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Inulin potential for encapsulation and controlled delivery of *Oregano* essential oil

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ABSTRACT

The ability of inulin, a prebiotic material, as encapsulation matrix was explored. Microcapsules of Raftiline were produced by spray drying inulin solutions at different solids content (5, 15 and 25%) at 120, 155 and 190 °C, according to a Central Composite Rotatable design. Produced capsules were analysed for morphology and size by SEM and physiochemical characterized by DSC, IR and RAMAN. Oregano essential oil was incorporated in the inulin solutions at 15% solids basis and the emulsions dried at the same conditions. The above mentioned methodologies were applied to evaluate the encapsulation ability and the changes induced by the presence of the EO in capsules morphology and structure. Furthermore the kinetics and amount of release was assessed by a spectrophotometric method. Results showed that it was possible to produce regular spherical inulin microcapsules (3–4.5 μm) for all the tested experimental conditions. According to IR and Raman results mainly drying temperature affected the structure of the capsules, three groups being clearly formed. These groups could be related to the morphology of inulin crystals. The EO was successfully encapsulated in the system as demonstrated by IR and Raman analysis. The differences found in the EO releasing amount, make clear that different degrees of core material retention is achieved, what should be related to structural changes in the matrix wall, denoting in some processing conditions interactions phenomena among inulin and EO. Those different releasing profiles patterns may be quite useful in finding different potential uses for the encapsulates.

1. Introduction

Inulin belongs to the group of fructans that naturally occurs as plant storage carbohydrate in many members of the Asteraceae family. It is a polymer of β(2→1)-linked D-fructose units, constituting chains of different lengths each of them with a terminal glucose unit. As summarized by Barclay, Ginic-Markovic, Cooper, and Petrovsky (2010), in this structure only one atom of the polymer backbone is attached to the fructose ring making inulin unusually flexible for a polysaccharide conferring its ability to assume a range of different structures.

Among the natural sources of inulin, industrial chicory (*Cichorium intybus* L., var. *sativum*) is one of the most important, due to its high content of inulin and the presence of a considerable fraction of inulin compounds with a high degree of polymerisation (Januário, 1999; Orafti, 1996; Van Loo, Coussement, De Leenheer, Hoebregs, & Smits, 1995). Inulin and fructooligosaccharides are actually important natural ingredients used in food industry by its very interesting and diversified technological and functional properties. These compounds behave as prebiotics what means they are non-digestible by the human organism only being degraded by certain colon bacteria (probiotics) like bifidobacteria (Gibson & Kolida, 2007; Roberfoid, 2007). This selective stimulation of those gut bacteria provides several benefits to the host health (Kaur & Gupta, 2002; Roberfroid, 2000). Additionally, to inulin are attributed important biological effects such as anticancer (Korbelik & Cooper, 2007) and immunomodulatory properties (Silva, Cooper & Petrovsy, 2004). Besides the uses in the food industry it has also important applications in pharmaceutical, as excipient, stabilizer and slow release drug delivery medium (Barclay et al., 2010).

Microencapsulation is a technology that allows sensitive ingredients to be physically entrapped in a homogeneous or

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heterogeneous matrix aiming at their protection. The encapsulation can be achieved by several processes such as spray-drying, spray-cooling, spray-chilling, freeze-drying, coacervation, etc.. (Gouin, 2004). However, mainly due to the low cost and available equipment spray–drying is the most commonly applied technology (De Vos, Faas, Spasojevic, & Sikkema, 2009; Gharsallaoui, Roudaut, Chambin, Voilley, & Saurel, 2007; Lopez-Rubio, Gavara, & Lagaron, 2006).

Spray-drying is a unit operation by which a liquid product is atomized in a hot gas current to instantaneously obtain a powder. The initial feed material can be a solution, an emulsion or a suspension and the final product obtained, depending also on operating conditions, can be a powder with particles ranging from 10 to 50 μm (Gharsallaoui et al., 2007).

Among others, the most common polymers used for food ingredients encapsulation include gelatine (Chiu et al., 2007; Shu, Yu, Zhao, & Liu, 2006), arabic gum (Brückner, Bade, & Kunz, 2007; Kanakdande, Bhosale, & Singhal, 2007), modified starches (Brückner et al., 2007; Kanakdande et al., 2007) and whey protein (Brückner et al., 2007).

Oregano is an aromatic plant quite widespread in Mediterranean countries, used as food ingredient due to the pleasant flavour. Besides, the antioxidant (Fasseas, Mountzouris, Tarantilis, Polissiou, & Zervas, 2008; Kulisic, Radonic, Katalinic, & Milos, 2004) and antimicrobial (Govaris, Solomakos, Pexara, & Chatzopoulou, 2009; Seydim & Sarikus, 2006) properties of *Oregano* essential oil (EO) makes this natural component also a good option as food preservative (Goulas & Kontominas, 2007) and health promoter. Carvacrol and thymol are the two main phenols that constitute oregano EO and primarily responsible for its antioxidant activity (Kulisic et al., 2004). However, EO composition can be changed as result of oxidation, chemical interactions or volatilization. To limit aroma degradation/loss during processing and storage and to control the delivery of the compound at the desired time and site encapsulation of volatile ingredients are beneficial prior to use in foods or beverages (De Vos et al., 2009; Madene, Jacquot, Scher, & Desobry, 2006).

Moreover and related to the prebiotic action it should also be noted the accrued interest of the potential use of inulin as wall material for encapsulation/protection of bioactive compounds susceptible to changes/degradation along the human digestive tract, since its release takes place only in the intestine, where they will be absorbed. Due to these functional properties, in addition to their technological properties and its low price (De Vos et al., 2009) drew the attention of inulin by the food industry. However till now in the food field, the use of inulin as wall material is almost unexploited. Saénz, Tapia, Chávez, & Robert (2009) report the ability of inulin for microencapsulation of bioactive compounds from cactus pear fruit.

To assess the ability of a microcapsule to act as an active agent carrier, diffusion of the selected compounds should be analysed. The rate of diffusion of a compound through the polymer wall of a microcapsule strongly depends on the preparation method of the microcapsules and on the permeability of the polymer membrane (Hansan et al., 2007).

The objective of the present work is to evaluate the potential of inulin as wall material for encapsulation and controlled release of *Oregano* essential oil. In order to understand both the characteristics of the wall capsules and the way the essential oil is thereat retained the use of several accessing methodologies are needed. In this scope morphological, physical and chemical properties of spray-dried materials accessed by scanning electron microscopy (SEM), differential scanning calorimetry (DSC), ATR-MID infrared spectroscopy and RAMAN were used as analytical tools.

2. Materials and methods

2.1. Materials

A commercial spray-dried inulin (Raftiline ST – >90% inulin; DP ≥ 10) obtained from chicory roots, was supplied by Beneo-Orafti (Tienen, Belgium).

Oregano (*Origanum vulgare* L.) was produced in Alentejo region in the south of Portugal. The essential oil was obtained by steam distillation of dry flowers and leaves and stored in the dark at refrigeration prior to the encapsulation process.

2.2. Production of inulin capsules with and without essential oil

Inulin solutions were prepared in hot water (80 °C) and dried in a LabPlant SD-04 (Leeds, UK) co-current spray dryer equipped with a 0.5 mm diameter nozzle. The pressure of compressed air for the flow of spray was adjusted to 1.9 bar. The inlet temperature varied between 120 and 190 °C according to the experimental design. A peristaltic pump feed the spray dryer at a rate of 5.8 mL min^{-1}. In encapsulation trials, the essential oil was added (15% inulin basis) to cooled inulin solutions and the mixture strongly homogenised with an Ultra-Turrax homogeniser (T25 Basic IKA LabortechNK, Germany) until a stable emulsion is obtained. Produced emulsions did not show phase separation or other visual changes during the feeding period prior to drying.

2.3. Experimental design

Feed suspensions, without and with essential oil incorporation, were prepared from different formulations according to a central composite face-centered design (CCFCD). The tested independent factors were inulin concentration and drying temperature (Table 1). Two central points were added in order to provide an estimate of the lack of fit of the second order statistical model obtained as well as the pure error of the experiments (Montgomery, 1996, p. 677).

2.4. Analysis of microcapsules powder

2.4.1. Morphological characterisation of microcapsules

The physical structure of dried powders was observed by SEM. Samples were set on metal pins with double-sided adhesive tape, then coated with a mixture of gold (95%) and palladium (5%) by the plasma deposition method in a coater Polaron E5350 and observed in a Jeol JSM-5410 scanning microscope. Microphotographs were obtained with an acceleration potential of 10 kV.

Particle size determination and particle size distribution were calculated from the measures of capsules diameters with SEM-AFORE software (v 5.0). Around one thousand measurements were performed for each sample.

Table 1
Experimental design. Coded and decoded values of independent variables.

Inulin (%)	Temperature (°C)	Inulin (%)	Temperature (°C)
−1	−1	5	120
−1	+1	5	190
+1	−1	25	120
+1	+1	25	190
−1	0	5	155
+1	0	25	155
0	−1	15	120
0	+1	15	190
0	0	15	155
0	0	15	155

2.4.2. Differential scanning calorimetry

Thermal characteristics were assessed by Differential Scanning Calorimetry (DSC) with a Shimadzu DSC − 50 calorimeter (model TA-50WSI, Shymadzu Corporation, Kioto Japan) calibrated using indium (m.p.: 156.6 °C, $\Delta H_f = 28.45\ Jg^{-1}$) and deionised water (m.p.: 0 °C, $\Delta H_m = 333\ Jg^{-1}$) as standards. The calibration procedure was completed with the scanning rate to be used in the trials. The samples (about 25 mg) were weighed directly in non-hermetic aluminium pans. An empty pan was used as reference. Nitrogen was used as carrier at a flow rate of 20 mL min^{-1}. The measurements were performed between 20 and 240 °C at a heating rate of 10 °C min^{-1}. Thermal transitions were evaluated in terms peak (Tp) transition temperature and enthalpy (ΔH). At least three replicates were tested.

2.4.3. Infrared spectroscopy

From Mid-IR spectra Inulin microcapsules with and without EO were acquired using a Spectrum BX (Perkin Elmer) (4000–600 cm^{-1}) FT-IR spectrometer; equipped with a 1 reflexion horizontal ATR (Golden Gate). All data were collected at a resolution of 8 cm^{-1} and 32 scans. 5 replicates from each sample were recorded.

2.4.4. Raman

Raman spectra (4000–600 cm^{-1}) from Inulin microcapsules with and without EO were measured with 300 scans, at a resolution of 4 cm^{-1}, using a Bruker RFS100/S FT-Raman spectrometer (Nd:YAG laser, 1064 excitation) at a power of 300 mV.

2.4.5. Release of EO from microcapsules

Oregano essential oil release profiles were obtained by a dialysis method (Nastruzzi, Esposito, Cortesi, Gambari, & Menegatti, 1994). 100 mg of microcapsules were added into a dialysis bag (molecular weight cut-off 3500; Cellu-Sep H1, Membrane filtration products, USA); the bag was subsequently placed into 100 ml of phosphate buffer solution (PBS − pH 5.8) with magnetic stirring. At regular time intervals, 1 ml samples were taken from the PBS and EO concentration was followed as a function of time by measuring the absorbance (Elisa Biotech Synergy HT) at appropriate wavelength (λ = 277 nm). True full replicates of the experiments were performed.

To quantify the EO, analytical curves of the compound in the release media were constructed. A known concentration of EO in PBS was scanned in the range of 200–600 nm by using UV visible spectrophotometer. For Oregano EO having concentration in the range 0.01–0.3 mg/mL, a sharp peak at 277 nm was noticed. The absorbance values at 277 nm obtained with the respective concentrations were recorded and plotted (Abs = 5.08 Conc (mg/mL) + 0.0925; $R^2 = 0.9962$). The absorbance measurements were carried out in a UV–VIS spectrophotometer (Jasco 560, Germany).

2.4.5.1. Kinetics release. In order to assess the transport properties of EO entrapped in inulin microparticles, mathematical models were used to describe the physical mechanism. Several authors have proposed mathematical relationships to describe compound release from microcapsules (Prata, Zanin, Ré, & Grosso, 2008; Romero-Cano & Vincent, 2002; Zhang, Grijpma, & Feijen, 2006), assuming some restrictions for polymeric matrices as described in a previous work (Beirão da Costa et al., 2012). Based on these works, a linear superimposition model was applied which describes the release of EO from spherical microparticles at time *t* by well-known equations:

$$\frac{M_t}{M_\infty} = 6\left(\frac{Dt}{\pi r^2}\right)^{0.5} - \frac{3Dt}{r^2} \quad \text{for} \quad \frac{M_t}{M_\infty} \leq 0.7 \tag{1}$$

and

$$\frac{M_t}{M_\infty} = 1 - 0.61\ \exp\left(-\frac{Dt\pi^2}{r^2}\right) \quad \text{for} \quad \frac{M_t}{M_\infty} \geq 0.7 \tag{2}$$

where M_t is the solute mass released at time *t*; M_∞ the solute mass released at infinite time when equilibrium is achieved; *r* is the radius and *D* can be calculated from the Eq. (1) or Eq. (2) by non-linear parameter estimation.

2.4.5.2. Statistics. All statistical analyses were carried out using the Statistica® 7 (Statsoft, Tulsa, OK, USA).

3. Results and discussion

3.1. Capsules morphology

Particle morphology can directly impact on other properties namely bulk density, rehydration characteristics and volatile loss (King, 1984) and so affect core protection and/or delivery. The effect of solids content and drying temperature on inulin capsules morphology is shown on Fig. 1.

According to Walton classification (2000), the obtained particles are clearly in the category of skin forming structures. In all tested conditions, as expected, smooth walled capsules were obtained, as inulin capsules were produced from inulin solutions. Our results are in accordance to those reported by Ronkart et al. (2007, 2009) referring to the effect of temperature preparation of inulin feed system, prior to spray-drying, on capsule wall surfaces. Inulin preparation at 40 °C results in a suspension leading to the production of rough surfaces capsules while at 95 °C a solution is obtained and smooth surfaces capsules produced.

Apparently, the solid content of the initial solution had a marginal effect on capsule wall characteristics. In contrast, as can be observed, as the drying temperature decreases some cavities are observed in wall surfaces. This effect is more pronounced at the lower drying temperature tested (120 °C). A similar feature was observed in maltodextrin particles also prepared by spray-drying (Alamilla-Beltrán, Chanona-Pérez, Jiménez-Aparicio, & Gutiérrez-López, 2005). Oakley (1997) justifies this phenomenon by the rapid evaporation and high pressure generated into the capsules, at higher temperatures, that allow particles to inflate. In opposition, when solutions are dried at low temperatures, water diffusion is slower and structures had more time to shrink and collapse.

Also, it is important to point out that in all tested formulations and for the different applied drying temperatures, the formed capsules showed no damaging, as it was not observed any blowholes or collapsed capsules, denoting that a resistant crust and consequently a resistant structure was created.

When EO is encapsulated in the referred inulin formulations, under the different drying conditions, the morphology of the capsules suffers modifications (Fig. 2). Despite continuous walls without blowholes or cracks is once more observed, the regular spherical shape of the formed structures is sometimes lost. This apparent pliability of the capsules wall seems to be more pronounced when drying is performed at lower temperatures (120 °C) and may be due to a plasticizing effect of the EO.

The experimental data of particles size, both with and without EO, were successfully described by second order models. The size of formed inulin capsules, without core material, is influenced by drying temperature and inulin content in feed solution ($p < 0.01$), both as linear and quadratic effects, being the interaction between the two independent variables marginally significant ($0.05 < p < 0.1$).

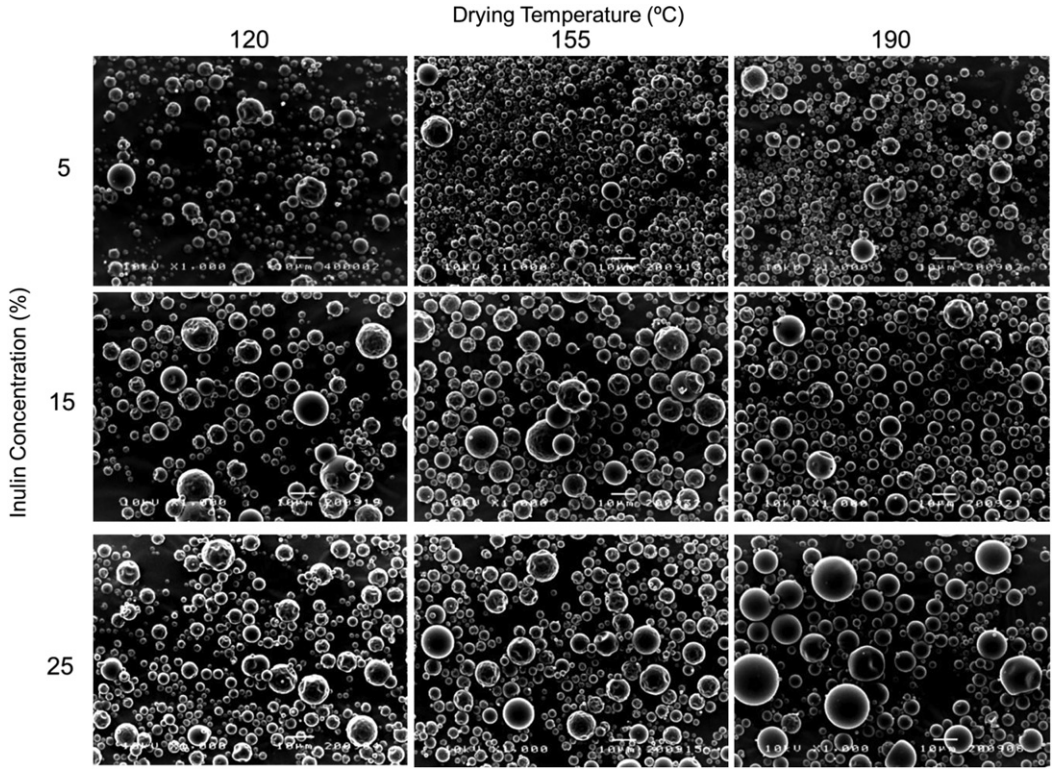

Fig. 1. Scanning electron microscopy microphotographs of Raftiline ST spherical capsules, formed by spray drying. ×1000 Magnifications.

In the same way, when EO is incorporated, both independent variables affected particles size although just quadratic term of temperature and linear term of inulin concentration are significant ($p < 0.05$).

The variation of average particles sizes of the formed structures are described in Fig. 3

The particle size distribution of inulin microcapsules was very narrow and the formed capsules were very small. Average size of

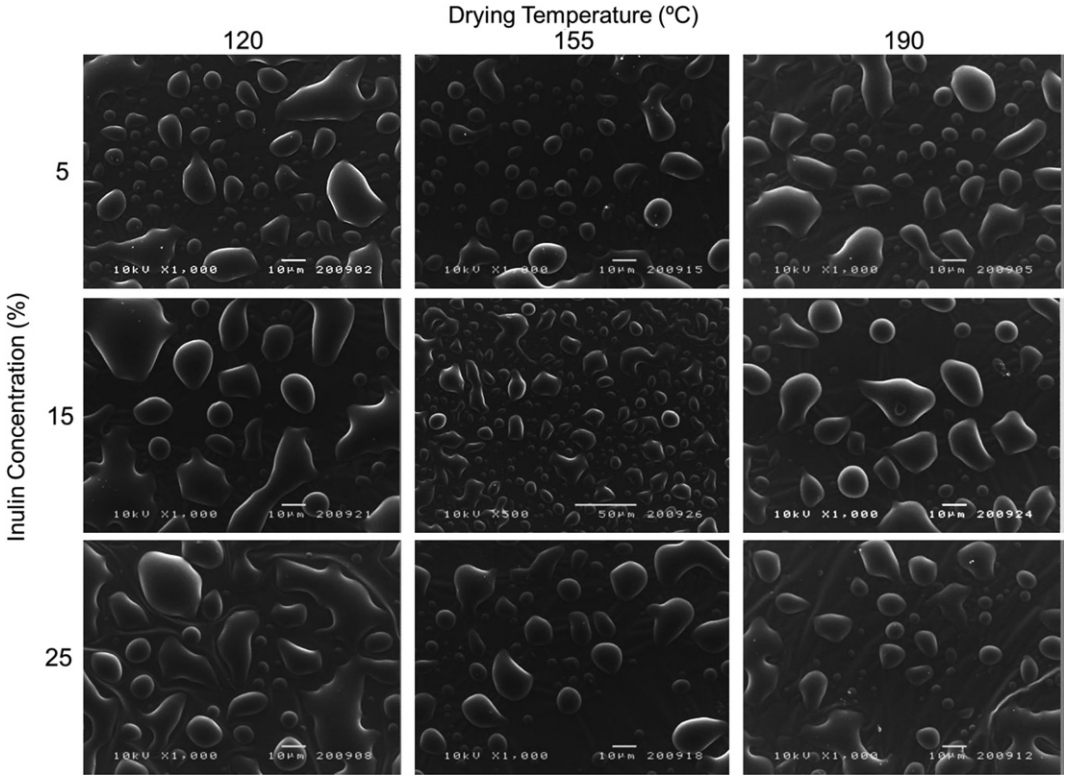

Fig. 2. Scanning electron microscopy microphotographs of Raftiline ST spherical capsules with Oregano essential oil, formed by spray drying. ×1000 Magnifications.

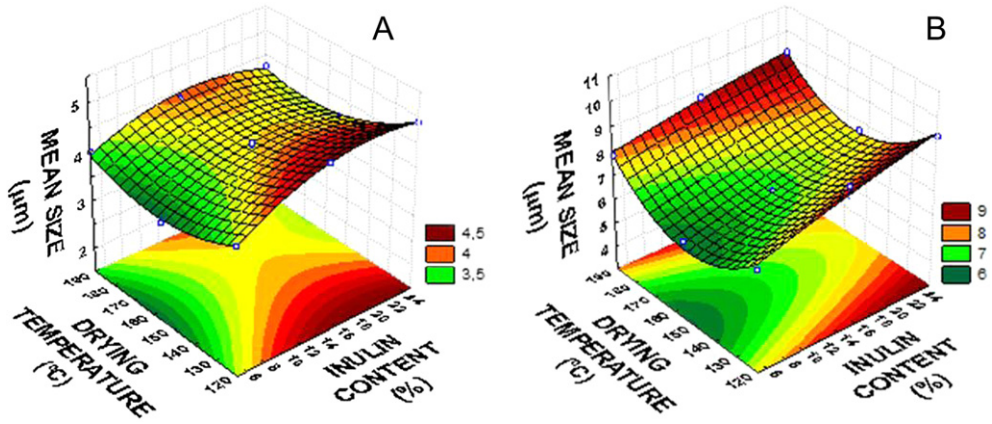

Fig. 3. Effect of inulin concentration and drying temperature on microcapsules size. **A** – Inulin capsules (R^2 = 0.97; R^2_{adj} = 0.93); **B** – Inulin capsules with EO (R^2 = 0.96; R^2_{adj} = 0.91).

formed capsules is inversely proportional to drying temperature being this effect softened for higher concentrations of inulin. When inulin concentration in the feed solution is below 10%, drying temperature showed a marginal effect in capsules diameters and the size of formed structures was quite small, under 3.5 μm (Fig. 3A). Larger capsules were formed from inulin solutions with an inulin content higher than 10% and when drying was performed below 150 °C. Nevertheless it seems to us that this variation is not important as the observed variation range from 3 to 4.5 μm of diameter.

The presence of EO in the spray drier feed emulsion leads to an increase in particles average diameter (6.3–9.2 μm) (Fig. 3B), the size of formed structures increasing with the inulin concentration in the medium. This tendency is observed independently of the drying temperature. Nevertheless, minimum values of particles size were found when drying was performed at intermediate temperatures (about 130–160 °C).

The effect of drying temperature on particles sizes was already reported by Walton (2000) pointed out an increase in particles diameter with increased drying temperature. Alamilla-Beltrán et al. (2005) also report the tendency to obtain particles with lower mean size when a 40% maltodextrin solution was spray dried at lower temperatures. In the present work, it was observed that the solids content of the feed solution was more important on capsules mean diameter than the drying temperature. Our results are in accordance to those reported by the above authors in the range of 130–190 °C however for lower drying conditions the effect of solids content overlapped that one.

The differences observed both on capsules morphology and capsules sizes are due to the different physical and chemical characteristics of the feed material. As known, an increase in solid content induces an increase in particle size, due to a rising in system viscosity and as a result, a higher residence time in the atomizer which render difficult droplet formation (Rosenberg, Kopelmen, & Talmon, 1990; Walton, 2000). A wall/core emulsion taking the place of a wall solution may reinforce this effect.

Most certainly, the formed crust properties are different depending on the drying parameters and solution solids content, namely in what concerns to pliability and porosity.

3.2. Physico-Chemical characterization of capsules

Thermal properties of materials, as glass and melting transition temperatures, are important tools to a better knowledge about structural features.

DSC analysis was performed both on Raftiline commercial powder and on capsules produced under the different conditions, with and without EO incorporation. Commercial Raftiline thermogram showed a glass transition (Tg) at 134 °C and two endotherms at 156 °C and 174 °C. This profile was also reported by Ronkart et al. (2007, 2009) for a similar inulin (Fibruline XL, Cosucra) the pattern being justified by the melting of two types of crystals, differing probably on shape, size and/or molecular weight. Those authors also refer that generally multiple endotherm behaviour is only attributed for semi-crystalline synthetic polymers and that the observed endotherms were an irreversible phenomena, corresponding not only to the melting of the crystals but also to the release of the water of crystallization (Ronkart et al., 2007).

Thermal analysis results of inulin microcapsules both without and with essential oil showed that the glass transition temperatures and thermal denaturation are found at the same range of temperatures of the dried inulin. Melting transitions temperatures are not significantly different in capsules with and without essential oil. However, probably due to some breaking effect during solubilisation and spray drying of inulin during capsules formation, some additional melting transition were identified at about 198 and 204 °C when compared to dried inulin thermogram. This feature suggests a reorganization of the material during capsules production by spray drying process.

Multivariate analysis of infrared spectra from inulin microcapsules (25% total solids) showed three distinct groups corresponding to each individual drying temperature, being samples dried at 155 °C between the groups of 120 °C and 190 °C (data not shown). Both techniques results induce to assume that some differences occur on the matrix structure as function of processing temperature, although those changes were not noticed on transition temperatures. This phenomenon may be explained by reorganization of inulin in the crystalline form when heated above Tg (Ronkart et al., 2007, 2009). So, capsules produced at 190 °C present differences in the organization state of wall probably due to a higher crystallinity degree of the polymer. When drying is performed at 120 °C none of the transition temperatures are attained while at 190 °C all of them were exceeded. At 155 °C, drying temperature is nearby inulin first endothermic transition peak.

The multivariate analysis of Raman spectra also shows a clear separation between samples with and without EO. The discrimination is done on the basis of the peaks 737 and 756 cm^{-1} (Fig. 4). Fig. 4B presents the Raman spectra from the essential oil, which is dominated by these two peaks. Regrettably Raman does not show if the OE is inside or on the surface of the capsules. It allows only

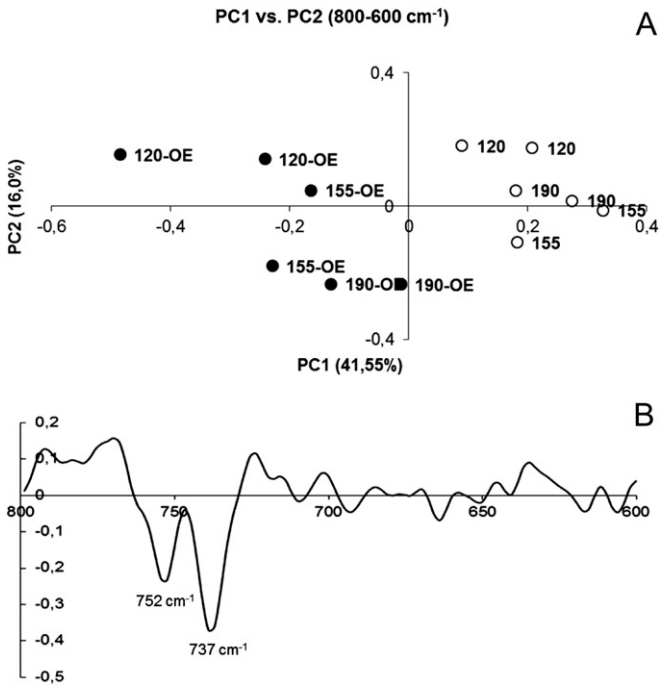

Fig. 4. PCA of Raman spectra from inulin capsules dried at 120 °C, 150 °C and 190 °C. A) Scores plot from samples with (●) and without (○) Oregano EO. B) PC1 loadings.

verifying the presence of the OE in the system. However, it is certain that there is a successful retention of the core material, for all drying conditions. Besides, in previous works (Beirão da Costa et al., 2012), by confocal laser scanning microscopy, it was observed that the oil particles are located inside the capsules instead of on the surface.

Changes are observed in Raman spectra at 754 cm^{-1} and 738 cm^{-1}. Looking to the spectra of encapsulates produced at 120 °C (Fig. 5B) the essential oil above mentioned distinctive peaks are quite visible. Instead, when encapsulation was performed at 190 °C (Fig. 5C) the spectra presents some changes in the region of 754–730 cm^{-1} indicating that interactions/modifications on the matrix – core system have occurred.

3.3. Release measurements

The contribution of volatile compounds to the aroma perceived in a food product will depend on their rate of release from the entrapment system (Baranauskienė, Venskutonis, Dewettick, & Verché, 2006). Controlled release of EO through different types of inulin microcapsules is shown in Fig. 6.

From Fig. 6 it can be seen that EO release from inulin microparticles is characterized by two different phases. Initially (0–75 min) a rapid release of EO is observed ("burst effect") followed by a period during which the release profile becomes constant (after 200 min) indicating a sustained release ("lag time").

The burst effect has been observed by other researchers in different polymeric matrices and can be due to volume expansion of polymer when immersed in liquid media. In aqueous liquid media hydrophilic polymers start to hydrate causing relaxation of the polymer chain. In the present case, it is hypothesized that this effect is the main responsible for the initial release of EO from inulin matrix; we believe that it is the EO that is not physically or chemically linked to matrix that will be released at this stage of the process.

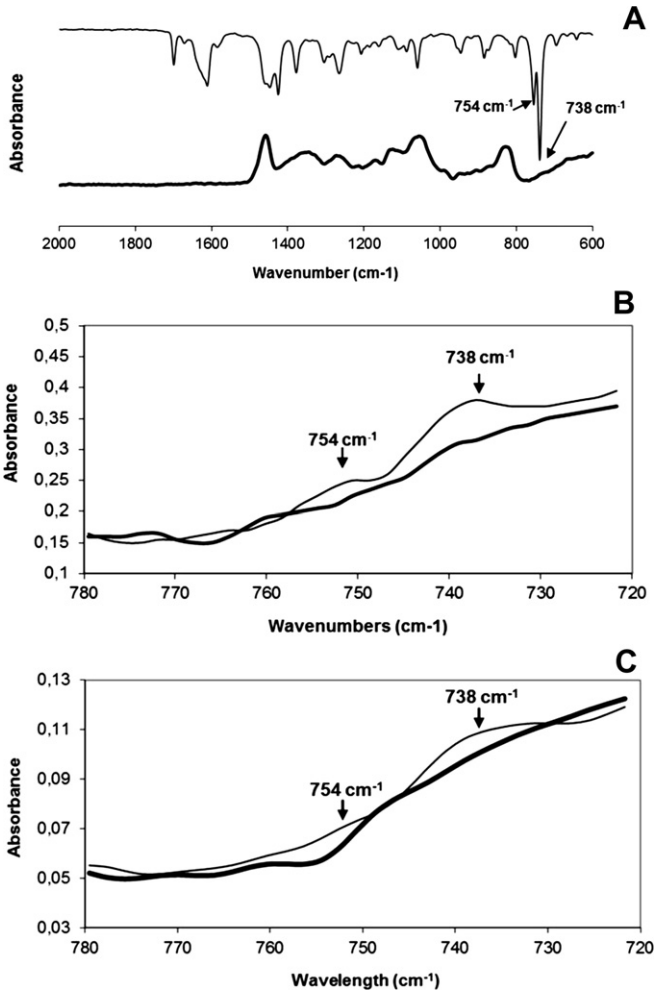

Fig. 5. Raman spectra from: **A** – Inulin capsules dried at 120 °C (—) and Oregano EO (—); **B** – Superimposed spectra from inulin capsules dried at 120 °C with (—) and without (—) EO; **C** – Superimposed spectra from inulin capsules dried at 190 °C with (—) and without (—) EO.

In contrast, the subsequent almost zero order EO release phase results from the overlapping of several effects, such as the increase in the diffusion pathways with time, which is at least partially compensated by the increase in device porosity (polymer degradation), and the maintenance of approximately linear EO concentration gradients over prolonged periods of time within the microparticles (Faisant, Siepmann, Richard, & Benoit, 2003).

In order to assess the transport properties of the EO inside the polymeric matrix, it is important to use a mathematical model that describes the physical mechanism without an unnecessary mathematical complexity. This can be done on the basis of experimental data observation and fitting procedures (Pinheiro, Bourbon, Vicente, & Quintas, 2013). A very good agreement between the model-generated and experimental values was found for all tested conditions, suggesting that this model is able to describe the experimental data and, hence, the physical mechanism of the transport phenomena involved here.

The total amount of EO released from both extreme drying conditions (120 °C and 190 °C) is dissimilar. Higher temperature leads to an increase in total diffused EO when compared to formulations dried at lower temperature being this difference more pronounced at 15% of solids. At this inulin concentration temperature has a more pronounced effect on core deliverance. As no significant differences were found in capsules size from samples of

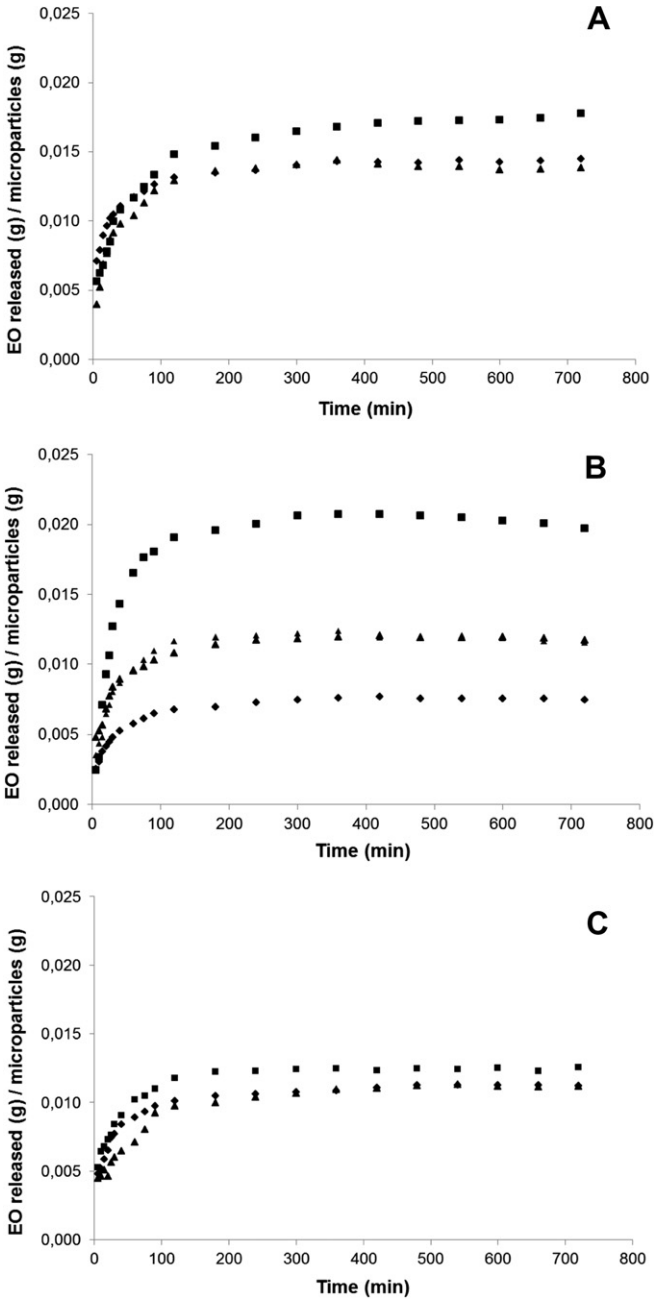

Fig. 6. Oregano EO release profiles at 25 °C from inulin microcapsules with different inulin concentrations and different drying temperature on microcapsules preparation: **A)** Inulin 5% **B)** Inulin 15% **C)** Inulin 25%. 120 °C (▲); 190 °C (■); 155 °C (◆).

the different drying conditions the dissimilarities in released EO are possibly due to differences in the wall structure or in the way the oil is retained rather than differences in total surface area. Besides it is also possible that the amount of retained oil in the matrix is not the same depending on drying temperature, for each solids concentration. Some studies refer the influence of drying temperature on encapsulation yield (Jafari, Assadpoor, He, & Bhandari, 2008) however, from previous results (Beirão da Costa et al., 2012) it is not expected that temperature has a significant influence on encapsulation efficiency as the antioxidant activity of capsules was shown not to be influenced by drying temperature.

In a previous study the values of all EO diffusion coefficients were estimated using Eq. (1) (Beirão da Costa et al., 2012). It was observed that the drying temperature applied to prepare inulin microparticles has significant effect on EO diffusivity from this system. It was also observed that increasing the drying temperature the diffusion coefficient decreases (from 7.72×10^{-16} to 2.96×10^{-16} m^2/s, from 6.46×10^{-16} to 4.13×10^{-16} m^2/s and from 8.76×10^{-16} to 8.21×10^{-16} m^2/s, for 5%, 15% and 25% of inulin content systems, respectively) and the effect of soluble solids on diffusion coefficient was more evident (Beirão da Costa et al., 2012).

Having these aspects in mind and according to the potential end use of encapsulates different processing conditions may be adopted.

4. Conclusions

Microcapsules of inulin ranging in size from 3 to 4.5 μm were developed by spray drying at different solids concentration and drying temperatures. It was always possible to produce smooth, regular and uninjured capsules. IR and Raman analysis showed that drying conditions clearly affected the capsules properties probably being related to the phase transitions of inulin. Encapsulation of oregano essential oil in those matrices conducted also to differentiated releasing profiles that are explained by the dissimilar properties of the wall. Those different patterns may be very useful in finding the most suited formulation and processing condition for a specific end use of encapsulates.

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