PAPER

MANGO-SEED EXTRACT AND SULPHITES AS PROMOTERS OF COLOR AND BIOACTIVE COMPOUNDS RETENTION DURING TRAY DRYING OF MANGO SLICES

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ABSTRACT

The study objective was to evaluate effect of mango-seed extract alone or in combination with sodium metabisulphite on the content of vitamin C, free phenols, six phenols compounds, and total carotenes, and color in mango slices dried at 60ºC until 15% moisture. From drying curves were calculated effective diffusivities [1.17-1.35x10^{-9} $m^2 \bullet s^{-1}$] and drying rate constants [1.53±0.90x10^{-3}-2.27±0.80x10^{-3}] using Midilli's model. Results showed that combination of mango-seed extract with sodium metabisulphite has an important role in retention of vitamin C and carotenes, and an enrichment of phenolic compounds was found in dried mango slices.

Keywords: dried mango; mango-seed extract, sodium metabisulphite, free phenols; total carotenes; vitamin C

1. INTRODUCTION

Mango (*Mangifera indica* L.) is one of the most important fruits grown commercially in tropical and subtropical regions in the world. Since mango fruit is highly perishable, it is generally transformed to a dried product (LIN *et al*., 2016) for prolonged shelf life. Fresh mango is characterized by its high content of phenolic compounds, vitamin C and carotenes. Since these compounds are antioxidants, they are able to impart beneficial properties for the consumer health (SIDDIQ *et al*. 2013).

Mangoes have phenolic compounds derived from phenolic acids (SCHIEBER *et al*., 2000) such as gallic acid, caffeic acid, *p*-coumaric acid, etc. Gallic acid has antioxidant, anti-inflammatory, and anticarcinogenic activity (VELDERRAIN-RODRÍGUEZ *et a*l., 2018). SCHIEBER *et al*. (2000) reported that mango pulp has 5.9 mg gallic acid•(100 g dry mass)$^{-1}$. Other phenolic acids found in mangoes are caffeic acid [6.6 mg•(100 g dry mass)$^{-1}$] and ferulic acid [8.9 mg•(100 g dry mass)$^{-1}$] (ABDALLA *et al*., 2007). In addition, methyl gallate is a potent cell protector against oxidative stress, reduces lipid peroxidation (LPO) and reactive oxygen species (ROS) (WHANG *et al*., 2005). Lee *et al*. (2010) reported that methyl gallate suppresses T regulatory cells (Treg) in mice's malignant tumors. Mangiferin is a C-glucosyl-xanthone found in some mango varieties, such as Tommy Atkins, Haden and Ubi. This compound has a wide range of biological properties, because it is gastroprotective, analgesic, antibacterial and cytoprotective (MASIBO and HE, 2008). SCHIEBER *et al*. (2000) reported that mango pulp has 3.8 mg mangiferin•(100 g dry mass)$^{-1}$. Vitamin C is one of the most abundant compounds in mango fruit and its concentration varies with the fruit maturity, as well as with the post-harvest handling and processing methods. ROCHA-RIBEIRO *et al*. (2007) reported a concentration of 94.0 mg AAE•(100 g dry mass)$^{-1}$ for vitamin C in fresh mango var Tommy Atkins grown in Brazil. Vitamin C decreases from thermal effects and exposure to air and light (LIU *et al*., 2014).

Mango is also a good source of carotenes. β-carotene is the most abundant in mango fruits (VARAKUMAR *et al*., 2011). β-carotene content is often used as an indicator of damage extent to mangoes during processing and storage (THARANATHAN *et al*., 2006). MANTHEY and PERKINS-VEAZIE (2009) reported a concentration of 32.9 to 59.1 mg of carotenes•(100 g dry mass)$^{-1}$ in mango Tommy Atkins Mexican mangoes.

During the mango convective drying process, considerable degradation of these bioactive compounds occurs (MÉNDEZ-CALDERÓN *et al*., 2018). Hence, to minimize bioactive compound degradation it may be beneficial to carry out mango pretreatments prior to the drying process (YAO *et al*., 2020). The pretreatment choice depends on the drying method and characteristics of desired product. Additionally, pretreatments may improve product quality by retaining color of fresh mango and reducing product darkening.

GUIAMBA *et al*. (2016) evaluated the retention of β-carotene and vitamin C (dehydroascorbic and ascorbic acids) in osmotically dried mango pretreated with calcium chloride or ascorbic acid. This study consisted in an initial osmotic dehydration using 45º Brix sucrose solutions added with 1% calcium chloride or 1% ascorbic acid. Then samples were dried in an air convection oven at 50ºC or 70ºC. The authors reported that both pretreatments significantly improved retention of vitamin C and *all-trans*-β-carotene in dried products. In other study, CHEN *et al*. (2007) performed mango drying experiments using hot drying air and freeze-drying in presence of 1% sodium hydrogen sulphite or 1% ascorbic acid. The authors reported that use of pretreatments during drying mangoes reduced carotenes degradation. This behavior also was observed by JIMÉNEZ-HERNÁNDEZ *et al*. (2017) when studied effect osmotic dehydration of mango slices in an emulsion based on inulin and piquin-pepper oleoresin. The results showed a retention of

68.8% of ascorbic acid and 95.5% of β-carotene at 30ºC. Also, this study found a strong decrease in retention of ascorbic acid (43.6%) when the process was carried out at 50ºC. While retention of β-carotene was 83.0%. Recently, DEREJE *et al*. (2020) dried mango slices using four pretreatments (lemon juice, salt solution dips, hot water blanching and control) and four drying methods (solar, tray, freeze and fluidized bed drying) to assess effect of pretreatments and drying methods on qualities of dried mango slices. The results showed that pretreatments and drying methods had significant effects on color antioxidants of the dried mango slices. The ascorbic acid and total phenol contents were affected by drying methods and had respective values of 33.18-41.24 mg AAE•(100 g dry mass)$^{-1}$ and 131.13-251.12 mg of gallic acid equivalents (GAE)•(100 g dry mass)$^{-1}$.
On the other hand, it has been reported that mango-seed extracts contain a significant amount of free phenols, 27.7±0.1 g GAE•(100 g dry mass)$^{-1}$ (Bernal-Mercado *et al*., 2018). For this reason, this same study used mango-seed extract as antioxidant agent of fresh-cut mango. The results showed that a solution mango-seed extract at 0.63% (*m*/*v*) contributed to preservation of fresh mangoes cubes due to increasing free phenols from 306.0 to 364.9 mg GAE•(100 g dry mass)$^{-1}$. Additionally, in this study found that mango-seed extract contains 60 mg of gallic acid•(100 g dry mass)$^{-1}$, 42.0 mg of mangiferin•(100 g dry mass)$^{-1}$, 77 mg of caffeic acid•(100 g dry mass)$^{-1}$ and 12.6 mg of *p*-coumaric acid•(100 g dry mass)$^{-1}$. Also, mango-seed extracts have been used to develop antioxidants films (ADILAH *et al*., 2018). To the best of our knowledge, there are no reports about use of mango-seed extracts as a fruit drying pretreatment.
Considering the above, aim of study was to evaluate effect of different pretreatments on retention of color and antioxidant compounds (free phenols, vitamin C and total carotenes) during tray drying of Tommy Atkins mango slices. Three pretreatments were used for tray drying of Tommy Atkins mango slices: 0.5% sodium metabisulphite (PT1), 1.44% mango-seed extract (PT2) and 0.5%/1.44% (*w*/*v*) sodium metabisulphite/mango-seed extract (PT3). HPLC was used to quantify the main phenolic compounds present in the dried products.

2. MATERIALS AND METHODS

2.1. Sampling procedure and sample preparation

Tommy Atkins mangoes (*Mangifera indica* L.) were obtained from Porfirio Díaz Market located in Huajuapan de León city, Oaxaca, México. Mango fruits were selected based on fruit size and pulp color as measured by CIELAB b* parameters, which defines yellow color. It was also verified that the fruits had no physical damage. Mangoes were peeled with a home peeler and slices of 6.0 ± 0.1 cm x 4.0 ± 0.1 cm (length x width) and 3.8 ± 0.4 mm thickness were obtained with a cutter.

2.2. Determination of mango physicochemical parameters

The physicochemical characteristics of fresh mangoes were evaluated from soluble solids content, moisture percentage, titratable acidity and pH, which are briefly described below.
Soluble solids content (AOAC 932.12). A drop of fresh mango juice was placed in Abbe refractometer and °Brix of sample was measured.
Moisture percentage (WROLSTAD *et al*., 2005). A known amount of mango pulp (3 g) was weighed into pre-weighed and dried crucibles. The samples were then placed in an oven

at 105°C for 24 h. After that, dried samples were placed in a desiccator for 30 min at room temperature and anhydrous conditions and were finally weighed.
Titratable acidity and pH (DEA *et al*., 2013). Mango pulp sample was blended for 2 min to homogenize the sample and the blend was filtered through a cotton plug. A potentiometer previously calibrated with standard solutions (pH 4 and 7) was used to measure sample´s titratable acidity. For titration 10 g of sample was used. A 0.1 N sodium hydroxide solution was continuously added, while the pH was measured until sample reached a pH of 8.3.

2.3. Preparation of the mango-seed extract

Tommy Atkins mango seeds were cut into small pieces, ground in a blender for 2 min and sieved through a #40 mesh to obtain a fine powder. Subsequently, 25 g of powder was weighed and mixed with 500 mL of 99.5% methanol and sonicated for 30 min at room temperature. Mixture was allowed to stand until a phase separation was observed. Supernatant was decanted and filtered through a Whatman filter paper #1. Pellet was treated with same extraction procedure three times. Following this, filtrates were collected, combined and evaporated on a rotary vacuum evaporator at 40°C until 11 g of solvent-free extract was obtained as a viscous reddish-brown liquid. Extract was dissolved in water to make a final volume of 100 mL in a volumetric flask. Finally, extract solution was stored at -20°C for preservation until further use.

2.4. Pretreatment of mango slices

700 g of mango slices were immersed for 3 min in 1.4 L of pretreatment solutions at 25ºC. Solutions were used only once, after they were discarded. Solutions used as pretreatment were 0.5% sodium metabisulphite solution (PT1), 1.44% mango-seed extract solution (PT2) and a combination of 0.5% sodium metabisulphite and 1.44% mango-seed extract solutions (PT3). Following this, slices were drained for 1 min. Mango slices without pretreatment was used as control.

2.5. Dryer and drying conditions

A tray dryer (SANTOS-SÁNCHEZ *et al*., 2012,) equipped with a tray rotating mechanism and a heating-air flow control, was used in this work to perform drying of 700 ± 8 g of mango slices. Mango slices were dried at 60°C, air velocity of 1.2 $m \bullet s^{-1}$ and 20 rpm tray rotation velocity. Moisture loss was quantified by weighing slices every 15 min until moisture content was about 15%. Drying time was around 110 min. For each determination, mass of three samples was measured.

2.6. Determination of effective diffusivity (D_{eff}) and drying rate constant (k)

Drying curves (moisture ratio *versus* drying time) were used to calculate effective diffusivity (D_{eff}) and drying rate constant (k). Drying curves were performed in triplicate. Moisture ratio (MR) of food slices can be predicted from Sherwood and Newman model, Equation 1. This equation relates MR with drying time (t), thickness of food slice (L) and effective diffusivity (D_{eff}). D_{eff} is related with Fourier number (F) through Equation 2. In this study, D_{eff} was calculated from curve slope expressed by simplified Sherwood and

Newman model, Equation 3 (ASHRAF *et al*., 2012). The k values were calculated from nonlinear regressions of Midilli equation (MIDILLI *et al*. 2002).

$$MR = \frac{MR_t - MR^*}{MR_o - MR^*} = \sum_{n=0}^{a} \frac{8}{\pi^2(2n+1)} \exp\left[-(2n+1)^2 \frac{\pi^2}{4} F\right] \quad (1)$$

$$F = \frac{D_{eff} * t}{L^2} \quad (2)$$

Where MR_t = moisture ratio at time t, MR_o = initial moisture ratio, MR* = equilibrium moisture ratio and F = Fourier number. Compared to values of MR_t and MR_o, the value of MR_e is relatively smaller, so the MR can be reduced to $MR = MR_t/MR_o$ (MEWA *et al*., 2018). Considering that for this study, n = 0 and diffusion occurs through two faces of mango slice (trays used for drying are perforated), MR can be expressed from Equation 3. D_{eff} values were obtained from slope of time *versus* ln *MR* curves.

$$\ln MR = \ln \frac{8}{\pi^2} - \left(\frac{\pi^2 D_{eff}}{4L^2}\right) t \quad (3)$$

Equation 4 was used to estimate k as well as n, a and b values, nonlinear regressions were applied to equation of MIDILLI *et al*. (2002), where MR is moisture ratio, t is the drying time, k is drying rate constant and a, b and n are the model constants. The regression was performed with InterReg 2014 (Kroll-Software).

$$MR = a \exp\left(-kt^n\right) + bt \quad (4)$$

2.7. Free phenols quantification of mango-seed extract and the mango slices

For free phenols determination, 0.2 g of mango seed was mixer with 3.1 mL of 60:40 % (*v*/*v*) ethanol:water or 2 g of mango pulp was mixed with 25 mL of 60:40 % (*v*/*v*) ethanol:water. The mixture was milled in a home blender for 1 min and subsequently filtered through cotton wool. Extracts were obtained in triplicate. Free phenols quantification was performed in a Biotek ELX-808 microplate reader using modified Folin-Ciocalteu method described by OCHOA-VELASCO *et al*. (2016). Extract or standard (40 μL) was pour in a microplate well with 40 μL of 0.1 M Folin-Ciocalteu reagent. The reaction mixture was allowed to stand for 3 min in microplate reader, and stirred for 15 s at low velocity. Subsequently, 40 μL of 0.5% sodium carbonate (*w*/*v*) was added and mixed by suction with a multichannel pipette. Mixture was allowed to stand for 30 min at 40°C, after which it was stirred at medium speed in microplate reader. Finally, sample absorbance was read at 765 nm. A calibration curve was prepared using gallic acid solutions with known concentrations. Free phenols content in mango-seed extract and mango samples was determined using calibration curve and was expressed in mg of GAE•(100 g dry mass)$^{-1}$. All measurements were made in triplicate.

2.8. Vitamin C quantification

The quantification of vitamin C was performed following procedure described by OCHOA-VELASCO *et al*. (2016). Briefly, 0.05 g of dried mango (0.3 g of fresh mango) was mixed with 1.00 mL of 1% metaphosphoric acid solution. Subsequently, mixture was sonicated in an 8510 sonicator (Branson Ultrasonics Co., USA) for 15 min at room temperature. Then mixture was centrifuged for 15 min at 900 *g*. Supernatant containing vitamin C was used for discoloration reaction of 2,6-dichloroindophenol sodium salt (DCIP) in a 96 well microplate plate. To carry out this reaction, 70 μL of extract was mixed with 70 μL of a solution of 30 ppm DCIP and allowed to stand for 1 min at room temperature in absence of light. Finally, mixture was stirred for 15 s and then absorbance at 515 nm was measured in a Biotek ELX-808 microplate reader. Vitamin C content in samples was determined using calibration curve and expressed in mg of ascorbic acid equivalents (AAE)•(100 g dry mass)$^{-1}$. All measurements were made in triplicate.

2.9. Phenolic compounds quantification by HPLC

A mixture of 0.2 g of dried mango and 2.5 mL of a 70:30% (*v*/*v*) methanol:water solution with 0.1% CH_3COOH was vortexed for 5 min. Subsequently, mixture was sonicated for 10 min at room temperature, followed by centrifugation for 10 min at 900 *g*. Supernatant was then removed and the pellet was re-extracted for a second time. Supernatants were pooled and dried on a rotary vacuum evaporator at 40°C. Extracts were obtained in triplicate. Dried extracts were redissolved in 500 mL of water and solution was cleaned up by eluting it through C18 SPE cartridges of 2.8 mL (Alltech, USA). The clean-up procedure consisted first in elution of 1 mL of deionized water through C18 SPE cartridge. Subsequently, extract solution was loaded into cartridge and eluted with 5 mL of water, followed by 1 mL of a 1:1 (v/v) methanol:water solution, and finally with 1 mL of methanol. Fractions were collected in HPLC vials. Phenolic compounds quantification was performed in an HPLC instrument equipped with a photodiode array detector (Water, alliance 2695, USA) and a C18 column of 4.6 mm x 250 and 5 μm inner diameter (Phenomex, USA). The following phenolic measuring standards were employed: mangiferin, methyl gallate, ferulic acid, gallic acid, caffeic acid and *p*-coumaric acid. A calibration curve was performed for each standard. A 10 μL injection volume and a 1 mL•min^{-1} flow was used. A binary mobile phase was used (A = 0.1% formic acid in water, B = 0.1% formic acid in acetonitrile) with the following gradient program: 0 min 10% B, 5 min 10% B, 15 min 80% B and 30 min 100% B, 35 min 10% B. The mangiferin was monitored at 365 nm and phenolic acids and methyl gallate were monitored at 280 nm for (Rocha-Ribeiro *et al*., 2008). All measurements were made in triplicate.

2.10. Total carotenes quantification

A modification of method described by WROLSTAD *et al*. (2005) was used for total carotene quantification. Briefly, 0.3 g of dried mango was weighed and ground in a mortar with 3 mL of water. For fresh mango samples, 1.0 g pulp sample was homogenized and mixed with 2 mL of water. Mixture was placed in a 10 mL amber vial with subsequent addition of 4 mL of 95% ethanol was added, followed by vortexing for 4 min. This procedure was performed in triplicate. Then the mixture was vacuum filtered through filter paper. Supernatant was then poured through a 25 mL filtration funnel with 10 mL of hexane, and then manually stirred. Filtration funnel was allowed to stand for 2 min.

Subsequently, ethanolic phase was removed. Absorbance of hexane phase was measured at 450 nm in a Lambda 32 UV-vis spectrophotometer (Perkin Elmer, USA). Sample measurements were performed in triplicate. Equation 5 was used for the calculation of total carotenes of sample expressed as mg of β-carotene•(100 g dry mass)$^{-1}$. All measurements were made in triplicate.

$$[\text{Total carotenes}] = \left(\frac{A_{450} \bullet \text{Vol}_{\text{solution}}(\text{mL})}{258.84\ \text{mL} \bullet \text{mg}^{-1} \bullet \text{sample mass (g)}}\right) \bullet 100 \qquad (5)$$

2.11. Sulphites quantification

Sulphites quantification in dried mango slices obtained from PT1 and PT3 pretreatments was carried out analogously to reported by LI and ZHAO (2006). Briefly, solutions used to prepare samples and standards were 0.1 mM ethylenediaminetetraacetic disodium salt (EDTA) solution, 1000 ppm tris(hydroxymethyl)aminomethane (Tris) buffer solution at pH 8 and 0.3 mM 5,5-dithio-bis-(2-nitrobenzoic acid) [DTNB, also called Ellman's Reagent]. 0.1 mM EDTA and Tris buffer solutions were prepared with deionized and degassed water for 25 min at 25ºC. Subsequently, EDTA 0.1 mM solution was used to prepare 1000 ppm sodium metabisulphite solution. Tris buffer solution was used to prepare 0.3 mM DTNB solution. Before being employed, all solutions were degassed for 15 min at 25ºC. On the other hand, around 1 g of dried mango slices (PT2 and PT3), were dry-milled in a mortar to form a homogeneous paste. 20 mg of this paste was mixed with 1 mL of 0.1 mM EDTA. Later, samples were shaken in a vortex for 2 min and degassed for 15 min at 25ºC on ultrasound, followed by a centrifugation for 5 min at 900 *g*. Supernatants were separated and then used to carry out colorimetric reaction with 0.3 mM DTNB. This consisted of a mix 60 μL of sample or standard with 60 μL of 0.3 mM DTNB in a 96-well plate. Also, two reaction blanks were prepared, the first was prepared by mixing 60 μL of sample with 60 μL of 0.1 mM EDTA, while the second blank was prepared by mixing 60 μL of 0.3 mM DTNB with 60 μL of 0.1 mM EDTA. Reaction mixtures were allowed to stand for 5 min at 25ºC, then stirred for 30 s in a BioTek® model ELX808 microplate reader and absorbance at 405 nm was measured. Calibration curve was built with five sulphites standards, which were prepared at concentrations in interval of 6 to 20 ppm from 1000 ppm sodium metabisulphite solution. All measurements were made in triplicate.

2.12. Color determination

A HunterLab Ultra ScanVis (USA) spectrophotometric colorimeter was used to determine CIELAB color parameters of mango samples. D65 illuminant was used with an observation angle of 10° and a 0.9525 cm slit. For each drying data point, the CIELAB color parameters (L*, a* and b*) of mango slices were measured at ten different points to obtain an average. Also, angle Hue* was calculated with Equation 6. Three mango slices were measured for every drying data point.

$$Hue^* = arc\ tan\left(\frac{b^*}{a^*}\right) \qquad (6)$$

2.13 Statistical analysis

A randomized experimental design was applied in this study, one-factor ANOVA tests and Duncan´s means comparison method were used with a significance level $\alpha = 0.05$ between treatments and variables. Design-Expert® v.6.0 software was employed for these analyses.

3. RESULTS AND DISCUSSION

3.1. Physicochemical parameters of the mango pulp

Soluble sugars content and titratable acidity are related to fruit ripeness stage. The determinations of these parameters for fresh mango pulp are listed in Table 1 and are similar to values reported by SIDDIQ *et al*. (2013) for ripe mango samples. Visual color scale reported by BRECHT (2010) for Tommy Atkins mango was used to report state of mango ripeness in this study. According to this scale, fresh mango fruits ripeness was 5. The L* value was 63.56±4.73, accounting for a low degree of darkness, while b* parameter (57.81±4.22) indicates an intense yellow color, which was greater than that reported by ROCHA-RIBEIRO *et al*. (2008) (Table 1).

Table 1. Physicochemical properties of fresh Tommy Atkins mango.

Parameter		This study	Literature
Mango weight (g)		671.46±80.47	-
Soluble solids (°Brix)		16.22±1.62	14-16[b]
Moisture (%)		88.30±1.09	-
pH		3.78±0.15	3.4±0.1[a]
Acidity %		0.47±0.07	0.9±0.0[a]
Color	L*	63.56±4.73	55.0-61.1[b]
	a*	11.45±1.95	11.5-14.4[b]
	b*	57.81±4.22	40.0-50.0[b]

Mean ± standard deviation, n = 3.
[a]Siddiq *et al*. (2013).
[b]Rocha-Ribeiro *et al*. (2008).

3.2. Physicochemical, chemical and antioxidant properties of mango seed

Tommy Atkins mango seeds of 24.6±7.5 g weight and a 6.5±0.5 cm length used for study had a 37.7±0.3% moisture content. Extraction yield was 12.2±0.3 g of extract•(100 g of seed mango)$^{-1}$, which was comparable with yields reported by DORTA *et al*. (2012) for mango-seed extracts var. Keitt using 50% aqueous acetone solvent and 60 min ultrasound, 12.0±1.0 g of extract•(100 g of seed mango)$^{-1}$. Concentration of feee phenols in mango seed extracts was 23.9±0.0 g GAE•(100 g dry mass)$^{-1}$. This concentration was superior to that reported by SOGI *et al*. (2013), for mango var. Tommy Atkins from USA, 20.03-11.23 g GAE•(100 g dry mass)$^{-1}$, which was previously dried using different drying methods

(freeze drying, tray drying, vacuum drying and infrared drying) to extraction. In this study, freeze drying allowed highest retention of phenols compounds in mango seed extract. On the other hand, BERNAL-MERCADO *et al*. (2018) obtained a total phenol content of 27.7±0.1 g GAE•(100 g dry mass)$^{-1}$ from mango var. Haden. This last extract was obtained from a maceration at 25ºC for 10 days in darkness. This indicates that extraction assisted by ultrasound, in addition to being fast, allows a high retention of phenolic compounds in extracts from mango seeds. Additionally, it is important to avoid drying mango-seed samples with hot air.

3.3. Drying curves

During the drying time from 0 to 15 min, free water heating and evaporation occur slowly for PT1 pretreatment. All drying curves presented a significant moisture reduction in range from 15 to 75 min, Fig. 1. This behavior can be explained considering that during this drying period mango slice surface is wet, thus forming a continuous free water film. Consequently, there is no resistance to water transfer from solid surface to surrounding air. On the other hand, drying rate decreased after 75 min of drying for all pretreatments and control, indicating start of a decreasing drying rate period.

3.4. Effective diffusivities and constant drying rate

Effective diffusivity, D_{eff}, for different drying pretreatments and control (data no showed) lied in range of 1.17-1.35 x 10^{-9} $m^2 \bullet s^{-1}$. These values are similar to those reported by DISSA *et al*. (2008) for 5 mm-thick mango slices, dried at 60°C. A comparative analysis of D_{eff} means attested that there are not significant differences between mango pretreatments.
Midilli's n, k and a constants were calculated with Equation 4. The determination coefficients (R^2) for all pretreatments were higher than 0.99. The b constant for this study was zero, and n and a constants were found in ranges of 1.42±0.09-1.54±0.13 and 7.16±0.04-7.75±0.90, respectively. Constant k is considered a measure of water evaporation rate from the mango slice. The k values for pretreatments and control were not significantly different as compared by the Duncan's mean comparison test ($p<0.05$) and were found in range of 1.53±0.90x10^{-3}-2.27±0.80 x 10^{-3}. The k values obtained in this study are similar to that reported by MURTHY and MANOHAR (2014) for slices of dried mango at 60ºC and air velocity of 2.25 $m \bullet s^{-1}$: k = 0.054, n = 1.022 and R^2 = 0.969. While n value, which corresponds to drying kinetics order, is higher in present study than in that reported by MURTHY and MANOHAR (2014). It should be noted that an order of drying kinetics close to unity is indicative that drying process depends almost exclusively on temperature and as n value increases, it is indicative that other variables are also contributing significantly to food drying process. These variables are drying air velocity, mango slices thickness, concentrations and types of pretreatments, among others.

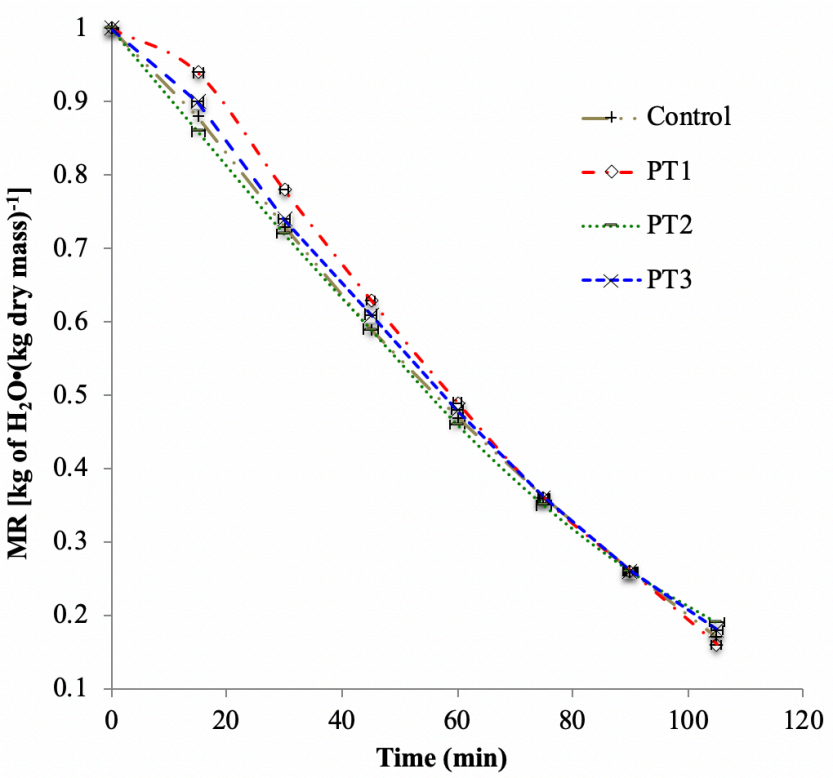

Figure 1. Drying curves for mango slices with different pretreatments. Control = Mango dried without pretreatment, PT1 = 0.5% (*w*/*v*) sodium metabisulphite, PT2 = 1.44% (*w*/*v*) seed extract mango, PT3 = seed extract mango/sodium metabisulphite [1.44%/0.5% (*w*/*v*)]. Each curve is mean of three drying replicates.

3.5. Free phenols

Fig. 2 shows free phenols content for dried and fresh mango slices. Free phenols content of fresh mango was 195.28±5.48 mg GAE•(100 g dry mass)$^{-1}$, which is within range reported by MANTHEY and PERKINS-VEAZIE (2009) of 171.8-257.3 mg GAE•(100 g dry mass)$^{-1}$, for a Mexican Tommy Atkins variety. From Duncan test comparisons, it can be implied that since control and PT1 pretreated mango slices (0.5% metabisulphite) are not statistically different ($p<0.05$), PT1 pretreatment did not have a significant effect on phenol retention (about 46%). This retention amount is similar to that reported by CHONG *et al.* (2013) (50.4%) who performed dried of mango slices using cold/hot air treatment. Treatment used by authors consisted of applying a flow of cold air (11.54±0.26°C) either at beginning or during dehydration process with hot air at 53.95±0.03°C.
On the other hand, dried samples which were pretreated with mango-seed extract (PT2 and PT3) had a much greater phenol content than corresponding values of both control and fresh mango samples (346.96±19.69, 368.00±11.84 mg GAE•(100 g dry mass)$^{-1}$, Fig. 2). This implies that, as opposed to bisulphites-only treatment (PT1), addition of mango-seed extract not only aided in retention of phenolic compounds but also in the increase of their concentration to 77.8 and 88.4% in PT2 and PT3 pretreatments, respectively. This effect appears to be due to the diffusion of phenolic compounds from pretreatment extract to the mango slices during the immersion period. It is also remarkable that in the case of PT3 pretreatment, addition of sodium metabisulphite caused a greater increase in total phenol content than that caused by use of the mango-seed extract alone (PT2) (Fig. 2). This seems

to indicate that sodium metabisulphite had a synergistic effect on retention and fortification of phenolic compounds during mango drying.

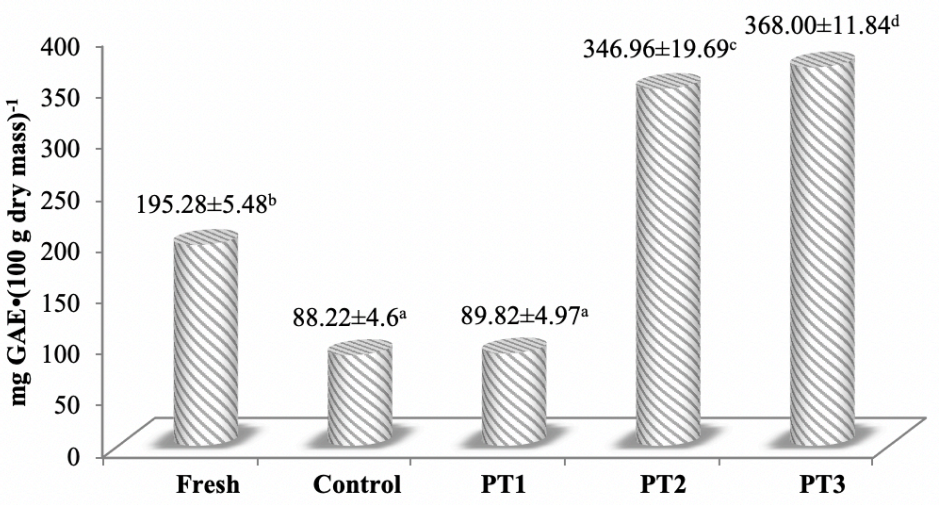

Figure 2. Free phenols content [mg gallic acid equivalents GAE•(100 g dry mass)$^{-1}$] in fresh mango and pretreated dried. Mean ± standard deviation, n = 3. Control = Mango dried without pretreatment, PT1 = 0.5% (*w*/*v*) sodium metabisulphite, PT2 = 1.44% (*w*/*v*) seed extract mango, PT3 = seed extract mango/sodium metabisulphite [1.44%/0.5% (*w*/*v*)]. Superscripts a-d showed a significant difference (α = 0.05) according to the Duncan test.

3.6. Profile of main phenolic compound in dried mango samples

The phenolic compound concentration profile of different pretreated samples was measured by HPLC for gallic acid, methyl gallate, mangiferin, caffeic acid, ferulic acid and *p*-coumaric acid (Table 2). Mangiferin concentrations were not affected by sample pretreatments with respect to control. Mango slices dried without pretreatment (control) had a similar behavior than mango sliced pretreated with metabisulphite (PT1), except for gallic acid. The PT1 pretreatment (0.5% sulphites only) caused a significant increase in the retention of gallic acid only (33.0%). On the other hand, PT2 pretreatment (1.44% mango-seed extract) significantly increased content of methyl gallate (27.4%), caffeic acid (70.9%), ferulic acid (244.4%), and *p*-coumaric acid (87%) with respect to control.

These results are also in agreement with total phenol assays in which PT2 samples had a higher phenolic content than both control and fresh samples, which confirms that PT2 dried products were enriched with phenolic compounds of mango-seed extract. This phenolic enrichment can be explained by considering that mango-seed extract solutions had a 10-fold higher concentration of free phenols than that present in fresh mango pulp, so molecular diffusion occurs from the extract to pulp by a concentration driving force.

In general, PT3 pretreatment samples (0.5% sulphites and 1.44% mango-seed extract) displayed greater increments in phenolic compounds concentration than those observed in PT2 pretreatment, with exception of methyl gallate, which is statistically equal in both treatments. In addition, PT3 samples presented a sharp increase in concentrations of gallic acid and caffeic acid with respect to PT2 pretreatment. This result agrees with free phenol

observations for PT2 and PT3 pretreatments, thus confirming synergic effect of bisulphites and mango extract on phenol enrichment of mango dried products.

Table 2. Phenolic compounds content in dried slices of Tommy Atkins mango quantified by HPLC.

Pretreatment	Compound [mg·(100 g dry mass)$^{-1}$]					
	Gallic acid	Methyl gallate	Mangiferin	Caffeic acid	Ferulic acid	*p*-Coumaric acid
Control	13.49±0.02^{a}	7.26±0.15^{a}	5.31±0.45^{a}	6.16±0.26^{a}	10.86±0.52^{a}	8.24±0.45^{a}
PT1	17.98±0.29^{b}	6.83±0.31^{a}	4.75±0.70^{a}	5.52±0.53^{a}	11.61±0.57^{a}	7.99±0.28^{a}
PT2	14.71±0.18^{c}	9.25±0.49^{b}	5.02±0.17^{a}	10.53±0.66^{b}	37.40±0.47^{b}	15.41±0.67^{b}
PT3	26.42±0.16^{d}	10.08±0.28^{b}	5.65±0.50^{a}	26.10±0.71^{c}	45.61±0.31^{c}	16.30±0.55^{c}

Control = Mango dried without pretreatment, PT1 = 0.5% (*w*/*v*) sodium metabisulfite, PT2 = 1.44% (*w*/*v*) seed extract mango, PT3 = seed extract mango/sodium metabisulfite [1.44%/0.5% (*w*/*v*)]. Mean ± standard deviation, n = 3. Superscripts a-d = mean difference significant in columns (α = 0.05) by Duncan test. In the calibration equation y = area and x = concentration of corresponding phenol compound.

3.7. Vitamin C content

Fresh mango samples had a vitamin C content of 135.59±3.40 AAE•(100 g dry mass)$^{-1}$ which is similar to that reported by ROCHA-RIBEIRO *et al*. (2007) (94.0 mg AAE•(100 g dry mass)$^{-1}$). Vitamin C contents in dried samples (Fig. 3) were greatly superior to those described by NDAWULA *et al*. (2004), who dried mango slices of 3-5 mm thickness in an open solar dryer. These authors reported a vitamin C content of 25.4 mg AAE•(100 g dry mass)$^{-1}$ and a 15.5% vitamin C retention in dried slices. Vitamin C content in PT2 (32.15±1.21 AAE•(100 g dry mass)$^{-1}$) pretreated samples (mango-seed extract only), was not significantly different than control (32.34±1.58 AAE•(100 g dry mass)$^{-1}$, $p<0.05$), thus indicating that PT2 pretreatment did not have a significant effect on retention of vitamin C in dried product. On the other hand, sulphites-added samples (PT1 and PT3) presented a higher vitamin C content than control samples, which indicates that bisulphites pretreatment efficiently promoted retention of vitamin C in dried mango (Fig. 3). Finally, PT3 pretreatment (combined sulphites and mango-seed extract) presented a 3-fold vitamin C content with respect to the control (96.52±5.09 AAE•(100 g dry mass)$^{-1}$, Fig. 3). Thus, similarly to phenol results, combined pretreatments of sulphites and mango-seed extract yielded an improved vitamin C retention in dried product (71.2%) as compared with individual pretreatments alone, which indicates a synergistic effect of such pretreatments on retention of antioxidant compounds during mango drying. However, in contrast to phenol results, sulphites pretreatment (PT1) was the most effective at maintaining ascorbic acid content of dried product.

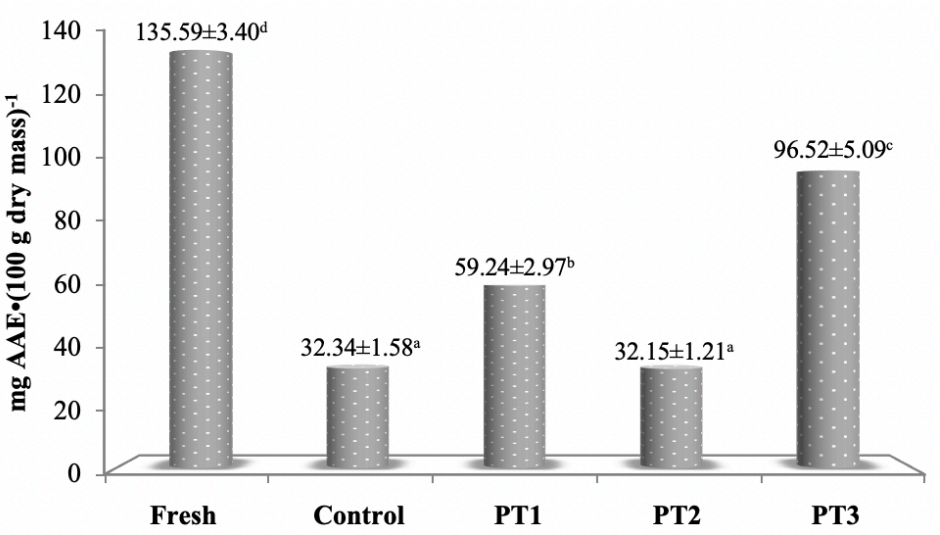

Figure 3. Content of vitamin C [mg ascorbic acid equivalents AAE•(100 g dry mass)$^{-1}$] in samples of fresh mango and pretreated dried. Mean ± standard deviation, n = 3. Control = without pretreatment, PT1 = 0.5 % (*w*/*v*) sodium metabisulphite, PT2 = 1.44% (*w*/*v*) mango seed extract, PT3 = [1.44%/0.5% (*w*/*v*)] mango seed extract/sodium metabisulphite. Superscripts a-d showed a significant difference (α = 0.05) according to the Duncan test.

3.8. Total carotenes content in dried products

The total carotenes content in fresh mango pulp was 25.54±0.81 mg of β-carotene•(100 g dry mass)$^{-1}$, Fig. 4, was within range reported by MANTHEY and PERKINS-Veazie (2009), who reported concentrations from 32.9 to 59.1 mg of carotenes•(100 g dry mass)$^{-1}$ in Mexican Tommy Atkins mangoes. Total carotenes content in sulphites-pretreated samples was 1.8 times higher than those reported by CHEN *et al*. (2007) for mango slices of 3 x 9 cm pretreated with 1% sodium bisulphite and dried with hot air at 60ºC. The variation in these results is probably due to different drying times and slice thickness. GUARTE *et al*. (2005) reported a carotene content of 6.80 mg•(100 g dry mass)$^{-1}$ for pulp mango, a very similar value to those measured for control and the PT2 samples in the present study.
From total carotene quantification of dried samples (Fig. 4), it was observed that mango-seed extract pretreatment (PT2) did not prevent carotene degradation since total carotene content with this pretreatment was similar than that of control [6.99±0.33 mg of β-carotene•(100 g dry mass)$^{-1}$, 7.06±0.321 mg of β-carotene•(100 g dry mass)$^{-1}$, respectively, Fig. 4]. Sulphites pretreatment (PT1) caused only a slight increase in total carotenes concentration with respect to control, which accounted for 29.3% of carotenes retention. On the other hand, PT3 combined pretreatment (sulphites and mango-seed extract) caused a remarkable increase in total carotenes content of dried sample with a 40.2% carotenes retention. Despite the fact that in this pretreatment carotenes retention was lower than 50%, total carotenes concentrations were 2.4 times higher than those obtained by CHEN *et al*. (2007) using a 1% sodium bisulphite treatment. In agreement with the previous results in this work, retention of carotenes was influenced synergistically by pretreatment with both the sulphites and mango-seed extract. Furthermore, the synergistic effect of combined pretreatments on carotene retention was more pronounced than with the other compounds, considering that in this case the individual pretreatments alone had little or no effect. A very high Pearson's correlation of total carotenes with vitamin C (r = 0.9580,

p = 0.0420) showed total carotenes are directly related with vitamin C content in mango slice during drying process.

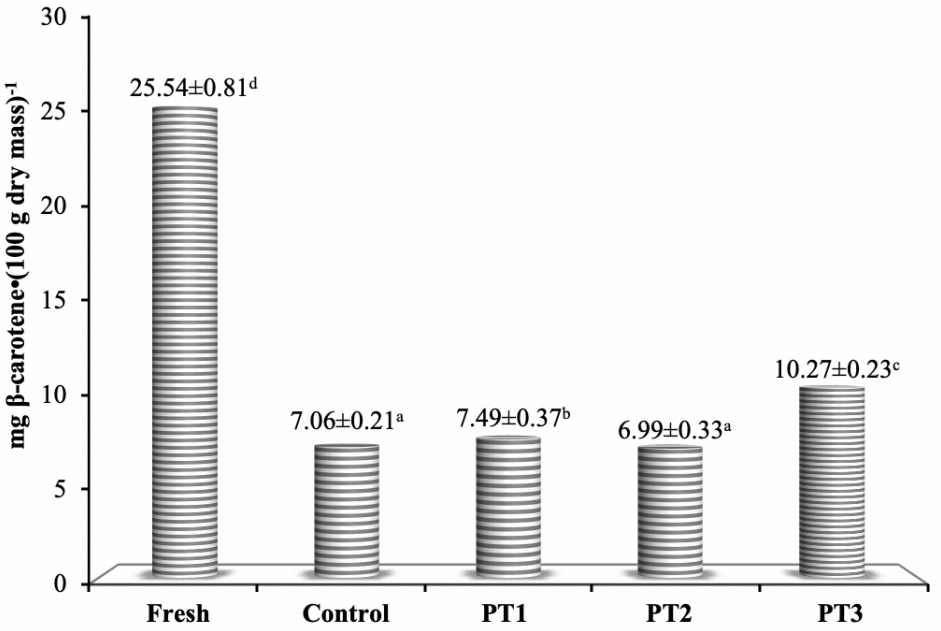

Figure 4. Content of total carotenes [mg β-carotene•(100 g dry mass)$^{-1}$] in samples of fresh mango and pretreated dried. Mean ± standard deviation, n = 3. Control = without pretreatment, PT1 = 0.5 % (*w*/*v*) sodium metabisulphite, PT2 = 1.44% (*w*/*v*) mango seed extract, PT3 = [1.44%/0.5% (*w*/*v*)] mango seed extract/sodium metabisulphite. Superscripts a-d showed a significant difference (α = 0.05) according to the Duncan test.

These results also could be ascribed to diffusion of antioxidant compounds from mango-seed extract/sodium metabisulphite solution to mango slice during pretreatment. This diffusion of antioxidant compounds could have contributed to maintain the solid structure of mango slices and thus reduce damage of mango pulp cells during drying process. The mango cells integrity during drying possibly reduced antioxidants compounds diffusion, including carotenes, from cell inside to mango slice surface. ADILETTA *et al*. (2016) provided evidence related to effect of pretreatments on solid structure preservation of foods during drying process. This study consisted in evaluating the pretreatment effect based NaCl 0.5% and trehalose 0.5% on eggplants drying. They observed by scanning electron microscopy (SEM) an increase in dried samples porosity, preserving its solid structure, while samples without pretreatment showed collapse and shrinkage phenomena. It is suggested that sodium metabisulphite behaves analogously to NaCl, promoting pores on the surface of mango slices during drying process, facilitating diffusion phenomena. Also, the combined effect of sodium metabisulphite with mango-seed extract, potency the decrease of antioxidant compounds degradation in mango slices.

3.9. Sulphites content in dried mango slices from PT1 and PT3

Sulphites are utilized as antioxidant additives for preventing oxidation, kept flavour and color, inhibit the growth of microorganisms that promote food spoilage, and also are anti-

browning agents for controlling enzymatic and non-enzymatic (Maillard) reactions (LOU *et al*., 2017). Otherwise, sulphites are allergenic components that can cause allergic reactions in asthma patients and people with diminished sulphite oxidase activity (SOUBRA *et al*., 2007). Additionally, these compounds can cause skin reactions (VALLY *et al*., 2009) and DNA damage (MENG *et al*., 2005). Hence, food safety organizations have considered an acceptable limit for sulphites in foods.

Sulphites concentrations in mango slices pretreated from PT1 and PT3 were 820.10±11.45 and 900.28±43.97 mg sulphites•(kg dry mass)$^{-1}$, respectively. Duncan's test showed a significant difference (α = 0.05) between samples PT1 and PT3. The latter showed approximately 9% more sulphites than PT1. Result indicates that mango-seed extract promoted an increase in sulphites diffusion towards the mango slice during immersion. Also, it is important to mention that sulphites concentrations in mango dried slices PT1 y PT3 are within the limit established by the Codex Alimentarius Commission, created by the World Health Organization and the United Nation's Food and Agriculture Organization (FAO), which supports a general maximum of 1250 mg•(kg SO_3^{-2})$^{-1}$ in dried fruits (LIAO *et al*., 2013).

3.10. Product color

The color parameters CIE L*a*b* and Hue° were determined for fresh and dried slices mango, Table 3.

Table 3. Color parameters CIE L*a*b* and Hue angle in dried slices and fresh mango.

Pretreatment	L*	a*	b*	Hue angle
Control	65.61±3.00^{b}	16.97±1.37^{c}	66.74±4.33^{b}	75.72±1.00^{a}
PT1	60.50±4.20^{a,b}	14.14±1.87^{b}	58.81±5.01^{a}	76.39±2.17^{a}
PT2	66.83±3.97^{b}	12.33±1.53^{a,b}	63.44±4.64^{a,b}	78.96±1.59^{b}
PT3	71.38±3.39^{b}	10.88±1.88^{a}	69.35±2.37^{b}	81.08±1.61^{b}
Fresh	63.56±4.73^{b}	11.45±1.95^{a}	57.81±4.22^{a}	78.84±1.43^{b}

Control = Mango dehydrated without pretreatment, PT1 = 0.5% (w/v) sodium metabisulfite, PT2 = 1.44% (w/v) seed extract mango, PT3 = seed extract mango/sodium metabisulfite [1.44%/0.5% (w/v)]. Mean ± standard deviation, n = 3. The letters a-c showed a significant difference in columns (α = 0.05) with Duncan test.

The values for L*, a*, b* and Hue° were found in ranges 60.50-71.38, 10.88-16.97, 57.81-69.35 and 75.72-81.08, respectively. Parameter L* indicates brightness degree of the sample on a scale from 0 (black) to 100 (white). The L* values allow affirm that pretreatment of mango slices with sulphites help to avoid the darkening of dried mango, as it expected. However, when sulphites are combined with extract of mango-seed, the effect is lost. This last behavior is attributed to the phenoloxidase enzymes present in seed mango extract that degrade the phenolic compounds to melanines. These compounds are responsible of darkening of mango slices during drying process. The temperature and sonication time used to obtain mango-seed extract, according to CHENG *et al*. (2013) do not inactivate phenoloxidase enzymes. These enzymes are inactivated only at temperatures above 62°C and ultrasound frequencies above 20 kHz.

Parameter a* indicates sample redness degree and as sample color is redder, a* has a bigger positive magnitude. Results obtained for a* show drying process induces an increase of redness in mango slices without mango-seed extract (control and PT1), Table 3. Parameter b* with positive values indicates yellowness degree of sample. Dried sample with 0.5% sodium metabisulphite and seed mango extract (PT3) show the higher valor of b*, 69.35±2.37. This result is concordant with carotenes content and indicates a combined effect protective of sodium metabisulphite and phenol compounds present in pretreatment (PT3).
Hue angle is one color property, defined as the degree to which a stimulus can be related with red, orange, green, yellow, green, blue and violet. A Hue° value of 90 represents a yellow tone. Therefore, from the data in Table 3 it can be affirm that samples pretreated with 0.5% sodium metabisulphite (PT2 and PT3) have a yellower tone than control and PT1 samples.

4. CONCLUSIONS

Midilli's model fitted very well to experimental data of mango slices dried without pretreatment and with the three pretreatments (PT1, PT2 and PT3) used in the present study. Comparative analysis of Midilli's constants, k, a and b using Duncan's means test showed pretreatments did not influence drying process of mango slices. Also, the results of antioxidant compounds quantification showed (1.44%/0.5%) mango-seed extract/sodium metabisulphite used as a pretreatment (PT3) has an important role on retention of vitamin C and carotenes in dried mango slices. The free phenols content was quadrupled compared to dried slices without pretreatment (control) and nearly were doubled compared to fresh mango. Mango slices pretreated with mango-seed extract were strongly enriched with gallic acid, caffeic acid, ferulic acid and *p*-coumaric acid, also with, methyl gallate to a lesser extent in relation to mango slices that did not receive a pretreatment (control). Furthermore, sulphites content of this dried product is within limit established by the Codex Alimentarius of FAO. Finally, this is the first study reporting combined use of mango-seed extract with sulphites as a pretreatment for drying mango pulp. Mango seed, which is generally an agro-industrial residue, used to obtain extracts rich in phenolic compounds and using these as a pretreatment for drying mango pulp and other foods could become commercially feasible in the proximate future.

ACKNOWLEDGMENTS

The authors thank Carol Ann Hayenga for her english assistance in the preparation of this manuscript and the reviewers for supplying suggestions and recommendations to accomplish standards of journal. Analleli Jiménez-Durán thanks the Consejo Nacional de Ciencia y Tecnología (CONACYT) from the Mexican government for its financial support through the M.Sc. scholarship Nº 483993. The Universidad Tecnológica de la Mixteca provided support.

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Paper Received February 1, 2020 Accepted July 4, 2020