ENCAPSULATION OF TOCOPHEROL AND TOCOTRIENOL IN VITAMIN-E USING SPRAY DRYING TECHNIQUE

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ABSTRACT

The encapsulation technology by spray drying technique was applied to protect vitamin E from unfavourable environment and to minimize the degree of vitamin E degradation. Vitamin E microencapsules were produced using a blend of maltodextrin and sodium caseinate as wall materials. The surface morphology of the microcapsules was observed by field emission scanning electron microscopy, which demonstrated a tendency of agglomeration. The outer surfaces of the capsules showed only a few pores or cracks. The particles sizes measured were the range of 13 μm to 29 μm. The tocopherol encapsulation efficiency and tocopherol retention after spray drying were measured by high-performance liquid chromatography. About 52-70% of tocopherol encapsulation efficiency has been achieved.

1. INTRODUCTION

Vitamin E is one of the most important phytonutrients in edible oils. The term vitamin E is used as a generic reference to tocopherol and tocotrienol compounds exhibiting the biological activity of α-tocopherol (Ball, 1988). α-tocopherol is a well-known fat-soluble antioxidant and is widely used in the food industry (Chang, et al., 2005). It is called antioxidant because of its ability of quenching or stabilizing free radicals that lead over time to degenerative diseases, including cancer and cardiovascular disease (Yoo, et al., 2006). Vitamin E can be degraded rapidly in the presence of oxygen, and free-radical mediated oxidative processes. These obstructions of vitamin E can be partially overcome by applying microencapsulation technology to protect α-tocopherol from unfavorable environment and to solubilize it in aqueous environment.

Microencapsulation is a process in which sensitive ingredients or 'core' materials are entrapped in a protective polymer encapsulating agent or 'wall' material (Hogan, et al., 2001). Spray drying technique has been widely applied in the food industry for encapsulating vitamins, minerals, and other sensitive ingredients (Yoo, et al., 2006).

Carbohydrate such as maltodextrin has been extensively used as encapsulating agents. Maltodextrins are non sweet nutritive polysaccharides consisted of: α(1-4)-linked D-glucose produced by acid or enzymatic hydrolysis of corn starch. Although maltodextrins do not promote good retention of volatile compounds during the spray drying process, they protect encapsulated ingredients from oxidation (Reineccius, 1991, Ré, 1998). Maltodextrins, however, require an additional emulsifying agent for the encapsulating of lipids, as the formation of a stable emulsion of small particle size is normally required for the preparation of a fat encapsulate (Risch & Reineccius, 1988). Sodium caseinate is used because of its excellent emulsifying and film forming abilities (Surh, et al., 2006). Sodium caseinate contributes opacity, solubility, heat stability, and most importantly, the superb water- and fat-binding characteristics that make them suitable as emulsifiers. It emulsifies so well because of its structure - a random coil with a hydrophobic head and hydrophilic tail.

The objective of the present study was to encapsulate mixed tocopherol and tocotrienol protecting against loss by oxidation using a hydrophilic matrix in order to produce microcapsules of antioxidant vitamin E for application in the food industry as fortification. The encapsulation was through spray drying technique. Microcapsules of vitamin E produced were water-soluble, which allowed more industrialized use of this vitamin in order to enrich foods such as, milk powder, flours, wholemeal, in short, into products with low water activity.

2. EXPERIMENT

2.1 Materials

Fully natural palm mixed tocopherol and tocotrienols concentrate was purchased from Super Vitamins Sdn. Bhd (Masai, Johor). Maltodextrin (DE 11-15) was purchased from Merck (Damstadt, Germany). Sodium caseinate (protein content: 88%) was obtained from DMV International (Veghel-The Netherlands). All other chemicals were of HPLC grade and were purchased from Merck (Darmstadt, Germany).

2.2 Methods

2.2.1 Preparation of emulsions. Wall material solutions containing blends of maltodextrin:sodium caseinate, both in the range of 18.5-24% w/w and 7-9% w/w, respectively were prepared in distilled water. The wall material solutions were homogenized at 3000 rpm for 20 minutes. A blend of palm natural tocopherol and tocotrienol mixed concentrate was emulsified into the wall material solution. Core/wall ratio of 0.6, 0.7 and 1.0 were used. In all cases, total solids content of feed solution was 22-40% (w/w). The emulsification was carried out at 3500 rpm for 30 minutes.

2.2.2 Spray drying. The emulsions were spray-dried using a pilot plant spray-drier (Pilot Spray Drying Plant PSD-00, Hemray Enterprise, Bombay, India) equipped with 0.5 mm diameter nozzle. The inlet and outlet temperature was maintained at 110 °C and 90 °C, respectively. The spray-dryer was operated at a feed rate of 10 ml/min and air pressure of 55 kgf/cm^2. The microcapsules were stored in a glass bottle at -20 °C prior to analysis.

2.2.3 Tocopherol content determination. Vitamin E microcapsules (2.0 mg) were dispersed in 1.0 ml of hexane in order to break the microcapsules under vortex agitation. The mixture was filtrated using a 0.45 μm polytetrafluoroethylene (PTFE) membrane before injection to the HPLC.

2.2.4 Encapsulation efficiency. Encapsulation efficiency was assessed by determining the total tocopherol content of the powder and surface tocopherol. The encapsulation efficiency was calculated according to McNamee, et al. (2001), as shown below:

$$\%EE = \frac{(TT - ST) \times 100}{TT}$$

Where:
TT= total tocopherol and ST=surface tocopherol.

Total tocopherol and surface tocopherol determination were developed based on the conditions described by Barbosa, et al. (2005) for bixin extraction from encapsulated bixin crystals with some modifications. Microcapsules (1.0 g) were dispersed in distilled water (20 ml) then homogenized for 1 min under vortex agitation. The mixture was transferred to a separation funnel then extracted with hexane (total of 40 ml).
Surface tocopherol was determined by direct extraction of 1 g of microcapsules with 5 ml hexane by vortex agitation for 30 s, followed by centrifugation (3000 rpm for 10 min). After phase separation, the liquid phase containing the tocopherol was collected and filtered through a PTFE membrane (0.45 μm) before analysed with HPLC. The analysis was carried out in duplicate.

2.2.5 HPLC analysis. The purity of tocopherol, either as liquid or spray-dried vitamin E microcapsules, was verified by HPLC. The analysis was carried out using a Waters HPLC system equipped with a photo-diode array detector (Waters, model 515). The equipment also included a Rheodyne 775i injection valve with a 20 μL loop and a column heater. Data acquisition and processing were performed using the CSW Switch software.
Separation of tocopherol from spray-dried microcapsules was achieved on a Synergy 4u hydro-RP 80A column (4 μm, 250 x 4.6 mm) detected at wavelength 292 nm with methanol/acetonitrile (50:50 v/v) as a mobile phase at flow rate of 1.0 ml/min and column

temperature set at 40 °C. The condition was previously evaluated by Aoun, et al. (2005). All solvents were HPLC grade.

2.2.6 Scanning electron microscopy. The surface morphology of the microcapsules was observed by a scanning electron microscopy (SEM), Jeol, model JSM-6390 LV, Japan, following methodology described by Farias, et al., (2007). The samples were placed on the SEM stubs (10 mm) using a two-sided adhesive tape. The specimens were subsequently coated with Platinum using an ion sputter coater. The coated samples were then analysed using the SEM operating at an accelerating voltage of 15 kV.

3. RESULTS AND DISCUSSION.

3.1 Encapsulation efficiency (EE)

In the present study, the encapsulation efficiency (EE) ranged from 52 to 70% (Table 1). Increasing core/wall ratio from 0.6 to 1.0 had little effect on EE of vitamin E microcapsules. This decrease in EE may have been due to instability of the oil droplets in the emulsion before the spray drying process (Dian, et al., 1996). It could also be due to the thinner layers of wall material between encapsulated oil droplets (Hogan, et al., 2001).

Table 1 Influence of core/wall ratio on the properties of emulsion powders prepared at a sodium caseinate/maltodextrin ratio of 1:3.

Core/wall ratio	Powder		
	Particle size (μm)	EE (%)	Retention (%)
0.6	13	70	51
0.7	29	66	60
1.0	23	52	63

3.2 Retention of tocopherol content

As shown in Table 1, about 51 to 63% of the initial tocopherol remained in the powder after spray-drying. The rest might have been destroyed during spray-drying which was carried out at a high temperature (110 °C). Retention of tocopherol content in the microcapsules were increasing with the increased of core/wall ratio. This may suggest that, the higher oil content in the emulsion, the higher percentage of tocopherol to be remained in the microcapsules.

3.3 Micrographs

The morphology of microcapsules can be observed in Figure 1 to 3. One reason for using SEM in studying microencapsulation is the need to visually determine the encapsulating ability of the wall materials. Indication of this ability could be further described by the retention and encapsulation efficiency of active materials in the microcapsules.

Morphologic analysis showed the size, the shape and common aspects of the microcapsules made from the blend wall materials and core/wall ratio of 0.6 (Fig. 1). Furthermore, the tendency of agglomeration of the smallest particles between themselves and the biggest ones was clearly observed.

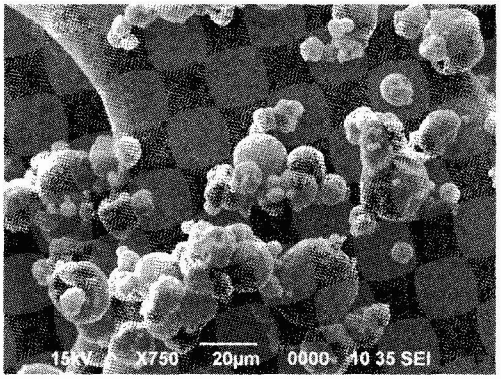

Fig.1. Scanning electron micrograph of spray-dried palm mixed vitamin E emulsion stabilized by wall materials combining sodium caseinate and maltodextrin prepared at a ratio of 1:3 and core/wall ratio of 0.6.

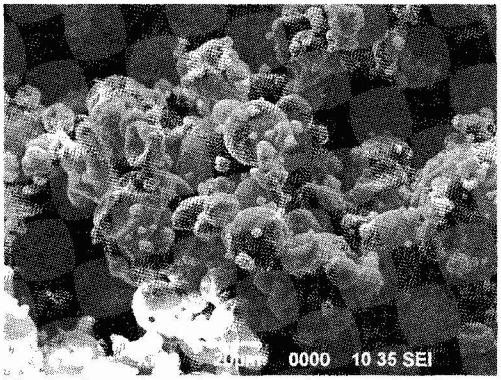

Fig.2. Scanning electron micrograph of spray-dried palm mixed vitamin E emulsion stabilized by wall materials combining sodium caseinate and maltodextrin prepared at a ratio of 1:3 and core/wall ratio of 0.7.

The difference in higher core/wall ratio i.e. 0.7 could be observed in figure 2. The outer surfaces of the spray-dried microcapsules are characterized by the presence of dents and these dents are most likely formed by shrinkage of the particles during drying and cooling. Similar dents were observed in the study of ascorbic acid encapsulation (Finotelli and Rocha-Leão., 2005) using maltodextrin and Capsul (National starch) as wall materials. Although the outer surfaces of the capsules had some dents, they showed only a few pores or cracks.

SEMs of powders prepared at a core/wall ratio of 0.6 appeared as discrete particles with both smooth and wrinkled surfaces (Fig. 1). However at a core/wall ratio of 0.7 and 1.0 (Fig. 2 and 3) powder particles appeared highly agglomerated may be due to high levels of surface oil (Hogan, et al., 2001).

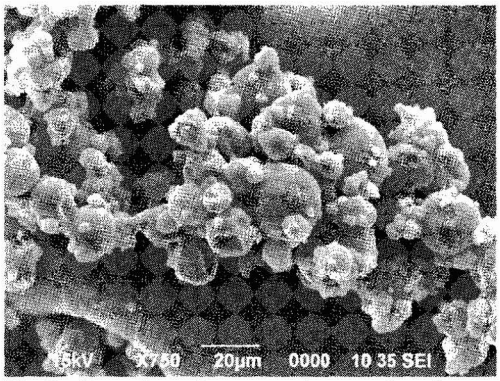

Fig.3. Scanning electron micrograph of spray-dried palm mixed vitamin E emulsion stabilized by wall materials combining sodium caseinate and maltodextrin prepared at a ratio of 1:3 and core/wall ratio of 1.0.

Dian, et al. (1996) reported that the higher the oil content of emulsions the more surface oil was found, with less oil being encapsulated. It could also be due to the effect of being insufficient wall material to encapsulate the oil, as the amount of oil was increased. However, according to Sankarikutty, et al. (1988) from their work some of the oil on the surface may be due to extraction of the oil from inside the microcapsules that release out through cracks. Development of cracks could be affected by the type of wall material used and also by the drying conditions employed, especially the rate of drying (Dian, et al., 1996).

Powder particle sizes are ranging from 13 to 29 μm and it was not affected by core/wall ratio. However, according to Hogan, et al. (2001) particle size of powder will be increased as a function of both total solids concentration (emulsion) and spray-drier nozzle atomizer.

CONCLUSION

Mixed tocopherol and tocotrienol were successfully encapsulated using spray-drying technique with blends of sodium caseinate and maltodextrin as a wall material with the percentage of encapsulation efficiency and retention of tocopherol were more than 50%. The vitamin E microcapsules can be readily used as food ingredient for application in food industry.

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