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THE INFLUENCE OF MIXING TIME AND COLLOIDAL SILICA ON THE LUBRICATING PROPERTIES OF MAGNESIUM STEARATE

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SUMMARY

The influence of mixing time and colloidal silica on the lubricating properties of magnesium stearate was studied using an instrumented single-punch machine. The lubricant was mixed with sodium chloride, anhydrous lactose, calcium citrate granulate and sodium chloride-colloidal silica-mixtures. Short mixing time decreased the negative effects of magnesium stearate on tablet strength and disintegration without reducing the lubricating efficiency. Addition of colloidal silica had a positive effect on the strength of lubricated sodium chloride tablets but increased the friction and did not improve the disintegration.

INTRODUCTION

Magnesium stearate is an efficient lubricant but may have negative effects on tablet disintegration or dissolution (Levy and Gumtow, 1963) as well as on tablet strength (Shotton and Lewis, 1964). These effects are dependent on the tablet ingredients and the lubricant concentration, but are also known to increase with prolonged mixing or with high mixing intensity (Bolhuis et al., 1975). It is a well-known practice to use short mixing times when admixing magnesium stearate, and it was recently reported that colloidal silica counteracts the negative effects of the lubricant (Lerk et al., 1977; Lerk and Bolhuis, 1977). As little information is available on the influence of mixing time or of silica additions on the lubricating effect of magnesium stearate, we found it interesting to investigate these aspects.

MATERIALS AND METHODS

Magnesium stearate USP (Unilever Emery, The Netherlands); sodium chloride USP, mean sieve diameter 0.15 mm and 0.40 mm, respectively; anhydrous lactose USP (Sheffield, U.S.A.); colloidal silica (Aerosil 200, Degussa, G.F.R.); calcium citrate granulate, prepared from calcium citrate (Merck, G.F.R.) moistened with 2% aqueous potato starch paste. After drying, 5% extragranular potato starch was added as a disintegrant.

Procedure

The materials were mixed with magnesium stearate and/or colloidal silica, in a cubic mixer (Erweka 3 litres) at 40 rpm for different times. The stearate and the silica were added through a 0.2 mm and a 0.7 mm sieve, respectively, to break up aggregates. The mixtures were compressed at a series of different loads between 35 and 265 MPa upper punch pressure, at a rate of 30 tablets per minute, in a single-punch press instrumented with piezo-electric load washers (Hölzer and Sjögren, 1977).

The tablet weight was calculated from the density of each mixture to provide tablets of 2.75 mm height at zero porosity using flat, 1.13 cm diameter punches. The die wall was conditioned by compressing at least 50 tablets before 10 recordings were made. The tablets were stored in sealed polyethylene bags for one week before determination of crushing strength (Schleuninger bardness tester mod. 2E/205) and disintegration time (USP, without discs). Tensile strength was calculated according to the method of Fell and Newton (1968). The friction was characterized by the force needed to eject the tablet calculated per unit contact area between the tablet and the die wall (Hölzer and Sjögren, 1978). All results are given as mean values of five determinations except disintegration which is given as a median of the same number.

RESULTS AND DISCUSSION

The influence of different concentrations of magnesium stearate and different mixing times on tablets prepared at 120 MPa is summarized in Table 1. Pure sodium chloride could be tabletted without lubricant but gave high friction values and the tablet machine emitted a groaning noise at high pressures. Magnesium stearate reduced the ejection force, and this effect was related to the concentration but not to the mixing time except at the lowest concentration. The porosity decreased with the magnesium stearate concentration because of a lower friction and an improved force transmission. Anhydrous lactose and the calcium citrate granulate could not be tabletted without lubricant. Addition of 0.25% or 0.5% magnesium stearate was insufficient to prevent adhesion of lactose to the punches. The picking was visually observed to be slightly reduced by prolonged mixing but the ejection force was not affected. At a lubricant concentration of 0.5% there was still a tendency for calcium citrate to adhere to the die-wall, which resulted in higher friction for one of the mixtures (25 min). Apart from these values, no influence of mixing time was observed. In general, it seems possible to obtain good lubrication even when the lubricant is poorly distributed in the mixture. This may be explained by magnesium stearate being a boundary type of lubricant, i.e. the polar part of the lubricant molecule adheres to the metal surfaces, forming a rather resistant layer, as demonstrated by Strickland et al. (1960). Differences in lubricant concentrations in subsequently compressed tablets, due to short mixing time, may therefore have little influence on the friction against the die-wall. The strength of sodium chloride tablets was reduced by addition of magnesium stearate, and both the amount and mixing time were important. The binding of the other two materials was very little affected by the lubricant, probably due to other binding mechanisms (Hersey et al., 1972; De Boer et al., 1978). The disintegration, on the other hand, was retarded for all materials and influenced by both concentration and mixing time. Tablets prepared from sodium chloride or calcium citrate mixed

TABLE 1

MIXTURES WITH MAGNESIUM STEARATE AND SODIUM CHLORIDE, ANHYDROUS LACTOSE AND CALCIUM CITRATE GRANULATE COMPRESSED AT 120 MPa

Substance	Magnesium stearate %	Mixing time min	Ejection force $kN \cdot cm^{-2}$	Porosity %	Tensile strength MPa	Disintegration time min
Sodium chloride (0.15 mm)	0	0	0.60	12.4	2.9	3.2
	0.1	0.25	0.24	9.5	1.8	3.7
	0.1	2.5	0.24	9.0	0.9	4.2
	0.1	25	0.19	9.0	0.7	5.0
	0.5	0.25	0.14	8.0	0.9	5.0
	0.5	2.5	0.12	8.0	0.6	8.0
	0.5	25	0.14	8.2	0.5	17.8
	2.5	0.25	0.10	6.6	0.6	30.5
	2.5	2.5	0.11	5.4	0.6	35.0
	2.5	25	0.11	5.2	0.6	39.0
Anhydrous lactose	0.25	0.25	0.13	18.3	1.6	6.3
	0.25	2.5	0.14	17.8	1.5	6.4
	0.25	25	0.16	18.3	1.5	6.9
	0.5	0.25	0.12	18.0	1.7	6.6
	0.5	2.5	0.11	17.1	1.6	6.8
	0.5	25	0.11	17.0	1.5	8.8
	2.5	0.25	0.11	15.5	1.4	15.4
	2.5	2.5	0.11	15.5	1.4	14.5
	2.5	25	0.11	15.2	1.3	19.8
Calcium citrate granulate	0.5	0.25	0.12	30.8	2.3	2.1
	0.5	2.5	0.12	30.5	2.0	2.8
	0.5	25	0.16	29.6	1.9	5.1
	2.5	0.25	0.09	29.3	2.1	>60
	2.5	2.5	0.11	29.6	2.1	>60
	2.5	25	0.11	28.6	2.0	>60

with magnesium stearate for 25 min disintegrated faster with increasing compaction loads and this may be due to disruption of a lubricant film around the individual particles by the stronger shear.

The effect of admixing colloidal silical was investigated on sodium chloride tablets as these were very sensitive to magnesium stearate additions. Table 2 summarizes the results of tablets prepared at 200 MPa. All tablets dissolved from the surface and did not disintegrate. The test therefore gave a good indication of the dissolution properties of the tablets. Admixing of colloidal silica improved the tablet strength but had no significant effect on the retardation of the tablet disintegration. Addition of colloidal silica decreased the lubricating efficiency and considerably higher magnesium stearate concentrations were needed to achieve the same reduction of friction. The interaction between colloidal silica and magnesium stearate was obtained independently of the order of admixing but was less pronounced if the sodium chloride was first mixed with the

TABLE 2

MIXTURES WITH CUBIC SODIUM CHLORIDE (0.40 mm) COMPRESSED AT 200 MPa

Magnesium stearate %	Colloidal silica %	Ejection force kN · cm^{-2}	Porosity %	Tensile strength MPa	Disintegration time min
–	–	1.31	5.0	1.3	4.2
0.1 [a]	–	0.18	3.6	0.5	8.8
0.5 [a]	–	0.09	3.5	<0.1	19.0
–	0.5 [a]	1.28	6.1	1.7	5.7
0.1 [a]	0.5 [a]	0.88	4.9	1.2	8.1
0.5 [a]	0.5 [a]	0.27	4.2	0.6	19.5
0.1 [a]	0.5 [b]	0.43	4.8	0.6	9.0
0.1 [a]	0.5 [c]	0.43	4.8	0.6	8.6
0.1 [b]	0.5 [a]	0.71	4.8	1.2	7.7

[a] Mixed with sodium chloride, 25 min.
[b] Admixed to mixture [a], another 25 min.
[c] Admixed to mixture [a], another 50 min.

lubricant. These results confirm previous observations regarding the strength of sodium chloride tablets (Lerk et al., 1977) but not regarding the disintegration or the dissolution (Lerk and Bolhuis, 1977). This discrepancy may be due to differences in concentrations or intensity of mixing.

Our study shows that both tablet strength and disintegration can be seriously affected by magnesium stearate, especially at longer mixing times. Short mixing time, resulting in a poor distribution of the magnesium stearate, did not impair the lubricating efficiency and it is therefore advisable to keep the mixing time and intensity as low as possible. Addition of colloidal silica improved the tablet strength of sodium chloride tablets but had a negative influence on the lubrication and did not improve the tablet disintegration. It is therefore doubtful if colloidal silica can be used as a general remedy for magnesium stearate problems in tablet formulations.

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