Formulating a Natural Colorant Containing Wax for a One-step Color-add Application for Fresh Citrus

Xiuxiu Sun

U.S. Horticultural Research Laboratory, USDA, ARS, 2001 South Rock Road, Fort Pierce, FL 34945; and Indian River Research and Education Center, University of Florida, 2199 South Rock Road, Fort Pierce, FL 34845

Elizabeth Baldwin

U.S. Horticultural Research Laboratory, USDA, ARS, 2001 South Rock Road, Fort Pierce, FL

Mark Ritenour

34945 Indian River Research and Education Center, University of Florida, 2199 South Rock Road, Fort Pierce, FL 34845

Robert Hagenmaier and Jinhe Bai¹

U.S. Horticultural Research Laboratory, USDA, ARS, 2001 South Rock Road, Fort Pierce, FL

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Abstract. In Florida, early season citrus fruits usually reach full maturity in terms of internal quality while their peel often does not turn to orange color after degreening due to insufficient buildup of carotenoids. For huanglongbing (HLB)-affected orange trees, the fruit may never turn orange during the entire harvest season, despite any cold weather. Improvement of early season citrus peel color is important to the citrus industry to better meet consumer expectations. Occasionally, packinghouses apply a dye, Citrus Red No. 2 (CR2), to improve the surface color of oranges, temples, and tangelos before applying a fruit wax to impart shine, retain moisture, and slow fruit senescence. In a previous report, we determined that paprika and annatto extracts are comparable to CR2 as natural colorant alternatives. In this research, the goal was to formulate a natural colorant [annatto, paprika, or paprika oleoresin (PO)]-containing carnauba wax coating. The coatings were first evaluated for color, shine, moisture retention, respiration rate, ethylene production, and internal gas content. Control fruit were coated with carnauba wax alone, or dyed with CR2 then coated with carnauba wax. The effects were assessed under different temperature and light exposure conditions to simulate commercial storage and marketing. The results showed that a one-step application of paprika-containing carnauba wax was comparable to the two-step (“CR2 then wax”) applications in improving fruit appearance and modification of internal gas composition.

Citrus HLB is a destructive citrus disease in Florida and other citrus production regions around the world. A primary symptom of HLB is that fruit of affected trees will remain green, and postharvest degreening will not induce their peel to turn orange due to insufficient carotenoid levels (Chen et al., 2016; Zhao et al., 2015). Some fruits, if

harvested late, will naturally degreen on the tree, but will remain yellow instead of completely turning orange (Chen et al., 2016; Zhao et al., 2015). The inability for HLB-affected fruit peel to turn orange even after degreening due to insufficient carotenoids is especially true of ‘Hamlin’s’ (Liao and Burns, 2012).

Before HLB was spread over the Florida citrus regions, only early oranges, temples, tangelos, and ‘K-Early’ citrus had color problems. The fruits were typically degreened and then treated with a dye, CR2, to adjust the color, which is the only dye allowed for this purpose (Hall, 2013). Previously, research in this laboratory searching for a CR2 alternative found that several oil-soluble natural colorants were comparable to CR2 as a color enhancer (Sun et al., 2015). However, postdye waxing was considered a necessity to reduce oxidation of carotenoid colorants (Sun et al., 2015).

Commercially, citrus fruits, regardless if degreening and dying were applied, are waxed to reduce water loss, lower the respiration rate, improve shine, and reduce spoilage (Lahlali et al., 2014; Mannheim and Soffer, 1996). Waxing also preserves higher levels of total phenolics during storage (Shen et al., 2013). Carnauba wax is a common coating used for such purposes in a variety of fruit, such as limes (Caron et al., 2015), apples (Jo et al., 2014), and orange (Sun et al., 2015).

The objective of the current research was to develop a natural colorant-containing carnauba wax so that the application of colorant and wax is a one-step process, instead of the current two-step (CR2 dye then waxing) process. The goal was to develop a coating that imparts improved peel color comparable to CR2, while imparting shine, retaining moisture, and modifying internal gases to extend storage life.

Materials and Methods

Colorant, solvent, and wax materials. Three food grade colorants were tested. Annatto suspension (8% bixin) was obtained from Food Ingredient Solutions, LLC (Teterboro, NJ); paprika O/S FANS445 from WILD Flavors Inc. (Erlanger, KY); and Durabrite® paprika oleoresin (PO) from Kalsec (Kalamazoo, MI). CR2 was obtained from JBT FoodTech (Lakeland, FL). The annatto, paprika, and PO dyes were all liquids for which the exact components are proprietary. Carnauba wax No. 4 was provided by Strahl & Pitsch, Inc. (West Babylon, NY). The oleic acid mixture (82% oleic, 12% linoleic, and 6% palmitic) was Palmac 760 from Condor Corp. (Englewood Cliffs, NJ), and the myristic acid mixture (90% myristic, 6% palmitic, and 4% lauric) was Hystrene 9014 from Humko Chemical Division, Witco Corp. (Memphis, TN).

Preparation of the colorant-containing wax. Natural colorant-containing wax coatings were prepared by incorporating the natural colorants during the initial processing of the wax mixture. Various carnauba wax + colorant formulations were tested in the preliminary experiments (Sun et al., 2015) and the following formula was selected for the research: 14.63% carnauba wax no. 4, 2.00% oleic acid, 0.73% myristic acid, 0.29% linoleic acid, 0.20% palmitic acid, 0.03% lauric acid, 2.44% morpholine, and 1.63% natural colorant solution or suspension (annatto, paprika, or PO), with the balance being water. The wax control without colorant was made with the same formulation except there was no colorant solution/suspension.

Absorption spectra test of the colorants. The colorants were dissolved in acetone and the absorbance was measured under spectrum mode using wavelength scanning from 190 to 800 nm (ultraviolet-2401 PC spectrophotometer; Shimadzu, Tokyo, Japan).

Evaluation of the color and gloss of the formulations by test paper. For color and gloss evaluations, 0.5 mL of each colorant-containing

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¹Corresponding author. E-mail: jinhe.bai@ars.usda.gov.

Fig. 1. Absorption spectra of CR2, annatto, and paprika/paprika oleoresin solutions in acetone (CR2 = Citrus Red No. 2).

Fig. 2. Changes of color (L^* , a^* , b^* , and $a^*:b^*$ ratio) and gloss values of five treatments on test papers over 7 d of exposure at 23 °C under 300 lux of standard fluorescent white light. Each data point represents the mean of 10 samples with the SD bar (CR2 = Citrus Red No. 2; A = annatto; P = paprika; PO = paprika oleoresin).

wax coating was spread on a 0.05-mm-thick and polished test sheet (Leneta Co., Mahwah, NJ) with a 4-mil castor (BYK-Gardner, Columbia, MD) with a speed of 1 $\text{cm}\cdot\text{s}^{-1}$. The CR2 control was applied to the test paper in two steps: first 0.5 mL of CR2 colorant (2 ppm in pine oil) was applied on the

paper and casted by using the 4-mil castor with a speed of 1 $\text{cm}\cdot\text{s}^{-1}$. After air drying at room temperature for 30 min, the wax without colorant was applied to cover the colorant in the same manner as described in the colorant-containing wax above. The papers were dried under hood at room temperature

for 3 h, and the color and gloss values were measured by a colorimeter and reflectometer as described below.

Fruit dying and waxing. Yellow (lacking orange color) 'Hamlin' oranges (*Citrus sinensis*) were harvested on 9 Mar. 2016 from the HLB-affected trees located in the USDA Picos Farm in Fort Pierce, FL. Fruits were directly used for dying and coating experiments without the typical degreening procedure used for traditional early fruit handling. Fruits (120) were selected for the experiment with 10 fruits (single fruit replicate) per treatment $\times$ 3 colorant treatments $\times$ 2 temperature/time combinations $\times$ 2 light exposures. A pilot-plant scale packingline located at the Indian River Research and Education Center was used for the washing, drying, and waxing processing. All fruits were washed for 1 min on the washer brush bed using detergent (Fruit Cleaner 395; JBT FoodTech), and dried using a forced air-drying tunnel at 50 °C for 2.5 min before applying the dye/wax treatments. PO-containing carnauba wax was selected based on the test sheet results which showed relatively stable red color and high shine. CR2 + wax (two-step applications) and non-colorant wax were used as positive and negative controls, respectively. For the CR2 positive control, fruits were dyed by following the simulated commercial procedure (Hall, 2013). Briefly, the fruits were dipped in 0.5% (v/v) of CR2 solution at 48.9 °C for 4 min. The CR2-dyed fruits were allowed to dry at 23 °C and 40% relative humidity (RH) for 3 h, and then carnauba wax was applied by the same procedure as other treatments described below. Colorant-containing or noncontaining waxes were applied by a 100-mL plastic sprayer (The Bottle Crew, Farmington Hills, MI) on the fruits laid on the rotating brush bed. The fruit were kept on the waxing bed for 2 min, and then were dried in the 50 °C heat tunnel for 2.5 min. A unique number was assigned to each fruit for tracking and initial color measurements were taken before fruits were stored. The following combinations of storage conditions were applied: storage at 20 °C for 7 d, or 5 °C for 14 d with or without light exposure at 300 lux using standard fluorescent white lights to simulate storage and marketing environments.

Color measurements. CIELAB was used for determining color using a colorimeter (model CR-300; Minolta, Tokyo, Japan). The instrument was calibrated daily using a white tile. Color was expressed as CIE L^* , a^* , b^* , and $a^*:b^*$ ratio, where L^* indicates lightness from 0 (black) to 100 (white). Positive a^* values indicates red color, whereas negative a^* values represents green color. Similarly, positive and negative b^* values indicate yellow and blue colors, respectively. A larger $a^*:b^*$ ratio indicates a darker orange color, and is generally recognized as the preferred color for oranges (Bai et al., 2009).

Gloss. A reflectometer (micro-TRI-gloss; BYK-Gardner, Silver Spring, MD) was used to evaluate shine of dye/wax treated sheets

and fruits. For the test sheets, the reflectance was measured at an angle of 20°, and expressed as gloss units (GU) (Bai et al., 2003a). For the citrus peel, a shield having a circular 19-mm-diameter aperture was attached to the reflectometer and GU was measured at 60° (Bai et al., 2003b). Ten measurements were made per test sheet/fruit.

Weight loss. The waxed fruit was weighed at the beginning and during storage at different temperatures and light conditions. Weight loss was expressed as the percentage loss of the initial total weight.

Respiration rate and ethylene production. Fruit was incubated in a 1-gallon glass jar for 30 min at 20 °C. Then headspace gas was taken from the jar by a 10-mL syringe. Ethylene concentration in the jar was analyzed using a gas chromatography (GC, Hewlett Packard HP 5890A; Hewlett Packard, Avondale, PA) equipped with a GSQ column and a flame ionization detector. The gas flow rate for He, H₂, and air were 10, 35, and 350 mL·min⁻¹, respectively. Temperatures of oven, injector, and detector were 90, 200, and 250 °C, respectively. The CO₂ concentration was determined by a same model GC fitted with a CTR column and a thermal conductivity detector. The gas flow rate for He and air were 80 and 350 mL/min, respectively. Temperatures of oven, injector, and detector were 70, 250, and 250 °C, respectively.

Internal gas combination. Samples for internal gas measurement were withdrawn by a 10-mL syringe from the central column/cavity of fruit submerged in water. The GC conditions for measurements of ethylene, CO₂, and O₂ were same as above.

Statistical analysis. JMP (version 11.2.0; SAS Institute, Cary, NC) was used for data analysis. Analysis of variance was applied based on a randomized complete block split-split plot model with the colorant as the main plot factor, storage temperature/time as the subplot factor, and light exposure as the sub-subplot factor. The multiple comparisons were applied with Tukey test when comparing selected treatment combinations (colorant × temperature/time × light).

Results and Discussion

Figure 1 shows the ultraviolet-visible absorption spectra of the colorant (CR2, annatto, paprika/PO) solutions in acetone. In comparison with CR2, which showed a single absorption peak at about 510 nm, annatto showed three peaks at about 440, 470, and 500 nm, and paprika and PO showed two peaks at about 440 and 470 nm, respectively (Fig. 1). The peak positions in the ultraviolet-visible spectrum of the carotenoids are characteristic and it provides information about the chromophore groups in the molecule (Scotter, 2009). The position of the peaks 276, 440, 468, and 503 nm are representative spectra for carotenoid bixin (Dias et al., 2011). The peaks at 501 and 470 nm are typical from cis-bixin conformation in chloroform; and those at 507 and

476 nm are for transbixin (Scotter et al., 1994). The wavelengths 472 and 508 nm (Fig. 1) were selected as the most appropriate for simultaneous quantification of carotenoid pigments fractions in the acetone extract of paprika/PO (Hornero-Mendez and Minguez-Mosquera, 2001). A quantitative determination of each extract to equivalent to CR2 in red color enhancement would provide useful information. However, we have very limited information on the extraction method, color ingredients, solvents, and concentrations, thus, there was no such quantitative information in the current research.

Figure 2 shows the CIELAB results and gloss property of CR2 + coating and the natural colorants (annatto, paprika, and PO)-containing coatings over time on test papers. Both annatto and PO-containing coatings exhibited relatively stable color profiles compared with CR2 with stable *L**, *a**, and *a*:b** ratio on the test paper. However, the annatto-containing coating exhibited substantially low gloss, while the gloss values in PO-containing coating were comparable to, or even better than the CR2 + wax, or wax alone treatments (Fig. 2). Test sheet coated with the paprika-containing coating initially exhibited high red color with high *a** value, however, it discolored consistently and the color effect was almost completely gone after 7 d (Fig. 2). This treatment also had poor gloss (Fig. 2). Overall, results from the sheet

test distinguished the PO wax mixture from the other formulations with high gloss and stable red/orange color (Fig. 2), thus, this color/wax combination was selected for the further fruit coating experiments.

The effect of PO-containing wax coating was confirmed by applying the treatment to orange fruit (Fig. 3): the color improvement (stable and high *a** and *a*:b** values) was comparable to the CR2 + wax treatment after 7 d storage at 20 °C and 14 d at 10 °C with or without light exposure (Table 1). Fruit treated with this PO wax mixture also showed similar respiration rate, ethylene production (all very low), and weight loss in comparison with the CR2 + wax and wax alone treatments (data not shown).

In comparison with wax alone treatment, all colorant-containing treatments significantly decreased the peel permeability, and caused lower internal O₂ (6.15% to 10.77%), and higher CO₂ concentrations (11.66% to 14.91%) (Table 2). However, there was no difference between different colorants (Table 2). Since both internal O₂ and CO₂ concentrations were in an acceptable range, physiologically, throughout the experiment, thus no adverse effects were expected (Bai and Plotto, 2011; Yan et al., 2016). Internal ethylene concentrations were unaffected by different colorant wax mixture applications, however, low temperature (10 °C) and dark combination storage conditions substantially

Fig. 3. Appearance of fruits (*Citrus sinensis* var. Hamlin) with different colorant treatments in the beginning and after storage at 20 °C for 7 d, or at 10 °C for 14 d under 300 lux of standard fluorescent white light exposure (CR2 = Citrus Red No. 2; PO = paprika oleoresin).

Table 1. Effect of colorant, storage temperature/time combination, and light exposure on orange fruit (*Citrus sinensis* var. Hamlin) color values.

Colorant	<i>a*</i>				<i>a*:b*</i>			
	20 °C for 7 d		10 °C for 14 d		20 °C for 7 d		10 °C for 14 d	
	Light	Dark	Light	Dark	Light	Dark	Light	Dark
Wax alone	11.71 b	10.41 b	11.91 b	9.02 b	0.19 b	0.18 b	0.16 b	0.13 b
CR2 then wax	15.81 ab	16.12 a	18.35 a	18.29 a	0.32 a	0.32 a	0.29 a	0.31 a
PO in wax	19.08 a	21.38 a	20.16 a	19.63 a	0.36 a	0.39 a	0.35 a	0.32 a
ANOVA (<i>F</i> value and significance)								
Colorant	40.88***				54.53***			
Temperature/time	0.30				3.94*			
Light or dark	0.18				0.09			

CR2 = Citrus Red No. 2; PO = paprika oleoresin; ANOVA = analysis of variance.

Mean values followed by different letters within a column indicate significant differences using Tukey test ($P < 0.05$). The asterisks on the top right of *F* value refer to statistical significance: *, **, ***significant at $P < 0.05$, 0.01, or 0.001, respectively.

Table 2. Effect of colorant, storage temperature/time combination, and light exposure on internal gas concentration of ethylene, CO₂, and O₂.

Colorant	C ₂ H ₄				CO ₂				O ₂			
	20 °C for 7 d		10 °C for 14 d		20 °C for 7 d		10 °C for 14 d		20 °C for 7 d		10 °C for 14 d	
	Light	Dark	Light	Dark	Light	Dark	Light	Dark	Light	Dark	Light	Dark
Wax alone	2.26 b	1.57 b	1.51 b	5.97 a	13.90	10.13	10.57	11.27	9.45	12.62	11.75	10.21
CR2 then wax	1.64 b	2.06 b	1.84 b	4.51 a	13.43	14.17	13.62	14.91	8.48	8.57	8.16	6.15
PO in wax	2.58 b	2.56 b	2.90 b	5.29 a	13.25	11.66	12.32	14.61	7.08	10.77	8.96	7.15
ANOVA (<i>F</i> value and significance)												
Colorant (C)		1.49				4.02*					4.19*	
Temperature/time (T)		15.75***				0.03					0.66	
Light or dark (L)		15.26***				0.01					0.08	
C × T		0.14				0.72					0.17	
C × L		0.27				1.05					0.43	
T × L		17.36***				3.99*					4.73*	

CR2 = Citrus Red No. 2; PO = paprika oleoresin; ANOVA = analysis of variance.

Mean values followed by different letters within a row indicate significant differences using Tukey test ($P < 0.05$). The asterisks on the top right of *F* value refer to statistical significance: *, **, ***significant at $P < 0.05$, 0.01, or 0.001, respectively.

induced the ethylene production in the fruit, and consequently led to high internal ethylene concentration in the fruit, regardless coating formulation (Table 2).

The interactions between temperature/time combinations and light were significant for ethylene, carbon dioxide, and oxygen (Table 2). An increase in temperature caused an accumulation of endogenous 1-aminocyclopropane-1-carboxylic acid (ACC), whereas ethylene production was greatly reduced (Yu et al., 1980). Elimination of high temperature stress caused more rapid recovery of ACC synthase activity compared with ethylene forming enzyme activity (Biggs et al., 1988). The mechanism of light-inhibited ethylene production in tobacco and rice leaves has been studied and the results indicated that light inhibited the conversion of ACC to ethylene (Kao and Yang, 1982). It has been suggested that the inhibitory effect of light is caused by oxidation of essential SH group(s) of the enzyme system converting ACC to ethylene (Gepstein and Thimann, 1980). This light-inhibiting process was combined with standard citrus degreening (Baldwin, 1993).

Paprika oleoresin (also known as paprika extract), an oil-soluble extract from certain varieties of red pepper (*Capsicum annuum* or *Capsicum frutescens*), has been primarily used as a color aid in food products such as cheese, sausage, snacks, salad dressing, and confectionary products (Nieto-Sandoval et al., 1999). It is composed of capsaicin and capsaanthin, the main coloring compounds, and capsaicin, the main flavoring compound giving pungency in high concentrations (Perez-Galvez et al., 2003). The overall quality of PO was revealed to be stable at 4 °C and 70% RH without adding antioxidants (Perez-Galvez et al., 2009).

In conclusion, the effects of natural colorant-containing wax coatings on citrus fruit peel color, shine, plant physiological, and food quality properties were investigated. Test paper and fruit surface evaluation showed that the PO-containing coating resulted in red/orange color when applied to both test papers and 'Hamlin' oranges, and this treatment was stable under both 10 °C and 20 °C with or without light exposure. It

significantly improved the 'Hamlin' red/orange peel color without causing any significant effect to weight loss, respiration, ethylene production, and internal gas (O₂ and CO₂) combination in comparison with fruit coated with wax alone, or processed with a "dye then wax" application. The experiments suggest that PO containing carnauba wax coating would be a successful application to enhance fruit appearance and storage life in citrus fruit. Future experiments could include a sensory evaluation component.

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