Alternatives to Shellac Coatings Provide Comparable Gloss, Internal Gas Modification, and Quality for ‘Delicious’ Apple Fruit

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Additional index words. Malus × domestica, shiny, firmness, modified atmosphere, volatile

Abstract. Zein, starch, polyvinyl acetate (PVA), carnauba, and carnauba-poly saccharide (CPS) coatings were compared with a commercial shellac coating using controlled atmosphere stored ‘Delicious’ apples (Malus × domestica Borkh.). Coated apples were stored in air at 2 °C for 2 weeks and then removed to 21 °C for an additional two weeks to simulate marketing conditions. Gloss, internal O2 and CO2 partial pressures, weight loss, flesh firmness, and contents of sugars, acids and volatiles were measured on 0, 2, and 4 weeks after coating treatment. Starch- and carnauba-coated apples had high initial gloss, similar to that found for shellac-coated fruit. Gloss of all coated fruit decreased similarly during the 4-week evaluation period, although all of the coated fruit were glossier than uncoated controls. For uncoated apples, the differences of O2 and CO2 partial pressure between internal and ambient atmosphere were ≈1 kPa at 2 °C, and these increased by a further 2 kPa after transfer to 21 °C. Fruit coated with shellac and starch had >10 kPa CO2, and <10 kPa O2 at 21 °C. Zein-, PVA- and carnauba-coated apples showed a reduced internal atmosphere (6–7 kPa CO2, 11–15 kPa O2). Internal partial pressures of O2 and CO2 were inversely related for most coatings, except for the CPS coating, for which partial pressures of both O2 and CO2 were low. Carnauba-, PVA-, and shellac-coated fruit lost less weight than uncoated fruit. Starch-, shellac-, and CPS-coated fruit were firmer than those from other coating treatments, and all coated fruit were firmer than uncoated control. Titratable acidity was higher in the fruit coated with CPS, starch, and shellac than in uncoated control. Ethyl alcohol and ethyl esters accumulated in starch-, shellac-, and CPS-coated fruit kept at 2 °C, but, levels of these volatiles decreased after transfer of fruit to 21 °C. Carnauba, PVA, and zein coatings compared favorably to shellac for gloss and other quality characteristics.

Most ‘Delicious’ apples marketed in the United States are coated with shellac or a mixture of shellac and carnauba wax. Shellac has a problem with discoloration (whitening) (Baldwin, 1994; Hagenmaier and Shaw, 1992) which limits marketability, and is sometimes associated with nonfood uses. The latter point might some day be viewed negatively by consumers; therefore, it would seem prudent to develop alternative coatings. Furthermore, shellac is currently not listed as “generally recognized as safe” (GRAS) by the U.S. Food and Drug Administration (FDA).

High gloss is considered by the industry to be beneficial for red apple sales. Recently, it has become common to see high gloss coatings on green apples as well. Reducing weight loss and respiration rate also helps extend shelf-life of apples previously held in controlled atmosphere (CA) or air. Coatings affect the internal atmosphere of fruit and, therefore, also can potentially reduce the respiration rate. Internal O2 and CO2 partial pressures of uncoated ‘Delicious’ apples at ambient temperature were 17–20 kPa and 2–4 kPa respectively (Alleyne and Hagenmaier, 2000; Bai et al., 1990). Coatings cause an increase in internal CO2, and a decrease in O2, partial pressures because of fruit respiration, in a manner similar to modified atmosphere (MA) packaging. Shellac coating on ‘Delicious’ apples raised internal CO2 to ≈10 kPa, and reduced O2 to ≈9 kPa at ambient temperature, and led to ethanol accumulation of ≈10 times that of uncoated control (Alleyne and Hagenmaier, 2000). The gas changes caused by shellac coatings were moderated by adding carnauba or candelilla wax to the formulations. Off-flavor was induced in ‘Starking Delicious’ apples stored in MA packaging at 8 °C, with 6 kPa CO2 and 7–9 kPa O2 partial pressure in the package (Ueda et al., 1993). Assuming an additional gradient of 1–3 kPa O2 and CO2 partial pressures for internal atmospheres relative to package atmosphere (Bai et al., 1990), these results suggested that the off-flavor developed because either internal CO2 exceeded 7–9 kPa, O2 declined to 5–7 kPa, or both.

In this research, we developed several edible and shiny coatings from alternative materials, observed how they affected internal gases, flavor compounds, and subsequently the quality of the coated apples, in an effort to find alternative coatings to shellac. The alternative materials included zein and starch, which are food ingredients with a wholesome image; polyvinyl acetate, which gives high gloss and is an approved food additive (Hagenmaier and Grohmann, 1999); carnauba wax, a natural plant wax; and a natural complex polysaccharide. Zein coatings have been used on candy, dried fruit, nuts, and meats (Baker et al. 1994). Zein also was evaluated on tomatoes resulting in a modified internal atmosphere, color change, inhibition of weight loss, and delayed softening (Park, 1991). Carnauba wax has been used commercially to coat apple, and does not discolor, but has less gloss than shellac. Preliminary experiments showed that application of carnauba coatings resulted in less modification of the internal atmosphere in coated fruit than did shellac, and was more effective in preventing weight loss. Polyvinyl acetate-coated apples had higher internal O2 partial pressures and less alcohol accumulation in fruit compared with shellac (Hagenmaier and Grohmann, 1999).

Material and Methods

‘Delicious’ apples (Malus × domestica Borkh.) were stored in commercial CA (1 kPa for both O2 and CO2 at 0.5 °C and 90% to 95% RH) in Washington State for 4–5 months, then transported to Florida in a refrigerated truck in Mar. 2000. Uniform (180–210 g) defect-free fruit were equilibrated at room temperature (25 °C) for 24 h, prior to application of coatings. Coatings were applied manually, using 0.5 mL/fruit, spread evenly over the fruit surface (surface area of ≈200 cm2) using latex gloved hands. This resulted in a coating thickness of ≈25 μm when wet, which becomes thinner as the coating dries. Each fruit was inspected for complete coverage. Instead of coating, water was used for control fruit. A pilot-plant scale conveyor dryer (Central Florida Sales and Service, Auburndale, Fla.) was used to dry fruit (including controls) at 50 °C for 5 min. All fruit (except those processed for initial, day 0 samples) were stored at 2 °C for 2 weeks, then transferred to 21 °C for a further 2 weeks, to simulate marketing conditions.

The treatments included experimental zein-, starch-, polyvinyl acetate (PVA)-, and carnauba-poly saccharide (CPS)-based formulations, as well as commercial carnauba- (Natural Shine TM 8000; EcoScience, Orlando, Fla.), and shellac-based (Apple Wax 55; EcoScience) coatings and uncoated controls. The main components of the experimental formulations (expressed as percentage by weight) were: 1) zein (8% defatted zein, 8% propylene glycol, 25% isopropyl alcohol, 25% etha-
nol and 34% water; 2) starch [11.7% potato starch (amyllopectin CLS; Abeve, Princeton, N.J.), 3.1% tapioca dextrin (K4484; National Starch and Chemical Co., Bridgewater, N.J.), 3.1% citric acid, 0.8% malic acid, 0.6% whey protein isolate (Bipro; Davisco Foods, Le Sueur, Minn.), 0.8% glycine, 10% isopropanol and 70% water]; and 3) PVA [20% polyvinyl acetate (Union Carbide, Danbury, Conn.), 2.2% citric acid, 0.8% propylene glycol, 57% isopropanol, and 20% water]. The CPS was donated by CH3O (Seattle, Wash.) for which the exact components are proprietary.

Gloss, internal O2 and CO2 partial pressure, weight loss, and flesh firmness were measured on 10 replicate fruit per treatment. Sugar, acid, and volatiles were determined using three composite replicates of three fruit each. Measurements were conducted initially (day of treatment), after 2 weeks of storage at 2 °C, and after removal from chilled storage plus a 2 weeks marketing period at 21 °C. Initial measurements were taken one day after coating treatment to be sure that the coatings had completely dried.

Flesh surface gloss was measured using a micro-TRI-gloss reflectometer (BYK-Gardner, Silver Spring, Md.) equipped with a shield having a circular 19-mm-diameter aperture (Hagenmaier and Baker, 1994), and expressed as gloss units (GU) at an angle of 60°. Ten measurements were made per fruit. The same fruit were used initially and at the end of the storage and marketing periods for gloss measurements.

Flesh firmness was assessed with a penetrometer (FT 327; McCormick, Facchini, Alfonse, Italy), equipped with an 11.1-mm-diameter cylindrical plunger. Two measurements were obtained per fruit from opposite sides where 16-mm-diameter peel discs were removed.

Samples for internal gas were obtained from the core cavity of fruit under submerged conditions (Alleyn and Hagenmaier, 2000). The CO2 and O2 partial pressures were analyzed using a gas chromatograph (HP 5890A; Hewlett-Packard, Avondale, Pa.) equipped with a thermal conductivity detector.

For weight loss determination, fruit were individually weighed initially and at the end of the storage and end of the marketing periods. Sugar, glucose, and fructose were analyzed using a Waters column at 90 °C, with a mobile phase of 100 µm ethylendiamine tetraacetic acid disodium-calcium salt (Ca EDTA), flow rate of 0.5 mL/min, and a Perkin Elmer LC-25 Refractive Index detector. Sucrose equivalents (SE) were used to show the relative sweetness, with coefficients of sucrose, glucose and fructose as 1.0, 0.74, and 1.73 respectively (Kruehler and Kayss, 1991).

For titratable acidity (TA) analysis, homogenates were titrated to pH 8.1 with 0.1 N NaOH, and the acidity was calculated as malic acid on weight basis (g/100 g) (Jones and Scott, 1984).

For volatile analysis, 50-g apple slices (core tissue removed) were homogenized with 25 mL deionized water and 25 mL saturated NaCl solution. Two mL of homogenate was transferred into a 6-mL vial sealed with a crimp-top and Teflon-silicone septum, flash frozen in liquid nitrogen, and stored at –80 °C prior to analysis. For GC analysis, sample vials were thawed under running tap water, heated rapidly to 80 °C and incubated for 15 min by a Perkin Elmer HS-6 headspace sampler heating block before the headspace sample was injected into the GC. The analysis was carried out using a gas chromatograph (Perkin Elmer model 8500) equipped with a 0.53 mm x 30 m polar stabilwax capillary column (1.0-µm film thickness; Restek, Bellefonte, Pa.) and a flame ionization detector. Oven temperature was held 40 °C for 6 min, then raised to 180 °C at a rate of 6 °C/min. The compounds were identified by comparison of retention times with those of authenticated standards and by enrichment of apple homogenate with authentic compounds. Concentrations were calculated by using regression equations, determined by injecting five different concentrations of each standard to obtain a peak height calibration curve as described by Nisperos-Carriedo et al. (1990). Identification of volatiles were periodically checked by spiking homogenate with standards. Volatile components that are abundant or that have been reported to have significance for apple or other fruit flavors (Mattheis et al., 1995) were analyzed including: ethanol, ethyl acetate, ethyl butyrate, butyl acetate, 2-methylbutyl acetate, and hexyl acetate.

PROC GLM of SAS Version 8 (SAS Institute, Cary, N.C.) was used for analysis of variance (SAS Institute, 1999). Mean separation was determined by the Scheffe’s test.

Results and Discussion

Uncoated fruit had low gloss with 3.7 gloss units (GU) initially, that subsequently decreased to 2.7 GU at the end of the total 4-week experiment (Fig. 1). Shellac-coated fruit had an initial gloss up to 11.3 GU, decreased to 10.1 GU after 2 weeks of storage at 2 °C, and further decreased to 7.3 GU in the following 2 week marketing period at 21 °C. Since ‘Delicious’ apples with 6 GU were shiny to the eye, the fruit coated by shellac maintained adequate gloss, even at the end of the marketing period. Starch- and carnauba-coated fruit showed high initial gloss values similar to shellac, while PVA-, zein-, and CPS-coated fruit showed moderate initial gloss (7.5–8.8 GU). The gloss of all coatings decreased during storage, but remained higher than the uncoated control. Since gloss decreased more when the initial value was higher, there were no significant differences among the different coating treatments at the end of 2 weeks storage at 2 °C and 2 weeks marketing at 21 °C (5.9–7.3 GU) (Fig. 1). All the coatings maintained substantial shine after the simulated marketing period (limit of noticeable shine is around 0.0 GU).

Internal (core) O2 and CO2 partial pressures in uncoated fruits at the end of 2 weeks storage (2 °C) were 20.1 and 1.1 kPa, respectively, and after 2 weeks at 21 °C, 18.1 and 2.8 kPa, respectively (Table 1). In coated fruits, more modification of the internal atmosphere occurred. Internal gas partial pressures of fruit coated by shellac were 9.0 kPa O2 and 10.1 kPa CO2 at the end of the 4-week experiment. The
high levels of CO2 observed in shellac and upper CO2 limit of atmosphere is 5 kPa for (Thompson, 1996 and industry sources). The duration. Zein, PVA, carnauba, and CPS coatings (Maynard et al., 1997b). However, higher CO2 often leads a more injury to the fruit, although LOL increased slightly at 32 °C. Elevated CO2 within 8 kPa did not affect LOL in both of the apple varieties mentioned above at 20 °C and affected LOL only slightly at 0 °C (Yearsley et al., 1997b). However, higher CO2 often leads to a lower LOL (Beaudry, 1993). In this work, the high levels of CO2 observed in shellac and starch coated fruits (10.1 and 11.3 kPa, respectively) could be injurious to the fruit, although the marketing period is relatively short in duration. Zein, PVA, carnauba, and CPS coatings resulted in lower levels of CO2 (5.9–7.4 kPa) compared to shelllac and starch. None of the internal O2 levels in coated fruit were low enough to cause anaerobic respiration. However, moderate O2 with a slightly high CO2, such as the combination of 6.0 kPa O2 and 5.9 kPa CO2 in CPS coating, might cause anaerobic metabolism.

CPS-coated apples had low O2 and CO2 partial pressures that were quite different from apples with other coatings. Unlike other coatings, for which the internal O2 and CO2 pressures, when added together, amounted to ≈18–21 kPa, this sum for CPS was only 14 kPa at 2 °C and decreased to 12 kPa after being removed to 21 °C (Table 1). Also, the internal CO2 at a given value of internal O2 was much lower for CPS than for the other coatings (Fig. 2). These observations suggest that the CPS coating tended to block pores in the fruit more than other coating treatments. The rationale for this conclusion follows.

If the dominant pathway for gas exchange between fruit and atmosphere is by permeation through intact portions of the skin, and if the respiratory quotient is near unity, then the CO2 pressure difference across the fruit skin is expected to be roughly 1/3 of the O2 pressure difference because CO2 permeabilities tend to be about three times O2 permeabilities (Banks et al., 1993; Cronin, 1985; Stanmett 1985). Thus, for example, if internal CO2 is 5 kPa and gas exchange is purely by permeation, the expected internal O2 concentration would be ≈6 kPa (15 kPa below ambient). If, on the other hand, all gas exchange is by diffusion through open pores, then at equal flux rates, the CO2 pressure difference would be expected to be ≈30% greater than the that of O2, because its diffusion constant in air is that much lower than that of O2 (Weast, 1988). For all coatings save CPS, the internal CO2 rose by about the same amount that O2 was lowered (thus resulting in the same total for internal CO2 and O2 pressures). This suggests that the gas exchange was partly by permeation and partly by diffusion. For CPS, the internal O2 fell by much more than the CO2 increased, which is what would be expected if less of the gas were exchanged through holes, thus the conclusion that this coating blocked pores (stomates, lenticels, stem and blossom scars). Meanwhile, the other coatings must have not entered into and blocked all pores as effectively as did the CPS coating. This is dependent on coating characteristics such as viscosity and surface tension as well as the peel anatomy of the fruit (Hagenmaier and Baker, 1993). Future research is warranted to understand how coating properties affect ability of coatings to block pores on fruit surfaces, and how percentage of pore blockage affects fruit internal atmosphere.

Weight loss of uncoated fruit was 2.1% after 4 weeks of storage (2 weeks at 2 °C + 2 weeks at 21 °C). The CPS coating was the least effective in inhibiting weight loss (2.2%), while carnauba wax was the most effective (1.3%), although the differences were not great. Other coatings showed intermediate weight loss control of 1.7% to 1.8% (Table 2). Weight loss is mainly caused by evaporation of water from the fruit. Coating, as an additional barrier to the peel, inhibited water loss (except for CPS). It has been reported (Hatfield and Kneze, 1988; Maguire et al., 2000) that 3% to 5% loss of weight can cause shriveling in apples. In this work, weight loss was below 2.2%, and no shrinkage was observed in any treatment. However, water loss can also cause softening of the flesh, ripening, and senescence, through ethylene production and other metabolic changes. Water evaporation and diffusion of O2 and CO2 through the coatings did not show strong relationships.

Initial firmness (force of resistance) of flesh was 65 N. Firmness decreased slowly at 2 °C and faster at 21 °C. After the 4 week storage period, the uncoated fruit averaged firmness values of 37 N (Table 2). All of the coated fruits maintained firmness values of 44 N or greater. Generally, the coating treatments that resulted in higher internal CO2 levels, lower internal O2 levels, or both (Table 1), also

**Table 1. Internal O2 and CO2 partial pressures of ‘Delicious’ apples coated with each of 6 formulations or uncoated, and kept at 2 °C for 2 weeks followed by 21 °C for 2 weeks**

| Treatment | O2 (kPa) | CO2 (kPa) | O2 (kPa) | CO2 (kPa) |
|-----------|----------|-----------|----------|-----------|
| Uncoated  | 20.1 a   | 1.1 c     | 18.1 a   | 2.8 c     |
| Zein      | 17.7 b   | 3.6 b     | 14.6 b   | 6.5 b     |
| Carnuba   | 14.8 c   | 4.6 b     | 14.5 b   | 6.3 b     |
| PVA       | 17.1 b   | 3.6 b     | 11.4 c   | 7.4 b     |
| CPS       | 10.3 d   | 4.6 b     | 6.0 d    | 5.9 b     |
| Shellac   | 11.8 cd  | 5.9 a     | 10.1 a   | 11.3 a    |
| Starch    | 13.5 c   | 4.6 b     | 9.9 c    | 11.3 a    |

1Initial (week 0) partial pressures at 5 °C: 20.0 kPa O2 and 1.8 kPa CO2.
2Mean value (n = 10) in same column that are not followed by the same letter show significant difference (P < 0.05).

**Fig. 2.** Relationship between internal O2 and CO2 of ‘Delicious’ apples coated with each of six coating formulations or uncoated, held for 2 weeks at 2 °C followed by 2 weeks at 21 °C. The linear regression line is for formulations other than the carnauba-poly saccharide (CPS), with a slope of –1.1 and an intercept of 21.0 (\( r^2 = 0.908 \)).
which should not have induced anaerobic metabolism, using accumulated ethanol as substrate for ester production. Ueda et al. (1993) reported that the ethanol evolution of ‘Starking Delicious’ apple, stored in MA packaging with 5–9 kPa O₂ and 5 to 6 kPa CO₂, increased shortly after packaging and decreased gradually after 4 weeks storage at 8°C, while control fruit remained low in ethanol evolution at first, and increased as senescence proceeded. Ethyl esters (for example, ethyl acetate and to a lesser extent, ethyl butyrate) showed a similar pattern to ethanol. However, the ‘Delicious’ apple aroma components butyl acetate, 2-methylbutyl acetate, and hexyl acetate generally increased when the fruits were removed from 2°C to 21°C. The concentrations of these aroma compounds, however, were generally lower in coated fruit than in controls (except for hexyl acetate), in contrast to ethanol and the ethyl esters. In banana, melon and strawberry fruit, the ester production, in conjunction to alcohol moieties, strongly reflected the alcohol content in the fruit (Ueda et al., 1992).

Conclusion

‘Delicious’ apples coated with zein-, starch-, carnauba-, CPS-, and PVA-based formulations showed intermediate initial gloss compared to shellac-coated and uncoated fruit, and ultimately a similar level of gloss compared with fruit coated with shellac, after storage for two weeks at 2°C followed by two weeks at 21°C. Shellac- and starch-coated fruit showed the greatest deterioration of gloss during storage compared to the other coating treatments, partially due to the fact that they had the highest initial gloss readings. The formulations of zein, carnauba, CPS and PVA provided more optimal internal gas levels in term of less CO₂ accumulation, and maintained good quality. The three coatings resulting in the lowest O₂ levels, retarded firmness, gave the highest TA and the lowest SE/TA (indicating delayed ripening) but also accumulated the most ethanol and ethyl acetate. This is interesting since low levels were not low enough to expect inhibition of ethylene production (and subsequent retardation of ripening). Ethanol has been shown to inhibit ethylene synthesis in tomatoes (Kelly and Saltveit, 1998) and these coated fruit had high ethanol levels. However, O₂ levels were not low enough to expect induction of anaerobic ethanol production. One explanation is that CO₂ levels may have induced fermentation, as has been suggested for blueberry, although levels of CO₂ were higher in that study (Beaudry, 1993). Alternatively, perhaps ethanol accumulated within the coating barrier.

Table 3. Concentration of volatile components (µg/100 g) in ‘Delicious’ apples coated with each of six formulations or uncoated, and kept at 2°C for 2 weeks followed by 21°C for 2 weeks

| Treatment | Ethanol | Ethyl acetate | Ethyl butyrate |
|-----------|---------|--------------|---------------|
|           | Week 2  | Week 4       | Week 2  | Week 4       | Week 2 | Week 4       | Week 2 | Week 4       |
| Uncoated  | 102 f   | 169 ef       | 1 f     | 6 ef         | 1 d    | 9 a          |
| Zein      | 127 ef  | 243 e        | 3 f     | 9 de         | 1 d    | 11 a         |
| Carnauba  | 545 cd  | 114 g        | 47 c    | 3 f          | 4 c    | 3 cd         |
| PVA       | 552 c   | 535 cd       | 3 f     | 3 f          | 1 d    | 4 c          |
| CPS       | 1996 b  | 305 de       | 151 b   | 9 de         | 5 c    | 2 d          |
| Shellac   | 2552 b  | 349 d        | 164 b   | 13 d         | 11a    | 4 c          |
| Starch    | 4652 a  | 19 f         | 294 a   | 13 d         | 9 ab   | 6 b          |
| Butyl acetate | 61 a  | 43 bc         | 67 a    | 1 d          | 2 c    |
| 2-methylbutyl acetate | 43 bc | 67 a | 1 d | 2 c |
| Hexyl acetate | 40 d | 43 bc | 53 b | 2 c | 4 ab |

*Mean value (n = 3) in same component that are not followed by the same letter show significant difference (P < 0.05).

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