



# Essential oil/alginate microcapsules; obtaining and applying

Mariia Kokina<sup>1</sup>, Mark Shamtsyan<sup>2</sup>, Cecilia Georgescu<sup>3</sup>, Monica Mironescu<sup>3</sup>, Viktor Nedovic<sup>1</sup>

<sup>1</sup>Department of Food Technology and Biochemistry, Faculty of Agriculture, University of Belgrade, 11080 Nemanjina 6, Belgrade, Serbia

<sup>2</sup>Department of Technology of Microbiological Synthesis, Saint Petersburg State Institute of Technology (Technical University), Moskovsky Prospect, Saint Petersburg, Russia

<sup>3</sup>Faculty of Agriculture Science, Food Industry and Environmental Protection, Lucian Blaga University of Sibiu, Bulevardul Victoriei, Sibiu, Romania

## \*Correspondence to

Mariia Kokina, Email: mariia.maak@gmail.com

Received 11 August 2018

Accepted 19 December 2018

Published online 10 January 2019

**Keywords:** Alginate, Essential oils, Encapsulation, Steam distillation, Volatile compounds

## Abstract

**Introduction:** Nowadays, plant extracts are highly applied in food industries either as sources of bioactive components or as an alternative to artificial additives. Therefore, food manufacturers are focused on innovative products, which can satisfy consumers' requirements.

**Objectives:** This study investigates the encapsulation of *Origanum majorana*, *Achillea millefolium*, *Foeniculum vulgare*, *Juniperus communis* and *Anethum graveolens* EOs in alginate capsules as a means of controlling the fast release of volatile constituents.

**Materials and Methods:** The EOs were obtained via steam distillation. Sodium alginate was chosen as a carrier because of its biodegradable and biocompatible properties. The paper describes the simple dripping technique used for the preparation of the alginate microcapsules with EO cores, and a possible application of the microcapsules as a natural flavor additive.

**Results:** Sensorial properties of the final product were subjectively analyzed and described. The changes of the taste and the flavour of candies in comparison with the control sample were significant. Nevertheless, the strong herbal odour was found as "uncommon in confectionary but pleasant."

**Conclusion:** It has been investigated, that the sodium alginate encapsulated EOs have to be added as a final step of a recipe to save its antimicrobial and antioxidant potential. Further assays need to be performed to investigate the recipe, which includes the EO alginate microcapsules in order to get a high-quality final product that can be used for commercial purposes.

## Citation:

Kokina M, Shamtsyan M, Georgescu C, Mironescu Mm, Nedovic V. Essential oil/alginate microcapsules; obtaining and applying. Immunopathol Persa. 2019;5(1):e04. DOI:10.15171/ipp.2019.04.

## Introduction

Essential oils (EOs) of plants are known to possess antimicrobial and antioxidant activity, which has been evaluated by many researchers (1,2). It is due to a secondary metabolism that produces a lot of specific compounds which are needed for the plant to survive in the ecosystem. Another critical feature of a secondary metabolism is an attraction for pollinators and protection against predators and infections. For example, high light or UV leads to the production of anthocyanins and flavones, but pathogen attack causes the production of chemicals such as pterocarpan and coumarin (3). Those substances in plant's extracts and EOs have a biological activity on human health as well. In contrast to their unique characteristics, EOs are very unstable and highly volatile at standard temperature and pressure. Therefore, usage of EOs involves

## Key point

It has been investigated, that the sodium alginate encapsulated EO has to be added as a final step of a recipe to save its anti-microbial and antioxidant potential. The reason is that the activity of cyclic hydrocarbons, which are the main compounds of essential oils, is limited by their instability and can be destroyed by a high temperature

their preparation in liquid forms: emulsions, micelles, liquid solutions; semi-liquid forms: gels, liposome; solid forms: capsules, films or spheres. Those techniques help to control the release of volatile components and to preserve them for different areas of application (4). Microcapsules are microparticles with the size in the scale of nanometers or millimetres that consist of core materials and coating layers (also named walls). By selecting the construction substances (core



element and coating layer), one can provide microcapsules with diverse characteristics. Microencapsulation is one of the most common methods applicable for EOs protection. By microencapsulation method solids, liquids or gases can be included in the small particles formed of layers of wall material around the desired core (5). The description of how alginates can be utilised as a “carrier” for unstable substances in the pharmaceutical industry and food production and numerous factors that might affect its release from alginate matrix have been well reviewed (6). Sodium alginate (SA) is the sodium salt form of alginic acid mainly extracted from the cell walls of brown algae, with chelating activity. When taken orally, sodium alginate binds to and blocks the intestinal absorption of various radioactive isotopes, such as radium Ra 226 (Ra-226) and strontium Sr 90 (Sr-90) (7). Besides, it is biodegradable and biocompatible natural material which absorbs water 200–300 times of its own weight (5). In addition, alginates have been shown as activators of human macrophages that may initiate a healing process. The previous study (8) suggests that sodium alginate causes natural immune responses through the activation of the protein complex that controls transcription of DNA, cytokine production and cell survival and possibly activates it the same pathways as pathogen identification. This work thus is based on the relevant interest to design a combination of SA with an antimicrobial and antioxidant food agent used as a core material. Therefore, the study focuses on the obtaining and applying the capsules from the emulsion of alginate solution and EO. A possible application of capsules in a food product, namely jelly, is analysed.

## Objectives

The study focuses on the obtaining and applying the capsules from the emulsion of alginate solution and EO.

## Materials and Methods

### Essential oils isolation

Aerial parts of plants *Achillea millefolium* (Yarrow), *Foeniculum vulgare* (Fennel), *Juniperus communis* (Juniper), *Origanum majorana* (Marjoram), and *Anethum graveolens* (Dill) were collected in different parts of Romania. The EOs were isolated at the chemical laboratory at the Faculty of Agriculture, Science, Food Industry, and Environmental Protection at the “Lucian Blaga” University of Sibiu, Romania. Steam distillation method with slight changes was used to obtain the EOs from air-dried plant materials (9). Different air-dried parts of the plant were placed in a Clevenger apparatus and the extraction was carried out for about four hours. The EOs were dried by using the anhydrous, sodium salt form of sulfuric acid and stored in dark glass bottles at room temperature until the experiment was done.

### Capsule preparation

The most common method to obtain sodium alginate microcapsules is by applying the dripping technique (5).

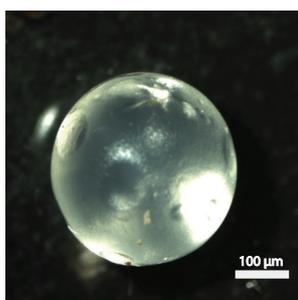
In this case, the alginate solution is extruded through a capillary at a moderate volumetric rate and left to drip by itself. Although this methodology has been proved for a long time, the formation of the microcapsules with desired shape and size characteristics often requires some expanded knowledge and equipment. In the present study, the experimental set-up was as follows: the syringe needles of outer diameter from 0.40 to 1.65 mm were used as dripping tips. The length of the needles was no more than 3 mm. The flow rate of alginate solution was adjusted to give an interval of 5 seconds between each drop so that the kinetic force generated during drop collection can be disregarded. The commercially available sodium alginate was chosen for this experiment. As it is commonly used for encapsulation purposes, the medium molecular weight sodium alginate was applied. The experiment was carried at room temperature. The EO was added to the Na-alginate solution, and the mixture was stirred manually for 5 minutes. For beads formation, the solution of calcium chloride (4%) was used as the alginate spherification bath. During the experiment, the distance between the dripping tip and the gelation bath was ranged from 5 to 10 cm. It is necessary, to maintain the microcapsules in the calcium chloride solution for approximately 30 minutes. Such an approach helps them to become more stable and firm. Afterwards, the beads were filtered from the solution, washed in distilled water and stored in the refrigerator until the microscopy evaluation. The size of particles formed through encapsulation may be scaled as a macro (>5000 µm); micro (1.0–5000 µm); and nano (<1.0 µm) (10).

### Microcapsules as a food additive

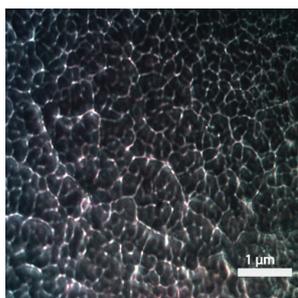
The encapsulated EOs in the food industry may provide the inhibition of pathogens or saprophytes. Moreover, according to their antioxidant activity, they could be a very efficient natural food preservative and, also, an excellent flavouring compound. The above-described properties may be used in the functional food production. Despite all these advantages, the capsules can affect the consumer characteristics and the organoleptic sensorial properties of the final product. Therefore, interactions between alginate EO capsules and other food ingredients and food additives need to be examined. In this study, microencapsulated EOs have been added to a jelly candy basic recipe at a concentration of 5% w/w, which represents the portion of approximately 30 g. Sensorial properties (the taste, flavour and appearance) of the final product were analysed by a panel formed by 12 persons.

## Results

Observations of shape and size were performed by using a standard microscope technique. The microscopy revealed that the beads have a spherical shape with a diameter of approximately 250–300 µm (Figure 1). At a higher magnification, the disordered structure of the surface becomes apparent (Figure 2) which can be due to the self-



**Figure 1.** Shape and size of the sodium alginate EO microcapsule



**Figure 2.** Structure of the upper layer of the sodium alginate EO microcapsule

agglomeration property of molecules of sodium alginate in the calcium chloride solution.

## Discussion

The chemical profile of the core material may also have an impact on the final membrane structure of the resulting microcapsules. The resulting diameter is in reasonable agreement with the described ratio (10) for micro sized capsules of 1.0–5000  $\mu\text{m}$ . The size of the microcapsules is an important characteristic that influences the sensory properties of foods. As it is reported (11), new extrusion technologies enable the production of more uniformly shaped microcapsules with a smaller dimension than those reached through the manual technique. After the examination of the sensorial properties of the jelly, the following summary can be drawn: The changes in the taste and the flavour of candies in comparison with the control sample were significant. Nevertheless, the strong herbal odour was found as “uncommon in confectionary but pleasant”. Noteworthy is the fact that microcapsules showed physical stability over four weeks with neither visible deformations of the shape nor significant variation of the size. However, the jelly mass had a slight viscosity increase, which in turn conducted to sticking mass to the forming surface. Further assays need to be performed to investigate the recipe, which includes the EO alginate microcapsules, in order to get a high-quality final product that can be used for commercial purposes.

## Conclusion

Sodium alginate EO microcapsules were obtained, and a microscopic characterisation was made. The study also

includes the preliminary results on the inclusion of EOs in a jelly candy recipe. The EO microcapsules described in this research represent a promising food additive for incorporation into functional foods, due to both bioactive and flavouring properties. The encapsulation into sodium alginate-based delivery systems of EOs thus was investigated as a method to improve the safety and the sensorial properties of foods through the addition of natural flavourings and preservatives. It has been investigated, that the sodium alginate encapsulated EO has to be added as a final step of a recipe to save its antimicrobial and antioxidant potential. The reason is that the activity of cyclic hydrocarbons, which are the main compounds of EOs, is limited by their instability and can be destroyed by a high temperature.

## Authors' contribution

CG, MM and MS; designed the study, developed the methodology. MK performed the analysis, collected the data and wrote the manuscript. All authors read and signed the final paper.

## Conflicts of interest

The authors declared no competing interests.

## Ethical considerations

Ethical issues (including plagiarism, data fabrication, double publication) have been completely observed by the authors.

## Funding/Support

None.

## References

- Georgescu C, Mironescu M. Obtaining, characterisation and screening of the antifungal activity of the volatile oil extracted from *Thymus serpyllum*. *Journal of Environmental Protection and Ecology*. 2011;12(4 A):2294-302.
- Orchard A, van Vuuren SF, Viljoen A, Kamatou G. The in vitro antimicrobial evaluation of commercially essential oils and their combinations against acne. *Int J Cosmet Sci*. 2018. doi:10.1111/ics.12456.
- da Silva JAT. Mining the essential oils of the Anthemideae. *African Journal of biotechnology*. 2004;3(12):706-20.
- Rezende Y, Nogueira JP, Narain N. Microencapsulation of extracts of bioactive compounds obtained from acerola (*Malpighia emarginata* DC) pulp and residue by spray and freeze drying: Chemical, morphological and chemometric characterization. *Food Chem*. 2018;254:281-91. doi: 10.1016/j.foodchem.2018.02.026.
- Wang L, Yang S, Cao J, Zhao S, Wang W. Microencapsulation of Ginger Volatile Oil Based on Gelatin/Sodium Alginate Polyelectrolyte Complex. *Chem Pharm Bull (Tokyo)*. 2016;64(1):21-6. doi: 10.1248/cpb.c15-00571.
- Paulo F, Santos L. Design of experiments for microencapsulation applications: A review. *Mater Sci Eng C Mater Biol Appl*. 2017;77:1327-40. doi: 10.1016/j.msec.2017.03.219.
- Kim MS, Jun SW, Lee S, Lee TW, Park JS, Hwang SJ. The influence of Surelease and sodium alginate on the in-vitro release of tamsulosin hydrochloride in pellet dosage form. *J Pharm Pharmacol*. 2005;57(6):735-42. doi: 10.1211/0022357056316.
- Yang D, Jones KS. Effect of alginate on innate immune activation of macrophages. *J Biomed Mater Res A*. 2009;90(2):411-8. doi: 10.1002/jbm.a.32096.
- Clevenger J. Apparatus for the determination of volatile oil. *Journal of Pharmaceutical Sciences*. 1928;17(4):345-9.
- Jafari SM, Assadpoor E, He Y, Bhandari B. Encapsulation efficiency of food flavours and oils during spray drying. *Drying Technology*. 2008;26(7):816-35.
- Manojlovic V, Rajic N, Djonlagic J, Obradovic B, Nedovic V, Bugarski B. Application of Electrostatic Extrusion - Flavour Encapsulation and Controlled Release. *Sensors (Basel)*. 2008;8(3):1488-96. doi:10.3390/s8031488.