

Encapsulation of soursop (*Annona muricata*) fruit pulp in calcium alginate hydrogels

Encapsulación de pulpa de guanábana (*Annona muricata*) en hidrogeles de alginato de calcio

Alex López-Córdoba^{1,2}, Edgar Mauricio Morales V.¹, Mónica María Pacheco V.¹, and Alba Sofía Navarro^{2,3}

ABSTRACT

Soursop (*Annona muricata*), so known as guanábana, constitutes a rich source of bioactive compounds which would have great potential to develop functional foods. In the current work, alginate hydrogels added of different concentrations of soursop pulp (5, 10 and 20 wt.%) were obtained by ionic gelation. Hydrogels showed diameters around 4 mm and high circularity. After drying, the systems exhibited values of water content between 8 and 10% and water activity between 0.3 and 0.4. Moreover, micrographs obtained by scanning electron microscopy showed that the incorporation of soursop pulp at 20% led to better preservation of the morphology of the beads. These preliminary results suggest that the calcium alginate beads containing soursop pulp would have useful applications as healthy food ingredients.

Key words: encapsulating systems, ionic gelation, alginate beads, functional ingredients.

RESUMEN

La guanábana (*Annona muricata*) constituye una fuente rica de compuestos bioactivos que pueden tener un gran potencial en el desarrollo de alimentos funcionales. En el presente trabajo se obtuvieron hidrogeles de alginato añadidos de diferentes concentraciones de pulpa de guanábana (5, 10 y 20%), mediante gelificación iónica. Los hidrogeles mostraron diámetros de alrededor de 4 mm y alta circularidad. Posterior al secado, los sistemas exhibieron valores de contenido de humedad entre 8 y 10% y de actividad acuosa entre 0,3 y 0,4. Micrografías obtenidas mediante microscopía electrónica de barrido mostraron que la incorporación de pulpa de guanábana al 20% permitió preservar mejor la morfología de las cápsulas. Estos resultados preliminares sugieren que las cápsulas de alginato de calcio conteniendo pulpa de guanábana pueden tener aplicaciones útiles como ingredientes alimentarios saludables.

Palabras clave: sistemas de encapsulación, gelificación iónica, cápsulas de alginato, ingredientes funcionales

Introduction

Soursop (*Annona muricata*) is a tropical fruit native from and common mainly in tropical America. This fruit is among the most important tropical fruits that have contributed to the economic growth of countries such as Brasil, Mexico and Venezuela.

Soursop fruit pulp is rich in minerals, vitamins and dietary fiber (Jiménez *et al.*, 2014). In addition, the pulp of this fruit has been proposed as a rich source of phenolic compounds, such as cinnamic acid derivative and p-coumaric acid, which have reported some potential health beneficial properties for humans (e.g., serving as antioxidants and protecting against chronic diseases such as cancer, brain dysfunction and atherosclerosis) (Jiménez *et al.*, 2014). Soursop fruits are commonly used for preparing juices, refreshing drinks, nectars, ice creams, sherbets and desserts.

Thus, the fresh fruit is often processed as a pulp and then preserved by pasteurization or freezing for industrial processing and commercialization. However, several active compounds from soursop fruit could be degraded by the effect of the applied treatment.

Ionic gelation is a simple and fast way of obtaining encapsulating systems, which allow protecting the encapsulated compound from factors that may cause deterioration such as temperatures, light, pH, moisture, and oxygen (López *et al.*, 2013, 2014). In this process, gel beads are formed by dropping an alginate aqueous solution containing active compounds into a divalent or polyvalent cation solution (e.g. Ca^{2+} and Zn^{2+}). Alginate hydrogels have been widely used as carriers of bioactive compounds such as natural antioxidants, cells, unsaturated oils, drugs, among others (Ćujić *et al.*, 2016; Deladino *et al.*, 2013; López *et al.*, 2014; Vicini *et al.*, 2015).

¹ GIADAI - Grupo de Investigación en Innovación, Desarrollo Tecnológico y Competitividad en Sistemas de Producción Agroindustrial, Escuela de Ingeniería Agroindustrial, Instituto Universitario de La Paz. Barrancabermeja (Colombia). alexlcordoba@gmail.com

² CIDCA - Centro de Investigación y Desarrollo en Criotecnología de los Alimentos, CONICET, Facultad de Ciencias Exactas (UNLP). La Plata (Argentina).

³ Facultad de Ingeniería, Universidad Nacional de la Plata. La Plata (Argentina).

In the current work, the ionic gelation technique is presented as a non-thermal alternative to protect soursop fruit pulp. To the best of our knowledge, this is the first study on the encapsulation of soursop fruit pulp within calcium alginate beads for potential use as functional food ingredients.

Materials and methods

Materials

Soursop pulp with 7°Brix (Carrusel, Cali, Colombia) was purchased from a local market in Barrancabermeja, Colombia. Sodium alginate with 90.8% of purity (Qingdao, China) was used as encapsulating agent and calcium chloride dihydrate ($\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$; Merck, Germany) as gelling reagent for hydrogels production.

Hydrogels preparation

Hydrogels with and without soursop fruit pulp were prepared by the ionic gelation technique as described in previous works (López *et al.*, 2013, 2014). For control hydrogels preparation, 1 g of sodium alginate was dissolved in 100 mL of water. The soursop pulp-containing hydrogels were obtained adding 5 g, 10 g or 20 g of soursop pulp into 100 mL of the alginate solution described previously. Once homogenized, the solutions were dipped into a calcium chloride solution (0.05 mol L^{-1}). The formed hydrogels were maintained in the gelling bath to harden for 15 min. Then, they were filtered and washed with distilled water.

Some hydrogel samples were dried in a convection oven (SanJor, Argentina) at 50°C for overnight previous to characterization measurement for comparison.

Hydrogels characterization

The shape and mean size of hydrogels were calculated analyzing photographs of at least 10 hydrogels with the

ImageJ processing software. Circularity was calculated as follows (Schneider *et al.*, 2012):

$$\text{Circularity} = 4 \pi (\text{area/perimeter}^2) \quad (1)$$

Moisture content (%) was measured gravimetrically by drying the samples in an oven at 105°C , until constant weight. Values of water activity (a_w) were determined using Rotronic Hygropalm equipment.

Scanning electron microscopy (SEM) analysis was performed using a FEI, Quanta 200 microscope (Netherlands). Beads were attached to stubs using a two-sided adhesive tape, then coated with a layer of gold (40-50 nm) and examined using an acceleration voltage of 20 kV.

Statistical analysis

Four different batches of hydrogels were prepared and characterization experiments of each batch were performed at least in duplicate. The data were reported as mean \pm standard deviation. The statistical analysis was performed using the Systat Inc. software (Evanston, USA). Analysis of variance (Anova) and Tukey pairwise comparisons were carried out using a 95% confidence level ($\alpha = 0.05$).

Results and discussion

Figure 1 shows digital images of calcium alginate hydrogels with and without soursop pulp.

Hydrogels without pulp showed mean diameters around 3.8 mm (Tab. 1). The samples containing 5 and 10% of soursop pulp showed similar diameter values than the control ones ($P > 0.05$), while the hydrogels containing 20% of fruit pulp showed a statistically significant increase in their diameter exhibiting values of around 5 mm ($P \leq 0.05$) (Tab. 1).

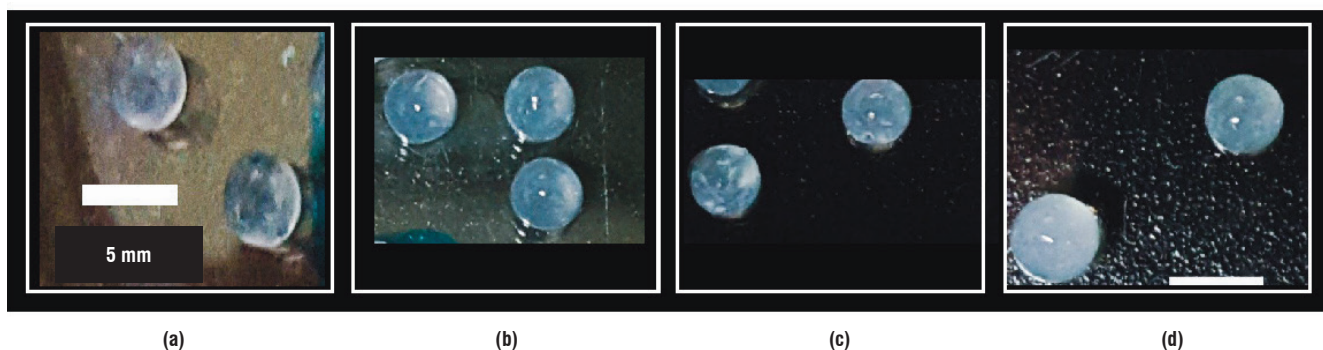


FIGURE 1. Digital images of the calcium alginate hydrogels: (a) control samples; (b), (c) and (d) hydrogels with 5, 10 and 20% of soursop pulp, respectively.

TABLE 1. Values of diameter and circularity of the alginate hydrogels with and without soursop pulp (\pm standard deviation).

Amount of pulp added (wt.%)	Diameter (mm)	Circularity
0	3.8 \pm 0.1 a	0.61 \pm 0.10 a
5	3.9 \pm 0.4 a	0.64 \pm 0.10 a
10	4.1 \pm 0.3 a	0.73 \pm 0.10 a
20	5.0 \pm 0.4 b	0.74 \pm 0.10 a

Different letters within the same columns indicate significant difference ($P \leq 0.05$).

On the other hand, all hydrogel systems showed similar circularity values ($P > 0.05$) (Tab. 1). According to Schneider *et al.* (2012) a circularity value of 1.0 indicates a perfect circle, while as the value approaches 0.0, it indicates an increasingly elongated polygon. In this sense, the high circularity of the samples could be attributed to that the operating conditions during ionic gelation were adequate and under control (López *et al.*, 2013).

Calcium alginate hydrogels commonly present high water content (c.a. 95%) and water activity (c.a. 0.98) (López *et al.*, 2013). Thus, the water content of hydrogels is often reduced by drying in order to avoid the spoilage of the encapsulated active compounds. After drying stage, all samples showed water content values between 8 and 10% (Tab. 2). With respect to the water activity, values around 0.3-0.4 were obtained (Tab. 2). It has been reported that in calcium alginate beads with a_w below 0.6, active compounds (*e.g.* natural antioxidants) could be conserved almost unaltered along storage (López *et al.*, 2013).

Figure 2 shows images of the encapsulating systems obtained by scanning electron microscopy.

It was observed that the morphology of the control samples (Fig. 2a) and the hydrogels with 5% (Fig. 2b) and 10% (Fig. 2c) of fruit pulp was irregular after drying process; while, in the systems containing 20% of fruit pulp the shape of the beads

TABLE 2. Values of water content and water activity of the alginate beads with and without soursop pulp after drying (\pm standard deviation).

Amount of pulp added (wt.%)	Water content (%)	Water activity (a_w)
0	9.7 \pm 0.7 ^a	0.35 \pm 0.07 ^a
5	8.7 \pm 0.1 ^a	0.35 \pm 0.07 ^a
10	10.0 \pm 0.8 ^a	0.36 \pm 0.06 ^a
20	9.9 \pm 1.6 ^a	0.36 \pm 0.06 ^a

Different letters within the same columns indicate significant difference ($P \leq 0.05$).

was better conserved (Fig. 2d). This behavior was attributed to that soursop fruit pulp was able to act as a structural support to control the shrinkage and to maintain the shape of the beads after drying.

Conclusions

Calcium alginate hydrogels containing different concentrations of soursop pulp were successfully developed by ionic gelation. The addition of soursop pulp did not show an effect on the circularity of the hydrogels. The applied drying process led to beads with water content and water activity characteristics of stable products.

Acknowledgements

The authors would like to thank the Instituto Universitario de La Paz (UNIPAZ) for their support and to the Argentinian National Research Council (CONICET).

Literature cited

- Ćujić, N., K. Trifković, B. Bugarski, S. Ibrić, D. Pljevljakušić, and K. Šavikin. 2016. Chokeberry (*Aronia melanocarpa* L.) extract loaded in alginate and alginate/inulin system. *Ind. Crops Prod.* 86, 120-131. Doi: 10.1016/j.indcrop.2016.03.045
- Deladino, L., A.S. Navarro, and M.N. Martino. 2013. Carrier systems for yerba mate extract (*Ilex paraguayensis*) to enrich instant soups. Release mechanisms under different pH conditions.

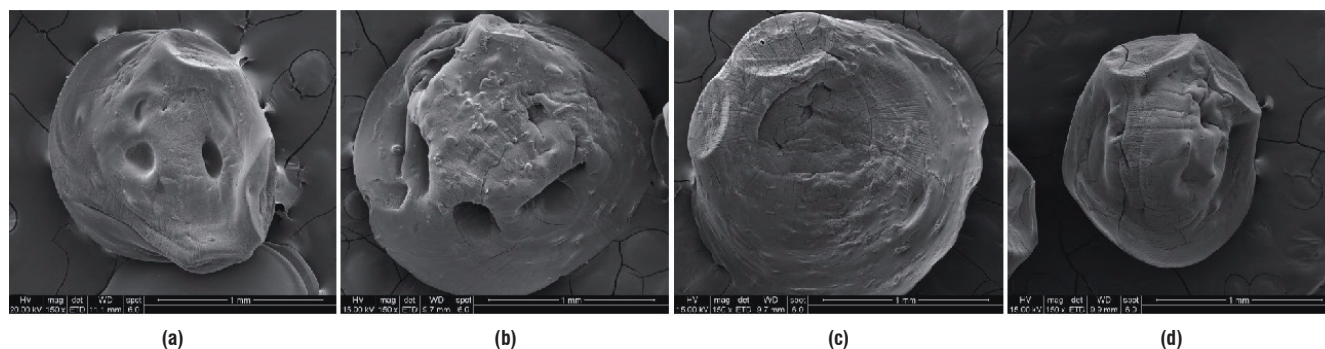


FIGURE 2. Images obtained by scanning electron microscopy of the calcium alginate hydrogels after drying process: (a) control samples; (b), (c) and (d) hydrogels with 5, 10 and 20 % of soursop fruit pulp, respectively.

- LWT - Food Sci. Technol. 53(1), 163-169. Doi: 10.1016/j.lwt.2013.01.030
- Jiménez, V.M., M. Gruschwitz, R.M. Schweiggert, R. Carle, and P. Esquivel. 2014. Identification of phenolic compounds in soursop (*Annona muricata*) pulp by high-performance liquid chromatography with diode array and electrospray ionization mass spectrometric detection. Food Res. Int. 65, 42-46. Doi: 10.1016/j.foodres.2014.05.051
- López, A., L. Deladino, and M. Martino. 2013. Effect of starch filler on calcium-alginate hydrogels loaded with yerba mate antioxidants. Carbohydr. Polym. 95(1), 315-323. Doi: 10.1016/j.carbpol.2013.03.019
- López, A., L. Deladino, and M. Martino. 2014. Release of yerba mate antioxidants from corn starch-alginate capsules as affected by structure. Carbohydr. Polym. 99(0), 150-157. Doi: 10.1016/j.carbpol.2013.08.026
- Schneider, C.A., W.S. Rasband, and K.W. Eliceiri. 2012. NIH Image to ImageJ: 25 years of image analysis. Nat. Meth. 9(7), 671-675. Doi: 10.1038/nmeth.2089
- Vicini, S., M. Castellano, M. Mauri, and E. Marsano. 2015. Gelling process for sodium alginate: New technical approach by using calcium rich micro-spheres. Carbohydr. Polym. 134, 767-774. Doi: 10.1016/j.carbpol.2015.08.064