Evaluation of a polyethylene–candelilla coating for ‘Valencia’ oranges[☆]

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Abstract

An experimental polyethylene–candelilla–wax coating formulation was evaluated in comparison to a high-gloss, shellac and wood resin citrus coating for storage of ‘Valencia’ oranges at 15–25°C. Oranges with the wax coating had relatively high flavor scores (8.9–10.4) even after 9–16 days storage at 15–25°C. By contrast, the high-gloss coating, shown to be typical of such citrus coatings, resulted in flavor scores as low as 3.7–4.1 after 9–16 days at 25°C or 16 days at 21°C. Flavor was especially low for fruit with internal O₂ < 1%. Flavor tended to decrease almost linearly with increasing ethanol content, which in turn was highly dependent on internal CO₂. Oranges with the shellac and wood resin coating temporarily had better gloss than fruit with the wax coatings, but this advantage may be lost after 8 days storage at 15–25°C. Published by Elsevier Science B.V.

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1. Introduction

The most commonly used coatings or ‘waxes’ applied to fruit in US citrus packinghouses have as their major ingredients a mixture of shellac and

wood resin. These coatings were designed primarily to impart high-gloss at the time of inspection by the buyer, usually after application of the coating.

When coatings are applied to fruit, they form an additional barrier through which gases must pass. Because coatings differ in gas permeance and ability to block openings in the peel, they have different effects on gas exchange (Hagenmaier and Baker, 1993). Citrus fruit with shellac- and wood resin-based coatings generally had lower internal O₂, higher internal CO₂ and higher ethanol content than fruit with wax coatings (Hagenmaier and Baker, 1994). High ethanol content, in turn, is an indication of off-flavor (Ahmad and

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Khan, 1987; Cohen et al., 1990; Ke and Kader, 1990). Shellac- and wood resin-based coatings also tended to increase prevalence of postharvest pitting (Petracek et al., 1997, 1998).

The purpose of this study was to evaluate an experimental wax coating on 'Valencia' oranges, with emphasis on flavor and gloss stability.

2. Materials and methods

2.1. Coatings

An experimental polyethylene–candelilla wax coating was made with 150 g polyethylene (grade A-C316 from Allied Signal, Inc., Morristown, NJ), 109 g candelilla wax (grade S.P. 75 from Strahl & Pitsch Inc., W. Babylon, NY), 47 g oleic acid (Emersol 6321, from Henkel Corp., Cincinnati, OH), 13 g myristic acid (Hystrene 9014 from Witco Corp., Memphis, TN and Emery 655 from Henkel Corp.) and 42 g of 30% NH_3 , and 865 g water. The polyethylene wax, candelilla wax, oleic acid, myristic acid, ammonia and 125 g water were heated in a pressure cell (Parr Instrument Co., Moline, IL) to 170°C, stirred at 250 rpm, an additional 550 g of water at 95°C was added, and stirred for another 5 min. The mixture was rapidly cooled to 55°C and the remaining water added to dilute the solids content to 26%. Turbidity of microemulsions was measured in units of nephelometric turbidity units (NTU) with the Ratio/XR turbidimeter (Hach Co., Loveland, CO). A coating preparation made April 18 was used for the experiments conducted on April 19–May 22, 1998.

The reference coating for these experiments (HIGLOSS) was a commercial high-gloss citrus coating used in citrus packinghouses in Florida and California. The principal components were shellac and wood resin in proportion 3:1, and it also contained, fatty acid soaps, propylene glycol and other minor ingredients, but no wax. The name of the product (which is not HIGLOSS) and the manufacturer are withheld on request. Four other commercial shellac–wood resin citrus coatings used for comparison were Britex 555 (Brogdex Co., Pomona, CA), FMC 590HS (FMC

Corp., Lakeland, FL), 'Luster Dry' (EcoScience, Orlando, FL) and B560-64 (EcoScience). An experimental shellac coating made in our laboratory consisting of 15.4% shellac (R52, Mantrose Haeuser, Attleboro, MA), 3.0% polyethylene glycol 600 (Union Carbide, Danbury, CT), 1.8% morpholine, 0.8% whey protein ('Bipro', Davisco, Le Sueur, MN), 0.5% oleic acid, 0.3% NH_3 , 150 ppm SE21 (10% polydimethyl siloxane antifoam preparation, Wacker Silicones Corp., Adrian, MI) and the balance water.

Oxygen permeances of coatings were measured with the Oxtran 100 (Modern Controls Inc., Minneapolis, MN). Coatings of thickness 8–25 μm were formed on polyethylene support films, and their thicknesses determined from weights and thicknesses of coated and noncoated support films. Permeance was determined at 30°C, 70% relative humidity from measurement of oxygen passing through coated and non-coated support film. The reported permeability values are equivalent to the permeances of coatings with thickness of 25 μm .

2.2. Fruit

The 'Valencia' oranges were all from Polk county, FL harvested between April and June, 1998. The fruit, not treated with ethylene for 'degreening', were cleaned with rotating polyethylene brushes, using fruit-wash detergent (Decco Kleen 241 or 278, (Elf Atochem, Monrovia, CA) and dipped in 1000 ppm imazalil (Fungaflor 500EC, Janssen Pharmaceutica, Titusville, NJ). Coatings were applied to fruit by hand (latex gloves); the mean amount applied was 0.25 g per orange. For each of the four experiments (April 29, May 3, May 12 and May 20), 20 fruit were treated with HIGLOSS coating and 20 with the wax coating, for each of three storage conditions (15, 21 and 25°C, with relative humidity of 25, 50 and 45%, respectively).

2.3. External quality

Reflectance measurements of fruit shine were measured in gloss units (G.U.) with the micro-TRI-gloss reflectance meter (BYK Gardner Inc.,

Silver Spring, MD) fitted with a shield having 19 mm diameter hole (Hagenmaier and Baker, 1994). For each experiment, ten measurements per fruit were made on each of the ten numbered oranges stored for 1 day and again after 9 days, and also for ten oranges stored for 1 day and again after 16 days. For comparison of different commercial coatings, gloss was ranked visually by ten untrained panelists, who arranged trays with five fruit according to relative gloss (no ties permitted).

Firmness was taken as the force in newtons (N) required to compress the fruit by 10 mm between two flat surfaces closing together at the rate of 20 mm/min (Model 1011, Instron Corp., Canton, MA). Firmness of each fruit was measured only once, as subsequent values were progressively lower, ten fruits per treatment. Weight-loss samples consisted of three trays of five oranges for each treatment, weighed 1 and 8 days after application of the coating.

2.4. Internal quality

Internal gas samples (ten fruits per treatment, each experiment) were withdrawn with a syringe (previously flushed with N₂ to remove traces of oxygen) from fruit held under water. For determination of O₂ and CO₂ concentrations, we used a loop (167 µl) for on-column injection into a Hewlett Packard 5890 gas chromatograph fitted with a CTR-1 column (2 m, 6 mm o.d., 6 ft long, Alltech, Deerfield, IL). Column flow rate was 140 ml/min. Temperatures were 40 and 120°C, respectively, for the column and thermal conductivity detector. Peak areas obtained from standard gas mixtures were determined before and after analysis of the samples. Oxygen concentration was calculated from the O₂–Ar peak area after correction for 0.93% Ar in the atmosphere.

Juice samples for ethanol were pooled from five pieces of fruit, the juice extracted with a rotary kitchen juicer, spiked with 1000 ppm n-propanol as internal standard, centrifuged and injected into the gas chromatograph using a FFAP column (Hewlett Packard) and flame ionization detector. Column flow was 4 ml/min. Column temperature was 55°C for injection, then increased at a rate of 3°C/min to 70°C and held for 1 min.

Juice flavor was evaluated after sniffing and tasting, by 14 trained panelists, who marked their rating on a 15-cm line, the low end of which was labeled ‘fermented, over ripe’ and the high end ‘very fresh or very good.’ For training purposes, juice from freshly harvested fruit and from stored fruit with elevated ethanol content was also presented.

Statistix 4.1 (Analytical Software, Tallahassee, FL) was used for computation of statistical parameters. Standard errors (S.E.) are shown as error bars on the graphs except when covered by the symbols. The changes in weight and gloss that occurred during storage were computed as mean changes on numbered fruit, and treated as paired comparisons. The reported probabilities (*P*) are for the null hypotheses.

3. Results and discussion

3.1. Coatings and internal gases

The polyethylene–candelilla-wax microemulsion used for this study was one of many wax microemulsion coatings formulated in our laboratory (Hagenmaier, 1998). The specific polyethylene wax (A-C316) used in that microemulsion had density and softening point considerably higher than polyethylene waxes generally used as fruit coatings. (Based on information from the manufacturer density was 0.98 g/cc and Mettler drop point was 140°C). This emulsion had pH 9.6 and turbidity of 834 NTU. Candelilla wax was incorporated into the formulation in order to reduce the melting point and hardness of the wax and also to reduce weight loss of the coated fruit. Of the waxes that can be used in fruit coatings, polyethylene is the most permeable to water vapor and candelilla (together with beeswax) is least permeable (Hagenmaier and Baker, 1997). Candelilla wax coatings tend to have low gloss, but this seems to have been compensated for by mixing it with polyethylene wax.

The mean oxygen permeabilities at 30°C of HIGLOSS, and the polyethylene–candelilla wax coating, in units of mol/(m s Pa), were $1.7 \pm 0.4 \times 10^{-15}$ and $0.07 \pm 0.01 \times 10^{-15}$, respectively.

The resistance offered to transport of a gas through a coating or film, which is the reciprocal of the permeance, is proportional to thickness of the film (Crank, 1964). The approximate thickness of both coatings on the ‘Valencia’ oranges was 2–3 μm , assuming uniform coverage of the fruit (200–300 mg of liquid coating at 20–26% solids content spread over $\sim 200 \text{ cm}^2$ surface area).

Internal gas concentrations were markedly different for the two coatings, and also depended somewhat on storage temperature (Table 1). Oranges coated with HIGLOSS and stored 9–16 days at 21–25°C had internal O_2 values of 0.7–0.9% and internal CO_2 of 17–22%. Oranges with the wax coating had internal O_2 of 8–11% and internal CO_2 of 5–7% at all storage temperatures. Those with HIGLOSS coating stored at 15°C were intermediate between these two groups.

There was no change in internal gas concentration from 9 to 16 (Table 1). It has been previously observed that internal gases reach a steady-state value within about 4 h after application of the coating (Hasegawa and Iba, 1980).

3.2. Internal quality

The flavor scores of stored fruit were markedly affected by coating and storage temperature (Table 2). Flavor scores were highest (8.9–10.5) for oranges with the experimental wax coating at all storage conditions (9–16 days at 15–25°C). Flavor scores were lowest (3.4–3.8) for the HIGLOSS coated oranges stored 16 days at 21°C and 9–16 days at 25°C. Thus, flavor was markedly reduced by non-refrigerated storage of ‘Valencia’ oranges. Although recommended stor-

Table 1

Mean internal gas concentrations of fruit after 9 and 16 days storage. Mean values for fruit harvested on April 29, May 3, May 12 and May 22 ^a

Storage temperature (°C)	CO_2 (%)				O_2 (%)			
	Wax		HIGLOSS		Wax		HIGLOSS	
	9 days	16 days	9 days	16 days	9 days	16 days	9 days	16 days
15	5.2 ^{aA}	5.2 ^{aA}	8.7 ^{aB}	9.6 ^{aB}	11.2 ^{Aa}	10.6 ^{Aa}	3.4 ^{aB}	2.5 ^{aB}
21	6.3 ^{aA}	6.8 ^{aA}	18.4 ^{bB}	17.4 ^{bB}	9.3 ^{aA}	8.5 ^{aA}	0.7 ^{bB}	0.8 ^{bB}
25	6.5 ^{bA}	6.7 ^{aA}	22.0 ^{bB}	21.3 ^{bB}	8.4 ^{aA}	7.6 ^{aA}	0.8 ^{bB}	0.9 ^{bB}

^a Different upper-case superscripts for CO_2 or O_2 in the same row, or different lower-case superscripts in the same column indicate significant differences ($P < 0.05$).

Table 2

Ethanol and flavor scores of juice after 9 and 16 days storage^{a,b}

Storage temperature (°C)	Flavor score				Ethanol (ppm)			
	Wax		HIGLOSS		Wax		HIGLOSS	
	9 days	16 days	9 days	16 days	9 days	16 days	9 days	16 days
15	10.4 ^{aA}	10.5 ^{aA}	8.8 ^{aB}	8.3 ^{aB}	1170 ^{aA}	1290 ^{aAB}	1790 ^{aBC}	2290 ^{aC}
21	9.4 ^{aA}	8.9 ^{aA}	5.1 ^{bB}	3.4 ^{bC}	1360 ^{aA}	1500 ^{aAB}	2880 ^{bB}	5360 ^{abC}
25	9.3 ^{aA}	9.9 ^{aA}	3.4 ^{cB}	3.8 ^{bB}	1730 ^{aA}	1670 ^{aA}	3760 ^{cAB}	7310 ^{bB}

^a For a given parameter (ethanol or flavor), different upper-case superscripts in the same row or different lower-case superscripts in the same column indicate significant differences ($P < 0.05$).

^b Mean values for fruit harvested on April 29, May 3, May 12 and May 22. Mean ethanol contents at day 0 for the four experiments were 450, 480, 680 and 730 ppm, respectively.

Fig. 1. Flavor versus ethanol after 9–16 days at 15, 21 and 25°C. Ethanol content before storage for the four experiments was 450–730 ppm.

age temperature for Florida oranges is 0–1°C (Grierson and Ben-Yehoshua, 1986), it is not uncommon for oranges to be stored at ambient indoor temperature during marketing; and in addition, oranges are often not refrigerated until transported from the packinghouse to warehouse. Therefore, flavor changes during unrefrigerated temperatures do seem relevant to fruit quality.

The HIGLOSS coated fruit stored at 21–25°C had high ethanol content (Table 2). The rate of increase of ethanol for these treatments was 420 ± 56 and 300 ± 45 ppm/day, respectively, at 25 and 21°C. By comparison, the ethanol content of the oranges coated with wax coatings, increased by only about a fifth of this rate, namely at 70 ± 21 , 60 ± 12 ppm/day, respectively, at storage temperatures of 25 and 21°C, indicating less fermentation with the wax coating. The rates of increase of ethanol at 15°C were 110 ± 14 and 44 ± 12 ppm/day, respectively, for HIGLOSS and wax coatings. Thus, although lowering the storage temperature to 15°C did prevent the deterioration in flavor observed with the HIGLOSS coating only after 1 week at 21–25°C, the rate of increase of ethanol content was somewhat higher than fruit with the wax coating. These rates of increase in ethanol content were determined by linear regression of ethanol from 1–16 days storage; somewhat higher values were observed for the first 9 days.

Flavor decreased linearly ($r^2 = 0.81$) with increasing ethanol in the range of about 800–5000

ppm (Fig. 1), taking into consideration data from all three storage temperatures, whether stored for 9 or 16 days, with wax or HIGLOSS coating. Over the range 800–5000 ppm, ethanol content appears to be a good estimate of flavor degradation. Baldwin et al. (1995) reported marked increases in flavor volatiles, especially ethanol, ethyl butyrate and ethyl acetate, in ‘Valencia’ oranges coated with a shellac-based citrus coating and stored at 16–21°C. High negative correlation between ethanol and flavor have also been reported for mandarin oranges (Ahmad and Khan, 1987) and ‘Valencia’ oranges (Ke and Kader, 1990).

Not shown in the tables and figures are the differences in means between the fruit harvested on the four dates. Regression analysis showed no dependence of ethanol, CO₂ or O₂ on harvest date except for the fruit with the HIGLOSS coating stored at 25°C, which tended to have higher CO₂ for fruit harvested at a later date. Fruit with this coating had internal CO₂ (mean of values measured after 9 and 16 days) of 15, 21, 22 and 29% for oranges harvested on April 29, May 3, May 12 and May 22, respectively. Thus, the HIGLOSS coating was somewhat less suitable as a coating as the season progressed.

Ethanol was especially high when internal O₂ < 1.0% (Tables 1 and 2). Thus, about 1.0% internal O₂ might be considered the internal gas equivalent of the minimum O₂ required in modified atmosphere packaging, the so-called ‘extinction point’ (Ko et al., 1996). This interpretation suggests an alternate way of reporting the data, given the fact that the mean values of O₂ were somewhat higher than the median values because of occasional samples with rather high internal values, caused by breaks in the skin too small to see. Accordingly, an alternate parameter might be the proportion of the samples having internal O₂ below the extinction point. Only one of 240 oranges with wax coating had internal O₂ < 1%, compared with 12, 70 and 66%, respectively, of the oranges stored at 15, 21 and 25°C with the HIGLOSS coating.

Linear regression of ethanol against internal CO₂ gave fairly good fit (Fig. 2). For example, for wax or HIGLOSS coated fruit stored 9 days at 25°C,

Fig. 2. Ethanol and internal CO₂ for oranges after storage for 9 days, combined results for storage at 21 and 25°C.

Ethanol (ppm) = $(1013 \pm 273) + (121 \pm 16)[CO_2]$, where, $[CO_2]$ is percent internal CO₂. For this equation, $R^2 = 0.89$ and $F = 56$. Because ethanol content increased with time and internal CO₂ did not, the regression parameters were dependent on storage time and temperature. The slope was 120 ± 15 ppm/(% CO₂) for 9 days at 21°C, 450 ± 106 for 16 days at 25°C and 280 ± 113 for 16 days at 21°C. These results are similar to those reported earlier, namely 140 ± 13 ppm after storage for 14 days at 23°C (Hagenmaier and Baker, 1994).

3.3. External quality

Probably the most important aspect of external quality is gloss. Gloss decreased with storage time

Fig. 3. Difference in reflective gloss (value for the shellac-resin coated minus value for the wax coating) at 15, 21 and 25°C. Mean results for the four experiments; each point is the mean of 400 measurements.

for all coatings and storage temperatures, at a faster rate for the HIGLOSS coating than the wax coating, and faster also for higher storage temperatures (Table 3). The HIGLOSS coating initially gave higher gloss than the wax coating, but this reversed after about 8 days storage at all storage temperatures (Fig. 3Table 3), after which the oranges with polyethylene–candelilla wax coatings had better gloss, in addition to better flavor (Table 2). Previous results from our laboratory have shown similar results with carnauba wax and polyethylene wax coatings (Hagenmaier and Baker, 1994).

The weight loss was markedly lower for the polyethylene–candelilla wax coating than the HI

Table 3
Mean changes in reflective gloss (in G.U.) during 8 or 15 days, each based on 40 paired comparisons of gloss on a numbered orange after storage for 1 day and again after 9 or 16days^a

Storage temperature (°C)	Wax		HIGLOSS	
	8 days	15 days	8 days	15 days
15	0.78 ^{cC}	1.09 ^{cB}	1.05 ^{bB}	1.40 ^{cA}
21	1.17 ^{bD}	1.36 ^{bC}	1.61 ^{aB}	1.86 ^{bA}
25	1.41 ^{aC}	1.66 ^{aB}	1.73 ^{aB}	2.07 ^{aA}

^a Different upper-case superscripts for CO₂ or O₂ in the same row, or different lower-case superscripts in the same column indicate significant differences ($P < 0.05$).

Table 4

Mean changes during storage (all were losses)^a

Storage temperature (°C)	Weight (%)				Firmness (N/day)	
	Wax 8 days	15 days	HIGLOSS 8 days	15 days	Wax	HIGLOSS
15	0.7 ^{Db}	1.5 ^{Bb}	1.0 ^{Cb}	2.2 ^{Ab}	0.19 ^{Ab}	0.36 ^{Ac}
21	2.4 ^{Ba}	4.3 ^{Ba}	4.6 ^{Ba}	8.3 ^{Aa}	0.60 ^{Aa}	0.88 ^{Ab}
25	2.7 ^{Ca}	5.3 ^{Ba}	5.2 ^{Ba}	10.3 ^{Aa}	0.93 ^{Aa}	1.23 ^{Aa}

^a The same fruit was weighed after storage for 1 day and again after 9 or 16 days. Firmness was measured on day 0 and also after 9 or 16 days storage, using different fruit each time. Different upper-case superscripts for the same parameter in the same row, or different lower-case superscripts in the same column indicate significant differences ($P < 0.05$).

GLOSS coating (Table 4). Mean rates of weight loss at 21–25°C were about 0.25%/day for the wax coating and about 0.47%/day for the HIGLOSS coating, and these rates were virtually the same whether calculated for a storage period of 8 or 15 days. Firmness, which decreased faster at higher storage temperature, was not highly correlated with weight loss (Table 4).

There was good correlation between weight loss and decrease in gloss ($r^2 = 0.85$, Fig. 4). As the fruit lost weight it tended to shrink and the peel become somewhat wrinkled and had a tendency to shed coating. These facts suggest that loss in weight is partly responsible for the observed decrease in gloss. Thus, the wax coating may have preserved gloss by slowing down the shrinkage rate.

3.4. Comparison of commercial coatings

Oranges coated with the HIGLOSS coating had ethanol content, internal gases and weight loss similar to those of fruit coated with four other shellac-wood resin citrus coatings and an experimental shellac coating (Table 5). These data indicate that the HIGLOSS coating was a fairly typical high-gloss citrus coating with respect to gas exchange and tendency to undergo anaerobic respiration. Therefore, it is likely that all of these shellac-wood resin coatings block gas exchange between fruit and atmosphere to such an extent that anaerobic respiration would occur if the coated oranges were stored at 21–25°C.

4. Conclusion

Compared with a typical shellac-wood resin citrus coating, 'Valencia' oranges with the experimental wax coating initially had lower gloss, but after storage for 9–16 days these had lower ethanol content, better flavor and higher gloss. The low flavor scores with shellac-wood resin apparently stemmed from the low O₂ permeance of this coating, which resulted in relatively high internal CO₂ and low internal O₂ of oranges with that coating. It would seem possible, by optimizing the wax formulation, to further improve its gloss. Changes in the wax formulation are unlikely to have much affect on interior gases, ethanol or flavor, because these are relatively insensitive to composition of the coating, provid-

Fig. 4. Regression of gloss decrease on weight loss, results of the individual experiments, oranges stored at 15, 21 or 25°C for 9 or 16 days.

Table 5

Properties of 'Valencia' oranges stored 8 days at 21°C, with various high-gloss citrus coatings^a and means for fruit harvested on June 16 and 17

Coating ^d	Weight loss (%/day)	Gloss rank ^b		Ethanol ^c (ppm)	Internal gases (%)		Percent samples O ₂ < 1%
		Day 1	Day 8		CO ₂	O ₂	
E560	0.63 ^b	5.2 ^a	5.8 ^a	2550 ^b	14.3 ^b	1.1 ^a	55 ^a
B555	0.82 ^a	5.1 ^a	3.6 ^c	2700 ^b	18.8 ^a	1.2 ^a	65 ^a
Luster	0.86 ^a	3.1 ^c	4.7 ^b	2890 ^{a,b}	14.3 ^b	0.5 ^a	85 ^a
F590	0.86 ^a	2.0 ^d	2.4 ^d	2820 ^{a,b}	14.2 ^b	2.0 ^a	60 ^a
HIGLOSS	0.82 ^a	4.0 ^b	3.8 ^c	3210 ^a	22.4 ^a	1.2 ^a	75 ^a
Experimental shellac	0.90 ^a	1.7 ^d	1.1 ^c	2570 ^b	21.5 ^a	0.7 ^a	90 ^a

^a Items in a column are not significantly different if they share a common superscript.

^b Mean rank, with 1.0 being the lowest possible gloss.

^c After 8 days storage. The mean value before storage was 1870 ppm.

^d All are commercial high-gloss coatings except the experimental shellac.

ing that it is composed of wax rather than shellac and wood resin (Hagenmaier and Baker, 1997).

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