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Effect of microwave treatment on metal-alginate beads

Laura Campañone [a,b,*], Estela Bruno [a,c,d], Miriam Martino [a]

[a] CIDCA (Centro de Investigación y Desarrollo en Criotecnología de Alimentos), CCT La Plata, CONICET, Facultad de Ciencias Exactas, UNLP, Calle 47 y 116, La Plata 1900, Argentina
[b] Departamento de Ingeniería Química, Facultad de Ingeniería, Universidad Nacional de La Plata (UNLP), Argentina
[c] Facultad de Ciencias Veterinarias, Universidad Nacional de La Plata, La Plata 1900, Argentina
[d] CIC (Comisión de Investigaciones Científicas de la Pcia. de Buenos Aires), La Plata 1900, Argentina

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ABSTRACT

Encapsulation is defined as a technique to protect solid, liquid or gaseous active compounds from interacting with the environment. Encapsulated minerals may be incorporated into food products submitted to microwave heating, thus it is interesting to study their interaction with microwaves due to the relevant dielectric properties of the ions during heating. In the present work, zinc sulphate heptahydrate, ferrous sulphate heptahydrate, calcium chloride and sodium alginate were used to test metal ion-alginate beads under microwave heating. The objective was to compare the behavior of free ions in solution with those trapped within the alginate matrix while being submitted to electromagnetic radiation. The beads were characterized before and after heating by texture profiles, differential scanning calorimetry and infrared spectroscopy (FTIR). During microwave heating, the presence of zinc and iron increased the inner temperature of the system; however calcium did not show the same behavior. These differences were attributed to the microstructure of the beads.

1. Introduction

Encapsulation is a process in which a thin polymer coating is formed around solid particles, liquid droplets or gases that are fully contained within the capsule walls. In some cases a minimum amount of the active material can remain exposed at the surface (Sahidi and Han, 1993; Deladino et al., 2008). In the food industry, encapsulation not only allows adding value to a food product, but also becomes a source of new additives with unique properties.

Alginic acid is a linear polysaccharide made up of α-L-guluronic acid and β-D-mannuronic acid. It is extracted from brown seaweeds and its sodium salt is a water-soluble polymer. Sodium alginate has been employed to make gel beads for the delivery of biomolecules such as drugs, peptides and proteins. Divalent cations bind preferentially to the guluronic blocks in the alginate in a highly cooperative way, thereby forming a gel (Wong et al., 2002).

Iron and zinc are two of the most important minerals for human nutrition. Iron plays an important role in human life, being essential in the oxygen transport and cellular respiration process. The human body takes this element from the diet by several ways according to its necessities. Besides, zinc is a trace element, indispensable for human health. It is essential for the body defense, plays an important role in the growing and division of the cells (Sarubin Fragaakis and Thomson, 2007).

Bearing in mind that these elements are needed for organic functions, their encapsulation would allow increasing their daily intake without modifying the original flavor of the preparations (Gouin, 2004; Tannenbaum and Young, 2005; Desai and Jin Park, 2005; Sun-Waterhouse and Wadhwa, 2013).

These capsulated metals can be incorporated in formulations like tea, soaps, mashed potatoes, etc. Generally, these products are supplied as in powder, and they are water reconstituted and microwave heated by the consumers. Although the use of microwave ovens is increasing both in industry and households, there is little known information on the behavior of metal ions within a gelling matrix submitted to microwaves.

In general, materials such as foods in which microwave energy is dissipated, are generally referred to as lossy materials. Water, the major constituent of foods, is the primary component responsible for dielectric heating. Because of their dipolar nature, water molecules try to follow the applied electric fields as they alternate at very high frequencies; such rotations increase temperature. Besides, ionic conduction could be attributed to salts, composed of positive and negative ions in dissociated form. The net electric field in the oven accelerates the particles and the temperature of them increases, interacting with their neighbors and transfer agitation or

* Corresponding author at: CIDCA (Centro de Investigación y Desarrollo en Criotecnología de Alimentos), CCT La Plata, CONICET, Facultad de Ciencias Exactas, UNLP, Calle 47 y 116, La Plata 1900, Argentina. Tel.: +54 2214890741.
E-mail address: lacampa@ing.unlp.edu.ar (L. Campañone).

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heat. This heat is then transferred to the other parts of the material (Datta and Anantheswaran, 2005; Datta et al., 2005).

The objectives of this work includes: (i) to investigate the change of the structural properties of beads caused by the thermal process through the FTIR, texture and thermal techniques (DSC); (ii) to compare the behavior of the free (solutions) and encapsulated ions in water solution through the measurement of the temperature increase during microwave heating.

2. Materials and methods

2.1. Preparation of beads

Beads were obtained with a peristaltic pump ((Minipuls 2 Gils, France) by dropping 2% sodium alginate solution (Aldrich, USA) through a syringe into the different gelling solutions. Tested solutions were (0.0055 g/mL) calcium chloride, 0.1 g/mL zinc sulfate heptahydrate (Biopack, Argentina) or an equivolumetric solution of 0.1 g/mL zinc sulfate heptahydrate and 0.1 g/mL ferrous sulfate heptahydrate (Anedra, Argentina). All the beads were maintained in the gelling bath to harden for 15 min. Beads containing, calcium (ACa), zinc (AZn), iron and zinc (AFeZn) were formed. A fraction of the last two types of beads were filtered and dipped in calcium chloride solution (0.0055 g/mL) for 30 min. Beads containing zinc and calcium (AZnCa) and three ions (AZnCaFe) were obtained. Then, beads were washed, filtered and allowed to stabilize in air for 15 min. They were kept at 4 °C for further analysis. Spherical beads were obtained with 4 mm diameter, measured using a Stereo Microscope Leica (Germany). As a summary, four types of capsules were prepared of Calcium Alginate (ACa), Zinc Alginate (AZn), Zinc–Calcium Alginate (AZnCa) and Zinc-Calcium-Iron Alginate (AZnCaFe).

2.2. Determination of bead mineral content

Content of sodium, zinc and iron encapsulated minerals was determined by flame atomic absorption spectrophotometry, according to specifications of the Standard Methods for the examination of water and wastewater APHA (1998) prior to acidic digestion of the samples according to the 25005 Method (AOAC).

2.3. Microwave heating

Water (control) and water containing encapsulated salts were heated in the present work. Also, salt solutions were heated to assess the behavior of the free ions. To compare the temperature increase between bounded and free ions, the same ratio of water/beads or water/solutions (W/W) was used. Two ratios, 15/35 (*R*1) and 25/25 W/W (*R*2), were used with a total volume of 50 mL.

A BGH microwave oven with nominal power of 1000 W and 2450 MHz frequency was used to heat the model systems. The heating time was 30 s. Inner temperatures were measured with an optical sensor (Fiso Technol. Inc., Canada) with an acquisition speed of 1 s.

2.4. Fourier Transform Infrared Spectrometry (FTIR)

The analysis was carried out with a Nicolet 380 FTIR spectrometer (USA). Disks were obtained by milling 5 mg of dried beads with 100 mg of KBr and were analyzed by transmission taking 64 scans per experiment with a resolution of 4 cm^{-1}. The analysis was performed on beads with and without microwave heating treatment in duplicate.

2.5. Differential Scanning Calorimetry (DSC)

Thermal analysis was performed on a DSC Q100 (TA Instruments, USA). The equipment was calibrated with the Indium standard. Samples of 15 mg were placed in aluminum capsules hermetically sealed and an empty aluminum capsule was used as the reference. Samples were heated from 20 °C to 290 °C at a scanning rate of 10 °C/min. The analysis was applied to dried beads with and without microwaves heating treatment. Assays were performed in triplicate.

2.6. Texture of beads

A TA.XT 2i texturometer (Stable Micro Systems, UK) was used to obtain the texture profiles (TPA). The analysis consisted of two consecutive cycles of capsule compression (Bourne, 2002). The capsules were placed in Petri dishes with a greater diameter than that of the probe (2.5 cm). The characteristic parameters of the different samples (hardness, cohesiveness and consistency) were obtained from the plot of force as a function of time provided by the equipment. The hardness is defined as the maximum force measured during the first compression, the cohesiveness was estimated as the relationship between the first and the second area obtained by the TPA and the consistency was obtained from the sum of the above mentioned areas.

The tests were conducted in triplicate and the values reported correspond to the average of 10 measurements.

2.7. Statistical analysis

The statistical study of the results was performed using the analysis of variance (ANOVA) with a significance level of 5% or *p*-value <0.05. Mean significant differences were determined using Duncan's multiple comparison test. The statistical analysis was carried out with Systat-software (SYSTAT, Inc., Evanston, IL, USA, 2001) version 10.0.

3. Results and discussion

Previous assays helped select a solution concentration of 0.1 g/mL for zinc sulphate and ferrous sulphate and 2% sodium alginate, based on the integrity and roundness of the beads. Calcium alginate concentration was selected following Anbinder et al. (2011). According to Gónzalez-Rodriguéz et al., 2002 ionic gelation depends on the nature and the ratio of the inorganic ion and the alginate. The authors found differences between the aspect and morphology of Ca^{2+} and Al^{3+} particles, namely that the Al^{3+} particles were disk-shaped with a collapsed center.

In the present work, metal content within the alginate matrix was determined in AZnCaFe beads. Mean values of Zn, Fe and Na (from the alginate salt) were 51.83 g/kg, 43.33 g/kg and 7.20 g/kg, respectively. These results showed that both ions could interact with alginate.

3.1. Microwave heating

In this work, the temperature increase values of salt solutions and encapsulated ions in water (in the same mass ratio: solution or encapsulated ions/water) were compared.

Fig. 1 a and b shows the temperature increase after 30 s of heating for salt solutions and capsules in water (*R*1 = 15/35), respectively. The different concentrations (*R*1 or *R*2) of solutions or capsules did not significantly affect the temperature increase ($p < 0.05$), suggesting that the lower ratio was enough to modify the inner temperature.