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The effects of compression rate and force on the compaction properties of different viscosity grades of hydroxypropylmethylcellulose 2208

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Abstract

The effects of compression speed and force on the compaction properties of four viscosity grades of hydroxypropylmethylcellulose 2208 (HPMC K100, HPMC K4M, HPMC K15M, HPMC K100M) have been assessed. The tensile strengths of their tablets, the energies involved in compaction, mean yield pressures, elastic recoveries and the contribution of the elastic and plastic energies to the gross energies have been evaluated using a compaction simulator. For each viscosity grade of HPMC, an increase in compression speed from 15 to 500 mm/s generally decreased the tensile strength of the tablets. The tensile strengths of HPMC K100 tablets were more sensitive to changes in compression speed than those of the other grades. Tablets of HPMC K100 had the highest tensile strength at any compression force or speed. An increase in compression speed from 15 to 500 mm/s resulted in an increase in the mean yield pressure of HPMCs. The highest elastic recoveries were found for compacts made at 500 mm/s at each viscosity grade (48.0, 28.1, 49.8 and 50.0% for HPMC K100, HPMC K4M, HPMC K15M and HPMC K100M, respectively). At each compression speed, HPMC K4M had the lowest elastic recovery. For each viscosity grade of HPMC, an increase in compression force from 5 to 10 kN resulted in an increase in elastic recovery; above a force of 10 kN the elastic recovery decreased for each HPMC except HPMC K4M. An increase in compression force and speed increased the percentage contribution of the elastic energies of the gross energies for the different grades of HPMC. For example, for HPMC K4M the percentage contribution of the elastic energies of the gross energies were 12.4 and 39.9% for compression forces of 5 and 20 kN, respectively, and for compression speeds of 15 and 500 mm/s were 16.1 and 34.2%, respectively.

Keywords: Hydroxypropylmethylcellulose 2208; Viscosity grade; Tensile strength; Compression force; Compression speed; Elastic recovery; Compaction energy; Strain rate sensitivity; Porosity

1. Introduction

The compaction properties of polymers such as cellulose ethers are important in the production of

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sustained release matrices. Even though polymers are widely used in oral sustained release drug delivery systems, the only polymer of pharmaceutical interest to be widely investigated for its compaction properties is microcrystalline cellulose (Roberts and Rowe, 1987; Udeala and Chukwu, 1989; Yu et al., 1989). Recently, investigators have studied the compaction properties of hydroxypropylmethylcellulose (Malamataris and Karidas, 1994; Malamataris et al., 1994; Nokhodchi et al., 1994, 1995a) and cellulose esters (Shivanand and Sprockel, 1992). The tensile strengths of tablets produced from different grades of HPMC of different particle sizes or methoxy/hydroxypropoxyl substitution increased with moisture content and reached a maximum at about 10% w/w moisture (Malamataris and Karidas, 1994). The particle deformation of HPMC was related to the moisture distribution which was moderated by the particle size and the methoxy/hydropropoxy substitution ratio but not to the viscosity grade (Malamataris et al., 1994). The changes in tensile strengths of HPMC tablets were related to changes in the compressional characteristics and the moisture distribution but not to the viscosity grades of HPMC (Malamataris et al., 1994). Particle size can affect compression properties of different viscosity grades of HPMC when a compression force of 10 kN and compression speed of 15 mm/s are used (Nokhodchi et al., 1995a). There is no documentation on the effect of compression speed and compression force on the compaction and compression properties of HPMC of different viscosity grades. This study examines whether changes in its viscosity grade, compression speed and compression force have any effect on the compaction properties of four grades of HPMC 2208.

2. Materials and methods

The 45–125 μm sieve fractions of four viscosity grades of hydroxypropylmethylcellulose 2208 (Methocel K100, Methocel K4M, Methocel K15M or Methocel K100M, Dow Chemicals, USA) were used. The fractions were dried at 70°C for 5 days. The true density of each viscosity grade of HPMC was determined using a Bechman air pycnometer model 930 and was calculated from a mean of five determinations.

2.1. Compression

Compression was carried out using a High Speed Compaction Simulator (ESH Testing Ltd Brierley Hill,West Midlands,U.K.) as modified at the School of Pharmacy, Liverpool John Moores University. A sawtooth time-displacement profile was used to control both upper and lower punches; 12.5 mm flat faced punches were used. Details of the simulator have been published elsewhere (Nokhodchi et al., 1995a,b,c). Constant weights of 400 mg were used for each sample. To evaluate the effects of compression force, pressures of 5, 10, 15 or 20 kN were used at a compression speed of 15 mm/s. To investigate the effects of compression speeds, speeds of 15, 140, 280 or 500 mm/s were used at a compression force of 10 kN. The die wall was cleaned with acetone and prelubricated with 4% w/w magnesium stearate in acetone before each compression. During compression, upper punch load and punch separation were monitored to an accuracy of $\pm$0.05 kN and $\pm$12 μm, respectively.

2.2. Tensile strengths

Tensile strengths of HPMC tablets were determined as described earlier (Fell and Newton, 1970; Nokhodchi et al., 1995a). The results are the means of four tablets.

2.3. Determination of porosity

Twenty four hours after ejection, the thicknesses and diameters of tablets were measured to $\pm$ 10 μm using a micrometer and the weight measured to $\pm$ 0.1 mg. The percentage porosity, ϵ, was calculated from equation 1:

$$\epsilon = [(V - V_0)/V] \times 100 \quad (1)$$

where V is the tablet volume and V_0 the volume of material at zero porosity. The results are the means of four tablets. The percentage porosities in the die at each compression force were also determined.

2.4. Energy and Heckel analysis

The manipulation of compression data and calculation of gross, plastic and elastic energies has been fully described earlier (Nokhodchi et al., 1995a). Regression analyses were carried out on the Heckel plots for data corresponding to forces between 20 and 75 MPa as described by Nokhodchi et al. (1995a). The mean yield pressures from four compressions at each of the four compression speeds were determined using the Heckel equation (Heckel, 1961). The results are therefore the means and standard deviations of four determinations.

2.5. Statistical analysis

All data were statistically analysed by two way analysis of variance and/or Tukey's multiple comparison test. Results are quoted as significant where $P < 0.05$.

3. Results and discussion

The effects of viscosity grade of HPMC and compression speed on tablet porosity are shown in Fig. 1. At each compression speed, the increase in viscosity grade resulted in an increase in the porosity of the HPMC compact. This confirms that the lower viscosity grade of HPMC can deform readily to fill inter-particulate voids (Nokhodchi et al., 1995a). Two way analysis of variance showed that this effect of viscosity grade on the porosity of tablets was significant. The porosities of matrices containing HPMC K15M or HPMC K100M were similar at all compression speeds (Tukey's test). There were no significant differences in the porosities of tablets made at 280 and 500 mm/s for each viscosity grade (Tukey's test). However, there was significant difference between the porosities of HPMC K100, HPMC K4M and the other two grades (Tukey's test).

The effects of compression speed and viscosity grade on the tensile strengths of HPMC tablets are shown in Fig. 2. For each viscosity grade, an increase in compression speed resulted in reductions in the tensile strengths of the tablets. This was probably due to there being less time available for the plastic deformation of the HPMC and hence for the formation of bonds, thus resulting in weaker compacts. It has been reported that viscoelastic materials (e.g. microcrystalline cellulose) are sensitive to compression speed, an increase in compression speed leading to a reduction in tablet strengths (Armstrong and Palfrey, 1989). These differences have been explained in terms of time of exposure to compression. Recently, Armstrong and Palfrey (1989) have shown that the primary effect of compression speed on tablet strengths was through a change in the porosity, which in turn is related to the consolidation mechanism. The decrease in tablet strengths with increased tableting speed is thus basically due to increased porosity (Fig. 1). Two way analysis of variance showed that both viscosity grade and compression speed had significant effect on the tensile strengths of the tablets. The tablets of each viscosity grade made at 15 mm/s had significantly (Tukey's test)

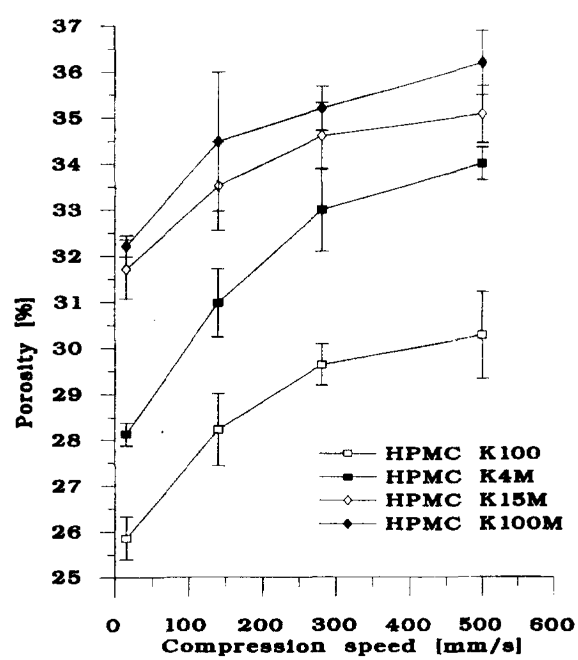

Fig. 1. The effect of compression speed on the porosity of HPMC (45–125 μm) tablets of different viscosity grades made at a compression force of 10 kN (results are the means and standard deviations of four determinations).

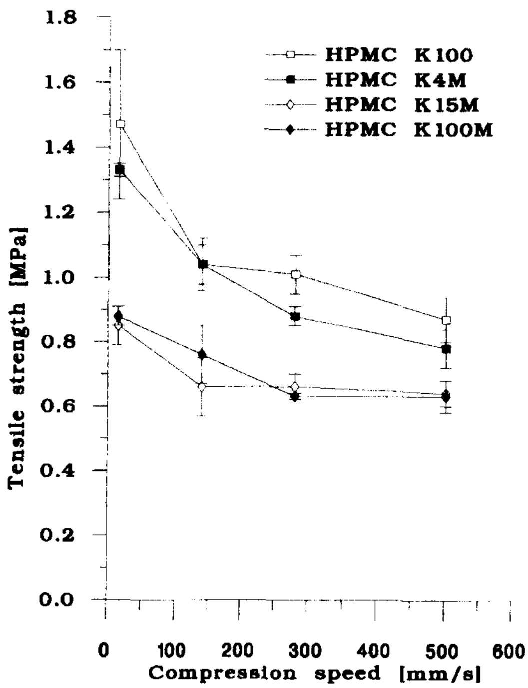

Fig. 2. The effect of compression speed on the tensile strengths of HPMC (45–125 μm) tablets of different viscosity grades made at a compression force of 10 kN (results are the means and standard deviations of four determinations).

higher tensile strengths than those made at 500 mm/s. Generally, therefore, the tensile strengths decreased with increase in compression speed. For HPMC K100 and HPMC K15M, there were no significant differences (Tukey's test) in the tensile strengths of tablets made at 140, 280 or 500 mm/s for each HPMC. On the other hand, for HPMC K4M the tensile strengths of tablets made at each compression speed could be clearly differentiated (Tukey's test). Generally, the effects of compression speed on the tensile strengths of tablets of HPMC K100 and HPMC K4M could not be differentiated from each other; neither could the strengths of HPMC K15M or HPMC K100M be differentiated (Fig. 2). Tablets containing HPMC K100 or HPMC K4M had higher tensile strengths than tablets containing HPMC K15M or HPMC K100M. Tablets with the highest tensile strengths were formed with HPMC K100 and HPMC K4M at the lowest compression speed.

Fig. 1 shows that an increase in compaction speed resulted in an increase in tablet porosity. This increase can probably be another reason for the reductions in tensile strengths with increase in compression speed. It has been shown that the viscosity grade of HPMC has no significant effect on the tensile strength of compacts in the absence of moisture at a compression speed of 1.5 mm/s (Malamataris and Karidas, 1994) but Fig. 2 indicates that the tensile strengths of HPMC tablets were indeed affected by the viscosity grade of HPMC, contrary to previous findings.

The effects of compression force and viscosity grade on the tensile strengths of HPMC compacts are shown in Fig. 3. Generally, HPMC K100 gave tablets with higher tensile strength than the other viscosity grades and the tensile strengths of tablets of all grades of HPMC increased with increase in compression force. Two-way analysis of variance and Tukey's test showed that the tensile strength of tablets made at different compression forces

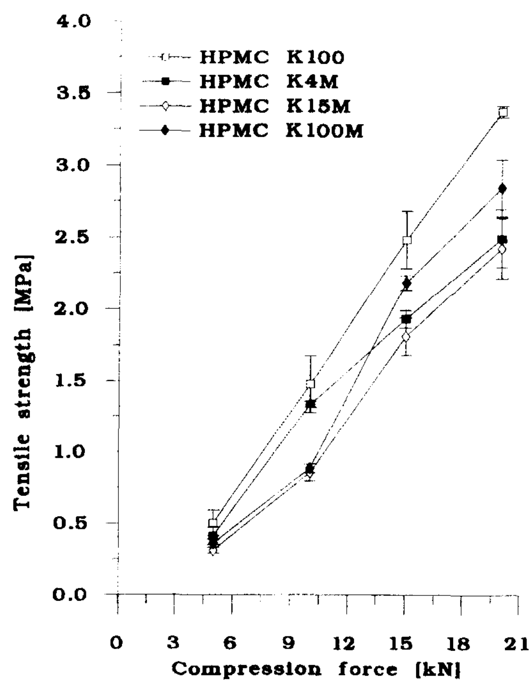

Fig. 3. The effect of compression force on the tensile strengths of HPMC (45–125 μm) tablets of different viscosity grades made at a compression speed of 15 mm/s (results are the means and standard deviations of four determinations).

Table 1
The effect of viscosity grade of HPMC (45–125 μm) on the constants A and B of equation 2 for compression forces between 5 and 20 kN and the correlation coefficients between porosity and logarithm of tensile strength

Materials	Constants		Correlation coefficient (r)
	A(1/MPa)	B(MPa)	
HPMC K100	0.089	2.59	0.996
HPMC K4M	0.090	2.59	0.985
HPMC K15M	0.100	2.94	0.998
HPMC K100M	0.098	3.04	0.999

could be clearly differentiated from each other for each grade of HPMC. The rank order of the polymers at compression forces of 15 and 20 kN was K4M ≈ K15M < K100M < K100. However, at lower compression forces (5 and 10 kN) there was no significant difference between the tensile strengths of HPMC K15M and HPMC K100M. It has been proposed that the relationship between the logarithm of tensile strength and porosity will be linear, since tensile strength is controlled by the porosity of tablets (Shotton and Ganderton, 1960). The porosities of tablets were very sensitive to compaction force. For instance, for HPMC K4M, the porosities 24 h after ejection were 38.1, 28.1, 20.3 and 18.1% for compression forces of 5, 10, 15 and 20 kN respectively. Attempts were made to fit the results of the tensile strengths of the tablets to equation 2,

$$Ln(\sigma_x) = B - A\epsilon \quad (2)$$

where σ_x is the tensile strength of tablets, A represents the slope of a straight line and B is the intercept. The regression constants and regression coefficients for relationship between porosity and the logarithm of tensile strengths for each HPMC are listed in Table 1.

Using the data listed in Table 1, the projected tensile strengths at zero porosity for HPMC K100, HPMC K4M, HPMC K15M and HPMC K100M were calculated as 13.3 ± 1.2, 13.3 ± 1.4, 18.9 ± 1.1 and 20.8 ± 1.1 MPa respectively. Even though HPMC K100 compacts had superior mechanical properties at all of the forces studied (Fig. 3), an examination of the projected tensile strengths at zero porosity would suggest that the value for HPMC K100M is higher than the other three grades.

Fig. 4 shows typical Heckel plots for each grade of HPMC. Increase in viscosity grade resulted in a decrease in relative density at any given applied pressure. This indicates that a decrease in viscosity grade facilitated powder consolidation under compression and was possibly due to an increase in plastic flow.

The effects of compression speed on the mean yield pressures of the compacts of HPMC were also investigated (Fig. 5). Two way analysis of variance showed that the mean yield pressures of the HPMCs were affected by compression speed and viscosity grade of HPMC. Generally, the mean yield pressures of HPMC increased as the compaction speed was increased up to 280 mm/s (Fig. 5). The increase in mean yield pressure with increase in the compression speed could be due to a reduction in the amount of plastic deformation as a result of the time dependent nature of plastic flow (Roberts and Rowe, 1987). Thus the time for bond formation is reduced. Alternatively, there

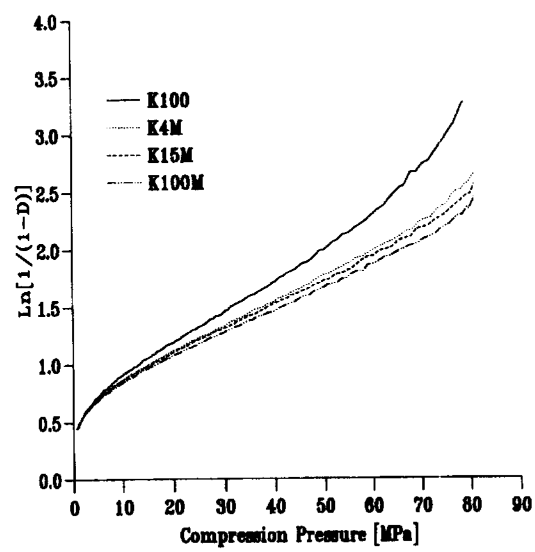

Fig. 4. The effect of viscosity grade of HPMC (45–125 μm) on the Heckel plot obtained at a compression speed of 15 mm/s.

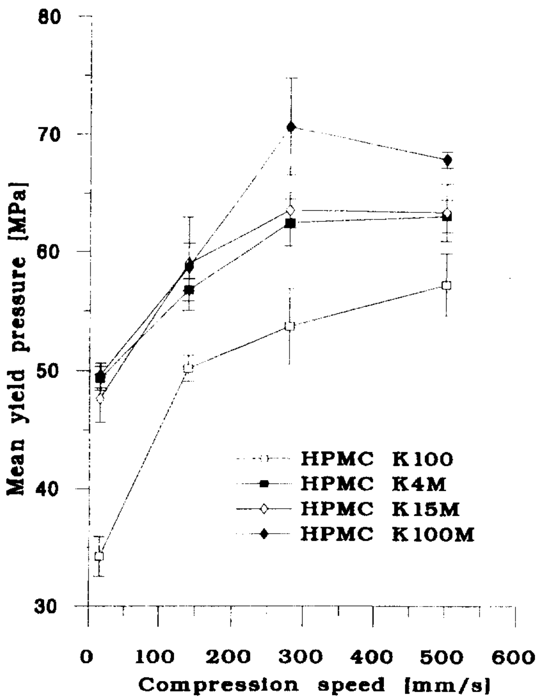

Fig. 5. The effect of compression speed on the mean yield pressures of HPMC (45–125 μm) tablets of different viscosity grades made at a compression force of 10 kN (results are the means and standard deviations of four determinations).

could be an increase in brittle behaviour (Roberts and Rowe, 1987). HPMC K100 produced tablets with mean yield pressures less than the other grades over a range of particle sizes from < 45 to 250–350 μm (Nokhodchi et al., 1995a). It appears that HPMC K100 has an optimal property in being soft enough to flow during compaction but strong enough to resist fracture when subjected to tensile strength testing at all compression speeds. The higher viscosity grades of HPMC are hard and less plastic and need higher pressure to deform. Therefore, the higher viscosity grades do not deform as readily as HPMC K100 during compaction and, for instance, HPMC K100M could not form tablets with as high tensile strengths as HPMC K100. However, Malamataris et al. (1994) reported that the mean yield pressures of HPMC tablets were not affected by viscosity grade in the absence of moisture; but these authors did not examine HPMC K100. Tukey's test showed that the mean yield pressures of HPMC K100 could be clearly differentiated from the other viscosity grades at all compression speeds in Fig. 5.

The effects of compression speed on the elastic recovery of the different HPMC grades are shown in Fig. 6. The compression speed of 500 mm/s produced the highest elastic recovery for each HPMC grade. This can be related to the tensile strength data (Fig. 2) where the lowest tensile strengths were obtained at 500 mm/s. The tablets formed at higher speeds were less able to resist the recovery of the elastic component of the material. There is the possibility that a shift in energy utilisation from plastic to elastic energy occurred with increasing compression speed. Tukey's test showed that elastic recovery was unaffected by compression speeds from 15 to 280 mm/s for each grade of HPMC. At a compression speed of 500 mm/s, the elastic recovery of HPMC compacts increased significantly (Tukey's test). In other words, the highest elastic recovery was found at 500 mm/s for each viscosity grade. HPMC K4M had the lowest elastic recovery (Fig. 6).

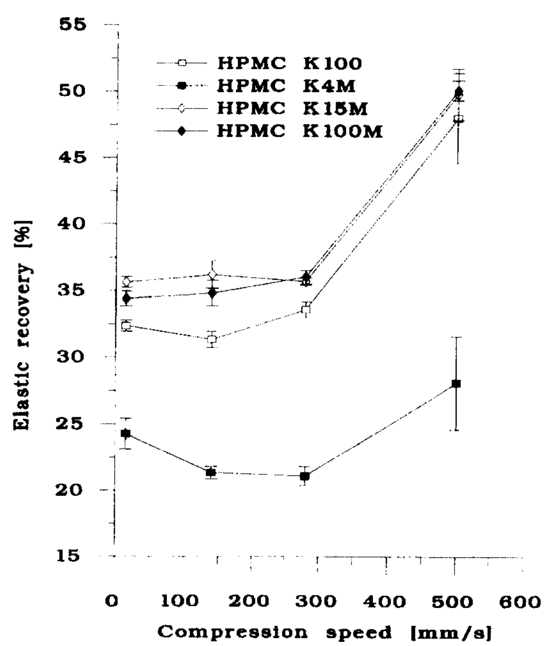

Fig. 6. The effect of compression speed on the elastic recoveries of HPMC (45–125 μm) tablets of different viscosity grades made at a compression force of 10 kN (results are the means and standard deviations of four determinations).

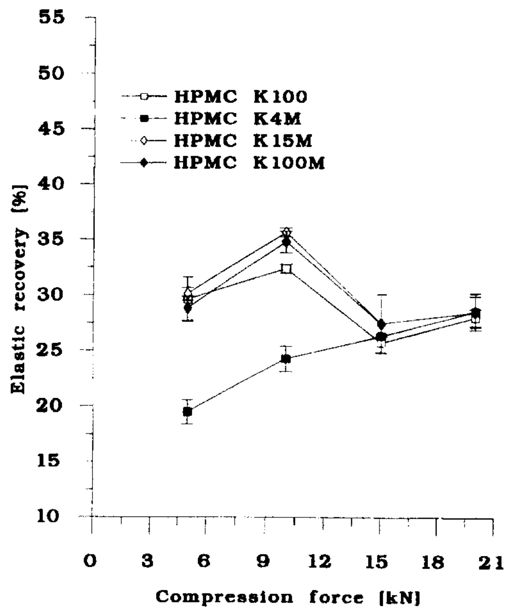

Fig. 7. The effect of compression speed on the elastic recoveries of HPMC (45–125 μm) tablets of different viscosity grades made at a compression speed of 15 mm/s (results are the means and standard deviations of four determinations).

The effect of compression force on the elastic recovery is shown in Fig. 7. The elastic recovery of HPMC compacts increased as the compression force was increased from 5 to 10kN. However, above 10 kN the elastic recovery of HPMC compacts decreased with the exception of HPMC K4M. Generally, the increase in elastic recovery can be ascribed to cellulose derivatives having a hollow microfibrillar structures (Marshall and Sixmith, 1974/1975) which do not collapse easily under pressure (Sixmith, 1977). Consequently, they exhibit a high degree of elastic deformation and recovery. However, due to numerous slip-planes and dislocations in cellulose polymers, (Lamberson and Raynor, 1976; Sixmith, 1977) they undergo extensive plastic flow and hydrogen bonding during compression. Therefore, although the elastic deformation increases to a maximum with increase in applied pressure (Fig. 7), large increases in interparticulate bonding, due to the formation of hydrogen bonds, limit the disruptive effects of elastic deformation. Thus, the elastic recoveries generally decreased for compaction forces above 10 kN. The initial increases in elastic recovery with compression force up to 10 kN were statistically significant (Tukey's test). The reductions in elastic recovery for HPMC K100, HPMC K15M or HPMC K100M at pressures above 10 kN were also significant (Tukey's test). HPMC K4M had the lowest elastic recovery in comparison with the other viscosity grades at low compression forces (5 and 10 kN), whereas at the highest compression force (20 kN) there were no significant differences between the elastic recoveries of the compacts of any of the HPMCs (Tukey's test).

Equations 3 and 4 were used to calculate strain rate sensitivity (SRS) from the slopes of the Heckel plots and the relative changes in values of tensile strength (B) of the compacts at 15 mm/s (low compression speed) and 500 mm/s (high compression speed) compression speed.

$$SRS = [(P_{y2} - P_{y1})/P_{y2}] \times 100 \quad (3)$$

$$B = [(T_{s1} - T_{s2})/T_{s1}] \times 100 \quad (4)$$

P_{y1} and P_{y2} are the mean yield pressure at low compression speed (15 mm/s) and high compression speed (500 mm/s), respectively, B is the percentage change in tensile strengths relative to T_{s1} where T_{s1} and T_{s2} are the tensile strengths of the tablets made at 15 and 500 mm/s, respectively. The results are shown in Table 2.

Materials which show higher strain rate sensitivity are those which deform plastically (Roberts and Rowe, 1985). HPMC K100, exhibited significantly higher values of SRS and B than those of the other grades. Therefore, the higher viscosity grades are less plastic than HPMC K100 (Table 2) and are consequently less sensitive to increases in compression speed.

Table 2
The effect of viscosity grade of HPMC (45–125 μm) on strain rate sensitivity (SRS) and changes in tensile strength (B) obtained at a compression force of 10 kN (the results are the means and standard deviations of four determinations)

Materials	SRS(%) ± S.D.	B(%) ± S.D.
HPMC K100	40.1 ± 4.1	41.4 ± 4.7
HPMC K4M	21.6 ± 2.1	32.9 ± 3.1
HPMC K15M	21.8 ± 2.2	23.9 ± 2.0
HPMC K100M	25.4 ± 2.1	25.2 ± 2.2

Table 3
The effect of compression force on the gross enrgies of HPMC (45–125 μm) tablets of different viscosity grades compressed at a compression speed of 15 mm/s (results are the means and standard deviations of four determinations)

Compression force	Gross energy (Joule ± S.D.)			
(kN)	HPMC K100	HPMC K4M	HPMC K15M	HPMC K100M
5	4.85 ± 0.25	4.67 ± 0.08	4.79 ± 0.14	4.53 ± 0.20
10	8.02 ± 0.11	8.02 ± 0.13	7.90 ± 0.19	7.86 ± 0.17
15	11.50 ± 0.31	10.90 ± 0.23	11.63 ± 0.34	12.05 ± 0.66
20	14.85 ± 0.59	13.59 ± 0.21	14.36 ± 0.51	13.78 ± 0.63

Table 4
The effect of compression force on the elastic energies of HPMC (45–125 μm) tablets of different viscosity grades compressed at a compression speed of 15 mm/s (results are the means and standard deviations of four determinations)

Compression force (kN)	Elastic energy (Joule ± S.D.)			
	HPMC K100	HPMC K4M	HPMC K15M	HPMC K100M
5	0.61 ± 0.05	0.58 ± 0.06	0.58 ± 0.07	0.57 ± 0.09
10	1.38 ± 0.22	1.29 ± 0.14	1.32 ± 0.24	1.18 ± 0.13
15	3.48 ± 0.37	3.34 ± 0.44	2.98 ± 0.64	2.95 ± 0.26
20	5.53 ± 0.33	5.43 ± 1.11	5.55 ± 0.65	5.24 ± 1.06

Table 5
The effect of compression force on the percentage contribution of the elastic energies to gross energies (EE/GE) of HPMC (45–125 μm) tablets compressed at a compression speed of 15 mm/s (results are the means and standard deviations of four determinations)

Compression force (kN)	Ratio of EE/GE (% ± S.D.)			
	HPMC K100	HPMC K4M	HPMC K15M	HPMC K100M
5	12.57 ± 1.03	12.41 ± 0.91	12.13 ± 0.54	12.51 ± 0.85
10	17.16 ± 1.01	16.06 ± 1.08	16.67 ± 0.96	15.03 ± 1.04
15	30.28 ± 3.20	30.64 ± 2.10	25.58 ± 0.10	24.49 ± 1.30
20	37.83 ± 2.12	39.92 ± 3.91	38.61 ± 0.50	38.01 ± 2.41

During compression, the gross energy of compaction comprises the friction between particles with the die wall, the amount of work required for the elastic and plastic deformation of the particles and for the formation of bonds between the particles. Table 3 shows the effect of compression force on the gross energy for the different viscosity grades of HPMC. An increase in compression force resulted in an increase in gross energy for each HPMC. There was little difference in the performance in each of the grades of HPMC (Table 3). However, the gross energy may be divided into two factors, the plastic and elastic energies, which permit a further analysis of behaviour during compression.

The effects of compression force and viscosity grade on the elastic energies are illustrated in Table 4. Two-way analysis of variance showed that the viscosity grade of HPMC did not affect the elastic energy. However, the elastic energies increased with increase in compression force. The percentage contribution of the elastic energies (Table 4) of the gross energies (Table 3) are shown in Table 5. It seems that with increasing

Table 6
The effect of compression force on the minimum porosity in the die during compaction of HPMC (45–125 μm) tablets of different viscosity grades compressed at a compression speed of 15 mm/s (results are the means and standard deviations of four determinations)

Compression force (kN)	Porosity (% ± S.D.)			
	HPMC K100	HPMC K4M	HPMC K15M	HPMC K100M
5	23.14 ± 0.61	24.40 ± 0.31	26.04 ± 0.85	28.64 ± 0.44
10	5.33 ± 1.05	8.01 ± 0.39	10.09 ± 0.78	11.14 ± 0.36
15	1.59 ± 0.78	3.26 ± 0.74	2.28 ± 0.63	2.65 ± 0.37
20	0.28 ± 0.34	2.12 ± 0.67	1.95 ± 0.61	2.09 ± 0.57

Table 7
The effect of compression speed on the gross energies of HPMC (45–125 μm) tablets of different viscosity grades compressed at a compression force of 10 kN (results are the means and standard deviations of four determinations)

Compression speed (mm/s)	Gross energy (Joule ± S.D.)			
	HPMC K100	HPMC K4M	HPMC K15M	HPMC K100M
15	8.02 ± 0.11	8.02 ± 0.13	7.90 ± 0.19	7.86 ± 0.17
140	10.80 ± 0.25	11.00 ± 0.33	11.15 ± 0.28	10.95 ± 0.42
280	12.99 ± 0.52	13.19 ± 0.43	12.86 ± 0.31	13.92 ± 0.39
500	13.85 ± 0.68	14.67 ± 1.08	13.62 ± 0.02	14.25 ± 0.85

Table 8
The effect of compress on speed on the contribution of the elastic energies to gross energies (EE/GE) of HPMC (45–25 μm) tablets of different viscosity grades compressed at a compression force of 10 kN (results are the means and standard deviations of four determinations)

Compression speed (mm/s)	Ratio of EE/GE (% ± S.D.)			
	HPMC K100	HPMC K4M	HPMC K15M	HPMC K100M
15	17.16 ± 1.01	16.06 ± 1.08	16.67 ± 0.96	15.03 ± 1.04
140	25.18 ± 1.95	25.36 ± 2.08	24.84 ± 0.31	26.58 ± 2.08
280	29.79 ± 1.50	29.79 ± 1.06	27.53 ± 0.09	31.47 ± 1.32
500	32.71 ± 1.65	34.22 ± 2.08	33.33 ± 0.95	33.12 ± 0.91

compression forces, the amount of elastic deformation contributing to the whole deformation process increased (Table 5), and relatively less of the total energy (gross energy) was used for the formation of bonds between the particles (De Blaey et al., 1971), because bond formation energies are implicit in the plastic energies whose contribution to the compaction process concomitantly decreased as the contribution by the elastic energies increased. Two-way analysis of variance showed that the effect of compression force on the percentage contribution of the elastic energies to the gross energies (Table 5) was significant for all grades of HPMC. The contribution of plastic energies to the compaction process may be determined by substracting the data in Table 4 from the data in Table 3. The viscosity grade of HPMC generally did not affect the plastic energy but, as with the elastic energies, increase in compression force resulted in its increase. However, the increase in compression force from 15 to 20 kN did not result in further increases in the plastic energies of HPMC K4M, HPMC K15M or HPMC K100M (Tukey's test). This indicates that the plastic energy probably no longer increases beyond the force at which the porosity of the tablet during compaction has reached a minimum (Table 6).

The effects of compression speed on the gross energies were also investigated (Table 7). Generally, the gross energies increased as the compression speed increased from 15 to 280 mm/s; further increase to 500 mm/s did not produce statistically significant increases in gross energy (Tukey's test). However, the gross energies were independent of the viscosity grade of HPMC. The contribution of elastic energies to the gross energies can be seen in Table 8. In a manner analogous to that seen for the effects of compression force on the elastic energies (Table 5), the contribution of elastic energies to the gross eneregies increased with increase compression speed. This increase in the contribution of elastic energies to gross energies with increase in compression speed was significant (two-way analysis of variance). This would be as a consequence of the reduction in time available for particle bonding due to reduction in dwell time as the speed of compression increased. As the dwell time became shorter, the stress relaxation will be correspondingly reduced and less particle-particle bonds will be formed. Obviously the contribution of the plastic energies to the gross energies concomitantly decreased although their absolute values increased. However, both the elastic and plastic energies were independent of the viscosity grade (two-way analysis of variance).

4. Conclusion

It has been reported that at a compression speed of 15 mm/s and a compression force of 10 kN HPMC K100 was the easiest of four polymers to compress (Nokhodchi et al., 1995a). This study confirms that at different compression forces from 5 to 20 kN and compression speeds from 15 to 500 mm/s, HPMC K100 is the easiest of four polymers to compress and that it displays more plasticity during compression than the other grades. However, when compacts were compared at zero porosity rather than equal forces, the highest viscosity grade (HPMC K100M) produced compacts of higher tensile strength compared to the other viscosity grades. This study also showed that the tensile strength of all viscosity grades of HPMC were affected by compression speed. The tensile strength of HPMC K100 was more sensitive to compression speed than the other viscosity grades. It must be kept in mind that when the compression speed of tableting machine is increased, the tensile strength of HPMC compacts decreased. Therefore, in order to obtain tablets with high tensile strength for sustained release drug delivery systems a low compression speed may be suitable. It is thus suggested that for compression of HPMC powders, the time during which the particulate system is under a compressive load may be an important influence.

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