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Evaluation of Caffeine Release from Hydrogel Colloidosome under Simulated oral Condition and Instrumental Analysis of the Microcapsule

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Abstract

In this research hydrogel-based colloidosome with shell of $CaCO_3$ microparticles was used in order to encapsulate caffeine as a model flavor compound. When the $CaCO_3$ particles were dispersed in sunflower oil and then water in oil emulsion was prepared, with adding D-gluconic acid δ-lactone, water droplets containing alginate slowly gelate with outer layer of $CaCO_3$ particles in micrometer hydrogels in the size of a few 10 μm without coagulation. $CaCO_3$ microparticles act as both cross-linker for the alginate and stabilizer of water in oil emulsion. After leaving for 48 h, the hydrogel colloidosomes sank to the bottom of the container due to gravity. The results of infrared spectroscopy showed that indicative peaks of caffeine functional groups appeared in the loaded colloidosome sample spectrum and shifted its position. The results of calorimetric tests showed that the difference between the melting point of colloidosome samples with varying amounts of calcium carbonate microparticles in their formulas are linked. The results of the X-ray diffraction test showed a change in the crystalline degree of caffeine. After 5 h, the colloidosome sample, continued the caffeine release in water up to 55% maximally, while using mouth conditions for this sample, release of the loaded caffeine increased 33%. Generally, the results of instrumental analysis and release test showed that caffeine in the hydrogel colloidosome was well encapsulated within the alginate hydrogel network and have proper release under simulated oral condition.

Keywords: Caffeine, Colloidosome, Encapsulation, Hydrogel, Release

Introduction

Flavors are one of the most valuable components of food formulas, and preserving them in foods is considered one of the most important concerns of manufacturers. One of the most significant ways to maintain the desired taste effect is to cover the flavor compounds (Madene, Jacquot, Scher, & Desobry, 2006). Controlled release is a method in which one or more components are

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available at a specific time and place and the desired speed. Using this technology, it is possible to use some additives sensitive to temperature and pH, including flavor compounds in food systems (Pothakamury & Barbosa-Cánovas, 1995). Colloidosomes are microcapsules with a hydrogel or hollow polymer nucleus, forming colloidal particles of the colloid shell, varying from nanometers to microns. Colloidal particles adhere to the surface of the nucleus through forces such as electrostatic or intermolecular interactions (Rosenberg, 2010). Advantages of using colloidosomes include the mechanical stability of the colloidal shell, the control of the shell cavities size, and the ability to release in response to external fields or changes in environmental conditions (Rosenberg, 2010). Taste release is a branch of food science that deals with the study of mechanisms affecting the release of aromatic compounds or the release of taste from food under certain conditions, especially release in the mouth while eating (Roberts, Pollien, & Milo, 2000). Flavor compounds are often added in a microencapsulation form to prevent chemical drop or change during the process and storage of food. In the food industry, hydration is the most common method for controlled release (Zuidam & Nedovic, 2010). This study aimed to present a simple, fast, and non-destructive method for caffeine microencapsulation, investigate the release of caffeine from the colloidosome in conditions similar to the human mouth, and finally study the possibility of caffeine microencapsulation in the hydrogel colloidosome by microcapsule analysis.

Materials and methods

In this study, Wang *et al*. (2006)(2006) method was used to produce porous calcium carbonate microparticles (Wang *et al*., 2006). To optimize colloidosomes production and select the best ratio of constituents, and modeling caffeine release, the parameters of Table (1) were used in the preparation of water emulsion in oil.

Table 1. Various parameters for preparing the colloidosome samples

Sample no.	1	2
Volume fraction of water (δ)	0.2	0.3
Volume of aqueous alginate solution (V_w, mL)	1.3	2.1
Weight of $CaCO_3$ microparticles (W_P, g)	0.05	0.08
WP/Vw (g/mL)	0.04	0.04

At first, porous CaCO3 microparticles were dispersed in 5 mL sunflower oil through stirring for 1 h. The defined amount of aqueous solution of 1 wt% sodium alginate was added into oil and water-in-oil emulsion was formed by stirring. Then 150 μL of freshly prepared 0.2 g/mL GDL aqueous solution was added to gelate the alginate core. The emulsion was shaken for 2 h and then left for 48 h in the experimental condition. Due to gravity the colloidosome hydrogel beads sank to the bottom of the container. After collection by centrifugation for 5 min in 4000 rpm and three times of wash with ethanol, the obtained colloidosome hydrogel beads were redispersed in water through shaking. In this study, caffeine was used as a model; because, in addition to being available and widely used in the food industry, it dissolves in water. Samples without calcium carbonate or caffeine-loaded sodium alginate beads were also prepared as controls (Moffatt, 1986). The infrared spectra of caffeine samples and its components in the range of 400-4000 cm^{-1} were measured using the Perkin Elmer spectrophotometer (Perkin Elmer Spectrum 65 FT-IR Spectrometer, USA) (William, 1996). Differential scanning calorimetry test was used in the temperature range of 0 to 400 °C with a temperature change of 10 °C/min in the presence of nitrogen gas (Mettler DSC instrument, UK) to investigate the thermal properties. In

order to qualitatively analyze the colloidosomes samples, the X-ray diffraction test was used with an X-ray diffraction instrument (XRD, 38066 Riva, d/G. Via M. Misone, 11/D (TN) Italy) at an angle of 2θ between 5 and 90 degrees. Applying a degree of an incision around the degree of incision of the mouth for a drink, which is usually considered 55 s^{-1}, the amount of caffeine released was measured at 37 °C and neutral pH. To evaluate the caffeine release in simulated oral conditions, the release rate was measured at three incision rates of zero, 50, and 100 s^{-1} and within 300 minutes with at least two repetitions.

Results and discussion

The infrared spectroscopy results of pure caffeine used in this study, sample number 1 of colloidosomes, and the same sample of colloidosomes in which caffeine was loaded are shown in Figs. (1), (2) and (3), respectively.

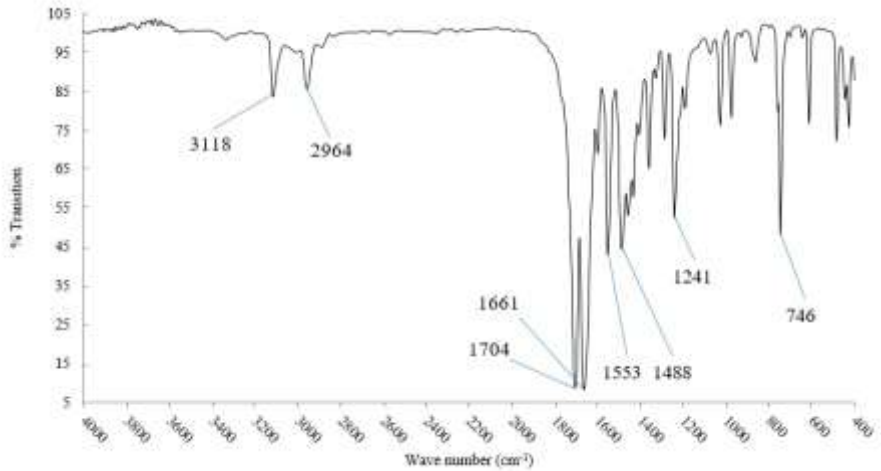

Fig. 1. The infrared spectroscopy of caffeine

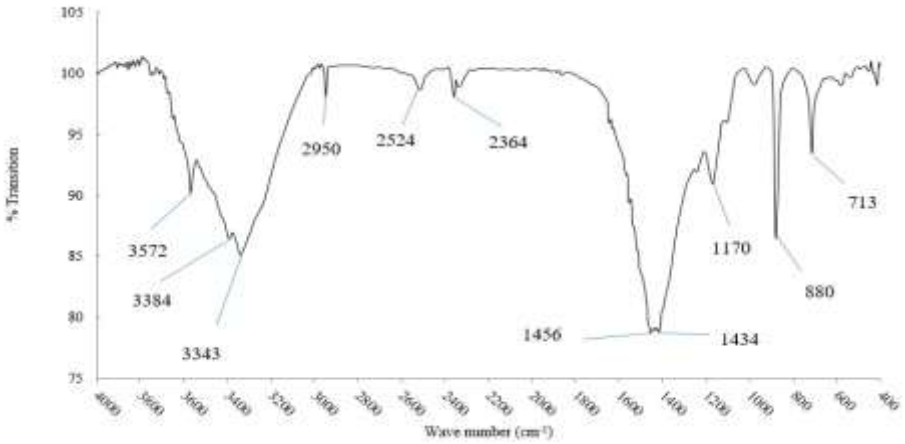

Fig. 2. Colloidosome Infrared Spectrum Sample 2

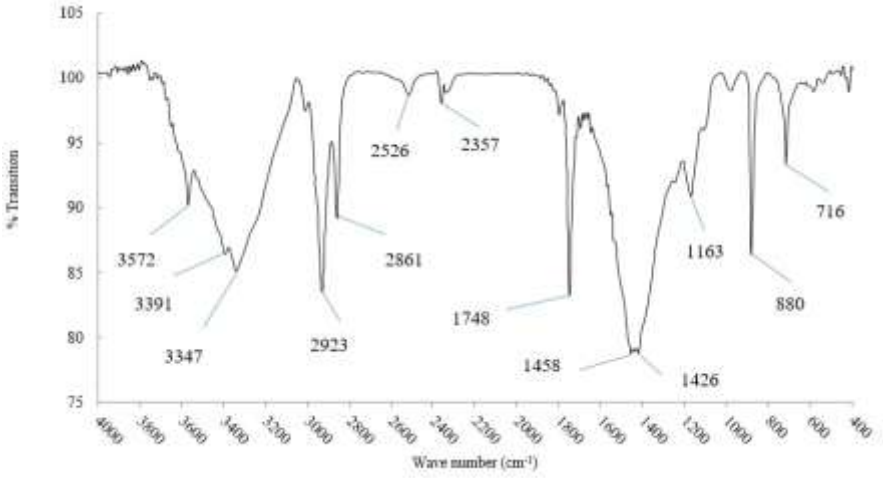

Fig 3. Colloidosome 2 infrared spectrum loaded with caffeine

In the infrared spectrum of caffeine, sharp peaks were seen that the peak related to hydrogen bonds of caffeine was in the wavenumber of 3118 cm^{-1}, the peak related to its aliphatic C–H bonds in the wavenumber 2964 cm^{-1}, and also the peaks related to C=O carbonyl in caffeine in 1661 and 1704 cm^{-1}, and C-N peaks in 1488 and 1553 cm^{-1}.

The intensity of the X-rays in sample number 1 at an angle of 29.4 degrees is maximum and equal to 56.05 percent, and in sample number 2, the maximum intensity at an angle of 29.3 degrees is equal to 95.45 percent. It should be noted that although caffeine has a crystalline structure, its crystalline nature is hidden within the hydrogel colloidosomes because the X-ray diffraction pattern of colloidal samples in which caffeine is loaded is amorphous (Fig. 4).

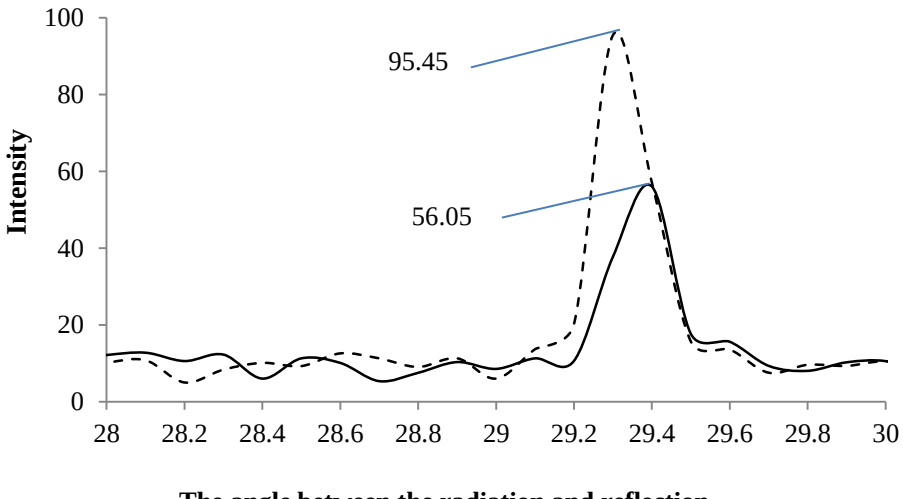

Fig. 4. X-ray diffraction diagram of colloid numbers 1 (fill line) and 2 (dashed line)

Conclusions

The infrared spectroscopy results showed that the significant peaks of caffeine functional groups appeared in the spectrum of the loaded colloidosomes sample, and its position was also shifted. It indicates a chemical interaction between caffeine and the colloidosomes sample and that the

caffeine in the colloidal hydrogel was microencapsulated. Also, while confirming the production of the desired microcapsules, the thermal test results showed that the difference in melting point between the colloidosomes samples was related to different amounts of calcium carbonate microparticles in their formula. The results of the X-ray diffraction test confirmed this fact, indicating a change in the crystalline degree of caffeine and its successful microencapsulation within the alginate hydrogel network.

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