6th WORKSHOP

Green Chemistry and Nanotechnologies in Polymer Chemistry

July 15-17, 2015

Polytechnic Institute of Bragança | PCT-TMAD Brigantia EcoPark

Bragança · Portugal

WORKSHOP PROCEEDINGS

Eds. - M. F. Barreiro, O. Ferreira, A.I. Pereira

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O16. MICROENCAPSULATION OF PHENOLIC EXTRACTS IN CALCIUM ALGINATE BEADS FOR NUTRACEUTICAL APPLICATIONS

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Introduction

Bioactive ingredients are generally prone to degradation, both during storage and food processing, as many of them are physically, chemically and/or enzymatically instable leading to their degradation or transformation with the consequent loss of bioactivity. To overcome these limitations microencapsulation emerges as a reliable response to protect and stabilize bioactives (or extracts containing them), also offering the possibility of a controlled or targeted delivery [1].

The encapsulation materials, productive process, microcapsule's morphology and ultimate application conditions are the most important factors to be taken into account when designing a novel microencapsulated product, together with stability and functional properties issues. Moreover, to obtain a successful product, the achievement of high encapsulation yields, process and release profile reproducibility and overcome microcapsule's aggregation, should be guaranteed.

For food applications the used materials must be considered "generally recognized as safe" (GRAS) and be, preferably, biodegradable. Both EU through the EFSA and the US through FDA have many strict rules about material usage for food applications. In this context, the most commonly used materials are natural or natural-derived polymers. Among them, carbohydrate polymers (e.g. starch and cellulose and their derivatives), plant exudates and extracts (e.g. gum, galactomannans, pectins and soybean polysaccharides), marine extracts (e.g. carrageenan and alginate), microbial and animal derived polysaccharides (e.g. xanthan, gellan, dextran and chitosan) and proteins were tested for these purposes. In what concerns microencapsulation processes, a set of techniques are available (e.g., coacervation, extrusion, emulsion based process, liposomes etc.). Nevertheless, spray-based processes are the most commonly used by academia and industry. They present the advantages of being flexible and economically competitive, allowing a continuous production [2].

In this work the use of microencapsulation to protect natural extracts will be demonstrated with a case study comprising the development of a nutraceutical formulation based on gelatine incorporating alginate microspheres enriched with bioactive phenolic extracts obtained from wild *Fragaria vesca* L.

Experimental

For the core material, a bioactive extract from the vegetative parts of wild *Fragaria vesca* L. obtained by infusion and characterized in terms of its qualitative and quantitative phenolic composition, was used. Microspheres were prepared by using an atomization/coagulation technique and alginate as the wall material. Optical microscopy (OM) and Scanning Electron Microscopy (SEM) analysis was used monitor the process and access morphology. FTIR was used to check the effective incorporation of the extract in the alginate matrix and HPLC-DAD to determine the encapsulation efficiency (EE). Furthermore, the developed nutraceutical formulation using *k*-carrageenan gelatine was evaluated for their antioxidant properties maintenance through DPPH scavenging activity and reducing power.

Results and discussion

The obtained extract was characterized as constituted by nineteen phenolic compounds, belonging to the groups of ellagic acid derivatives, flavonols and flavan-3-ols. Quercetin *O*-glucuronide, followed by (+)-catechin, were the major compounds found.

Immediately after the atomization process, OM analysis showed light brown coloured spherical microspheres, proving efficient extract incorporation with homogeneous distribution within the microspheres. EE determination, done by HPLC-DAD, and based on the analysis of quercetin *O*-

glucuronide present both in the coagulation and washing solutions, gave a value around 97%. The FTIR analysis confirmed the presence of the infusion extract inside the microspheres.
SEM analysis, performed with the freeze-dried microspheres form, showed a spherical shape and a rough surface, being also observed round cavities due the proximal presence of other particles during the drying process. Fig. 1 shows microcapsules at different preparation stages.
Concerning the gelatine preparation it was observed that microspheres integrity was not affected by the used temperature (100 ºC). In fact incorporated microspheres acquired a perfect round shape possible due a prompt rehydration. After freeze-drying treatment the shape was also maintained. Black dots could also be observed inside the microspheres corroborating the presence of the extract. Neither the control nor the gelatin with microencapsulated extracts showed antioxidant activity in DPPH and reducing power assays, demonstrating the efficiency of the encapsulation in the protection of the bioactive extract.

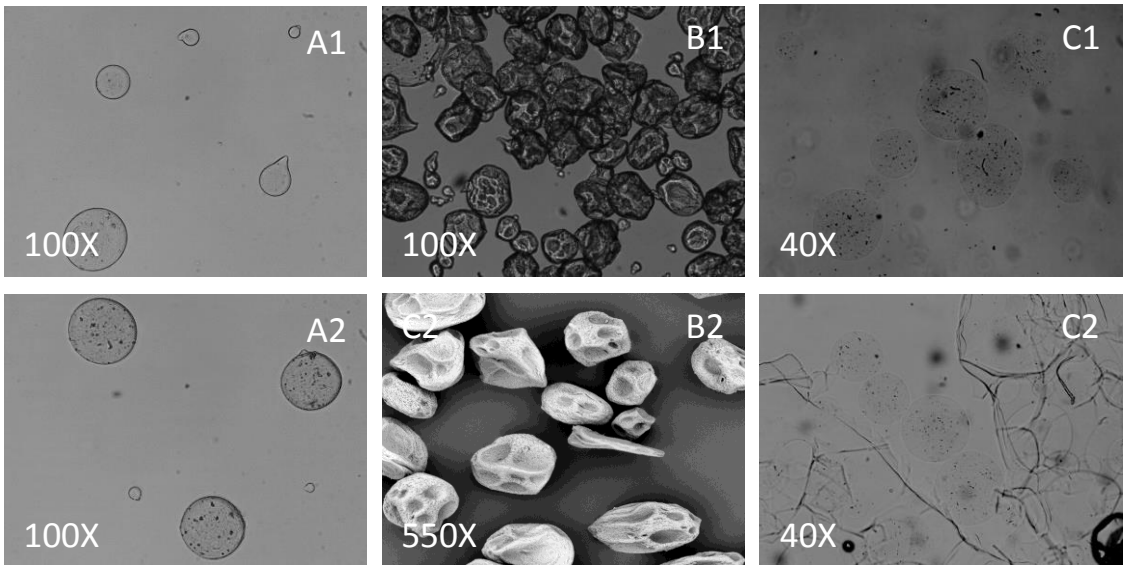

Fig.1. OM analysis with magnifications of 100X of the microspheres immediately after atomization (A1) and after 4 hours coagulation period under stirring at 400 rpm (A2), OM analysis of lyophilized microspheres (B1), and SEM analysis with magnification of 550X (B2). OM analysis with magnification of 40X of k-carrageenan with microencapsulated infusion extract before (C1) and after (C2) lyophilisation.

Conclusions

The microencapsulation technique of atomization/coagulation was effectively applied to produce microspheres enriched with the antioxidant extract of the infusion of wild *F. vesca* (a EE of 97% was reached). This is an innovative study on the development of nutraceuticals based on *F. vesca* extracts. Further studies will be performed to assess the controlled release of the bioactive extract, using an *in vitro* gastrointestinal model.

Acknowledgements

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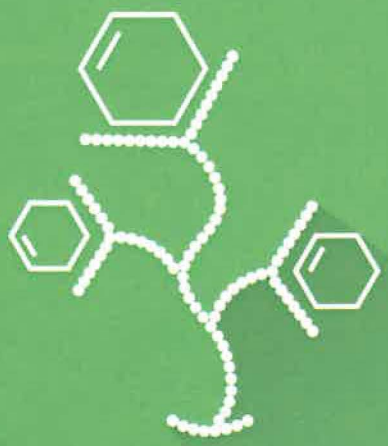

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CERTIFICATE OF ATTENDANCE

This is to certify that

Isabel Ferreira

has attended the 6th Workshop on Green Chemistry and Nanotechnologies in Polymer Chemistry held from July 15 to 17, 2015, in Bragança, Portugal.

Filomena Barreiro
(Chair of the Organizing Committee)