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# THE EFFECT OF AGGLOMERATION METHODS ON THE MICROMERITIC PROPERTIES OF A MALTODEXTRIN PRODUCT, MALTRIN 150™

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## ABSTRACT

Maltrin M150 is a fine powder of maltodextrin which is a carbohydrate product made by controlled hydrolysis of corn starch. Agglomerated Maltrin was prepared using a fluidized bed granulation process and a roller compaction method, respectively. The micromeritic properties of these two granular products were compared. Three different sizes of granules (20/30, 40/50 and 80/100 mesh size) were used in the evaluation. Granules produced by the fluidized bed method showed a relatively low bulk density as compared to the roller compacted granules.

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As the granule size was reduced, the roller compacted granules showed a decrease in bulk density while an increase in bulk density was seen in the fluidized bed granulated product. A better flowability of the roller compacted granules was demonstrated by a higher flow rate and a lower compressibility index. For a given compression pressure, roller compacted granules produced compacts with a lower tensile strength. A significant work-hardening effect was exhibited by the roller compacted product.

## INTRODUCTION

Direct compression is a widely used method for tablet manufacturing. The advantages of this method over wet granulation have been well documented[1]. The important role of a direct compression tablet excipient in this method has also been discussed[1]. To be an ideal excipient for direct compression the material should possess the attributes of flowability and compressibility[2]. Therefore, the selection of an adequate agglomeration method in the production of a directly compressible tablet excipient would be critical to impart both compressibility and flowability to the final product. Maltrin M150 is a fine maltodextrin powder with poor flowability. In order to develop a maltodextrin product for direct compression a granular form would be more acceptable. Agglomerated products were prepared by a fluidized bed method and a roller compaction process respectively. The purpose of the present study is to investigate the influence of the agglomeration process on the micromeritic properties of the granular products. The influence of granule size on these properties was also investigated.

## EXPERIMENTAL

### Materials

Maltrin M150 and M550 were supplied by Grain Processing Corporation, Muscatine, Iowa. Maltrin M550 is an agglomerated form of maltrin M150 produced by a fluidized bed granulation method using water as the binding agent.

### Methods

Roller Compaction - Maltrin M150 was roller compacted using a roller compactor[(a)] with an air pressure of 30 psi, roller speed at 7 rpm, horizontal feed setting at 1.6 and vertical feed setting at 1.4. Granules were formed by breaking the compacts through an oscillating granulator[(b)] equipped with a 20-mesh stainless screen. From the roller compacted product and the fluidized bed granulated Maltrin M550, the 20/30, 40/50 and 80/100 mesh size granules were obtained by mechanically sieving the granules through a stack of standard sieves.

Physical Characterization of Granules - The bulk density was determined by pouring a sample into a 100 ml cylindrical graduate and then measuring the volume occupied by the weight of the sample. The tapped density was determined using a motorized tap density tester[(c)] set to operate for 200 cycles. The compressibility index was subsequently calculated[3].

The flow rate of the granules through a circular orifice 1.5 cm in diameter was measured using a recording powder flowmeter[4]. 100 grams of sample were used for each measurement.

The moisture content of the granules was determined by drying the samples at 105°C under vacuum for 24 hours.

Powder Compression and Compact Tensile Strength - A weighed quantity of granules was compressed using the Carver Press[(d)] equipped with flat 1/2" punches and die which were prelubricated with a 5% magnesium stearate suspension in chloroform. The duration for compression at a given pressure was 5 seconds. Five replicate compacts were prepared at a range of compression pressures. The weight of the compacts was determined (±0.001 g) and the dimensions of the compacts were measured (±0.01 mm) using a hand micrometer 24 hours after compression. The apparent density of the compacts was subsequently computed and the relative density was given by the ratio between the apparent density of the compact and the particle density of the material which was determined using an air comparison pycnometer[(e)]. The porosity of the compact was computed using the relative density of the compact. A plot of the calculated compact relative density against the applied pressure was obtained using the Heckel relationship[5,6]. The slope of the linear portion of the plot was determined using the least square linear regression method. The reciprocal of the slope is referred to as the mean yield pressure of the material. The force which caused the tensile failure of the compact under diametrical compression was measured using a stress-strain analyzer[7]. The tensile strength of the compact was subsequently calculated[8].

## RESULTS AND DISCUSSION

Some physical characteristics of the two agglomerated products are listed in Table 1. The roller compaction was found to produce granules with a much higher bulk density as compared to the fluidized bed granulation. Figures 1 a,b and 2 a,b show the morphology of granules prepared by these two methods. A high degree of densification was seen in the roller compacted granules which exhibited a very low degree of intragranular porosity. The fluidized bed method produced highly porous granules which were formed by the binding of individual particles through crystalline bridge formations of the water soluble saccharides. Due to the random nature of the agglomeration and fluidization process, the fluidized bed granules are very irregular in shape and extremely rough on the surface. The effect of granule size on bulk density varies depending on the method of granule preparation. In the case of fluidized bed granules, as the granule size was reduced a closer packing of the smaller granules in the sample resulted in a decrease of intergranular void space and an increase in bulk density (Table 1). However, a reduction in the size of the roller compacted granules resulted in a lower bulk density. This is probably due to an increase of the intergranular void spaces in the sample as a result of breaking down the highly compacted large granules.

For both types of granules an excellent to good flowability was predicted from the value of the compressibility index that was determined[3]. A marked difference in flowability between these two types of granules was shown by the flow rate determined using a flowmeter.

A

B

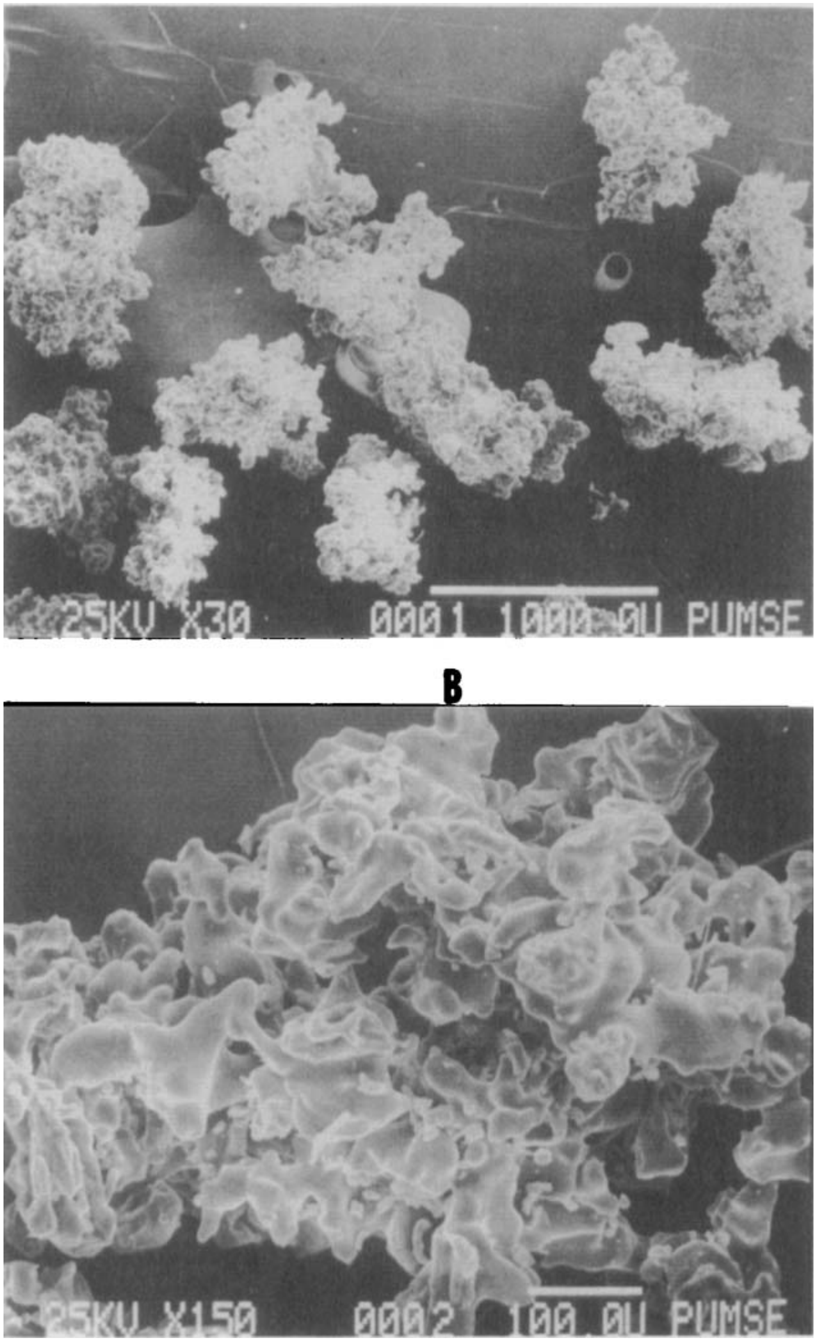


FIGURE 1

SEM photomicrographs for the fluidized bed granules. Key: Magnification (A) x30 and (B) x150.

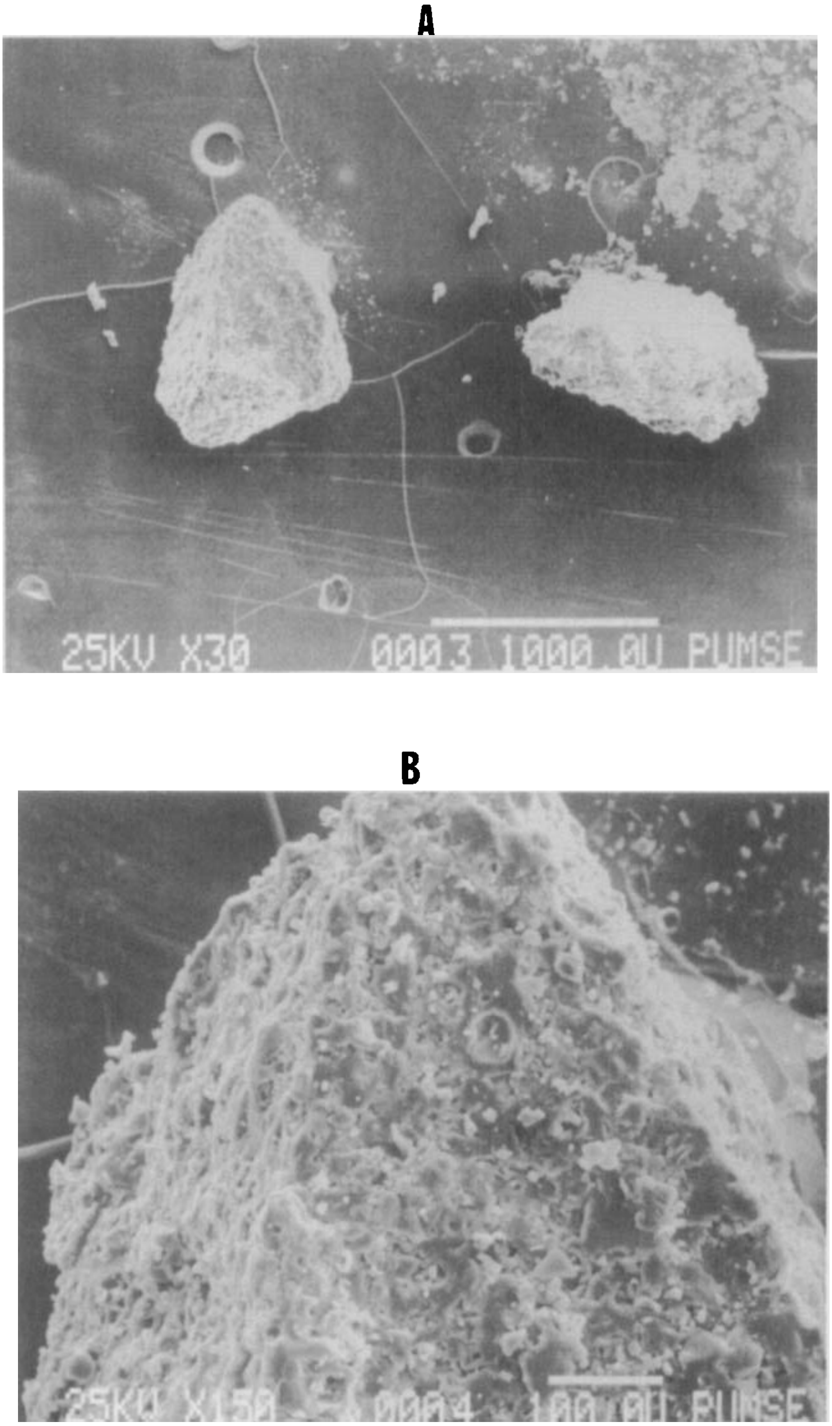


FIGURE 2

SEM photomicrographs for the roller compacted granules. Key: Magnification (A) x30 and (B) x150.

TABLE 1

Physical Characterization of Granules

| | Method of Agglomeration | | | | | |
|---|---|---|---|---|---|---|
| | Fluidized Bed | | | Roller Compaction | | |
| Granule Size (Mesh Size) | 20/30 | 40/50 | 80/100 | 20/30 | 40/50 | 80/100 |
| (Mean Size, μm) | 717.5 | 358.5 | 163.0 | 717.5 | 358.5 | 163.0 |
| Moisture Content* (% w/w) | 2.8 | 3.0 | 2.9 | 3.1 | 2.9 | 2.8 |
| Bulk Density* (g/cc) | 0.2871 | 0.3046 | 0.3659 | 0.6743 | 0.6317 | 0.4762 |
| Tapped Density* (g/cc) | 0.2941 | 0.3261 | 0.4226 | 0.6897 | 0.6570 | 0.5401 |
| Compressibility* Index (%) | 2.4 | 6.6 | 13.4 | 2.2 | 3.9 | 11.8 |
| Flow Rate* (g/sec) | 7.82 | 9.03 | 12.14 | 28.42 | 27.73 | 21.20 |
| (cc/sec) | 27.24 | 29.65 | 33.18 | 42.15 | 43.90 | 44.52 |

* The mean of three measurements.

The roller compacted granules exhibited a better flowability than the fluidized bed granules in terms of gravimetric flow rate and volumetric flow rate. For the fluidized bed granules a reduction in granules size resulted in an increase in both flow rates. The roller compact granules yielded a higher gravimetric flow rate with a larger size but a higher volumetric flow rate was associated with a

TABLE 2

The Mean Yield Pressure for the Granules

| Granule Size (Mesh) | Mean Yield Pressure ($Kg/cm^2$) Fluidized Bed | Roller Compaction |
|---|---|---|
| 20/30 | 1234.6 (0.9929)* | 1459.9 (0.9893) |
| 40/50 | 1215.8 (0.9975) | 1419.5 (0.9918) |
| 80/100 | 1243.9 (0.9981) | 1426.2 (0.9954) |

* The Correlation Coefficient

smaller granular size. It appears that in addition to the granule size, the bulk density of the granule also has a positive effect on the granule flow rate.

Table 2 lists the mean yield pressure of the granules. A higher yield pressure was determined for the roller compacted granules indicating that the material becomes more resistant to deformation after the roller compaction. The influence of granule size on the yield pressure appears to be insignificant.

Figure 3 shows the tensile strength of compacts formed with different size fractions of granules under a range of compression pressures. Stronger compacts were formed with the fluidized bed granules, the strength increasing with decrease in granules size. The compression of the roller compacted granules gave rise to compacts with lower tensile strength. This loss of compressibility can be explained in terms of work-hardening which has been

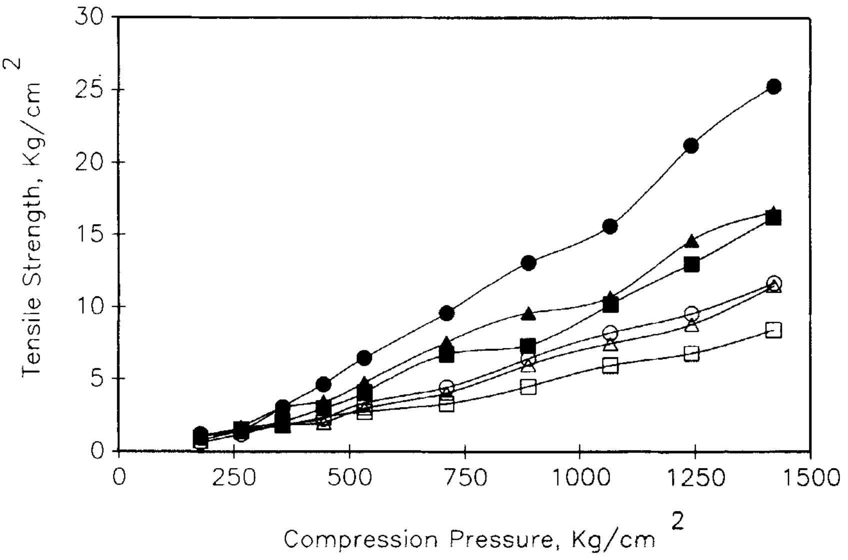


FIGURE 3

The relationship between compression pressure and tensile strength of maltodextrin compacts prepared with various granules. Key: Fluidized bed granules: (■) 20/30 mesh, (▲) 40/50 mesh, and (●) 80/100 mesh; Roller compacted granules; (□) 20/30 mesh, (△) 40/50 mesh, and (○) 80/100 mesh.

reported for a variety of tablet excipients[9,10]. This effect is further illustrated by the finding that for the same porosity of the compact, the roller compacted granules achieved a lower compact tensile strength (Figure 4). The fluidized bed granules were formed mainly by crystal bridging and the particles were not subjected to extensive stress during the process; thereby the compressibility of the material was still retained by the product. A relationship between the granule size and the compact tensile strength has been noted (Figures 3 and 4). The influence of granule size on the compact tensile strength may be due to the difference in granule crushing strength which is a function of the granule

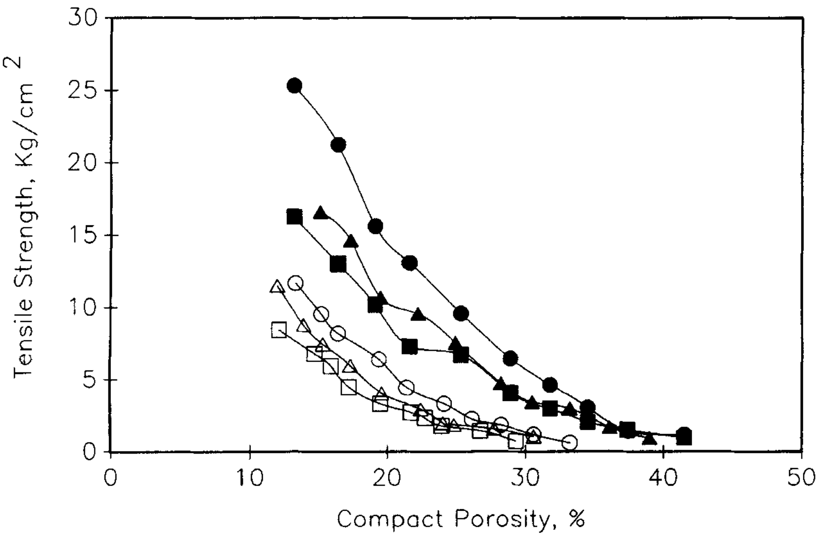


FIGURE 4

The relationship between porosity and tensile strength of maltodextrin compacts prepared with various granules. Key: Fluidized bed granules: (■) 20/30 mesh, (▲) 40/50 mesh, and (●) 80/100 mesh; Roller compacted granules: (□) 20/30 mesh, (△) 40/50 mesh, and (○) 80/100 mesh.



size[11]. Granules of a larger size were more resistant to deformation under compaction; thereby less interparticulate bonds were formed within the compact which consequently exhibited a lower tensile strength.

## CONCLUSION

Two agglomeration methods have been evaluated for the production of a granular maltodextrin product. Roller compaction produced very dense granules and the fluidized bed method yielded granules with a very low bulk density. Since extensive deformation took place for the maltodextrin powder during roller compaction a marked reduction in compressibility was shown by the roller compacted granules. This work-hardening effect caused a significant increase in the granule yield pressure and a pronounced decrease in compact tensile strength. Granules produced by the fluidized bed method remained highly compressible. In conclusion, fluidized bed granulation is an acceptable method for the production of granular maltodextrin. Roller compaction of maltodextrin should be used for producing dense granules but it is noted that a reduction in compressibility is possible.

## ACKNOWLEDGMENT

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## NOTES

(a) Chilsonator, Model DM, The Fritzpatrick Company, Elmhurst, Illinois.

(b) Stokes Oscillating Granulator, Model 43A, F.J. Stokes Machine Company, Philadelphia, Pennsylvania.

(c) Vanderkamp Tap Density Tester, Van-Kel Industries, Inc., Chatham, New Jersey.

(d) Carver Laboratory Press, Model C, Fred S. Carver, Inc. Menomonee Falls, Wisconsin.

(e) Beckman Air Comparison Pycnometer Model 930, Beckman Instruments, Inc., Fullerton, California.

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