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PHYSICAL STABILITY OF β-CAROTENE ENCAPSULATED WITH DIFFERENT WALL MATERIALS

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ABSTRACT

β-carotene microcapsules were prepared by spray drying using potato starch, maltodextrin and gelatin as wall materials. All the microcapsule samples were found to be light coloured with slight difference in colour values (*L**, *a**, *b**, *C** and *H**) irrespective of different wall materials used. The bulk density and tapped density of microencapsulated β-carotene powder varied from 0.28 to 0.33 g/cm^3 and 0.34 to 0.39 g/cm^3, respectively The β-carotene microcapsules with maltodextrin as wall material showed good flow characteristics while those with potato starch and gelatin as wall materials showed fair flow characteristics. The β-carotene content of all microencapsulated samples was decreased during storage for 60 days. At ambient conditions the retention of β-carotene was less (63-73 %) as compared to refrigerated conditions (70-81 %). Half life of β-carotene at refrigeration conditions (4 ± 1ºC) was found around 9 to 15 months whereas at ambient conditions (25 ± 5ºC) it was around 7 to 11 months. Among the wall materials, maltodextrin was found to have good potential to serve as a wall material based on the physical stability of encapsulated β-carotene compared to potato starch and gelatin.

INTRODUCTION

β-carotene has been found in most yellow, orange, dark green leafy vegetables and fruits such as kale, pumpkin, spinach, papaya, apricots, and peaches. Carrots are said to be a major source of β-carotene. Carotene, a yellow orange pigment, is present in the chromoplasts in fresh carrot. Maintenance of the naturally colored pigments in stored foods has been a major challenge in food processing (Ihl *et al.*, 1998).

β-carotene possesses 50 per cent Vitamin A activity (Delgado Vargas *et al.*, 2000) and its demand has increased due to its reported anticancer (Nishino *et al.*, 1999), free radical quencher and other biological antioxidant activities (Murakoshi *et al.*, 1999). The high degree of unsaturation in β-carotene structure renders it extremely susceptible to oxidation.

One of the approaches that can be used to improve the stability and retention of β-carotene is microencapsulation. Microencapsulation is defined as a process in which tiny particles or droplets are surrounded by a coating, or embedded in a homogeneous or heterogeneous matrix, to give small capsules with many useful properties. Microencapsulation can provide a physical barrier between the core compound and the other components of the product. Microencapsulation by spray-drying has been successfully used in the food industry for several decades (Gouin, 2004). In the present work, an attempt has been made to microencapsulate β-carotene by spray drying using potato starch, maltodextrin and gelatin as wall materials. The microcapsules were evaluated for flow properties and colour values. The stability of microencapsulated β-carotene was also evaluated in terms of retention of β-carotene during storage and its half life period. Thereby to investigate the ability of potato starch, maltodextrin and gelatin as a good wall material for microencapsulation of β-carotene.

MATERIALS AND METHODS

Pure *trans* β-carotene was obtained as a complementary sample from DSM Nutritional Products India Ltd., Thane. The potato starch, maltodextrin, gelatin and mannitol used were of food grade. All other reagents were of analytical grades and obtained from Himedia, Mumbai.

Preparation of sample

The microencapsulation of β-carotene was done by the method given by Kshirsagar *et al.* (2014).

Pure *trans* β-carotene was added to the solution of potato starch (30% w/w), maltodextrin (30% w/w) and gelatin (10% w/w, due to limit of viscosity) in distilled water. The mixture was then homogenized to obtain an aqueous emulsion (feed liquid) and immediately fed to the spray-dryer (Labultima, LU-228). The inlet and outlet air temperature were maintained at 170 ± 5ºC and 95 ± 5ºC, respectively. All the experiments were performed in triplicate. The spray-dried powders were collected, kept in plastic bags wrapped with aluminum foil and stored in desiccators containing silica gel at room temperature.

Colour determination

The colour scanning machine (Model: Colour Flex EZ) was used for the measurement of colour of microencapsulated β-carotene powder (Klaypradit and Huang, 2008 and Drusch *et al*., 2006b).

Flow properties of microencapsulated β-carotene powder

Flow properties (bulk density, tapped density, Carr's index and Haussner's ratio) of microencapsulated β-carotene powder was determined by following the methods adopted by Drusch *et al*. (2006a and 2006b).

Determination of β-carotene content

The β-carotene content in the micro-encapsulated powder was determined following the method of Ax *et al*. (2003).

Stability of β-carotene within the microcapsules

Degradation of β-carotene in microencapsulated powder was analyzed at an interval of 10 days according to Glassgen *et al*. (1992). The percentage retention of β-carotene in microencapsulated powder was calculated by the formula (analyte at 'X' storage time) x 100/ (analyte at zero storage time). A semi-log plot of percentage retention of all these analytes vs. time according to Cai and Corke (2000) was done to obtain the rate constant (*k*) as the slope of the graph. Half-life ($t_{1/2}$) for the retention β-carotene in microencapsulated powder was calculated from the rate constant as 0.693/*k*.

SEM analysis

Particle size and structure of spray-dried microcapsules were evaluated with scanning electron microscope, (Model: Hitachi S-4800 Type-II) using the methods described by Rosenberg *et al*. (1985) and Krishnan *et al*. (2005). The microcapsules were mounted on specimen stubs with double sided adhesive carbon tapes. The specimen was coated with gold and examined at 5 kV. Type 55 Polaroid film was used to produce micrographs.

Statistical analysis

The data obtained was analyzed statistically to determine statistical significance using methods given by Panse and Sukhatme (1967). The analysis of variance revealed at significance of $p < 0.05$ level, S.E. and C.D. at 5 % level was mentioned wherever required.

RESULTS AND DISCUSSION

Colour values

The changes in colour values (*L**, *a**, *b**, *C** and *H**) of microencapsulated β-carotene powders with respect to different wall materials are presented in Table 1.

The difference in wall material had significant effects ($p < 0.05$) on *L** values. The colour values of microencapsulated β-carotene powders *viz*., *L**, *a**, *b**, *C** and *H** were observed with different wall materials *viz*. potato starch (95.969, -1.217, 2.871, 3.118 and 112.999), maltodextrin (96.137, -1.240, 3.400, 3.619 and 110.065) and gelatin (96.510, -0.455, 4.826, 4.847 and 95.420) respectively. It was observed that among the different microencapsulated β-carotene powders, the gelatin treated powder gave the highest value of *L**, it being visually the lightest. The *a** coordinate which quantifies the red intensity, showed negative values for all the samples, except

Table 1: Colour values of microencapsulated β-carotene powder

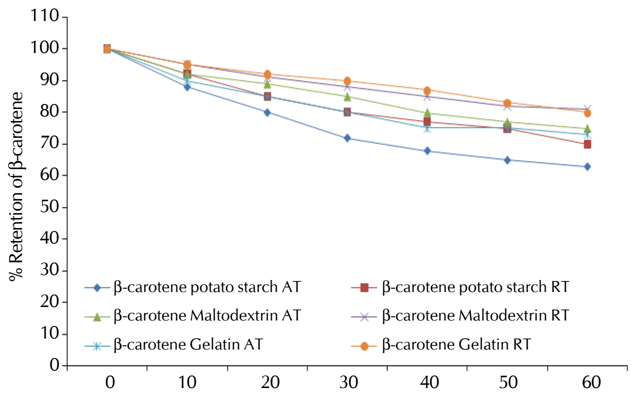
Wall materials	Colour values				
	*L**	*a**	*b**	*C**	*H**
Potato starch	95.969	-1.217	2.871	3.118	112.999
Maltodextrin	96.137	-1.240	3.400	3.619	110.065
Gelatin	96.510	-0.455	4.826	4.847	95.420
SE±	0.054	0.097	0.033	0.057	0.071
CD at 5%	0.166	0.369	0.103	0.187	0.218

Table 2: Flow properties of microencapsulated β-carotene powder

Wall materials	Bulk Density (g/cm^3)	Tapped Density (g/cm^3)	Carr's Index (%)	Hausner's ratio
Potato starch	0.28	0.34	17.65	1.21
Maltodextrin	0.33	0.38	13.16	1.18
Gelatin	0.30	0.37	18.91	1.23
SE±	0.004	0.003	-	-
CD at 5%	0.013	0.009	-	-

Table 3: Kinetic degradation data for microencapsulated β-carotene powder during storage

Storage conditions	Wall materials	Carotene degradation kinetics k (10^{-3} day^{-1})	Haif life$t_{1/2}$ (months)
Ambient temperature	Potato starch	3.34	7
	Maltodextrin	2.08	11
	Gelatin	2.28	10
Refrigerated temperature	Potato starch	2.58	9
	Maltodextrin	1.53	15
	Gelatin	1.62	14



AT- Ambient Temperature, RT- Refrigerated Temperature

Figure 1: Retention of β-carotene in microencapsulated powder during storage

gelatin sample which showed slightly higher value. It can be seen that all samples had a negative *a** value. The *b** coordinate which quantifies the yellow intensity, also showed a higher value for the gelatin sample. All samples showed positive *b** values.

It can be seen that all the samples were lighter. The difference in the colour parameters of powder samples may be attributed to the colour of different coating material and also due to the effects of the heat to which the samples were subjected in the spray drying process (Parize *et al*., 2008). Yousefi *et al*. (2011) also reported better colour properties obtained by using maltodextrin as wall material.

Flow properties of microencapsulated β-carotene powder

The bulk density and tapped density of microencapsulated β-carotene powder varied from 0.28 to 0.33 g/cm^3 and 0.34 to 0.38 g/cm^3, respectively (Table 2). The data showed that bulk density and tapped density of potato starch treated samples were 0.28 and 0.34 g/cm^3, respectively and maltodextrin treated samples were 0.33 and 0.38 g/cm^3, respectively whereas gelatin treated samples were 0.30 and 0.37 g/cm^3, respectively.

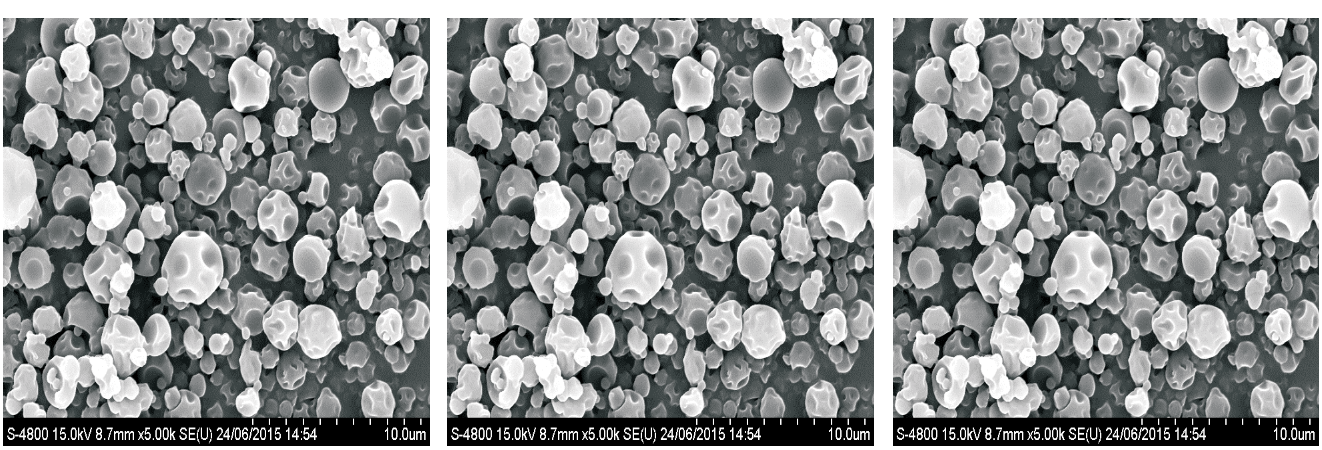
The high values of bulk density and tapped density were observed in case of maltodextrin which may be due to more spherical and porous form of microcapsules giving rise to higher surface area thereby resulting into greater bulk densities. The lower values of bulk densities for potato starch and gelatin microcapsules may be attributed to their larger particle size. These results were in good agreement with Desobry *et al*. (1997). Kshirsagar *et al*. (2014) reported that bulk density of spray dried powder was affected by the particle size and sphericity of granules.

The data revealed that Carr's index of powder sample with potato starch, maltodextrin and gelatin as wall materials were 17.65, 13.16 and 18.91 % respectively, while their Hausner's ratio were 1.21, 1.18, 1.23 respectively. As per the indices of Carr's index (13.16 %) and Hausner's ratio (1.18), the flow properties of microencapsulated β-carotene powder with maltodextrin as wall material showed good flow characteristics. The Carr's index and Hausner's ratio of microencapsulated β-carotene powder with potato starch and gelatin as wall material were recorded between 16 to 20 % and 1.19 to 1.25 respectively, which showed fair flow characteristics.

Storage studies of microencapsulated β-carotene powder

The carotene contents of powders (Fig. 1) were decreased at the end of 60 days storage period at both ambient (25 ± 5ºC) as well as refrigerated conditions (4 ± 1ºC). At ambient conditions the retention of β-carotene was less (63-73 %) as compared to refrigerated conditions (70-81 %). Among all the three wall materials used for microencapsulation, maltodextrin and gelatin samples showed better retention of β-carotene i.e. 81 and 80 % respectively at refrigerated conditions compared to potato starch (70 %).

The present findings are more or less similar to the findings reported by Sutter *et al*. (2007) and Desobry *et al*. (1997). Ersus and Yurdagel (2007) also reported that storage at refrigerated temperature showed better retention of anthocyanin in microencapsulated black carrot powder than at ambient temperature. Kshirsagar *et al*. (2014) reported that better retention of β-carotene in microencapsulated powders



(A) Potato starch (B) Maltodextrin (C) Gelatin

Figure 2: Sem photomicrographs of microencapsulated β-carotene powder coated with (A) Potato starch, (B) Maltodextrin and (C) Gelatin As wall material

was attributed to particle size and surface β-carotene.

Kinetic degradation of microencapsulated β-carotene powder

The kinetics of degradation of β-carotene were monitored over the storage period, and the rate constant and half-life values of reactions were determined. Most works indicated that carotenoids undergo first order degradation reaction (Wagner and Warthesen, 1995; Desroby *et al*., 1997, 1999), generally occurring in two distinct periods an initial fast rate degradation period up to around 4 to 6 weeks and a slower rate degradation period at longer times (Rodríguez-Heuzo *et al*., 2004). The data presented in Table 3 revealed that an increase in storage temperature led to an increase in rate constants. Half life of β-carotene at refrigeration conditions (4 ± 1ºC) was found around 9 to 15 months whereas at ambient conditions (25 ± 5ºC) it was around 7 to 11 months. Among all wall materials, maltodextrin and gelatin showed better half life value of 15 and 14 months respectively, as compared to potato starch (9 months) under refrigerated conditions of storage. It is well known that stability of microencapsulated product is influenced by the composition of wall material (Moreau and Rosenberg, 1998).

Wagner and Warthesen (1995) also reported degradation of α and β-carotene during storage of the powders at temperatures ranging from 37 to 65°C, followed first-order kinetics and both degraded at the same rate. Increasing the proportion of carrier decreased the carotene degradation rate and also decreased surface carotene.

Desobry *et al*. (1997) reported that oxidation of β-carotene microcapsules followed first order kinetics with an initial fast first order reaction followed by a second much slower first order reaction period. Ersus and Yurdagel (2007) also reported higher anthocyanin degradation (i.e. low $t_{1/2}$) in microencapsulated black carrot powder at ambient temperature than refrigerated temperature.

SEM analysis

Results (Fig. 2) clearly showed significant differences in size and shape of microcapsules. Microcapsules from potato starch showed rounded shape, smooth surface with no obvious dents. The granular of potato starch were homogenous having size ranging from < 5 to 28 μm. The microcapsules from maltodextrin showed spherical shapes with smooth and some dented surfaces. The granules of maltodextrin were more heterogeneous and consisted of very small granule size of < 3 μm and few large size of 8-20 μm, whereas microcapsules from gelatin showed spherical shapes with smooth outer surface. The formation of dents on surface of spray dried particles was attributed to the shrinkage of the particles during the drying process (Rosenberg *et al*., 1985 and 1990). Similar results were reported by Loksuwan (2007) and Hejri *et al*. (2010). The SEM also indicated the suitability of maltodextrin and gelatin as a wall materials for microencapsulation of β-carotene as compared to potato starch.

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