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Effect of wall materials in spray-dried microencapsulation of fucoxanthin

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Abstract

Fucoxanthin as a seaweed xanthophyll is abundant in edible brown algae. Fucoxanthin showed numerous excellent bioactivities and pharmacological benefits. Its durability and bioavailability were very important during processing and storage when it is used as a colorant and bioactive ingredient in food applications. However, fucoxanthin is very susceptible to external factors in environmental conditions such as heavy metals, high temperature, light and oxygen. In order to improve its bio-accessibility, the same proportion of 12% of wall materials such as maltodextrin, trehalose, Arabic gum, modified starch, inulin; inlet/outlet drying temperature 150°C/80°C and feed flow rates 10 mL/min were investigated on spray drying of fucoxanthin. The spray-dried powder was then checked for microencapsulation efficiency, particle size and bulk density. Storage stability was also observed at 4 week-interval for 24 weeks of storage at ambient conditions**.** Results showed that Arabic gum was the best selection among wall materials. By spray drying with Arabic gum, the spray-dried fucoxanthin powder showed the highest microencapsulation efficiency (94.27±1.17%), particle size (6.79±0.05 µm) and bulk density (0.43±0.02 g/mL). The best fucoxanthin stability (90.46±0.15%) was also noticed in Arabic gum during 24 weeks of storage. Meanwhile, inulin was not suitable as a carrier for microencapsulation of fucoxanthin. The findings of this research suggested that proper carrier was very essential for the microencapsulation validity of thermal-sensitive constituents.

1. Introduction

Fucoxanthin is a marine carotenoid mostly derived from brown algae, microalgae, seaweeds, and diatoms. Its structure included an allenic bond and a 5,6-monoepoxide. It revealed therapeutic advantages in accelerating biotransformation, deterring ghrelin, boosting Leptin and regulating the digestive and immune response. Fucoxanthin contributes an excellent anti-obesity ability (Shoketsu and Hiroshi, 2017). Its anti-obesity property was based on the suppression of fat absorption and synthesis, cholesterol and fatty acid decomposition (Jeon *et al.*, 2010; Matsumoto *et al.*, 2010; Peng *et al.*, 2011). Fucoxanthin improved the enzymatic kinetic of carnitine palmitoyltransferase for fat-biotransformation (Ha and Kim, 2013). Fucoxanthin displayed anti-obesity capacity in several clinical tests via its property to uncoupling protein in the white grease cell-induced lipid combustion (Hossain *et al.*, 2019). Therefore, fucoxanthin was very effective in weight gain prevention. Remarkable weight loss and decrease in belly perimeter were clearly observed after taking fucoxanthin during the 16-week clinical test by 151 persons (Abidov *et al.*, 2010). Fucoxanthin was also helpful in colorectal cancer prevention via food intake (Terasaki *et al.*, 2021). Different literature also reported on its functional properties such as photoprotection (Chen *et al.*, 2019), anti-diabetes (Mikami *et al.*, 2017), antioxidation (Sachindra *et al.*, 2007), anti-angiogenesis (Sugawara *et al.*, 2006), anti-inflammation (Shirator *et al.*, 2005), anti-cardiovascular (Grasa-Lopez *et al.*, 2016). Fucoxanthin showed significant improvement in antioxidant attributes such as catalase, superoxide dismutase, and glutathione (Gumus *et al.*, 2018). Fucoxanthin was proven to reduce reactive oxygen species, like hydrogen peroxide, superoxide, and hydroxyl radical (Orazio *et al.,* 2012; Riccion, 2012). Fucoxanthin could be utilized as an alternative for the ameliorative therapy of reproductive vulnerability (Wang *et al.*, 2020).

The exploitation of fucoxanthin as a functional food and nutrient supplement was restricted owing to its

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hygrophobicity, sensitivity, and low bioavailability (Hashimoto *et al.*, 2012; Chunyan *et al.*, 2020). Fucoxanthin was extremely sensitive to acidity due to the electrophilicity of its polyunsaturated bonds when it was exposed to gastric acid in the stomach. Fucoxanthin was mostly absorbed in the small intestine (Xiaowen *et al.*, 2018). Unbeneficial factors like oxidation, photo-degradation, thermal sensitivity, and heavy metal in the processing and preservation significantly affected fucoxanthin properties (Pinto *et al.*, 2003; Hii *et al.*, 2010; Zhao *et al.*, 2014). In order to enhance its stability and bioavailability, spray-dried microencapsulation using biopolymers would be an alternative that could resolve these matters. Microencapsulation via spray drying was a promising alternative to convert bioactive ingredients into dehydrated powder, granule or agglomerate form with long-term stability. This microencapsulation is composed of three major steps: vapor, vapor-hot air exposure and moisture removal (Khalid *et al.*, 2018). Feed intake should be in solution, emulsion or suspension (Gharsallaoui *et al.*, 2007). The microencapsulated powder had extended durability with junior decomposition of thermal-sensitive constituents. Moreover, lightweight**,** convenient packaging, and cost-saving were other benefits of microencapsulated powder in logistics (Kha *et al.* 2010; Tan *et al.*, 2011). During spray-dried microencapsulation, carriers played an important role in limiting the exposure of the bare components with oxidation, photo-degradation, and moisture absorption (Poshadri and Kuna, 2010). Under low moisture content and water activity, physicochemical quality decompositions in spray-dried fucoxanthin powder would be effectively controlled. Different carriers such as polysaccharides, oligosaccharides, and proteins were proven to be effective in the coating of carotenoids (Montenegro *et al.*, 2007; Anarjan *et al.*, 2012; Bustos-Garza *et al.*, 2013; Mahfoudhi and Hamdi, 2014). Maltodextrin, Arabic gum and whey protein isolate was proven to be effective carriers in preventing fucoxanthin against thermal degradation and gastric acidity while improving its absorption behavior in the human colon (Xiaowen *et al.*, 2018).

Maltodextrin originated from starch hydrolysis consisting of D-glucose units and α (1→4) glycosidic bonds. Maltodextrin was always the first choice because of its cheap price, high solubility and versatile function suitable for microencapsulation of bioactive substrate (Nguyen, 2021). Its high solubility was highly preferred due to the decreased viscosity of feed dispersion suitable for atomization (Vidovic *et al.*, 2014). Trehalose was a non-reducing disaccharide with ideal physicochemical attributes for microencapsulation (Cerimedo *et al.*, 2008). Arabic gum was a hydrocolloid consisting of polysaccharides and glycoproteins commonly used in spray drying due to its high solubility, low viscosity, protective colloid functionality and stable emulsion in preparation (Krishnan *et al.*, 2005). It contributed to the protected characteristic in colloidal emulsion (Shaddel *et al.*, 2018). Starch was known as the major component in cereal grains with a broad variety of applications in the food sector. However bare starch did not always satisfy physicochemical characteristics owing to its delicate-molecular structure (Singh *et al.*, 2010). Bare starch was not appropriated for microencapsulation during spray drying because it caramelized, enclosed to the spray-dryer wall, and stranded the nozzle due to its heterogeneous stage during spray drying (Bayram *et al.*, 2005). Modified starch revealed the film-forming capability to cover microencapsulated components (Pegg and Shahidi, 2007). Inulin was a promising wall material due to its competitive price and nutritional aspect (Stevens *et al.*, 2001). It was commonly utilized as a carrier for probiotic microencapsulation to enhance its thermo-resistance and prolong the stability of probiotic products (Fritzen-Freire *et al.*, 2012; Pinto *et al.*, 2012; Avila-Reyes *et al.*, 2014; Nunes *et al.*, 2018). The purpose of this study was to improve the stability and bio-accessibility of fucoxanthin, the microencapsulation by spray drying in different biopolymers such as maltodextrin, trehalose, Arabic gum, modified starch, inulin was investigated. Based on the microencapsulation efficiency, particle size, bulk density and storage stability of the spray-dried powder, the appropriate wall material would be selected for spray drying of fucoxanthin.

2. Materials and methods

2.1 Experimental design

Fucoxanthin extract (purity of >99%) was purchased from Nasol Co. Ltd., Ha Noi, Vietnam. Maltodextrin 20DE as a carrier agent originated from Roquette (France). Each carrier of 120 g was weighed and dissolved in 1000 mL distilled water. Fucoxanthin extract was mixed with each carrier solution by a mixer at 1000 rpm for 30 minutes until the fucoxanthin was absolutely scattered to a uniform suspension. A mini spray dryer (model B-290, Buchi, Switzerland) was equipped with two-fluid nozzle. The spray dryer operated under the same proportion 12%, inlet/outlet drying temperature 150°C/80°C and feed flow rates 10 mL/min of different wall materials such as maltodextrin trehalose, Arabic gum, modified starch, inulin. The microencapsulated powders were collected at the funnel of the cyclone separator and kept in aluminum foil at room temperature. All batch preparations were conducted in triplicate. The spray-dried powder was then examined microencapsulation efficiency, particle size

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and bulk density. Storage stability was also monitored in 4 week-interval during 24 weeks of storage.

2.2 Measurements of technical parameters

2.2.1 Microencapsulation efficiency

Microencapsulation efficiency (%) was estimated by the ratio between the content of fucoxanthin in specific powder and the total content of fucoxanthin used (Ravi and Baskaran, 2015).

2.2.2 Particle size

Particle size (μm) was measured by using a Mastersizer 3000 (Malvern, United Kingdom).

2.2.3 Bulk density

Bulk density (g/ml) was estimated by the volume of a known weight of powder in a graduated glass cylinder. 10 g of microencapsulated powder was gently loaded into a 100 ml graduated cylinder. The marked volume was indicated to measure the bulk density as weight/volume (Chegini and Ghobadian).

2.2.4 Storage stability

Storage stability (%) was estimated by the content of fucoxanthin left in the microencapsulated powder at time *t*, and the content of fucoxanthin in the microencapsulated powder at the beginning. These measurements were conducted by High-performance liquid chromatography (Agilent 1300 HPLC) analysis (Wang *et al.*, 2012).

2.3 Statistical analysis

The experiments were run in triplicate with different groups of samples. The data were presented as mean ± standard deviation (SD). Statistical analysis was performed by the Statgraphics Centurion version XVI.

3. Results and discussion

3.1 Microencapsulation efficiency of fucoxanthin powder by specific wall materials

Microencapsulation efficiency was specified as the mass of the main matter that was microencapsulated inside a carrier, with respect to the content of the main matter that was utilized for microencapsulation. With the accelerating degree of the main matter, there was a downtrend of reduction in microencapsulation efficiency (Nitamani *et al.*, 2021). Microencapsulation efficiency was believed as the most essential factor in demonstrating the effectiveness of respected carriers as well as the functionality of the microencapsulated constituent. The microencapsulation efficiency of fucoxanthin powder by different specific wall materials is presented in Figure 1. The results showed a significant difference in the microencapsulation efficiency of the fucoxanthin powder by changing the carriers. The microencapsulation efficiency of the fucoxanthin powder was highest at Arabic gum (94.27±1.17%) while the lowest was at inulin (64.15±1.37%). The result was contrary to another finding, fucoxanthin microcapsule with Arabic gum had the lowest microencapsulation efficiency 86.48±0.71% (Xiaowen *et al.*, 2018). However, the results obtained in this study were similar to findings by microencapsulating fucoxanthin with casein and chitosan (Koo *et al.*, 2016), chitosan-glycolipid hybrid nanogel (Ravi and Baskaran, 2015). Carriers or wall materials were utilized to protect the bioactive components against external conditions like hot temperature damage, photo-degradation and oxidation (Tonon *et al.*, 2010). Lycopene powder microencapsulated by Arabic gum during spray drying showed excellent microencapsulation efficiency (Nunes and Mercadante, 2007). Maltodextrin revealed various benefits like low viscosity, perfect oxidative stability, and low cost; but it displayed low emulsifying ability (Carneiro *et al.*, 2013). A combination of Arabic gum, maltodextrin and modified starch showed a synergistic effect when compared with a singular treatment (Przybysz *et al.*, 2016). Maltodextrin with a lower dextrose equivalent induced better microencapsulation capacity (Negrao-Murakami *et al.*, 2017; Haghighat-Kharazi *et al.*, 2019). Maltodextrin and Arabic gum in individual or combination were used as wall materials during spray drying of jambolan juice. Arabic gum was recommended as an appropriate carrier agent supporting the spray drying process to achieve the best physicochemical attributes of its powder (Rakesh *et al.*, 2020). Lutein microencapsulated powder prepared by trehalose produced spherical granules with less minor shrinkage compared to maltodextrin. Trehalose or modified starch revealed fair microencapsulation efficiency due to its amorphous status after spray drying. Inulin provided 75.2±0.7% of microencapsulation

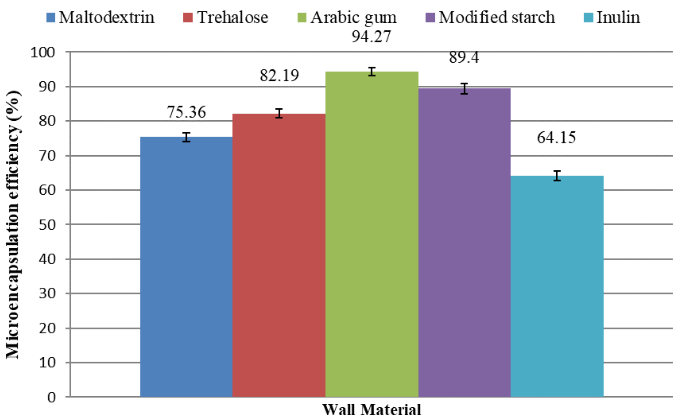

Figure 1. Microencapsulation efficiency (%) of fucoxanthin powder by specific wall materials.

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efficiency for lutein spray-dried powder (Zhuang *et al.*, 2020). A solid lipid nanoparticle-microcapsule delivery system implementing mixed lipids of palm stearin and cholesterol coated with gelatin/Arabic gum to load lipophilic fucoxanthin was formulated. The microencapsulated efficiency of fucoxanthin microcapsules was 96.24±4.60% (Chen *et al.*, 2022).

3.2 Particle size of fucoxanthin powder by specific wall materials

The largest particle size was recorded at Arabic gum (6.79±0.05 µm) while the lowest particle size was noticed at inulin (2.14±0.03 µm) (Figure 2). This could be explained due to the higher viscosity of Arabic gum compared to modified starch, maltodextrin, trehalose and inulin. With the lower viscosity of wall materials, with atomization energy remaining constant, the atomized droplet size might decrease, thus decreasing the final particle size. This result was similar to another study, the fucoxanthin microcapsule with Arabic gum had a particle size of 7.18±0.08 µm (Xiaowen *et al.*, 2018). Particle size not only affected bulk powder characteristics but also strongly correlated to powder processing and stream, with inferior powder stream speeding up the hazard for inferior tablet stuff uniformity and restriction on manufacture bandwidth (Nyqvist, 1982). Powder stream enhanced with ascendant particle size because of accelerated relative strength that fixed cohesion and friction strength (Lantz *et al.*, 1990). Powder with tiny particle size downtrend to contain higher total solid on compaction than greater particles of similar carrier with equal bonding force and facial roughness (Sun, 2011). Greater particle size will enhance powder current while falling compaction characteristics. The variation in powder particle size among carriers could be derived from the different physicochemical properties of spray suspension feedings (Alyssa *et al.*, 2019). The particle size was varied by the carrier and approach (Gharsallaoui *et al.,* 2007). Wall material caused no statistically significant difference in particle size. Particle size received from spray-dried microencapsulation is based on the physical properties of wall materials as well as the viscosity of the feed input to the spray dryer (Naji-Tabasi *et al.*, 2021).

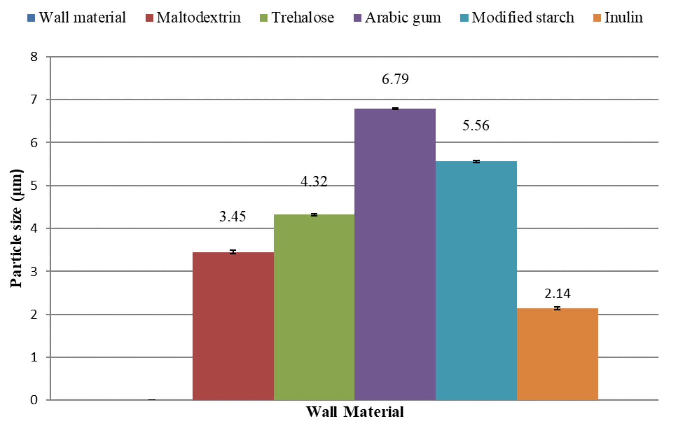

Figure 2. Particle size (µm) of fucoxanthin powder by specific wall materials.

3.3 Bulk density of fucoxanthin powder by specific wall materials

The effect of different wall materials such as maltodextrin, trehalose, Arabic gum, modified starch, inulin on the bulk density of fucoxanthin powder was illustrated in Figure 3. There was a significant difference in bulk density by specific wall materials. The highest bulk density was observed at Arabic gum (0.43±0.02 g/ml) while the lowest bulk density was noticed at inulin (0.20±0.03 g/ml). The high bulk density of fucoxanthin powder in Arabic gum was greatly beneficial for packaging, storage and transportation. Bulk density was a major parameter in packaging, preservation and transportation (Mayor *et al.*, 2011). It could be greatly influenced by different drying variables (Sagar *et al.*, 2020). It reflected the compaction of granulate solids (Phisut, 2012). Interlocking characteristics of irregularly shaped granules induced a lower bulk density (Lantz *et al.*, 1990). The biggest advantage of granules having higher density was space-saving with a huge amount in smaller bags in comparison with powder in lower density. Moreover, higher bulk density provided a lower amount of air filled in the gap between granules resulting in minor oxidative rancidity (Helena *et al.* 2013). The change of wall material from maltodextrin to Arabic gum resulted in a higher bulk density of chokeberry juice powder. There was an inverse correlation between bulk density and powder solubility (Ciou *et al.*, 2021).

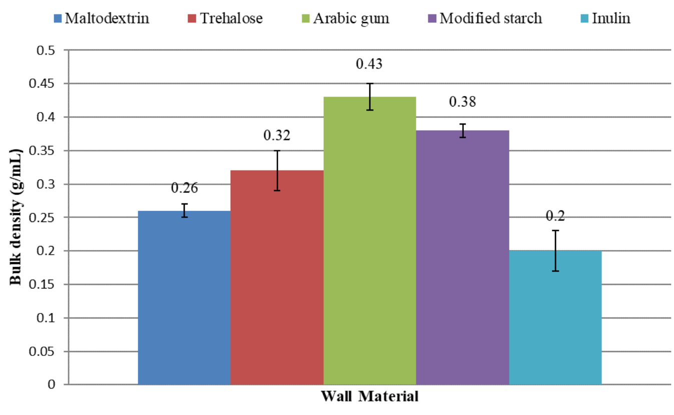

Figure 3. Bulk density (g/mL) of fucoxanthin powder by specific wall materials.

3.4 Stability of microencapsulated fucoxanthin powder by specific wall materials during storage

The major drawback of fucoxanthin was its instability in acidity, glow, oxygen, metal and fluctuating temperature. Bioactive ingredients like fucoxanthin powder should be effectively preserved against

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degradation in long-term storage. Inulin showed the least fucoxanthin stability with only 84.12±0.19% retention while microencapsulation with Arabic gum revealed the best protection of fucoxanthin with 90.46±0.15% retention after 24 weeks of storage (Figure 4). It could be due to a more uniform and less cracked texture (Kanakdande *et al.*, 2007). The excellent protective characteristics of the film-forming and emulsifying attributes of Arabic gum could be explained by its high-branched heteropolymer including arabinogalactan proteins, glycoproteins and arabinogalactans. Arabic gum effectively maintained β-carotene due to the protective effect of carotenoid from oxidation during spray drying (Kha *et al.*, 2015). Maltodextrin and Arabic gum stabilized the thermal retention of fucoxanthin in the spray-dried microcapsule (Xiaowen *et al.*, 2018). A combination of maltodextrin and Arabic gum (1/7 w/w) maintained the highest fucoxanthin stability during preservation (Oliyaei *et al.*, 2020). Trehalose of modified starch failed to support enough safeguard for the core ingredient in the extended preservation while inulin offered extra effective defence (Zhuang *et al.*, 2020).

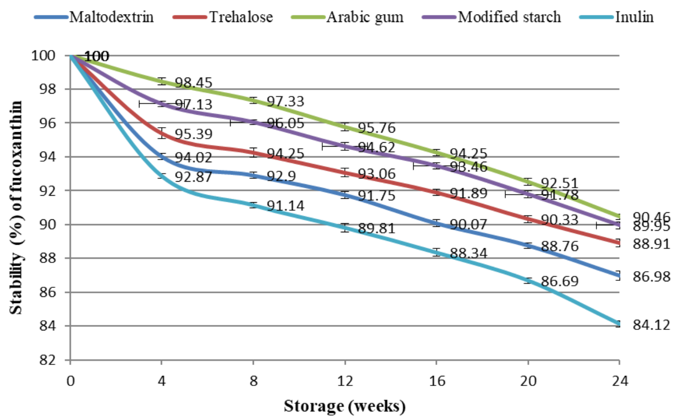

Figure 4. Stability (%) of fucoxanthin powder by specific wall materials during storage (weeks).

4. Conclusion

Findings revealed that Arabic gum was the most suitable carrier for microencapsulation of fucoxanthin. This coating material contributed as a physical barrier against the invasion of heat, light and oxygen to finally defend the core ingredient. Microencapsulation is an effective approach to prevent rapid degradation of sensitive compounds and increase the bioavailability of fucoxanthin.

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