Drug Development and Industrial Pharmacy, 24(7), 613–621 (1998)

RESEARCH PAPER

Effect of Compression Speed and Pressure on the Physical Characteristics of Maltodextrin Tablets

M. C. Monedero, M. R. Jiménez-Castellanos,*
M. V. Velasco, and A. Muñoz-Ruiz

Dpto. Farmacia y Tecnología Farmacéutica, Facultad de Farmacia, C/ Tramontana s.n. 41012, Sevilla, Spain

ABSTRACT

The present paper studies the effect of applied pressure (0–300 MPa) and compression speed (8 and 40 cycles/min) on the physical characteristics of four varieties of maltodextrins for direct compression. The materials were tableted by using a single-punch tablet machine. On the basis of the mechanical properties it seems to be reasonable to propose a limit in the plastic deformation and consequently bonding between particles. This limit could be approximately 90 MPa, and it was almost independent of the variety compressed. Disintegration behavior and, most probably, release properties are related with this limit; i.e., mechanical parameters and disintegration time increased as applied pressure was increased up to this limit. Above this limit, no differences of note were found. The different movement of particle layers in the single-sided eccentric profile led to the computation of differences between upper and lower tablet hardness surfaces, which were indicative of a consolidation mechanism. The differences obtained were indicative of the plastic deforming nature of maltodextrins.

INTRODUCTION

It is well known that some tablet formulations are susceptible to changes in the speed at which they are compressed and that this may lead to difficulties in the production of tablets (1). The time-dependent phenomena including the punch velocity effect, are important in the compaction of solids because of the time-dependent nature of plastic deformation, which is necessary to obtain tablets with desirable properties (2).

The studies of the effect of speed of applied pressure on the physical properties of materials are related to

*To whom correspondence should be addressed.

breaking strength of plastic and fragmentating materials. The breaking strength increases when the punch velocity decreases; for plastic materials, this is mainly because of time dependence (3–5). It has also been demonstrated that the axial recuperation of tablets increases when the punch velocity decreases (6,7).

Marshall et al. (7) studied the effect of compression speed and applied pressure on a drug (ibuprofen) using a compaction simulator. The density and breaking strength increased and the porosity of tablets decreased when the punch velocity decreased. These results show that the consolidation mechanism for this material is a balance between elastic and plastic deformation, depending on the principal mechanism of punch velocity.

In previous works we found that the thickness of maltodextrin tablets decreased as tablet strength was increased from 4 to 8 kp. In the case of Maltrin® QDM 500, this decrease became more apparent as breaking strength increased from 6 to 8 kp. Also, all tablets showed clear dependence on the friability with breaking strength. However, the disintegration properties of pure maltodextrin compacts were, in general, short (8). Although, Maltrin QDM 500 was a more stress-dependent material than M 510, the latter on the basis of differences of yield pressure values between Heckel tablet-in-die and ejected-tablet methods, showed an instantaneous rate of stress relaxation close to the rate of the slow decompression stress–time profile performed (9).

The present paper studies two effects, compaction speed and applied pressure, on the physical characteristics of four varieties of maltodextrins using a single-punch tablet machine.

MATERIALS AND METHODS

Maltrin is the registered trademark for a family of maltodextrins composed of water-soluble glucose polymers obtained from the reaction of starch with acid and/or enzymes in the presence of water. The hydrolysis results in a carbohydrate mixture of various saccharides with a dextrose equivalent of less than 20 (10).

Four direct-compression excipients were used: maltodextrin Maltrin M 150, batch P-1291; M 510, batch A3533; QDM 500, batch 083916V; and QDM 550, batch 357-96 (GPC, Muscatine, IA). All excipients were stored under controlled humidity conditions (relative humidity [RH] = 40%) and room temperature (20°C) before use.

Tablets were prepared by using a single-punch tablet machine (Bonals, AMT 300, Spain) at six pressures between 0 and 300 MPa to produce 200-mg tablets. The device was equipped with a forced feeding system. For every pressure, two compression speeds were used (8 and 40 cycles/min). Tablets were compressed after the die and punches were lubricated with a chloroformic solution of stearic acid (5% w/v).

The physical tests were performed according to European Pharmacopoeia II. The weight uniformity of the tablets was determined using Mettler AE 100 analytical balance (Mettler Instruments, Greinfesee, Switzerland). The weight data from the tablets were analyzed for sample mean, standard deviation, and coefficient of variation. Breaking strength of tablets was determined on 10 tablets using a commercially available tester (Schleuniger-2E, Dr. K. Schleuniger, Geneva, Switzerland). In every case the tensile strength value was calculated according to (11)

$$\sigma = \frac{2P}{\pi D t}$$

where P = breaking force, D = diameter of tablet, and t = thickness of tablet.

The individual crown-to-crown measurement for the thickness and diameter of 10 tablets was determined using a micrometer (Mitutoyo 102-101, Tokyo, Japan). Friability was evaluated from the weight loss of 20 tablets tumbled at 100 revolutions in an Erweka TA-3 (Erweka, Heusenstamm, Germany) friability tester. Disintegration testing (six tablets) was performed at 37°C in HCl 0.1 N medium using an Erweka ZT-3 apparatus without disks.

The diameter of the Vickers indentation was measured using a Zwick 3212 hardness testing machine (Norma UNE, Spain, 1986). Preliminary tests were performed to select an applied force that ensured a permanent indentation in the tablet surface. A load of 1 kg was selected to obtain the indentations in tablet surfaces. For Maltrin QDM 500 at lower applied pressures, the load was reduced to 0.5 kg. These tablets, which were weaker than the others, need lower load to produce similar indentation sizes. Contact time between the indentor and tablet surface was fixed at 10 sec.

Three indentations were made in the upper (U) and lower (L) surfaces, and two indentations were made in the side of each tablet. Three tablets were measured in each tablet batch.

RESULTS AND DISCUSSION

Table 1 shows the values of weight uniformity of the tablets compressed at two different compression speeds. Although in general, the tablet weight variation was low, Maltrins M had lower values than Maltrins QDM. This

Table 1

Weight Uniformity (mg), Standard Deviations, and Coefficients of Variation (%) of Different Batches of Maltodextrin Tablets Obtained at Two Compression Speeds (cycles/min)

	Speed	30 MPa	60 MPa	90 MPa	120 MPa	150 MPa	300 MPa
M 150	8	196.7	196.2	199.4	197.6	197.6	197.4
		(1.2)	(1.5)	(1.4)	(3.3)	(1.2)	(2.3)
		0.61%	0.76%	0.70%	1.67%	0.60%	1.16%
	40	197.7	196.6	197.6	197.7	198.1	197.4
		(2.2)	(1.8)	(0.6)	(1.2)	(1.3)	(1.0)
		1.11%	0.91%	0.30%	0.60%	0.65%	0.50%
M 510	8	205.3	206.5	210.5	209.4	206.2	203.9
		(0.7)	(1.0)	(2.3)	(2.3)	(1.6)	(3.0)
		0.34%	0.48%	1.09%	1.09%	0.77%	1.47%
	40	204.8	206.8	206.7	205.1	205.8	203.1
		(2.4)	(1.0)	(1.9)	(1.0)	(1.4)	(0.8)
		1.17%	0.48%	0.91%	0.48%	0.68%	0.39%
QDM 500	8	217.1	222.4	217.7	208.7	211.4	203.0
		(7.5)	(5.3)	(5.1)	(2.4)	(4.7)	(2.6)
		3.45%	2.38%	2.34%	1.14%	2.22%	1.28%
	40	201.9	206.5	205.8	208.7	202.2	198.0
		(1.1)	(1.2)	(2.0)	(2.7)	(2.6)	(4.4)
		0.54%	0.58%	0.97%	1.29%	1.28%	2.22%
QDM 550	8	203.2	202.4	196.7	192.4	188.7	185.7
		(1.5)	(2.5)	(1.9)	(3.0)	(2.6)	(2.1)
		0.73%	1.23%	0.96%	1.55%	1.37%	1.13%
	40	209.1	208.0	211.6	208.9	211.8	212.9
		(2.0)	(6.1)	(1.9)	(1.3)	(1.4)	(2.6)
		0.95%	2.93%	0.89%	0.62%	0.66%	1.22%

can be explained because Maltrins QDM, which were obtained by fluid bed, presented more irregular and wrinkled particles than Maltrins M, which were obtained by spray-drying (12). Therefore, the flow of Maltrin QDM particles was more irregular, causing more dispersion of weight values (13,14).

The low height of the adjusted die for 200 mg tablet weight gave no differences of note in tablet weight for the two compression speeds in this study. Also, the differences in compression speed, which were not great enough, tended to produce tablets with similar weight. Higher height of the die and larger differences in compression speed will probably produce tablets with different weight variation, as has been reported by Fassihi et al. (15).

The thickness of maltodextrin tablets (Table 2) obtained a high compression speed was greater than the thickness of tablets obtained at low compression speed. However, the compression speed did not seen to have an important effect on thickness of tablets.

Figures 1 and 2 show friability versus applied pressure. All of the maltodextrins had satisfactory friability values: less than 1% in the range of 90–300 MPa. This supports the findings of Mollan and Çelik (14) for five varieties of maltodextrin. The friability values, as expected, were higher for tablets compressed at the fast compression speed. A characteristic behavior could be observed: the lowest friability values were obtained in the intermediate range from 90 to 120 MPa. These profiles suggest the presence of a stress limit for the bonding of maltodextrin particles, at least for the tablet surfaces that the friability test attempts to quantify.

At low compression speed, the friability of QDM varieties was lower than that of the M series. The different manufacturing processes (14) could explain the differences observed in these values.

The tensile strength values (Table 3) at high compression speed were lower than those at low compression speed, and were in agreement with Katikaneni et al. (16) results, obtained for ethylcellulose tablets. The differences, however, were smaller because of the narrow range of compression speed in the eccentric press. In general, tablet tensile strength increased with applied pressure.

Table 2

Thickness (mm) and Standard Deviations of Different Batches of Maltodextrin Tablets Obtained at Two Compression Speeds (cycles/min)

	Speed	30 MPa	60 MPa	90 MPa	120 MPa	150 MPa	300 MPa
M 150	8	3.351	3.140	2.707	2.598	2.504	2.391
		(0.006)	(0.005)	(0.021)	(0.014)	(0.008)	(0.028)
	40	3.422	3.001	2.778	2.739	2.650	2.424
		(0.031)	(0.012)	(0.010)	(0.020)	(0.020)	(0.014)
M 510	8	3.697	3.196	2.967	2.816	2.690	2.548
		(0.008)	(0.005)	(0.008)	(0.008)	(0.007)	(0.020)
	40	3.591	3.339	3.012	2.840	2.699	2.510
		(0.030)	(0.005)	(0.006)	(0.004)	(0.092)	(0.015)
QDM 500	8	3.662	3.323	3.169	2.869	2.825	2.639
		(0.006)	(0.007)	(0.007)	(0.007)	(0.006)	(0.009)
	40	3.532	3.194	2.865	2.846	2.711	2.459
		(0.003)	(0.003)	(0.003)	(0.003)	(0.007)	(0.020)
QDM 550	8	3.301	2.970	2.744	2.589	2.513	2.295
		(0.008)	(0.008)	(0.008)	(0.017)	(0.007)	(0.040)
	40	3.556	3.249	3.213	2.783	2.877	2.573
		(0.007)	(0.010)	(0.005)	(0.008)	(0.023)	(0.029)

The breaking strength of maltodextrin tablets observed by Mollan and Çelik (14) tended to increase up to a certain limit, approximately 200 MPa; above this value, tensile strength was the same or even lower than the maximum obtained at approximately 200 MPa.

The results in this paper for M 510 at low compression speed support this phenomenon. The other maltodextrins, however, showed an increase in the whole range of applied pressure (up to 300 MPa). This different behavior can be explained on the basis of the differ-

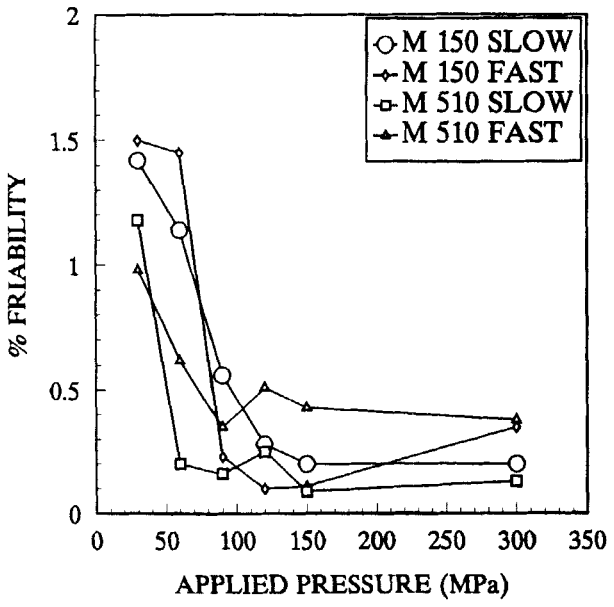

Figure 1. Friability versus applied pressure of Maltrin M.

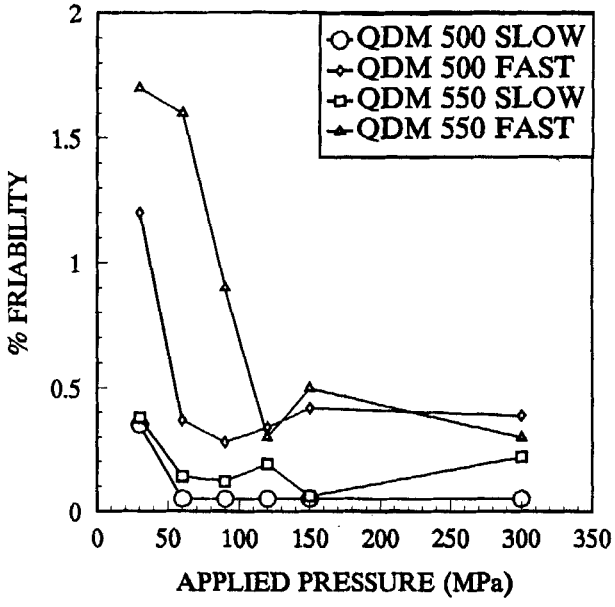

Figure 2. Friability versus applied pressure of Maltrin QDM.

Table 3

Values of Tensile Strengths (MPa) and Standard Deviations of Different Batches of Maltodextrin Tablets Obtained at Two Compression Speeds (cycles/min)

	Speed	30 MPa	60 MPa	90 MPa	120 MPa	150 MPa	300 MPa
M150	8	0.40	0.75	2.02	2.86	3.78	4.55
		(0.02)	(0.03)	(0.09)	(0.12)	(0.09)	(0.18)
	40	–	0.74	1.52	2.16	2.56	4.36
			(0.05)	(0.09)	(0.02)	(0.06)	(0.08)
M 510	8	0.27	1.44	2.58	3.53	4.77	4.52
		(0.01)	(0.02)	(0.06)	(0.08)	(0.11)	(0.16)
	40	0.36	0.93	2.10	2.62	3.67	4.83
		(0.02)	(0.03)	(0.07)	(0.05)	(0.09)	(0.21)
QDM 500	8	0.58	1.21	1.85	2.53.	2.97	–
		(0.04)	(0.05)	(0.06)	(0.07)	(0.12)	
	40	0.64	0.81	1.23	2.65	2.03	4.45
		(0.05)	(0.01)	(0.02)	(0.05)	(0.08)	(0.15)
QDM 550	8	0.51	1.83	1.90	3.22	3.63	5.05
		(0.04)	(0.09)	(0.05)	(0.08)	(0.15)	(0.17)
	40	0.29	0.88	1.71	2.49	2.24	3.67
		(0.03)	(0.05)	(0.03)	(0.11)	(0.16)	(0.09)

ent compression profiles used in both studies. Thus, Mollan and Çelik (14) used a constant displacement–time compression profile (100 mm/sec), corresponding to contact times around 50 msec. This time is significantly shorter than the one used in the eccentric press even for the fast compression speed. According to Marshall et al. (7), as the punch velocity is decreased, there is a reduction in the limiting compact porosity and an increase in the maximum tensile strength of the obtained compacts. In our case the lower punch velocity tended to increase this limit probably above the maximum applied pressure used in this study.

In relation to the mechanical properties, it seems that maltodextrin particles at tablet surfaces were more prone to show a more limited bonding or compact porosity than particles inside the tablets. The friction effect on tablet surfaces compressed without lubricant, however, could be an important factor in obtaining these results.

The effect of polymerization degree, humidity, and storage conditions could also play an important role in the tensile strength of maltodextrins used in this study (13,17).

Tables 4–7 show average indentation diameters and Vickers hardness obtained from the three indentations, one central and two laterals, at the upper (V) and lower (L) tablet surfaces. There is no clear trend in the indentation hardness values at the upper or lower tablet surface. The maximum values did not necessarily appear in the central indentation. These results support the finding of Vázquez and Romano (18) and Pesonen and Paronen (19) for several pharmaceutical materials.

The hardness is a function of the compressed material. This is especially true for microindentation measurement; in this case, microhardness can be related to parameters of crystal structure (20). Tablet hardness, however, depends basically on the degree of densification at the measurement surface. The boundary around the indentation, including nonspecific intermolecular forces as well as other types of bonds between particles in compressed powders, determines the local indentation hardness. According to our results, no differences of note were found for the hardness values at the different locations on the tablet surfaces, i.e., the upper and lower hardness values are the average of the corresponding three indentations for each surface.

Theoretically, a tablet can be considered rather homogeneous, at least for axial particle layers inside the tablets (21), when tablet edges are not computed in the whole surface value. Tablet border is more affected by the die wall friction, which can disturb the hardness surface value. According to these considerations, it seems reasonable to accept the surface hardness from the three

Table 4

Values of Indentation Diameters (ID, mm) and Vickers Hardness (VH, MPa) of Different Batches of Maltrin M 150 Tablets Obtained at Two Compression Speeds (cycles/min)

Speed			30 MPa	60 MPa	90 MPa	120 MPa	150 MPa	300 MPa
8	U	ID	0.748	0.639	0.532	0.501	0.416	0.355
			(0.02)	(0.04)	(0.03)	(0.04)	(0.03)	(0.01)
		VH	17.53	24.17	34.90	39.66	57.61	77.99
			(1.01)	(2.92)	(3.88)	(6.19)	(9.13)	(6.09)
	L	ID	0.738	0.642	0.533	0.527	0.397	0.355
			(0.04)	(0.533)	(0.03)	(0.07)	(0.03)	(0.01)
		VH	18.10	24.07	34.62	37.00	62.81	81.87
			(2.02)	(3.21)	(3.51)	(9.67)	(8.22)	(7.32)
40	U	ID	0.764	0.561	0.560	0.507	0.466	0.360
			(0.07)	(0.04)	(0.03)	(0.04)	(0.02)	(0.01)
		VH	18.01	31.47	32.37	38.56	45.33	75.33
			(3.53)	(4.40)	(2.87)	(5.90)	(4.18)	(4.27)
	L	ID	0.787	0.539	0.554	0.504	0.452	0.359
			(0.09)	(0.04)	(0.02)	(0.02)	(0.02)	(0.01)
		VH	16.33	34.11	31.96	38.71	48.30	47.97
			(3.92)	(4.76)	(2.36)	(2.94)	(5.49)	(4.07)

indentation measurements. It should be noted that lateral opposite indentations are located at a distance of 2.25 mm from tablet edges.

As expected, indentation hardness increased as applied pressure was increased. In general, M 150 showed higher values than the M 510 and QDM 500. Maltrin QDM 550 showed an intermediate behavior. The differences between Vickers hardness of M 150 and the others diminished and applied pressure increased. In this sense, the maltodextrin is less sensitive to applied pressure. These results are also substantiated by the tensile strength values as well as previous works that demonstrated a higher tendency of M 150 to fragment (8).

When single-sided profiles are used, which are produced in the eccentric press, the differences between upper and lower surface hardness could be used to estab-

Table 5

Values of Indentation Diameters (ID, mm) and Vickers Hardness (VH, MPa) of Different Batches of Maltrin M 510 Tablets Obtained at Two Compression Speeds (cycles/min)

Speed			30 MPa	60 MPa	90 MPa	120 MPa	150 MPa	300 MPa
8	U	ID	0.764	0.633	0.586	0.525	0.508	0.453
			(0.03)	(0.02)	(0.03)	(0.03)	(0.03)	(0.03)
		VH	16.78	24.43	28.69	38.16	38.16	48.23
			(1.23)	(1.56)	(2.74)	(4.25)	(4.25)	(6.30)
	L	ID	0.957	0.682	0.580	0.533	0.494	0.485
			(0.04)	(0.02)	(0.02)	(0.02)	(0.02)	(0.02)
		VH	10.70	21.02	29.20	34.59	40.26	46.81
			(1.05)	(1.35)	(2.23)	(3.19)	(3.07)	(3.46)
40	U	ID	0.853	0.697	0.597	0.566	0.521	0.453
			(0.01)	(0.02)	(0.01)	(0.02)	(0.03)	(0.02)
		VH	13.44	20.17	27.52	30.59	36.36	47.80
			(2.46)	(1.71)	(1.24)	(1.70)	(4.00)	(3.43)
	L	ID	0.718	0.646	0.581	0.572	0.526	0.451
			(0.03)	(0.02)	(0.03)	(0.02)	(0.04)	(0.02)
		VH	19.00	23.47	29.21	30.00	35.89	48.0
			(1.28)	(2.03)	(2.89)	(2.41)	(5.08)	(3.30)

Table 6

Values of Indentation Diameters (ID, mm) and Vickers Hardness (VH, MPa) of Different Batches of Maltrin QDM 500 Tablets Obtained at Two Compression Speeds (cycles/min)

Speed			30 MPa	60 MPa	90 MPa	120 MPa	150 MPa	300 MPa
8	U	ID	0.936	0.757	0.666	0.636	0.584	0.513
			(0.05)	(0.03)	(0.02)	(0.06)	(0.03)	(0.03)
		VH	11.18	17.08	22.11	24.74	28.87	37.69
			(1.01)	(1.06)	(1.49)	(4.33)	(2.59)	(5.25)
	L	ID	1.013	0.755	0.666	0.634	0.566	0.488
			(0.09)	(0.02)	(0.02)	(0.07)	(0.04)	(0.02)
		VH	9.54	17.19	22.13	24.98	31.00	41.26
			(0.98)	(1.52)	(1.30)	(4.92)	(4.79)	(3.28)
40	U	ID	1.077	0.738	0.607	0.558	0.517	0.430
			(0.06)	(0.05)	(0.02)	(0.03)	(0.02)	(0.02)
		VH	8.44	18.35	26.70	31.70	36.80	53.33
			(1.32)	(2.01)	(1.84)	(3.72)	(3.17)	(6.16)
	L	ID	1.003	0.735	0.612	0.555	0.517	0.418
			(0.01)	(0.06)	(0.02)	(0.03)	(0.02)	(0.01)
		VH	9.73	18.10	26.25	31.96	36.79	56.00
			(1.15)	(1.64)	(2.25)	(2.88)	(2.65)	(2.79)

lish the consolidation mechanism of the materials that undergo compression (22). The movement and deformation of particle layers during single-sided compression is different. The portion of densification which results from die filling and rearrangement is higher in the case of fragmenting materials (23). The differences between these processes at the upper and lower tablet surfaces could result in a great hardness variation. Plastic deforming materials, however, resulted in more similar tablet surface hardness (22). Also, indentation hardness is an easy (although indirect) method of predicting pressure transmission and distribution in tablets during compaction. The plastic deforming materials transmitted the applied pressure more effectively and uniformly in the

Table 7

Values of Indentation Diameters (ID, mm) and Vickers Hardness (VH, MPa) of Different Batches of Maltrin; QDM 550 Tablets Obtained at Two Compression speeds (cycles/min)

Speed			30 MPa	60 MPa	90 MPa	120 MPa	150 MPa	300 MPa
8	U	ID	–	–	0.584	0.525	0.487	0.393
					(0.02)	(0.02)	(0.04)	(0.03)
		VH	–	–	28.80	35.55	42.07	64.81
					(2.38)	(2.12)	(7.75)	(13.7)
	L	ID	–	–	0.577	0.510	0.484	0.402
					(0.02)	(0.03)	(0.04)	(0.02)
		VH	–	–	29.40	37.96	42.61	61.45
					(1.52)	(4.37)	(7.80)	(9.44)
40	U	ID	–	–	–	0.521	0.544	0.406
						(0.02)	(0.05)	(0.04)
		VH	–	–	–	36.19	34.16	60.82
						(3.25)	(8.92)	(12.4)
	L	ID	–	–	–	0.510	0.528	0.379
						(0.02)	(0.03)	(0.06)
		VH	–	–	–	37.75	35.52	73.32
						(3.61)	(5.33)	(2.74)

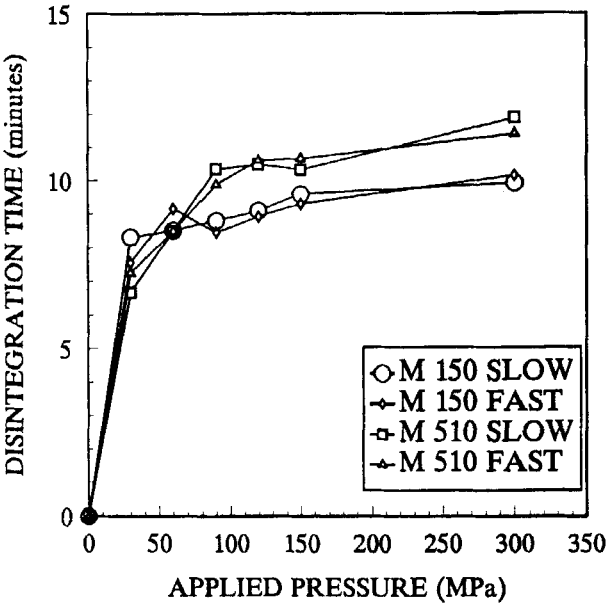

Figure 3. Disintegration time versus applied pressure of Maltrin M.

whole tablet (22). On the basis of the differences observed (Figs. 3 and 4), the maltodextrins can be considered as plastic deforming materials, with values similar to those obtained for microcrystalline celluloses and starches (24), and lower than those obtained for lactose-based direct-compression excipients (22).

Figures 3 and 4 show a linear increase between the disintegration time and applied pressure until 90 MPa. Above this pressure, the disintegration time showed only small increases. These results could be explained because the mechanism of disintegration is not controlled by the porosity of tablets, but by a gelose layer that surrounds the tablet when this is plunged into water (14). For this reason the different maltodextrins had very similar values, in spite of the different way the values were obtained.

We agree basically with this hypothesis; however, the plastic-limiting deformation of maltodextrin (9) should be attained to observe this gel formation. In the range of applied pressure up to 90 MPa, the expected behavior can be observed, i.e., disintegration times increase with an increase in applied pressures.

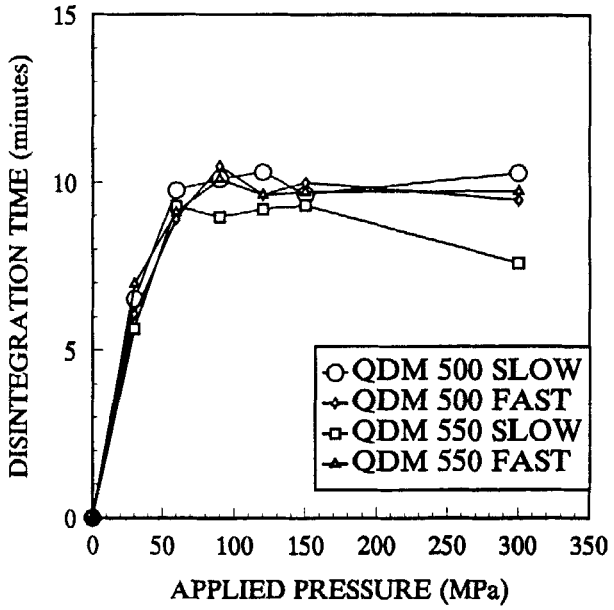

Figure 4. Disintegration time versus applied pressure of Maltrin QDM.

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