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Salicylic acid, cinnamaldehyde, and thymol incorporated into cassava starch coatings for mango preservation

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Ácido salicílico, cinamaldehído y timol incorporados a revestimientos de almidón de yuca para la conservación del mango

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ABSTRACT

Keywords:
Instrumental texture
Shelf life
Titratable acidity
Tommy atkins
Total soluble solids

Mango is a fruit consumed all over the world. There are some methods used during storage such as modified atmosphere, hot steam, irradiation, wax coating, and immersion in hot water to extend mango shelf life. However, heat treatment accelerates maturation and reduces organoleptic quality. Edible coatings are also used to extend the shelf life of food. Edible coatings are layers made of proteins, polysaccharides, lipids, antimicrobial components, antioxidants, or a mixture of them. Additives with antimicrobial, antioxidant, or other properties, are added to coatings to improve their functionality. Coatings improve food quality by slowing down unwanted changes and extending shelf life. Knowing that starch is not the best material for preparing edible coatings, in the present work, it was modified by adding salicylic acid or a cinnamaldehyde-thymol mixture to the cassava starch coating-forming solution. Cassava starch or chitosan coatings were applied to Tommy Atkins mangoes. Total soluble solids, titratable acidity, weight loss, and instrumental texture (firmness) were determined over four weeks of storage at 12 °C and 90% of relative humidity. Mangoes coated with cassava starch containing salicylic acid (SSA) had the highest weight loss, while fruits coated with starch-cinnamaldehyde-thymol (SCT) had the lowest weight loss during the whole storage time. The titratable acidity showed a decrease throughout the four weeks of storage. However, in the third week of storage, mangoes coated with chitosan, SSA, and SCT samples ripened more slowly, as indicated by higher acidity than uncoated samples. The SSA and chitosan-coated mangoes showed statistically similar penetration forces that were also higher than the SCT and uncoated samples. Cinnamaldehyde-thymol improved the hydrophobic characteristics of the starch coatings and therefore, it reduced the weight loss of mango during storage.

RESUMEN

Palabras clave:
Textura instrumental
Tiempo de vida
Acidez titulable
Tommy atkins
Sólidos solubles totales

El mango es una fruta que se consume en todo el mundo. Para prolongar su vida útil durante su almacenamiento, se usan atmósferas modificadas, vapor caliente, irradiación, recubrimientos con cera o inmersión del mango en agua caliente. Sin embargo, el tratamiento térmico acelera la maduración y reduce la calidad organoléptica. Los recubrimientos comestibles también se usan para prolongar la vida útil de los alimentos. Los recubrimientos son capas hechas de proteínas, polisacáridos, lípidos, componentes antimicrobianos, antioxidantes o una mezcla de ellos. Aditivos con propiedad antimicrobiana, antioxidante u otra propiedad, se añaden a los recubrimientos para mejorar su funcionalidad. Los recubrimientos mejoran la calidad de los alimentos al ralentizar los cambios no deseados y prolongar su vida útil. Conociendo que el almidón no es el mejor material para preparar recubrimientos comestibles, en el presente trabajo, se modificó agregando ácido salicílico o una mezcla de cinamaldehído-timol a las soluciones formadoras de recubrimientos de almidón de yuca. Se aplicaron recubrimientos de almidón de yuca o quitosano a mangos Tommy Atkins a los cuales se les determinó sólidos solubles totales, acidez titulable, pérdida de peso y textura instrumental (firmeza) a lo largo de cuatro semanas de almacenamiento a 12 °C y 90% de humedad relativa. Los mangos recubiertos con almidón de yuca que contenían ácido salicílico (SSA) presentaron la mayor pérdida de peso, mientras que los frutos recubiertos con almidón-cinamaldehído-timol (SCT) presentaron la menor pérdida de peso a lo largo de todo el tiempo de almacenamiento. La acidez titulable mostró una disminución a lo largo de las cuatro semanas de almacenamiento. Sin embargo, en la tercera semana de almacenamiento, los mangos recubiertos con muestras de quitosano, SSA y SCT maduraron más lentamente, como lo indica una mayor acidez que las muestras sin recubrir. Los mangos recubiertos con SSA y quitosano mostraron fuerzas de penetración estadísticamente similares que también fueron más altas que las muestras SCT y sin recubrimiento. El uso de revestimientos a base de quitosano o almidón de yuca, este último conteniendo ácido salicílico o cinamaldehído-timol retrasó la maduración del mango. El cinnamaldehído-timol mejoró las características hidrófobas del revestimiento de almidón y, por lo tanto, redujo la pérdida de peso del mango durante el almacenamiento.

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Mango is among the five most consumed tropical fruits worldwide (Caballero *et al.*, 2015). Tommy Atkins is the variety of mango most exported from Ecuador. To be exported, modified atmosphere, hot steam, irradiation, wax coating, and immersion in hot water are used. Mango is normally immersed in hot water to extend its shelf life (National Mango Board, 2020). However, heat treatment accelerates maturation and reduces organoleptic quality.

The short shelf life of mango fruits, susceptibility to chilling injury and postharvest diseases are common postharvest problems that need to be considered for expanding the international mango trade (Singh *et al.*, 2013). Edible coatings become a technological alternative that may reduce mango deterioration during storage. Edible coatings are made from proteins, polysaccharides and lipids. Starch is the most important material used in the formulation of biodegradable films and edible coatings (Chiumarelli *et al.*, 2010). Plasticizers and other additives are utilized to modify the physical and functional properties of edible coatings (Shah *et al.*, 2016).

The use of edible coatings along with essential oils (EOs) has importance because of EOs extend the shelf life of food (Sung *et al.*, 2013). EOs and their active components extracted from aromatic and medicinal plants have antibacterial and antifungal properties (Maurya *et al.*, 2021). Some examples of the EO constituents are carvacrol (found in thyme and oregano), carvone (dill seed), cinnamaldehyde (cinnamon), citral (lemongrass), p-cimene, eugenol (clove), limonene (citrus fruits), menthol (peppermint) and thymol (thyme and oregano) (Kawacha *et al.*, 2021). EOs have been used together with starch coatings to preserve fruits: cassava starch and cinnamaldehyde-thymol in fresh-cut mango (Santacruz, 2021), cassava starch and thyme in apples and persimmons (Sapper *et al.*, 2019), and sweet potato starch and cumin essential oil in pears (Oyom *et al.*, 2022). In addition to EOs, salicylic acid (SA) can be used in the formulation of edible coatings to delay fruit ripening. SA has been used to control fruit decay of guava (Lo'ay and El Khateeb, 2011), apricot (Ezzat *et al.*, 2017) and papaya (Castro *et al.*, 2017). Edible coatings based on chitosan (Chien *et al.*, 2007), arabic gum (Khaliq *et al.*, 2015) and alginate (Robles-Sánchez *et al.*, 2013) have been used in mango, however, no studies on the use of edible coatings based on cassava starch containing SA, cinnamaldehyde or thymol on Tommy Atkins mango have been performed.

In recent years, chitosan has had considerable interest in the industry due to its biodegradability, non-toxicity (Wang *et al.*, 2020a), and antimicrobial properties (Wang *et al.*, 2020b). Studies of chitosan coatings to preserve mango have been performed (Yin *et al.*, 2019; Tavassoli-Kafrani *et al.*, 2020). However, the low availability and high cost of chitosan in the Ecuadorian market (Salas, 2011) compared to other materials may reduce its application. Therefore, essential oils and salicylic acid together with cassava starch could be a good choice for the formulation of edible coatings thanks to its availability and relatively low price (Souza *et al.*, 2012).

In the present work, Tommy Atkins mangoes were coated with either chitosan or cassava starch. Starch coatings together with SA or a mixture of cinnamaldehyde-thymol were utilized. A comparison of total soluble solids, titratable acidity, weight loss, and instrumental texture (firmness) of coated fruits with the two coating materials along four weeks of storage at 12 °C was done.

MATERIALS AND METHODS

Chitosan, degree of deacetylation 95% and Mw of 149 kDa, was donated by Universidad Pública de Navarra, Spain. Cinnamaldehyde, thymol, tween®-20, glycerol, and glucose were obtained from Merck (Germany). Cassava starch (La Pradera, Ecuador) and Tommy Atkins mangoes were obtained from a local market in Manta, Ecuador. Mangoes with a degree of ripening of two (Báez, 1998) were selected according to the size and without any injuries. Afterward, 48 mangoes were washed with water (Santacruz, 2021) and 12 fruits were used for each treatment (Table 1). The coated mangoes were obtained by immersing the fruits in three different solutions (Table 1), followed by drying at room temperature (approx. 25 °C) and storage for four weeks at 12 °C and an RH of 90%. Analyses of weight loss, total soluble solids, titratable acidity, and instrumental texture were performed in triplicate every week.

Table 1. Composition of coating forming solution

Treatment	Chitosan (%, w/v)	Cassava starch (%, w/v)	Salycilic acid (mmol L^{-1})	Cinnamaldehyde (%, w/v)	Thymol (%, w/v)
Uncoated	-	-	-	-	-
Chitosan	1	-	-	-	-
SSA[1]	-	0.5	2	-	-
SCT[2]	-	0.5	-	0.15	0.15

[1]Starch + salicylic acid; [2]Starch + cinnamaldehyde + thymol

Coating forming solution preparation

Chitosan coating forming solution was prepared by dissolving 1% (w/v) of chitosan in 1% (v/v) acetic acid solution. 1% (w/v) of Tween 20, 0.5% (w/v) glycerol and 0.5% (w/v) of glucose were added before the solution was homogenized by using an ultraturrax (Polytron, Switzerland) at 11000 rpm for 4 min.

Starch coating forming solution was prepared according to Santacruz *et al.* (2015). A 0.5% (w/v) cassava starch suspension in water was heated from room temperature up to 90 °C, under stirring, where it was kept for 5 min. 1% of Tween®-20, 0.5% (w/v) glycerol, and 2 mmol L^{-1} of SA were added before cooling. Once the solution was cooled to room temperature, glucose (0.5%, w/v), 0.15% (w/v) cinnamaldehyde (>95%) and 0.15% (w/v) thymol (98.5%) were added. Finally, the coating forming solution was homogenized by using an ultraturrax (Polytron, Switzerland) at 11000 rpm for 4 min.

Weight loss, titratable acidity, and total soluble solids of coated mangoes along storage time

Weight loss was calculated by the following equation.

$$\%WL = \frac{(W0 - Wt)100}{W0}$$

Where: WL: Percentage of weight loss, W0: Fruit weight at time zero, Wt: Fruit weight at any storage time.

Titratable acidity was determined by titration with 0.01 M NaOH solution according to the AOAC method (1990), the results of three measurements were reported as a percentage of citric acid. Total soluble solids were determined according to the AOAC method (1990). Three fruits were disintegrated using a household blender and the obtained juice was filtered with textile and analyzed with a refractometer (Atago, Japan). The results of three measurements were reported as °Brix.

Instrumental texture analysis

Puncture tests were performed in triplicate at the central part of three fruits according to Castro *et al.* (2014). A Shimadzu texturometer (Model EZ-LX, Japan) together with a stainless-steel probe of 3 mm diameter and 8 cm length were utilized. The probe was inserted 15 mm into the fruit at a speed of 10 mm s^{-1} and the maximum penetration force was recorded.

Statistical analyses

All measurements were performed in triplicate. ANOVA and Tukey test with a significance level of 5% were run using InfoStat statistics software (Infostat version 2014, Argentina).

RESULTS AND DISCUSSION

Weight loss, titratable acidity, and total soluble solids of coated mangoes along storage time. Mangoes coated with cassava starch containing salicylic acid (SSA) showed the highest weight loss, whereas fruits coated with starch-cinnamaldehyde-thymol (SCT) showed the lowest weight loss throughout the whole storage time (Table 2). SCT mangoes showed a weight loss that varied between 4.4 and 15.7%, which was statistically different than SSA samples. After the first week of storage, no difference in weight loss was found among uncoated mangoes and mangoes coated with either chitosan or SCT. The hydrophilic nature of starch may contribute to losing water in a higher amount in mangoes coated with SSA compared to uncoated samples. The use of cinnamaldehyde-thymol together with starch (SCT) led to lower weight loss compared to

SSA. The hydrophobic properties of cinnamaldehyde-thymol (Man *et al.*, 2019) may be the reason for such behavior. The incorporation of EOs into the coating-forming solution can confer water-resistance properties to coatings because this oil has a hydrophobic nature (Sánchez-González *et al.*, 2011).

Table 2. Weight loss and total soluble solids of coated mangoes. Coatings based on either chitosan or cassava starch and stored for 28 days at 12 °C and 90% relative humidity.

Treatment	Days of storage (week)									
	0		7 (1)		14 (2)		21 (3)		28 (4)	
	WL (%)	SS (°Brix)	WL (%)	SS (°Brix)	WL (%)	SS (°Brix)	WL (%)	SS (°Brix)	WL (%)	SS (°Brix)
U	0	6.4±0.3 a	5.0±0.2 ab	6.0±0 a	9.6±0.1 a	10.8±0 b	13.8±0.1 a	14.5±1.8 a	17.8±0 a	14.3±0.1 b
C	0	6.4±0.4 a	4.9±0.6 ab	7.1±0.3 a	9.3±1.1 a	12.9±0.4 c	13.3±1.5 a	11.2±2.3 a	17.2±1.9 a	10.2±0.3 a
SCT	0	6.4±0.3 a	4.4±0.2 a	5.8±2.3 a	8.4±0.3 a	13.8±0.3 c	12.0±0.5 a	13.5±0.9 a	15.7±0.7 a	11.2±1.9 a
SSA	0	6.4±0.3 a	6.3±0.6 b	5.4±0.3 a	12.3±0.8 b	9.6±0.8 a	17.5±1.1 b	10.4±0.8 a	22.4±1.4 b	9.5±0.1 a

The same capital letters in a column indicate no statistically significant difference (P<0.05). U=Uncoated, C=Chitosan, SCT=Starch+cinnamaldehyde+thymol, SSA=Starch+salicylic acid, WL=Weight loss, SS=Soluble solids

Titratable acidity showed a decrease along the four weeks of storage. Biochemical changes, e.g., ascorbic acid content on mangoes during ripening may lead to a reduction of titratable acidity (Pandarinathan and Sivakumar, 2010). There was no difference in titratable acidity among samples up to the second week of storage (Table 3). However, on the third week of storage, mangoes coated with chitosan, SSA, and SCT samples, ripened more slowly as indicated by higher acidity than uncoated samples. On the fourth week of storage, only mangoes coated with SSA showed lower acidity than the uncoated sample. A decline in acidity demonstrates advancement of maturation (Maftoonazad *et al.*, 2008), thus the coated fruits contributed to delaying the fruit maturation/ripening. Higher acidity in coated fruits may be the result of the formation of carboxylic acid by dark fixation of CO_2 (Maftoonazad *et al.*, 2008).

Table 3. Titratable acidity and maximum force of coated mangoes. Coatings based on either chitosan or cassava starch and stored for 28 days at 12 °C and 90% relative humidity.

Treatment	Days of storage (week)									
	0		7 (1)		14 (2)		21 (3)		28 (4)	
	TA (%)	MF (N)	TA (%)	MF (N)	TA (%)	MF (N)	TA (%)	MF (N)	TA (%)	MF (N)
U	3.5±0.2 a	27.8±1.8 a	2.2±0 a	26.9±0.9 a	2.2±0 a	24.7±3.6 ab	0.8±0.2 b	9.4±0.4 c	1.7±0.4 a	7.1±0.9 b
C	3.5±0.2 a	27.9±1.4 a	2.5±0.3 a	27.6±0.2 a	2.4±0.3 a	24.7±0.1 ab	1.4±0 a	15.5±3.3 ab	1.1±0.1 ab	11.6±0.9 a
SCT	3.5±0.2 a	27.9±1.2 a	2.3±0.4 a	27.3±0.2 a	2.9±0.2 a	21.0±1.3 b	1.4±0 a	13.5±0.7 bc	1.2±0 ab	7.1±0.9 b
SSA	3.5±0.2 a	27.7±1.2 a	2.4±0.4 a	27.5±0.2 a	2.7±0.9 a	27.3±0.6 a	1.4±0.1 a	19.1±1.2 a	1.0±0 b	10.0±0.7 a

The same letters a column indicate no statistically significant difference (P<0.05). U=Uncoated, C=Chitosan, SCT=Starch+cinnamaldehyde+thymol, SSA=Starch+salicylic acid, TA=Titratable acidity (percentage of citric acid), MF=Maximum force (Newtons)

The four treatments showed an increase in total soluble solids along the storage time. Total soluble solids had no differences among samples up to the first week (Table 2), however, an increase for the four samples during the second week of storage was noticed. At the end of storage, the uncoated sample reached the highest value of total soluble solids (approximately 14 °Brix), which was statistically different than the three coated mangoes (total soluble solids between 9 and 11 °Brix). No statistical difference was found among total soluble solids for coated mangoes. The low value of mangoes coated with SSA suggests that SA may reduce the rate of ripening. This ripening reduction may be probably through inhibition of ethylene biosynthesis (Yin *et al.*, 2013). In fact, Lo'ay (2017) found that an exogenous supply of SA delays the ripening of grapes.

Texture analysis

Results of puncture tests showed that the maximum force of penetration decreased during the storage time for all samples. Mangoes coated with SSA and chitosan showed statistically similar forces of penetration which were also higher than SCT and uncoated samples (Table 3). SSA and chitosan coatings led to mangoes with low total soluble solids and low penetration forces which may be the result of a low respiration rate (Cissé *et al.*, 2015) and an inhibition of ethylene production by the presence of SA (Hayat *et al.*, 2007). Chitosan coating caused substantial delays to some processes involved in ripening most notably weight loss, total soluble solids, and texture of the fruit. These effects may be linked to the reduced rates of ethylene production and respiration that might be the result of lower internal oxygen levels (Jitareerat *et al.*, 2007).

CONCLUSIONS

The use of coatings based on either chitosan or cassava starch containing either salicylic acid or cinnamaldehyde-thymol delays changes in weight loss, soluble solids, titratable acidity, and firmness of mango along four weeks of storage at 12 °C. Cinnamaldehyde-thymol improves the hydrophobic characteristics of the cassava starch coating compared to salicylic acid and therefore reduces the weight loss of stored mango. For practical applications, the use of starch coatings with salicylic acid together with cinnamaldehyde-thymol to control the ripening of mango could be analyzed in future research.

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