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MORPHOLOGICAL CHARACTERIZATION OF MALTODEXTRIN DERIVATIVES USING SCANNING ELECTRON MICROSCOPY

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Abstract

This paper deals with the study of the significance of the morphological characteristics of some maltodextrin derivatives in the preparation of tablet dosage forms. Six maltodextrin derivatives, M-040, 100, 150, 180, 500, and 700, with varying dextrose equivalent (DE) values and varying degrees of agglomeration were studied for their suitability in the preparation of the tablets based on the morphological characteristics observed by scanning electron microscopy. A comparison of micromeritic properties, moisture absorption, and morphological characteristics of various maltodextrin derivatives is reported.

Key Words: Maltodextrin, excipients, electron microscopic evaluation.

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Introduction

Maltrodextrins ((C₆H₁₀O₅)ₙ), are non-sweet bionutritive saccharide polymers that contain a number of D-glucose units linked by α-1-4 bonds [8]. They are prepared by the hydrolysis of corn starch with appropriate acid and/or enzymes. The physical properties of maltrodextrins are mainly determined by the degree of starch hydrolysis which is represented by the dextrose equivalent (DE) values of the product. The DE value is defined as the amount of reducing sugars present and expressed as a percentage of dextrose in the dry substance. A high DE value indicates a low average degree of polymerization and a high percentage of saccharide [6]. Maltodextrins have been recognized as a safe and effective food ingredient for human consumption. Pharmaceutically, one of the maltodextrin products (Soludex™, a maltodextrin derivative) has been shown to have compressible excipient properties that are comparable to those of spray-dried lactose and microcrystalline cellulose which are extensively used as directly compressible tablet adjuvants/diluents [11]. Several maltodextrin derivatives are available which can be used as excipients in the preparation of tablet dosage forms. The knowledge and control of the particle size and the size range is of profound importance in pharmacy [9]. Clinically, the particle size of drug and/or excipient can affect the drug release from tablet dosage forms [14]. In the manufacturing of tablet dosage form, the control of particle size and the knowledge of bulk/tap densities, packing properties, flow properties, moisture affinity, morphological characteristics, etc., become necessary in achieving the proper flow and mixing of the ingredients [5]. The objective of this study is to examine the morphological characteristics of maltodextrins with various DE values and various degrees of agglomeration. The relationship between morphological characteristics of maltodextrin derivatives and micromeritic properties is also investigated.

Materials and Methods

Materials

Maltodextrin derivatives [M-040 (DE = 4), M-
100 (DE = 10), M-150 (DE = 15), M-180 (DE = 18), M-500 (DE = 10, agglomerated) and M-700 (DE = 10, superagglomerated) supplied by Grain Processing Corporation, Muscatine, IA; spray-processed lactose (Mendell Inc., Patterson, NY; methods (Foremost) supplied by Wisconsin Dairies, Baraboo, WI; and microcrystalline cellulose (Emcocel) supplied by Vankel Industries, Edison, NJ, was used to determine bulk density and tapped bulk density of maltodextrin and control (lactose and microcrystalline cellulose). Individual powder samples weighing between 10 to 30 grams (W) was transferred to the cylinders, and the initial volume was recorded (Vo). The tester was run for 100 tappings at a time, and the volume was measured. The tappings continued until a constant volume (Vc) was recorded (approximately 300 tappings). The bulk density (W/Vo) and tapped bulk density (W/Vc) were calculated. For each sample, the procedure was repeated 4 times and the results are reported as the average of the four trials. The flow properties of all the powders were studied by the cone funnel method to determine the angle of repose. The angle of repose is defined as the angle between the horizontal and the slope of a heap of particles dropped from some elevation [3]. The angle of repose was calculated and reported as the average of three trials. Micromeritic properties were studied for all the six maltodextrin derivatives with lactose and microcrystalline cellulose as controls.

Moisture absorption studies of various maltodextrins. The general method used to find the amount of moisture absorbed by maltodextrin powder was as follows: two grams of maltodextrin powder was kept at 60°C over a period of 3-4 days until a constant weight was obtained. The constant weight sample was transferred to a pre-equilibrated desiccator maintained at 100% humidity [12, 13]. After 2, 4, 8, 24, 48, and 72 hour time intervals, a sample was taken out and covered with aluminum foil (preweighed) to avoid air contact. The covered sample was weighed accurately and immediately transferred to the desiccator. The samples were run in triplicate for the maltodextrin derivatives except M-180. The reported results are average of the three samples.

Effect of moisture content on micromeritic properties of maltodextrins. Adequate quantities (approximately 10 grams) of four maltodextrin derivatives were dried to a constant weight and then transferred to desiccators pre-equilibrated for 100% R.H. At 2, 4, 8 hour intervals, moisture uptake was measured and the tapped bulk density [13] of the sample was determined by tapping the cylinder 300 times (covered with aluminum foil to avoid air contact). The angle of repose (flow properties) was determined by the fixed funnel and cone method [4]. At each time interval, samples in triplicate were used and average values are reported. Four derivatives [M-040 (DE = 4), M-150 (DE = 15), M-500 (DE = 10, agglomerated), and M-700 (DE = 10, superagglomerated)] were chosen as representative maltodextrin derivatives with different DE values and degrees of agglomeration.

Scanning electron microscopy (SEM) studies. Sample preparation for the maltodextrin derivatives was as follows: Aluminum specimen mounts were sonicated for 5 minutes in acetone to clean them. A small piece of double sticky tape was put onto the specimen mount which was then dipped into the powder sample. The samples were then sputter coated with gold/palladium using a Hummer-6 sputter coater supplied by Technics, Alexandria, VA, and viewed in a Super IIIA model SEM (International Scientific Instruments, Milpitas, CA).

Results and Discussion

Micromeritic properties

The aerated bulk density, tapping bulk density, and angle of repose values for maltodextrin derivatives are given in Table 1. It is observed that for maltodextrins, the aerated tapped densities increased with an increase in DE values. However, for M-500 and M-700 (superagglomerated), there is a significant decrease in aerated and tapped densities. M-500 and M-700 have a very fluffy nature and therefore, have lower bulk density. In addition, M-500 and M-700, both have lower angles of repose compared to the rest of the powder samples. The higher the flowability of a powder, the lower is the angle of repose. The angle of repose is a direct indication of the potential flowability of a material [3].

Moisture absorption by various maltodextrins.

It is known that the presence of moisture causes a deterioration of all powder and granular flow properties, due to the rise in internal cohesion [1]. The amount of moisture absorbed by various maltodextrins is shown in Figure 1. It is observed that the amount of moisture absorbed by various maltodextrins ranged between 6 to 16% in first 2 hours, and 36 to 48% in 48 hours for various maltodextrin derivatives (Fig 1). All the samples became a wet or pasty mass by 48 hours showing that all maltodextrin could become a pasty mass irrespective of type of maltodextrin used in 48 hours exposure to 100% relative humidity (RH). Though, initially, the M-150, M-500 products absorbed less moisture compared to M-040 and M-700, by the end of 48 hours, both agglomerated and non-agglomerated products became a pasty mass. In contrast, the controls, spray-dried lactose and microcrystalline cellulose, absorbed less than 5% and 10% moisture respectively in 48 hours without forming a pasty or wet mass showing their advantageous characteristics over maltodextrins as far as moisture uptake is concerned.

Effect of moisture absorbed on micromeritic properties of maltodextrins

The values of tapped bulk density and angle of repose for various maltodextrins are given in Tables 2 to
SEM characterization of maltodextrins

Table 1. Micromeritic Properties of Maltodextrins.

<table>
<thead>
<tr>
<th>Maltodextrin</th>
<th>Aerated BD</th>
<th>Tapped BD</th>
<th>Angle of Repose</th>
</tr>
</thead>
<tbody>
<tr>
<td>M-040</td>
<td>0.403±0.003</td>
<td>0.553±0.007</td>
<td>48.63±1.92</td>
</tr>
<tr>
<td>M-100</td>
<td>0.466±0.005</td>
<td>0.578±0.005</td>
<td>47.43±1.76</td>
</tr>
<tr>
<td>M-150</td>
<td>0.499±0.005</td>
<td>0.632±0.006</td>
<td>42.74±2.09</td>
</tr>
<tr>
<td>M-180</td>
<td>0.526±0.003</td>
<td>0.682±0.019</td>
<td>50.81±0.39</td>
</tr>
<tr>
<td>M-500</td>
<td>0.277±0.007</td>
<td>0.338±0.012</td>
<td>39.12±0.71</td>
</tr>
<tr>
<td>M-700</td>
<td>0.115±0.002</td>
<td>0.133±0.001</td>
<td>40.81±1.13</td>
</tr>
<tr>
<td>Control</td>
<td>0.696±0.009</td>
<td>0.792±0.005</td>
<td>44.09±1.40</td>
</tr>
<tr>
<td>Lactose</td>
<td>0.300±0.001</td>
<td>0.409±0.005</td>
<td>45.97±2.22</td>
</tr>
<tr>
<td>Microcrystalline Cellulose</td>
<td>0.300±0.001</td>
<td>0.409±0.005</td>
<td>45.97±2.22</td>
</tr>
</tbody>
</table>

Figure 1. Moisture absorption (%) studies of various maltodextrin derivatives and controls, lactose and microcrystalline cellulose (MCC). at 100% relative humidity.

Table 2. Effect of moisture content on micromeritic properties of maltodextrins: M-040.

<table>
<thead>
<tr>
<th>Moisture Content %</th>
<th>Tapped Bulk Density gm/cc</th>
<th>Angle of Repose (degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>0.553±0.058</td>
<td>30.29±1.50</td>
</tr>
<tr>
<td>2.79</td>
<td>0.563±0.079</td>
<td>31.30±0.80</td>
</tr>
<tr>
<td>5.71</td>
<td>0.575±0.014</td>
<td>36.90±1.75</td>
</tr>
<tr>
<td>7.70</td>
<td>0.619±0.024</td>
<td>37.40±1.25</td>
</tr>
</tbody>
</table>

Table 3. Effect of moisture content on micromeritic properties of maltodextrins: M-150.

<table>
<thead>
<tr>
<th>Moisture Content %</th>
<th>Tapped Bulk Density gm/cc</th>
<th>Angle of Repose (degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>0.632±0.050</td>
<td>31.00±1.70</td>
</tr>
<tr>
<td>3.85</td>
<td>0.658±0.078</td>
<td>34.31±2.15</td>
</tr>
<tr>
<td>6.91</td>
<td>0.675±0.032</td>
<td>35.91±1.25</td>
</tr>
<tr>
<td>8.75</td>
<td>0.692±0.045</td>
<td>37.40±1.17</td>
</tr>
</tbody>
</table>

Table 4. Effect of moisture content on micromeritic properties of maltodextrins: M-500.

<table>
<thead>
<tr>
<th>Moisture Content %</th>
<th>Tapped Bulk Density gm/cc</th>
<th>Angle of Repose (degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>0.338±0.015</td>
<td>31.00±1.10</td>
</tr>
<tr>
<td>4.48</td>
<td>0.350±0.025</td>
<td>30.50±2.15</td>
</tr>
<tr>
<td>5.07</td>
<td>0.370±0.052</td>
<td>31.29±1.75</td>
</tr>
<tr>
<td>10.27</td>
<td>0.400±0.020</td>
<td>35.70±2.70</td>
</tr>
</tbody>
</table>

Table 5. Effect of moisture content on micromeritic properties of maltodextrins: M-700.

<table>
<thead>
<tr>
<th>Moisture Content %</th>
<th>Tapped Bulk Density gm/cc</th>
<th>Angle of Repose (degrees)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>0.133±0.001</td>
<td>41.15±1.25</td>
</tr>
<tr>
<td>5.92</td>
<td>0.150±0.015</td>
<td>43.25±2.20</td>
</tr>
<tr>
<td>6.99</td>
<td>0.160±0.004</td>
<td>47.19±2.15</td>
</tr>
<tr>
<td>14.40</td>
<td>0.190±0.010</td>
<td>50.20±1.10</td>
</tr>
</tbody>
</table>
When the dry powder is exposed to moisture, water molecules are first adsorbed onto the surfaces to form a monomolecular layer and then a second layer, or a multilayer, forms which prevents the evaporation of water from beneath it [7]. The monomolecular layer is subject to both surface binding and diffusional forces, the latter tending to cause moisture transfer into the material [2, 15]. Since M-500 and M-700 powder particles have hollow spaces and openings as shown in Figures 6 and 8, the water, therefore, easily diffuses into these particles causing softening of the particle structure and could cause other adverse effects [2].

Morphological characteristics

The scanning electron micrographs of maltodextrin derivative M-040 and M-100 are shown in Figures 2 and 3, respectively. It is observed that both these derivatives with DE values of 4 (M-040) and 10 (M-100) have particles of different shapes and sizes with no definite characteristics. The particles have a very high affinity for moisture so there are some particles sticking to each other.

A scanning electron micrograph of a single particle of M-180, a maltodextrin derivative with a DE value of 18, is shown in Fig. 4. It is observed that this particle is perfectly spherical with a smooth surface. Most of the particles in this powder have been found to have spherical shapes which is advantageous for their application as tablet excipients.

Maltodextrin is marketed in two different agglomerated forms, M-500 agglomerated (DE = 10, Figures 5 and 6) and M-700 superagglomerated (DE = 10, Figures 7 and 8). Figures 5 and 7 show the powder particles of M-500 and M-700, respectively, while Figures 6 and 8 show individual particles of M-500 and M-700.

It is observed that M-500 particles are much larger in size than M-100 particles (Fig. 3). The particles seem to be fused with each other forming larger particles. Again, the particles do not show any specific size or shape. The individual agglomerated particle, when viewed at higher magnification (Fig. 6), shows evidence of several particles being fused together. None of the particles retain their original shape. The agglomeration process leads to higher particle size and significantly affects the tapped bulk density of these particles as compared to M-040, M-100, or M-150 (Tables 2, 3, 4). The process improves the flow properties of this maltodextrin derivative.

In the case of the superagglomerated maltodextrin product M-700, significant differences in micromeritic properties have been observed. The scanning electron micrographs in Figures 7 and 8 showed that most of these particles are hollow in nature. The particles have close to spherical shapes and are linked with each other at one peripheral point. Subsequently, other particles aggregate around its periphery forming a bigger particle, but its hollow structure is retained during the process.

Most of the particles have surface holes indicating the possibility that particles attached at that point might have been dislodged. There are several smaller individual particles with holes representing those particles which may have been dislodged from bigger particles during handling or processing. Figure 8 shows an individual particle of M-700 indicating fusion of 2 or 3 spherical particles with at least three holes from where 3 particles might have been dislodged. The big particles seem to be hollow and there is another smaller particle peeping out of his home. These hollow and fluffy particles significantly decreased the tapped bulk density from 0.51 to 0.13 gm/cc. The volume occupied by these particles is enormously higher than same weight of M-040, M-100, M-150, or M-180. There is a decrease in the flow properties after superagglomeration (for M-700) which might be attributed to higher surface area available for interaction with moisture in air during testing leading to higher angle of repose values. M-500 and M-700 have very

Figure 2. Scanning electron micrograph of M-040 (DE = 4) powder showing particles with different shapes and sizes. Bar = 100 µm.

Figure 3. Scanning electron micrograph of M-100 (DE = 10) showing individual particles of different sizes and shapes not interlinked with each other. Bar = 100 µm.
SEM characterization of maltodextrins

**Figure 6.** Scanning electron micrograph of a single agglomerated particle of M-500 agglomerated maltodextrin derivative with DE = 10. The bigger particles show interlinking of several particles with different shapes and sizes. Bar = 10 µm.

**Figure 4.** Scanning electron micrograph of a single particle of M-180 (DE = 18) showing spherical shape and smooth surface of the particle. Bar = 10 µm.

**Figure 5.** Scanning electron micrograph of M-500 agglomerated (DE = 10) showing agglomerated particles of different shapes and sizes are linked together to form bigger particles. Bar = 10 µm.

**Figure 7.** Scanning electron micrograph of M-700 superagglomerated derivative with DE = 10. The agglomerated particles show a hollow-empty particle closer to spherical shape linked with each other at one of the peripheral points. Bar = 100 µm.

**Figure 8.** Scanning electron micrograph of a single particle of M-700 superagglomerated derivative with DE = 10. The two magnified particles show a hollow space inside in which another spherical particle is located. The holes on the surface represent the sites of attachment from which other particles might be dislodged. Bar = 100 µm.
high moisture absorption values (Fig. 1) which again can be attributed to significantly higher surface areas for interaction with moisture.

The scanning electron micrographs of maltodextrins show interesting morphological properties which can be correlated with their micromeric and compressional properties. The micrograph of M-040 shows several particles with uneven surfaces and the particles appear to be dense. No hollow structures have been seen. This is reflected in its micromeric properties with high values of aerated and tapped bulk densities and higher value of angle of repose showing improper flow properties. The angle of repose is a parameter used to express frictional forces between particles and the surface on which these are flowing. Higher angle of repose reflects the presence of uneven particles which is confirmed by SEM characterization. The flow properties affect the compressional properties of powders (5). In the case of M-500 and M-700, which are agglomerated and superagglomerated products of M-100 with DE = 10, there was improvement in flow properties with lower values of angle of repose. During the superagglomeration process, spray-drying technique is used and hot air, which is blown-in during the process, creates hollow structures in the particles. The micrograph of M-700 clearly shows such structures and it appears that particles become attached to each other forming an agglomerate. Due to their fluffy nature, the particles have very high specific surface values (surface area per unit weight). These morphological properties clearly explain the significantly low values of aerated and tapped bulk densities (Table 1). The angle of repose values for these two maltodextrin derivatives are lower as compared to M-100 (non-agglomerated DE = 10 maltodextrin) or other maltodextrin derivatives. The lower value of angle of repose shows improvement in flow properties and lowering of frictional forces. It can be attributed to the smooth surface and spherical shape of the particles and agglomerates formed during the superagglomeration process. The morphological studies clearly shows a correlation between surface characteristics and micromeric properties such as bulk densities, angles of repose, and specific surfaces. Bulk density and angle of repose affects the compressibility of powders [5]. We have discussed the micromeric and compressional characterization of maltodextrins elsewhere [10]. Maltodextrins have very useful compressional properties but due to their high affinity for moisture might pose processing and stability problems. The agglomeration and superagglomeration processing of maltodextrins increased their moisture absorption. Nevertheless, with their directly compressible excipient properties, these need further detailed evaluation to be recommended as tablet excipients, and moisture interaction needs to be studied carefully before use.

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References


Discussion with Reviewers

E. Mathiowitz: Why do you get hollow structures for some of the particles (Fig. 6)?

Authors: The super agglomeration process involves spray-drying of the maltodextrin slurry. The particles, during the processing, become fluffy, spherical and get attached to each other forming agglomerates. Due to the voids, the bulk density is significantly reduced and powder becomes light and fluffy. The hollow structures are due to expansion of air trapped within the particles during processing.

M. Rosenberg: Would it be correct to say that DE values have no effect on bulk density of agglomerated dextrins?

Authors: Yes, the bulk density can be altered by agglomeration processes using spray-driers. DE values show no correlation with bulk density of agglomerated products.