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Measurement and Influence Factors of the Flowability of Microcapsules with High-content β-Carotene*

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Abstract The flowability of five kinds of microencapsulation powders, with different β-carotene contents and by two alternative particle-forming technologies *i.e.* spray-drying and starch-catching beadlet technology, was measured. The actual flow properties of the five powders were compared based on bin-flow test, and three flow indexes (Hausner ratio, repose angle and flow index) were measured. It was found that the repose angle is the most suitable index to reflect the flowability of these powders for the particle properties would not be altered due to compaction or tapping during the measuring process. Particle size and particle size distribution play most important roles in the flowability of these granular materials, which was also influenced by other factors like shape, surface texture, surface roughness, *etc.* Microcapsules with wall material of gelatin and a layer of modified starch absorbed on the surface showed excellent flowabilities and good mechanical properties, and they are favorable for tabletting to supply β-carotene.
Keywords β-carotene, microcapsule powders, flowability, Hausner ratio, repose angle

1 INTRODUCTION

Carotenoids, such as β-carotene, have long been demonstrated to be capable of providing some medical or health benefits, including the possible prevention or treatment of skin cancer and cardiovascular disease [1—4]. Moreover, β-carotene is still an important biological compound for its provitamin A activity[5].

With the increasing knowledge of the positive functions of β-carotene, more and more people take interest in some foods or pharmaceuticals containing β-carotene ingredients, such as beverages, baked goods, oils, capsules and tablets. Usage of tablets made of high-content β-carotene granule or powder to provide this active material is very effective and conventional. Besides high-content, some properties, such as particle size, size distribution, surface texture, surface energy, moisture content, flowability, compactibility, morphology of the powder or granule, are also very important for the formation and characterization of the tablet. Among them, the flowability and compactibility are two essential factors to ensure a successful process for preparing tablets[6—8].

In recent years, there appeared some kinds of β-carotene powders/beadlets in the market. These products, contained different compositions or produced by different particle-formed methods, were not all fit for being compressed into a tablet. In this work, the flowability of different microencapsulated powders with high β-carotene content is studied to make sure which kind of β-carotene microcapsules is suitable for tabletting dosage. In detail, Hausner ratio, flow index and repose angle of different β-carotene microcapsules are chosen as three flow indexes for comparing with their actual-flow properties based on bin-flow test, so as to find the most congruent flow index to reflect its flowability. Furthermore, the reasons for the discrepancies of flowability through different indexes and factors influencing the β-carotene microcapsule powders flowability are also discussed.

2 MATERIALS AND METHODS

2.1 Materials

The test materials consist of five kinds of pharmaceutical or food powders with high-content β-carotene, in which three kinds contain 10% of β-carotene and the rest two content 20% of the same active ingredient. Meanwhile, they were produced by two processes—spray-drying and starch-catching beadlet technology. Spray-drier GLZ-5 was from Shanming Machine Co., Fujian, China. When particles were produced with starch-catch beadlet technology[9], the prepared emulsion was sprayed from a revolving spray head into a fluidized bed with modified starch particles operated at 0℃. After all the emulsion had been collected by the fluidized starch particles, the entire mixture was dried with air of moderate temperature, then the dried mixture was screened and the β-carotene-containing particles retained on the screen were collected.

Five kinds of samples were produced by Xinchang Pharma. Factory, Zhejiang Medical Co. Porcine gelatin were from Roussellet (Wenzhou, China), modified starch were supplied by National Starch&Chemical Trading Ltd., and sucrose was purchased from the local market.

The components and the operation methods of the powders as well as the main components, including its β-carotene content, microcapsule wall material, $D_{90\%}$, $D_{10\%}$, $D_{50\%}$ and spans of the particle sizes of five different particles were listed in Table 1.

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Table 1 The main components, drying methods and other physical properties of the five kinds of β-carotene microencapsulated powders

	Content of β-carotene	Wall material of the microcapsules	Methods of forming powder	Mean particle size, μm	$D_{90\%}$	$D_{10\%}$	$D_{50\%}$
SD-10S	10%	modified starch, sucrose	spray-drying	240.0	361.1	123.5	240.0
SD-10G	10%	gelatin, sucrose	spray-drying	115.6	180.5	17.5	114.2
SD-20G	20%	gelatin, sucrose	spray-drying	120.4	203.2	24.5	118.4
SC-10GS	10%	gelatin, starch, sucrose	spray and starch-catching beadlet technology	242.9	324.4	165.2	249.0
SC-20GS	20%	gelatin, modified starch, sucrose	spray and starch-catching beadlet technology	281.4	371.6	194.0	277.4

	Span	Sphericity (ψ)	ρ_B, kg·cm^{-3}	ρ_P, kg·cm^{-3}	Hausner ratio	Water content, %
SD-10S	0.99	0.82	335	1121	1.20	5.9
SD-10G	1.43	0.90	563	1315	1.25	7.1
SD-20G	1.51	0.92	446	1297	1.13	6.0
SC-10GS	0.64	0.95	685	1467	1.12	6.9
SC-20GS	0.64	0.95	608	1370	1.09	6.1

Note: The five kinds of powders also contain a spot of emulsion as ascorbate palmitate, anti-oxidizer vitamin E, *etc.* $D_{90\%}$ is the diameter for which 90% of the samples is smaller, $D_{10\%}$ is the diameter for which 10% of the samples is smaller, $D_{50\%}$ is the diameter for which 50% of the samples is smaller, and Span $=(D_{90\%}-D_{10\%})/D_{50\%}$. Particle density ρ_P measured by gas flow method.

2.2 Methods

2.2.1 *Particle physical properties analysis*

In order to study the surface properties of particles, scanning electronic microscopy (FEI, USA) was used. Samples were placed on a brass cylinder and coated with gold/palladium thin layer with a fine-coat ion sputter JFC1100.

Ten particles were observed by optical microscopy, fitted with a CCD monochrome camera connected to a MATROX grabbing board. The size and shape descriptors characterizing each particle as in its silhouette were calculated. Two parameters, the silhouette breadth (B, smallest dimension) and length (L, largest dimension), were noted, and the degree of sphericity ψ is defined as

$$\psi=\frac{L}{B} \tag{1}$$

Particle size and particle size distribution were analyzed with coulter LS-230 laser particle size analyzer.

2.2.2 *Descriptions of the flowability of particles*

(1) Hausner ratio

Hausner ratio (HR) is defined as[10]

$$\mathrm{HR}=\frac{\rho_{\mathrm{tapped}}}{\rho_{\mathrm{aerated}}} \tag{2}$$

where ρ_{aerated} (aerated bulk density) of a powder is determined by allowing the dispersed powders to settle in a container under the influence of gravity; ρ_{tapped} (tapped bulk density) is obtained by tapping the container mentioned above. The values of HR could distinguish the powder flowability of the free-flowing, easy-to-fluidize group from that of the cohesive, difficult-to-fluidize group.

Densities of the β-carotene microencapsulated powders were examined as suggested by the European Pharmacopoeia's Technical Procedure. Five runs were handled on each sample for the aerated density and the tapped density. The average values were used for the aerated and tapped bulk density, respectively.

(2) Flow index

Jenike shear cell was used for measuring the angle of wall friction[11]. The standard shear test technique[12] was used in this experiment. A flow function is a plot of unconfined yield strength (UYS) versus the major consolidating stress (MCS). It gives the stress needed for the testing microencapsulated powder flowing, and the inverse slope of each flow function was defined as the flow index. The instantaneous flow functions reflect the wall friction and the internal friction among particles. It represents the strength developed within a powder when consolidated, which must be overcome in making the powder flow. Higher flow index value indicates better flowability of powder, in the contrast, lower flow index reflects that the powder is prone to be cohesive[13].

(3) Repose angle

The traditional method for measuring static repose angle α as shown in Fig.1 is suitable for free-flowing granules but not for cohesive powders[14]. Some modified experimental apparatus was used to determine the repose angle of cohesive powders, too[15,16].

In order to measure both free-flowing and cohesive powder with the same apparatus, we framed an equipment as Fig.2, which was the assembly of a screen cover, a screen, a spacer ring, a chute attached to a vibrator of variable amplitude and a stationary funnel. The funnel was made with polished stainless steel and specially designed to be steep and glabrous enough for the purpose that no particles would aggregate in it as