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Carotenoids microencapsulation by spray drying method and supercritical micronization

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ABSTRACT

Carotenoids are used as natural food colourants in the food industry. As unstable natural pigments they need protection. This protection can involve the microencapsulation process. There are numerous techniques that can be used for carotenoid protection, but two of them –spray drying and supercritical micronization – are currently the most commonly used. The objective of this paper is to describe these two techniques for carotenoid microencapsulation.

In this review information from articles from the last five years was taken into consideration. Pigments described in the review are all carotenoids. Short summary of carotenoids sources was presented. For the spray drying technique, a review of carrier material and process conditions was made. Moreover, a short description of some of the most suitable processes involving supercritical fluids for carotenoids (astaxanthin, β-carotene, lutein and lycopene) encapsulation was given. These include the Supercritical Antisolvent process (SAS), Particles from Gas-Saturated Solutions (PGSS), Supercritical Fluid Extraction From an Emulsion (SFEE) and Solution Enhanced Dispersion by Supercritical fluids (SEDS).

In most cases the studies, independently of the described method, were conducted on the laboratory scale. In some a scale-up was also tested. In the review a critical assessment of the used methods was made.

1. Introduction

The colour of food is seen as an indicator of the quality and acceptability of the product. Most consumers assess and buy foods by the "eyes"; hence the colour of food is increasingly seen as an important quality and acceptability indicator of the product. In recent years increased consumer interest in foods containing natural ingredients can be observed. This forces manufacturers to abandon using synthetic colourants, but allows them more creativity in designing food. Therefore the study of characteristics of the carotenoids and their interactions in food products is essential (Galaffu, Bortlik, & Michel, 2015; Solymosi, Latruffe, Morant-Manceau, & Schoefs, 2015).

The most popular natural pigments used in the food industry are carotenoids, anthocyanins, betalains and chlorophylls. Carotenoids are yellow-orange-red colourants which can be found in fruits and vegetables, some fungi and algae. Carotenoids have many health functions. As antioxidants they may reduce the risk of certain chronic diseases, such as cardiovascular diseases and age-related macular disease, and prevent damage to the antioxidant. They also help strengthen the immune system from attacking the infection, and support the proper functioning of the reproductive system (Vila, Chaud, & Balcao, 2015; Galaffu et al., 2015).

The basic problem of using natural active substances including carotenoids as colourants in industrial application is their low resistance to changes in pH, heating and light exposure. The presence of oxygen and oxidizing agents can also lead to their decay. Method that can be used to protect natural origin compounds is microencapsulation. Application of this method allows one to obtain a product with a lower volume, which reduces transport and storage costs, also facilitating its dosage. More importantly, the encapsulated material is protected from external factors, which results in the stabilization of unstable substances. Nowadays, the most commonly used microencapsulation techniques are spray drying and methods with supercritical CO_2. The spray drying method allows one to obtain a product with the desired physical properties by applying variable drying parameters such as temperature, feed flux and by changing the type of carrier material or pre-treatment of the raw material (Chranioti, Nikoloudaki, & Tzia, 2015; Domian, Brynda-Kopytowska, & Oleksza, 2015; Janiszewska, 2014).

In this review the basic information about carotenoids and its sources are presented. The main topic of the presented review is the

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protection of selected carotenoids by microencapsulation using the spray drying process and supercritical micronization techniques.

1.1. Carotenoids

Carotenoids are the most commonly used and known natural pigments used in the food industry. All carotenoids are isoprene colourants, which are divided into carotenes, which contain only carbon and hydrogen, and xanthophylls, which are made up of carbon, hydrogen and oxygen (Fernández-García et al., 2012; Vila et al., 2015; Rutz, Borges, Zambiazi, da Rosa, & da Silva, 2016).

Carotenoids are lipid-soluble, yellow-orange-red colourants which can be found in fruits and vegetables, some fungi and algae. Their molecules are built from isoprene units. Most of them have 40 carbon atoms connected with a system of double bonds. This combination of double bonds determines the anti-oxidant activity, light absorbing and its biological properties (Vila et al., 2015; Galaffu et al., 2015; Solymosi et al., 2015).

The carotenoids most generally known and used in the food industry are: β-carotene, lycopene, capsanthin, lutein and bixin/norbixin (annatto) (Fernández-García et al., 2012; Galaffu et al., 2015; Solymosi et al., 2015; Vila et al., 2015).

In general all carotenoid pigments can be found in different types of sources (vegetables, fruits, algae), and have to be supplemented by humans and animals, because they do not have the ability to synthesize them themselves (Saini, Nile, & Park, 2015).

The very good development of fruit and vegetable as sources of carotenoids and their chemical structure and changes can be found in articles by Saini et al. (2015) and Fernández-García et al. (2012) for selected groups of vegetables and fruits, and Provesi and Amante (2015) for pumpkin, with a summary of carotenoid concentrations in the majority of pumpkin varieties.

A comprehensive summary on algae as a source of carotenoids with a description of quantity and quality is presented in articles written by Christaki, Bonos, Giannenas, and Florou-Paneri (2013), Jung, Ok, Park, Kim, and Kwon (2016) and Paliwal et al. (2016). The authors not only present the quality of carotenoids extracted from a given source, but also give a very detailed quantitative statement.

A detailed list of selected sources in which carotenoids can be found and extracted from is summarised in Table 1.

Unfortunately carotenoids have a low resistance to changes in pH, heating and light. Moreover, the presence of oxygen and oxidizing substances can lead to their decomposition. However, they are stable during heating, sterilization and freezing processes, and they are soluble in oil but not in water (Galaffu et al., 2015; Solymosi et al., 2015).

Method that can be used for protection of natural carotenoids is microencapsulation.

Table 1
Selected sources of carotenoids (summary).

Source	Carotenoids	References
Macroalgae		
Phaeophyta (brown algae) e.g., *Laminaria* spp.	Fucoxanthin, β-carotene, violaxanthin	Christaki et al., 2013
Chlorophyta (green algae) e.g., *Ulva fenestrare*	α-Carotene, neoxanthin, violaxanthin, siphonoxanthin	Christaki et al., 2013
Rhodophyta (red algae) e.g., *Porphyra* spp.	β-Carotene, lutein, zeaxanthin	Christaki et al., 2013
Chlorella spp.	Astaxanthinlutein, β-cryptoxantin, canthaxanthin	Jung et al., 2016 Paliwal et al., 2016
Microalgae		
Dunaliella salina	β-Carotene, α-carotene, zeaxanthin	Christaki et al., 2013
Hematococcus pluvialis	Astaxanthin, canthaxanthin, lutein	Christaki et al., 2013
Spirulina spp.	β-Carotene, zeaxanthin	Christaki et al., 2013
Fruits and vegetables		
Acerola	Lutein, β-carotene, α-carotene, β-cryptoxanthin	Saini et al. (2015)
Apple	Lutein, zeaxanthin, β-carotene	Saini et al. (2015)
Apricot	Lutein, zeaxanthin, β-cryptoxanthin, β-cryptoxanthin	Saini et al. (2015)
Banana	Lutein, β-carotene, α-carotene	Saini et al. (2015), Fernández-García et al. (2012)
Broccoli	Lutein, β-cryptoxanthin, β-carotene	Saini et al. (2015), Fernández-García et al. (2012)
Brussels	Lutein, β-cryptoxanthin, β-carotene	Saini et al. (2015), Fernández-García et al. (2012)
Carrot	Lutein, β-carotene, α-carotene	Saini et al. (2015), Fernández-García et al. (2012)
Cauliflower	Lutein, β-cryptoxanthin, β-carotene	Saini et al. (2015)
Corn	Lutein, zeaxanthin, β-cryptoxanthin	Sain et al. (2015), Fernández-García et al. (2012)
Mango	Lutein, zeaxanthin	Saini et al. (2015), Fernández-García et al. (2012)
Papaja	Lutein, zeaxanthin	Saini et al. (2015)
Peach	Zeaxanthin, β-cryptoxanthin	Saini et al. (2015)
Pumpkin	β-Carotene, α-carotene	Provesi and Amante (2015); Saini et al. (2015), Fernández-García et al. (2012)
Tomato	β-Carotene, α-carotene, lutein, lycopene	Saini et al. (2015), Fernández-García et al. (2012)
Lettuce	Lutein, β-carotene	Saini et al. (2015), Fernández-García et al. (2012)

*For more specific references see articles: Saini et al. (2015). Carotenoids from fruits and vegetables: chemistry, analysis, occurrence, bioavailability and biological activities. Food Research International, 76, 735–750.
Fernández-García et al. (2012). Carotenoids bioavailability from foods: from plant pigments to efficient biological activities. Food Research International, 46(2), 438–450.

1.2. Microencapsulation

Microencapsulation is a process which involves covering a liquid, solid or gaseous substance in surrounding material (carrier) (Chranioti et al., 2015; Domian et al., 2015).

After the process microcapsules can be obtained in different structure, usually obtained single capsule in which it is possible to see the outer layer and inside the active ingredient and microspheres, in which the active ingredient distributed throughout the carrier matrix can be seen (Vila et al., 2015).

There are numerous microencapsulation methods that can be used, but before choosing the best one several factors must be taken into consideration: different chemical and physical properties of the encapsulated substance and core material, non-reactivity with core material, its stability for a long time and future usage. Also important are the size of the capsule and its final form (Kandansamy & Somasundaram, 2012; Özkan & Ersus, 2014).

One of the oldest and also currently the most often used for carotenoid microencapsulation techniques is spray drying. This method allows one to obtain a product with the desired physical properties by applying variable drying parameters such as temperature and feed flux and by