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Original Research Article

Chemical composition and phenolic compounds profile of cladodes from *Opuntia* spp. cultivars with different domestication gradient

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ABSTRACT

The *Opuntia* genus, whose origin is in Mexico where several species grow wild, is taxonomically diverse and has become an important crop worldwide. The aim of this work was to assess the molecular composition of five *Opuntia* species and three different varieties of each taxonomically identified species with different domestication gradients (from the wildest to the most domesticated *O. ficus-indica*). Data were subjected to chemometric evaluation using principal component analysis (PCA), which showed that *O. ficus-indica* seems to fall far outside of the current domestication gradient that has been proposed, and its ancestor is still unclear. Phenolic compounds showed major (eucomic acid, kaempferol 3-O-robinobioside-7-O-arabinofuranoside, isorhamnetin 3-O-galactoside, and isorhamnetin 3-O-rhamnoside-7-O-(rhamnosyl-hexoside)) and minor compounds that were present only in wild (kaempferol 3-O-arabinofuranoside) or domesticated (quercetin 3-O-rhamnosyl-(1→2)-[rhamnosyl-(1→6)]-glucoside) species. This information could be very useful for authentication of *Opuntia* species and the identification of species with the highest potential as sources of compounds with nutritional and therapeutic properties.

1. Introduction

The genus *Opuntia* belongs to the Cactaceae family, subfamily *Opuntiodeae*. *Opuntia* presents high adaptation capacity to extreme environmental conditions (high temperature, drought, UV radiation) and is distributed in arid and semiarid regions (Stintzing and Carle, 2005; Reyes-Agüero and Aguirre, 2011). The *Opuntia* genus is taxonomically diverse and widely distributed in the Americas; 78 species are native to Mexico where the richness of wild and semi-domesticated variants and cultivars can be found (Reyes-Agüero et al., 2005). Since ancient times in Mexico, young cladodes known as "nopalitos" and fruits (prickly pear) were consumed as food (Stintzing and Carle, 2005). Since the beginning of the 1950s, *Opuntias* have spread and currently cover about 66,000 ha of commercial plantations in Mexico (Bellón et al., 2009); however, several Mexican *Opuntia* species are still mainly located in their own wild habitat such as backyards, plantations, and agricultural terraces. Nowadays, the most propagated *Opuntia* for commercial production, distributed worldwide, is the domesticated *O. ficus-indica* (Griffith, 2004).

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Domestication is an evolutionary process in which humans transform natural plant populations by encouraging the dominance of more useful physiological, chemical, or genetic features of wild plants and by discouraging or eliminating minor variants. *Opuntias* have been domesticated or naturally selected by humans for food, ornamental uses and medicinal purposes (Stintzing and Carle, 2005). The effect of domestication in *Opuntia* has been reflected in improved attributes in fruits such as size, shape, color, texture, and flavor. In *Opuntia ficus-indica*, the effect of domestication has resulted in oval or rhombic cladodes; an abundance of glochidia, a red fruit color; and fewer and smaller spines (Reyes-Agüero and Aguirre, 2011). For these morphological characteristics, domestication gradients have been proposed starting with the wildest species of *O. streptacantha*, continuing with variants of *O. megacantha* and *O. chaveña*, and ending with the most domesticated variants from two species, *O. albicarpa* and *O. ficus-indica* (Reyes-Agüero et al., 2005).

It is well known that the chemical composition of cladodes is modified by maturity stage, harvest season, environmental conditions, post-harvest treatment and type of species (Guevara-Figueroa et al., 2010; Contreras-Padilla et al., 2011; Hernández-Urbiola et al., 2011). Therefore, for comparative analysis it is important to select plant material cultivated under the same environmental conditions and at the same developmental stage. On the other hand, there is a lack of information about the effect of domestication at the molecular and biochemical level. Thus the aim of this study was to evaluate proximate composition, mineral content, phenolic compounds, and antioxidant activity of fifteen taxonomically identified *Opuntia* spp. with different domestication gradients. In an attempt to classify the *Opuntia* samples, the data were subjected to chemometric evaluation using principal component analysis (PCA). *Opuntia* species were classified into seven groups by PCA. Interestingly, the chemometric evaluation indicated that *O. ficus-indica* seems to fall far outside of the current domestication gradient that has been proposed for it, and its ancestor is still unclear. In addition, the phenolic compound composition was investigated using LC-MS/MS.

2. Materials and methods

2.1. Chemicals and reagents

Gallic acid (≥98%), Folin-Ciocalteu phenol reagent, sodium carbonate (Na_2CO_3, 99.95–100.05%), quercetin (≥98%), aluminum chloride ($AlCl_3$, ≥99.9%), 1,1-diphenyl-2-picrylhydrazyl, 6-hydroxy-2,5,7,8-tetramethylchroman-2-carboxylic acid (DPPH, ≥97%), and dextrose (≥99.5%), anthrone–H_2SO_4 (≥97%), HNO_3 (70%), HCl (≥37%), absolute ethanol were purchased from Sigma–Aldrich Chemical Co. (St. Louis, MO, USA). All solvents were ACS grade. Ultra-pure water (18.2 MΩ/cm resistance) was obtained from a Milli-Q-plus purification system (Millipore Corp., Bedford, MA, USA). Acetonitrile and formic acid were UHPLC–MS grade (Sigma–Aldrich).

2.2. Plant material

Fifteen varieties of five *Opuntia* species representative of the domestication gradients described by Reyes-Agüero et al. (2005) were selected. These included the wildest *O. streptacantha* and *O. hyptiacantha*, the intermediate domestication *O. megacantha* and *O. albicarpa*, and the most domesticated *O. ficus-indica* (Supplementary data, Table S1). At one extreme, *O. streptacantha* has abundant, long thick spines, while at the other extreme, the domesticated *O. ficus-indica* has few, short, thin spines and is the species of major national and international importance.

Opuntia spp. young cladodes or "nopalitos" were collected in April 2010 at the *Opuntia* Germoplasm Bank from the Agrobotanical Garden located in "El Orito", Zacatecas, México (22° 44.7′ N, 102° 36.4′ W). The donor *Opuntia* plants were grown under the same natural environmental conditions (temperature, precipitation and soil). Three young cladodes were harvested from healthy plants with homogenous coloration and mean length of 15–20 cm. Cladodes were washed and frozen under liquid nitrogen, the spines were removed and ground in a mill (KRUPS GX4100, Mexico), and then stored at −80 °C. Samples were freeze-dried (LABCONCO, Kansas, MO, USA) and dry material was sieved through a mesh 80 and stored in plastic bags at 4 °C until use.

2.3. Proximate composition analysis

Total nitrogen content was determined by the micro-Kjeldahl method (AOAC, 2007, method 12.960.52), and total protein content was calculated using a 6.25 factor. Fat content was determined by the Soxhlet method (AOAC, 2007, 996.01 method). Crude fiber and ash contents were obtained according to AOAC (2007) methods 991.43 and 900.02, respectively. All determinations were analyzed at least in triplicates.

2.4. Mineral content determination by ICP-MS (inductively coupled plasma mass spectrometry)

For mineral content analysis, hydrolysis of the samples was performed according to the optimized method in the National Laboratory of Medical and Environmental Biotechnology (LANBAMA). Samples (0.1 g) were hydrolyzed using a HNO_3:HCl (1:3) mixture, on a hot plate at 250 °C for 5 min, diluted with ultra-pure water and filtered through 0.45 μm paper (Whatman Int. LTD, Maidstone, UK). Samples were analyzed in a Varian 820-MS ICP mass spectrometer (Agilent Technologies, Santa Clara, CA, USA).

2.5. Total sugars, titratable acidity and pH

Total soluble sugars were determined using the anthrone–H_2SO_4 reagent method (Irigoyen et al., 1992). Dextrose was used as a reference compound and results were expressed as mg of dextrose/100 g of sample. Titratable acidity and pH were measured according to the AOAC (2007) method. For pH measurement, samples (1 g) were added to 10 mL of ultra-pure water, homogenized and measured with a pH meter 210 (Hanna Instruments, Carrolton, TX, USA). Acidity of samples (1 g) was titrated to a pH 8.2 using a 0.1 N NaOH solution (Sigma–Aldrich). Results were expressed as % citric acid = [(N*V*mEq citric acid)/W], where N was the concentration of NaOH; V, the volume of NaOH used for titration; W, the weight of sample; and mEq, the citric acid milliequivalents (0.064).

2.6. Phenolic compound extraction procedure

The phenolic compound extraction procedure was carried out as previously reported with some modifications (Guevara-Figueroa et al., 2010). Briefly, samples (1 g) were added to 100 mL of absolute ethanol pre-chilled at −20 °C, mixed at 4 °C for 2.5 h and centrifuged at 13,000 × g at 4 °C. Supernatants were filtered (No. 0.45 μm filter, Whatman) and used for phenolic compound determinations. Independent extraction procedures (triplicates) were performed for each sample. All determinations were made within 24 h after extraction.

2.7. Total phenolic contents

Phenolic compounds were analyzed using the Folin-Ciocalteu method as reported (Guevara-Figueroa et al., 2010). The ethanolic