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Osmotic pretreatment to assure retention of phenolics and anthocyanins in berry jams

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ARTICLE INFO

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ethanol (PubChem CID: 702)
hydrochloric acid (PubChem CID: 313)
potassium chloride (PubChem CID: 4873)
sodium acetate (PubChem CID: 517045)
pectin (PubChem CID: 441476)
distilled water (PubChem CID: 962)
sodium carbonate (PubChem CID: 10340)
cyanidin 3-O-glucoside (PubChem CID: 441667)
gallic acid (PubChem CID: 370)

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ABSTRACT

A Rotational Central Compound Design was used to optimize time and sucrose concentration for osmotic pretreatment of blueberries and strawberries to preserve anthocyanin and phenolic compound content and color during subsequent jam preparation. No significant differences were found for color, but statistically significant differences in the phenolic compounds and anthocyanins content in the resulting jam were observed. Superposition of the resulting response surfaces allowed for prediction of optimized osmotic pre-treatment time and sucrose concentration. The predicted values were confirmed in triplicate. The results indicate that pretreatment times of 242 and 219 min and sucrose concentrations of 1.65 and 1.46 M recovered the greatest amount of the target compounds in blueberry and strawberry jams, respectively.

1. Introduction

Blueberry (*Vaccinium corymbosum*) and strawberry (*Fragaria x ananassa*) are important sources of bioactive chemicals such as flavonoids, stilbenes, tanins, and phenolic compounds (Tulipani et al., 2008). These compounds may protect against degenerative diseases, and their effects on health have been commonly attributed to their antioxidant properties (Seeram, 2008).

Although these fruits may be consumed raw, their preservation in processed food, such as jams, jellies, and cakes is attractive as it improves shelf life and allows for year-long enjoyment (Olsson et al., 2004; Côté, Caillet, Doyon, Sylvain, & Lacroix, 2010; Garzón, Riedi, & Schwartz, 2009; Scibisz & Mitek, 2009). However, preparation of jams (Figuerola, 2007) brings a number of challenges, one of which is to preserve the beneficial compounds in the raw fruit, while simultaneously inhibiting spoilage (Kowalska, 2005). Duration of heat treatment, identity and amount of ingredients, and pretreatment of the fruit are all reasonable preparation steps to optimize. Indeed, heat treatment and addition of sugar and citric acid can affect jam quality and final concentrations of flavones and anthocyanin (Scibisz & Mitek, 2009; Wu, Frei, Kennedy, & Zhao, 2010).

A common pretreatment method is osmotic dehydration, which involves immersion of the fruit in a hypertonic solution (Castañeda, Arteaga, Siche, & Rodriguez, 2010). This process may preserve desirable characteristics of the fruit later used in jam making by decreasing water activity in the fruit (Rahman, 2008; Phisut, 2012). The driving force of the process is the difference in solute concentration between the solution and interstitial fluid which causes the exit of water from and the entrance of solutes to the cells until osmotic equilibrium is reached (Phisut, 2012; Rahman, 2008).

Research suggests that osmotic dehydration of strawberries with sucrose before jam preparation stabilizes anthocyanin more than the

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mere addition of sucrose during jam preparation (Watanabe, Yoshimoto, Okada, & Nomura, 2011). However, the identity and concentration of solute (typically sucrose); temperature; immersion time; pressure; ratio of food to solution; and the structure, permeability, shape, and size of raw food all have an influence on the process (Osorio et al., 2007). In the present work, the concentration of phenolic compounds, anthocyanin content and color were optimized in the prepared jam by varying the sucrose concentration and time of osmotic pretreatment.

To design the optimization efficiently, response surface methodology (RSM) was used. This technique allows for varying of multiple parameters simultaneously with a defined set of experiments. The results are used to prepare a surface predicting the response of the dependent variables in the experiment. The main advantage of RSM is use of minimum experimental runs necessary for statistical validity. RSM is faster and gives more information than the classical one-variable-at-a-time approach or the use of full factorial designs (Ozdemir, Ozen, Dock, & Floros, 2008).

2. Materials and methods

2.1. Raw material

Blueberries (*Vaccinium corymbosum*, Biloxi variety) and strawberries (*Fragaria* × *ananassa*, Tioga variety) were obtained from Virú and Trujillo Provinces, respectively, Department of La Libertad, Peru. Fruits were harvested at commercial ripeness and shipped under refrigeration to the Department of Agroindustrial Engineering at the National University of Trujillo. Fruit was packed in 300 g clamshell containers and stored in the dark at 2.0 ± 0.5 °C and 95 ± 2% RH. The following day, samples of the berries were pulped using a blender to measure pH, °Brix, and titratable acidity using a portable pH meter (Hanna Instruments, Nusfalau, Romania) and a refractometer (Atago, PAL-1, Fukaya, Japan). Titratable acidity was determined according to the AOAC official method 942.15 (AOAC, 1999) and the results were expressed as both the percentage of citric acid in the products and in the dried matter.

2.2. Jam preparation

A single lot of blueberries (14.0 ± 0.2°Brix, pH =3.0 ± 0.1 and 0.78 ± 0.04% of titratable acidity) or strawberries (6.0 ± 0.3°Brix, pH =3.6 ± 0.2 and 0.60 ± 0.05% of titratable acidity) was used to prepare all jam samples. Berries without bruises, signs of dehydration, softening or contamination were cleaned, disinfected, and added 1:3 w/v to sucrose solutions (between 0.88 and 2.04 M for blueberries and between 0.88 and 1.76 M for strawberries). The samples were incubated at 25 ± 1 °C and under agitation (40 rpm, vertical stirrer IKA® EUROSTAR 20 Digital, Königswinter, Germany) for various time points between 60 and 360 min (Queiroz, Oliveira, Pinho, & Ferreira, 2009; Watanabe et al., 2011). After impregnation, the excess sucrose solution was removed by draining. Then, the fruit was lightly homogenized in an Ultra Turrax (T25 model; Janke and Kunkel Co., Staufen, Germany), at 3000 rpm x 1 min, to obtain a thick pulp.

Jam was prepared by combining 60% wet fruit pulp, 40% sucrose (w/w) and high methoxyl pectin (4 g/kg of pulp). The main source of high methoxyl pectin is citrus peel (Aglupectin HS-MR; gel strength: 145–155; degree of methoxylation: 64–67). The combined mixture was heated in a stainless steel container until it reached 62°Brix and a temperature greater than 85 °C. Aliquots (250 g) of the prepared jam were added to round glass jars (212 mL, twist off cap, 73.5 mm highx69.7 mm wide). The jars were hermetically sealed and cooled to 15 °C for at least 2 h. The samples were stored at 5 °C for at least 24 h before being used in the analysis.

2.3. Physicochemical analysis

Jam was further homogenized (5000 rpmx3 min) for the evaluations of content of anthocyanin, phenolic compounds and color (lightness) according to the experimental design.

2.3.1. Total monomeric anthocyanins

Acidified ethanol (0.01 M solution of HCl in ethanol; 40 mL) was added to 10.0 g homogenized jam. The extract was kept cool overnight, and filtered the next day using S & S 520 (Schleicher & Schuell - Whatman filter paper grade 520, Maidstone, United Kingdom) (Wicklunda et al., 2005).

The total monomeric anthocyanins (TA) in the extracts were estimated using the pH-differential method (Lee, Durst, & Wrolstad, 2005) using hydrochloric acid / potassium chloride buffer (0.025 M, pH 1.0) and sodium acetate buffer (0.4 M, pH 4.5). Briefly, 0.2 mL of the filtrate was mixed with 1.8 mL of one of the buffer solutions and the absorbance was measured at 510 nm and 700 nm on a UV–vis spectrophotometer (PLUS 250, Jena, Germany).

The concentration of anthocyanins in the extract was calculated and expressed as cyanidin-3-O-glucoside equivalent per g of jam according to Eq. (1) (Giusti & Wrolstad, 2001).

$$\text{TA (mg/100 g)} = \frac{(A * M_W * \text{DF} * 100)}{M_A * L} \quad (1)$$

where:
$A = (A_{510} - A_{700})_{pH\ 1.0} - (A_{510} - A_{700})_{pH\ 4.5}$; M_w= molecular weight (449.2 g/mol); DF= dilution factor (50); M_a= extinction coefficient 26,900 L/(cm*mol) (Jurd & Asen, 1966); L= path length (1 cm).

2.3.2. Phenolic compounds

Homogenized jam was diluted with ethanol in a flask (2 g jam/10 mL solution). The extract was kept cool overnight, and filtered (S & S 520) the next day. Phenolic compound content was determined using the Folin-Ciocalteu assay (Singleton & Rossi, 1965) with a slight modification.

Briefly, 20 μL of diluted extract was mixed with 100 μL of Folin-Ciocalteu reagent and 1580 μL distilled water. The mixtures were vortexed (Thermo Scientific™ A LP Vortex Mixer, FBKT17302, Darmstadt, Germany), kept in the dark at room temperature for 20 min, and then transferred into a 40 °C water bath with 300 μL addition of 20% sodium carbonate (w/v) for another 20 min. Samples were immediately cooled in an ice bath for 3 min, and the A_{765} of the samples was measured using the UV–vis spectrophotometer (PLUS 250, Jena, Germany). With gallic acid as the standard, a calibration curve (Ab =2.5676*c+0.5686; r^2 =0.9905; Ab: absorbance; c: concentration of gallic acid) was prepared with 0.05, 0.1, 0.15, 0.20, 0.25, 0.30, 0.35, 0.40, 0.45 and 0.50 mg/mL gallic acid solutions, and the results were expressed in gallic acid equivalents (mg GAE/g jam) (Piljac-Zegarac, Valek, Martinez & Belščak, 2009).

2.3.3. Color analysis

A colorimeter (Minolta CR-400 Chroma Meter, Osaka, Japan) having an 8 mm diameter viewing area was used for chromatic analyses following the CIE-1976 norms (Commission International de l'Eclairage,l'Eclairage, 1995). Lightness, red/green and yellow/blue are represented by L*, a* and b*, creating a three-dimensional color space, where a* and b* are two color axes (Goncalves et al., 2007).

2.4. Experimental design

A rotatable central composite design (RCCD) with 22 full factorial points, 3 central points and star points was used to select the time and concentration of osmotic pretreatment for blueberries and strawberries (Supplemental Information Tables 1 and 2). Analysis of variance