gloss

It is important to consumers that fruit should have high gloss (Hagenmaier and Baker, 1994). After coating application, the gloss value of fruit coated with 100% PE, 75% PE, 60% PE, 0% PE and non-coated fruit were 4.29, 4.71, 4.40, 3.12 and 2.66 G.U., respectively. Using ranking method by panelists, the results

showed that the gloss was in order of 75% PE > 60% PE > 100% PE > 0% PE > non-coated fruit. During storage for 21 days, peel gloss decreased with storage time for all coated fruit (Figure 2a). Fruit coated with 75% PE and 60% PE coatings had good gloss and appearance throughout the storage period. Fruit coated with



**Figure 2.** Changes in gloss score of the peel (a), total soluble solids (b), titratable acidity (c), TSS/TA ratio (d), pH value (e) and vitamin C content (f) of tangerine fruit cv. Sai Num Pung coated with various coatings during storage at  $25\pm 2^{\circ}\text{C}$  and  $87\pm 4\%$  RH for 21 days.



100% PE coating initially gave high gloss but decreased after 16 days. Candelilla wax (0% PE) tended to have low gloss, but it was improved when mixed with the polyethylene wax. 'Valencia' orange coated with polyethylene-candelilla wax coating and stored 8 days at 15-25°C had better gloss, in addition to better flavor (Hagenmaier, 2000).

#### **Total soluble solids, titratable acidity and pH values**

TSS, TA, TSS/TA and pH values of tangerine fruit during storage for 21 days are shown in Figures 2b, 2c, 2d and 2e. Coating treatments did not influence TSS, TA, TSS/TA and pH values but there were significant changes during storage. TSS, TSS/TA and pH values of all coated fruit increased but TA decreased during storage. Ethanol and acetaldehyde content are important in determining flavor. Other factors, such as TSS, TA and their interaction with volatile compounds also play a role in orange flavor. TSS and TA can enhance the sensory quality of fresh fruit (Ke and Kader, 1990).

#### **Vitamin C content**

Vitamin C content of tangerine fruit decreased about 11.08-13.88% during storage for 21 days (Figure 2f). Coating treatments did not have influence on vitamin C content. It has been previously reported in 'Avana' mandarins and 'Okitsu' satsuma fruit about a loss of 13% of vitamin C content in segments after 12 days of storage at 4°C under MAP conditions (Piga et al., 2002).

### **CONCLUSION**

Results from this work showed that 60% PE (polyethylene-candelilla wax microemulsion) could be a good coating for tangerine fruit cv. 'Sai Num Pung' because it can reduce percentage of weight loss, maintain high internal O<sub>2</sub> and low internal CO<sub>2</sub>, maintain low ethanol content, impart surface gloss and retain good quality for about 21 days of storage.

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