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Press Chamber Coating as External Lubrication for High Speed Rotary Presses: Lubricant Spray Rate Optimization

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ABSTRACT Lubrication of the tooling (punches and dies) is necessary to produce tablets. The most commonly used lubricant is magnesium stearate. Adding and blending magnesium stearate to the tablet mass often has negative effects on the properties of the compressed tablets (e.g., decreasing the tensile strength of the tablet). To avoid these negative effects, external lubrication systems were developed. This study investigated the functionality and the influence of a new press chamber coating system called the PKB II. The major difference between the PKB II and previous systems is its ability to spray a mixture of powdered magnesium stearate and air directly onto the punches and dies which was determined to allow the running of the rotor at higher speeds. The data showed a clear correlation between the spray rate of the lubricant and the concentration of the magnesium stearate per tablet. The PKB II was designed to allow for adjustments, in order to optimize the spray rate, by using the ejection force. The concentration of magnesium stearate was reduced to approximately 0.04% per tablet, using the PKB II. Additionally, the most common negative effects, such as the decrease in tablet tensile strength, were avoided by using this system.

KEYWORDS Press chamber coating, PKB, Tablet lubrication, Lubricant concentration, Magnesium stearate, External lubrication, Rotary tablet press

INTRODUCTION

Lubrication of the press chamber, including the punch and die system, is necessary to produce tablets (Gruber et al., 1988; Miller & York, 1988; Moody et al., 1981). The friction of the tablet between punches and die has to be minimized (Führer et al., 1970a; Lachman et al., 1976; Miller & York, 1988; Moody et al., 1981; Stainforth et al., 1989). High friction causes higher abrasion of material up to rotary press damage. Sticking of tablet masses at punches and dies disturbs smooth production (Moody et al., 1981; Stainforth et al., 1989). In most cases the lubricant, generally magnesium stearate (Miller

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& York, 1988; Riepma et al., 1993; Shangraw, 1987), is previously added to the tablet mass (internal lubrication). Incident of high friction and sticking decreases by coating the press chamber with unbounded lubricant. Additionally, magnesium stearate coats the particles of tablet mass and causes decrease of interparticulare interaction (Miller & York, 1988; Riepma et al., 1993). Thus, tablet tensile strength can be reduced, depending on ingredients of tablet mass (Bolhuis & Lerk, 1977; Bolhuis et al., 1975, 1980; De Boer et al., 1978; Führer et al., 1970b; Lerk et al., 1977). High concentrations of lubricant and long mixing times can accumulate negative effects on tablet properties (Bolhuis et al., 1975; Jarosz & Parrott, 1984; Laich & Kissel, 1997; Ragnarrson et al., 1979). Choice of the right lubricant concentration is extremely important. The normal range of magnesium stearate concentration is from 0.25% to 1.5% for most tablet blends (Bolhuis et al., 1975; Gruber et al., 1988; Laich & Kissel, 1997; Miller & York, 1988).

To avoid negative effects of magnesium stearate, external lubrication systems were developed. The lubricant is applied directly to the press chamber before die filling. Different methods of external lubrications are described by literature, e.g., wick-lubrication or spraying lubricant suspension (Gruber et al., 1988; Laich & Kissel, 1997, 1998a, 1998b; Mohrle, 1980; Ritschel & Bauer-Brandl, 2002). This study investigates the modern method of applying powdered lubricants using press chamber coating. The study was accomplished using the presently marketed PKB II (Press-Kammer-Beschichtung [German]) by Fette GmbH, Germany, as press chamber coating system. Forerunner models (Gruber et al., 1988; Laich & Kissel, 1997, 1998a, 1998b; Mohrle, 1980; Ritschel & Bauer-Brandl, 2002) are not described in detail in this study. The simplified control system of the PKB II and the uninterrupted lubricant deposition enables high rotor speed of tablet presses (Hinzpeter et al., 1997).

The aim of this study was to optimize lubrication spray rate using PKB II and to investigate the influence of different spray rates on tablet properties.

MATERIALS AND METHODS

Materials

All materials were kindly supplied by the manufacturers: α-lactose-monohydrate (Tablettose 70™, Meggle, Germany), mannitol (Pearlitol 200SD™, Roquette, France), sorbitol (Neosorb P60W™, Roquette, France), pregelatinized maize starch (Lycatab C™, Roquette, France) and magnesium stearate (Magnesium stearate veg™, Baerlocher, Germany) as lubricant.

Press Chamber Coating System

The pulverized lubricant was deposited on the material contacting surfaces of all punches and dies by the press chamber coating system PKB II designed by Fette GmbH, Germany. Spray rates ranged from 100 to 750 g/h magnesium stearate depending on the selected excipients.

The design of the PKB II is shown in Fig. 1. The powdered lubricant was deposited in the PKB II storage container. Two screw feeders, fitted at the bottom of this container, carried the magnesium stearate out of it. The lubricant dropped into a free fall tower and was mixed with air homogeneously (Hinzpeter et al., 1997).

The mixture was transported into the discharge nozzle under air pressure and blown uninterrupted on the punches and dies. The thin slot nozzle sprayed the lubricant both up onto the upper punch and down inside the die and onto the lower punch. The spray

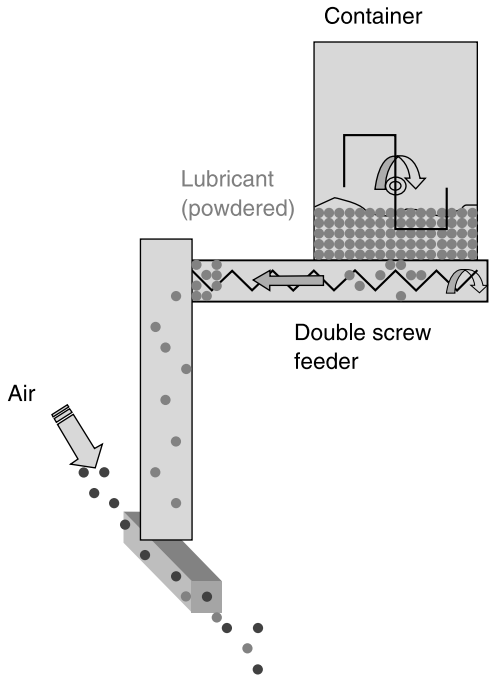

FIGURE 1 PKB II, Illustration of the Functional Unities Storage Container, Screw Feeder, and Air-Mixing Zone.

nozzle was fixed between ejection area and die feeding (Fig. 2). A directly adjacent suction nozzle exhausted the surplus of magnesium stearate. The detailed construction of the spray and exhausting nozzle is described in the patent specification (Hinzpeter et al., 1997) and illustrated as cross section in Fig. 3.

Spray rate was controlled by the K10SU controller (K-Tron Soder, Switzerland). Continuous discharge of lubricant out of the storage container was adjusted gravimetrically. The K10SU controller readjusted the spray rate of magnesium stearate by varying the speed of the screw feeders.

Tableting

The tablets were compressed at a Fette P1200 (Fette GmbH, Germany) rotary press. The press was fitted with 24 EU19 pairs of punches. The round punch tip had a diameter of 9.0 mm and a convex radius of 15.0 mm. The rotor speed of the P1200 was adjusted to 60 rpm. The tablet masses were compressed with a main compression force of 314.4 MPa (20 kN) and a precompression force of 62.9 MPa (4 kN). Exceptionally, the starch derivative Lycatab C™ was compressed with a main compaction force of 251.5 MPa (16 kN). Due to comparability of ejection forces, the tablets were compressed at same edge height (1.6 mm). So the tablet masses varied and can be read out of the corresponding figures.

Tableting Parameters

Each of the four tablet masses was compressed using different spray rates of magnesium stearate. To optimize the lubricants spray rate, ejection force was

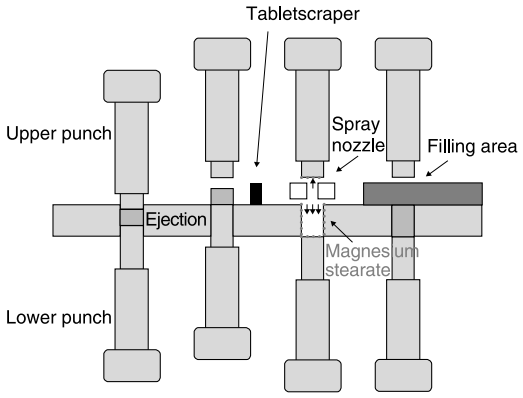

FIGURE 2 **Spray Nozzle, Adjustment at the Press Between Ejection, and Filling Area.**

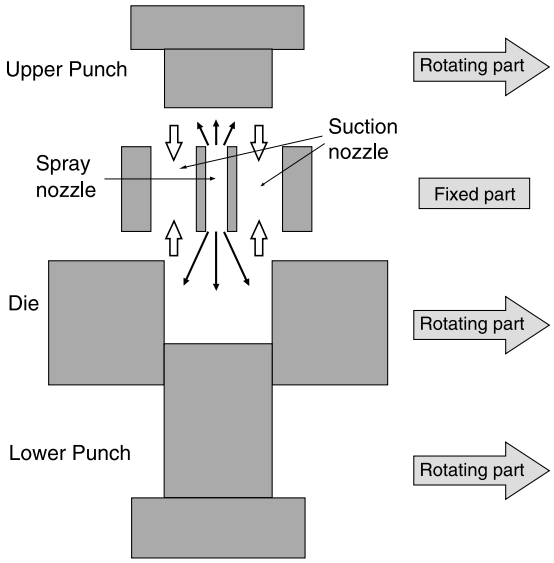

FIGURE 3 **Functional Diagram of Spray and Suction Nozzle. The Small Arrows Illustrate the Dispersion of Magnesium Stearate Onto the Upper and Lower Punch and the Die Walls by the Spray Nozzle. The Suction Nozzle Exhausts Magnesium Stearate (Large Arrows) While Punches and Dies Rotate Below and Above the Fixed Nozzle System.**

detected (Al Shammat et al., 1979; Führer et al., 1970b; Mitrevej & Augsburger, 1982; Schrank-Junghäni et al., 1984). An increasing ejection force suggests a cumulative friction and danger of sticking. The ejection force of 120 tablets per sample batch was analyzed.

Tablet mass, crushing force, height, and diameter of sample batches consisted of 50 tablets were measured by Erweka Multi Check®, respectively. Furthermore, the tablet tensile strength (TS) was calculated using the tablets crushing force (F), height (a), and diameter (d) (Fell & Newton, 1970).

$$TS = \frac{2 * F}{\pi * a * d}$$

The authors evaluated this formula with flat tablets. Due to the convex punch set, we calculated the tensile strength as follows: Instead of the measured height (a) we calculated the equivalent height (a_{eq}) of flat tablets with the same volume. The other parameters of the formula were not changed.

Determination of Magnesium Stearate

The concentration of magnesium stearate of five tablets per sample batch was detected by atom

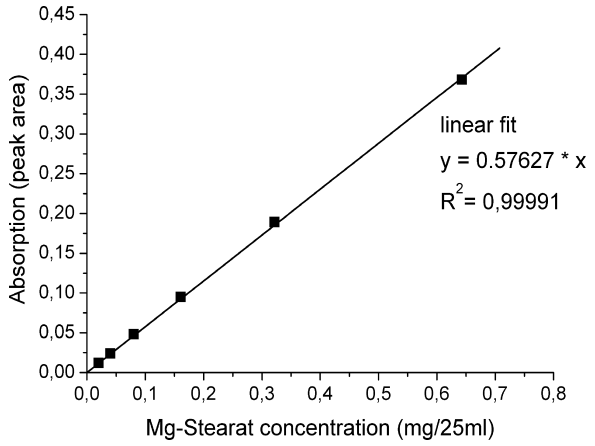

FIGURE 4 **Calibration Curve of Magnesium Stearate.**

absorption spectrometry (AAS). The test serials were run on a Varian SpectrAA-10 plus spectrometer. A magnesium lamp was fixed and the wavelength was set to 285.2 nm to reach an optimum working range between 0.003 µg/mL and 1.0 µg/mL (Varian, 1989). An air-acetylene mixture was used as burnable gas for the flame absorbance. Each tablet was solved in 25.0 mL solvent consisting of 60% hydrochloric acid (2 molar), 20% *n*-butanol, and 20% ethanol. Concentrations of magnesium atoms were measured and magnesium stearate content was calculated via calibration curve. Due to optimization, the AAS calibration was compiled daily. Figure 4 exemplify the calibration data for the mannitol tablets. All other calibrations were similar. All measured data was in the range of the calibration curve with a correlation coefficient (R^2) of 0.99991.

RESULTS AND DISCUSSION

Lubricants Spray Rate Optimization

The optimum of lubrication was characterized by absent sticking, adequate friction, and minimum amount of magnesium stearate. The optimized lubricant spray rate was determined by detecting the ejection force. Increasing ejection force indicated sticking and high friction (Schmidt & Tenter, 1989). Compressing lactose, mannitol, or sorbitol produced a strong increase of ejection force at lower spray rates (Figs. 5–7). The optimum spray rate was reached at the lowest ejection force and thus the lowest friction.

The minimum ejection force was reached at a spray rate of 200 g/h by compressing lactose (Fig. 5).

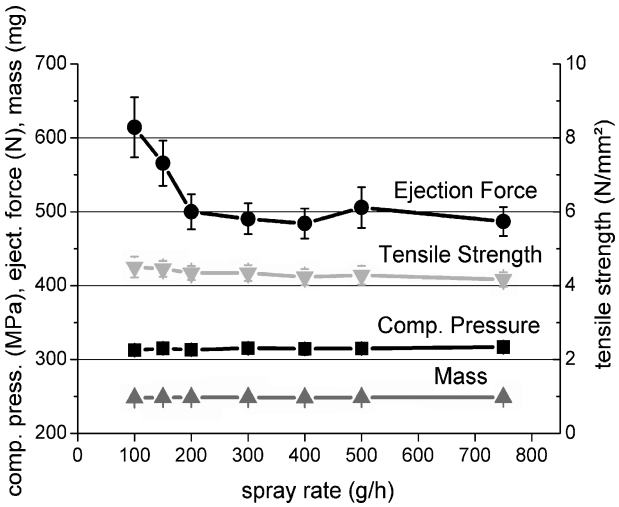

FIGURE 5 **Compressional Pressure, Ejection Force, Tablet Mass, and Tensile Strength of α-Lactose Monohydrate (Tablettose 70) Tablets as a Function of Magnesium Stearate Spray Rate.**

Higher spray rates caused no significant enhancement. So the optimum spray rate of magnesium stearate was 200 g/h.

Mannitol is known to require high lubricant concentration (Gullatz, 1996). Even with a high spray rate of 1000 g/h, a minimum ejection force was not detected (Fig. 6). However, increasing spray rates caused a decrease of the gradient. Although higher spray rates still caused lower ejection forces, an acceptable level of ejection force was reached at a spray rate of 500 g magnesium stearate per hour.

A similar effect was detectable for sorbitol (Fig. 7). An optimum was reached at a spray rate of 500 g/h. At this spray rate, the ejection force reached an acceptable

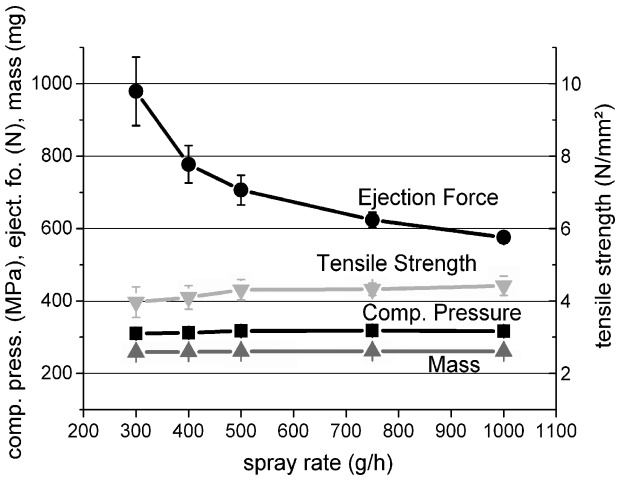

FIGURE 6 **Compressional Pressure, Ejection Force, Tablet Mass, and Tensile Strength of Mannitol (Pearlitol 200SD) Tablets as a Function of Magnesium Stearate Spray Rate.**

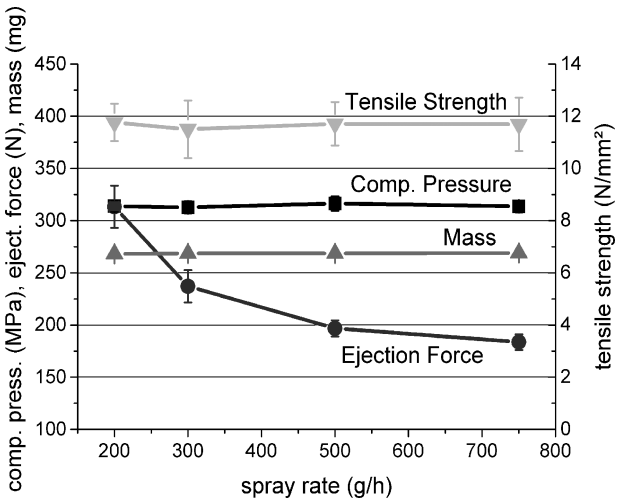

FIGURE 7 **Compressional Pressure, Ejection Force, Tablet Mass, and Tensile Strength of Sorbitol (Neosorb P60W) Tablets as a Function of Magnesium Stearate Spray Rate.**

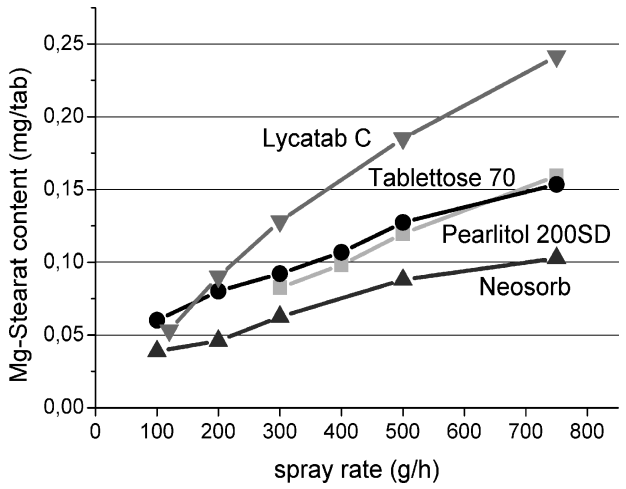

FIGURE 9 **Magnesium Stearate Concentration of Each Tablet (mg) as a Function of Spray Rate.**

level of 200 N. Higher spray rates caused only a slight decrease of ejection force.

Starch products are known for low lubricant needs (Bolhuis & Lerk, 1973). Therefore, it was not amazing that gradient of ejection force was not detectable (Fig. 8). The pregelatinized maize starch required marginal concentrations of magnesium stearate; a spray rate from 100 to 200 g/h was sufficient.

Lubricant Concentration

By compressing all excipients, a nearly linear dependency between spray rate and magnesium stearate concentration of tablets was detected (Fig. 9).

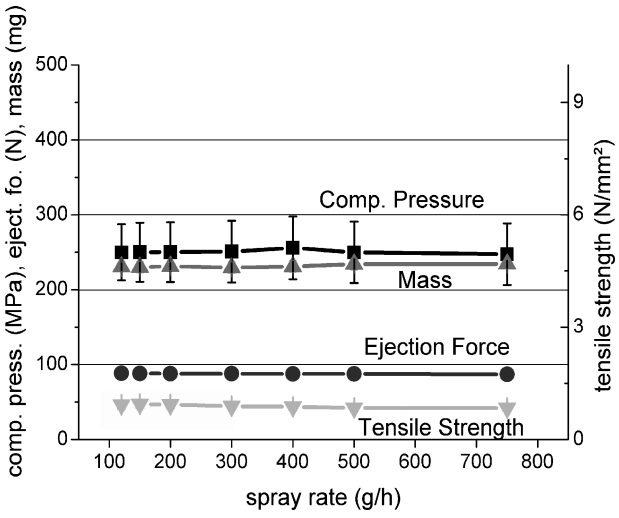

FIGURE 8 **Compressional Pressure, Ejection Force, Tablet Mass, and Tensile Strength of Pregelatinized Maize Starch (Lycatab C) Tablets as a Function of Magnesium Stearate Spray Rate.**

Magnesium stearate concentration in lactose and mannitol tablets seemed to be congruent (Fig. 9). At an optimized spray rate of 200 g/h in case of lactose tablets, the magnesium stearate concentration was 0.080 mg per tablet measured by AAS (Fig. 9). This is equivalent to a concentration of 0.032% in each tablet.

The magnesium stearate content of mannitol tablets, which required a spray rate of 500 g/h, is 0.12 mg and 0.046% in each tablet, respectively.

All values of magnesium stearate content of sorbitol tablets are lower compared to the other excipients (Fig. 9). At the optimized spray rate of 500 g/h, the concentration is similar to lactose tablets at 200 g/h, 0.088 mg, and 0.033% per tablet, respectively.

The gradient of magnesium stearate content of Lycatab C™ tablets is higher compared to tablets compressed with the remaining excipients (Fig. 9). The value of magnesium stearate concentration at a required spray rate of 200 g/h is 0.090 mg or 0.039% per tablet. Inspite of the different gradient, these values are in the same range comparing the other excipients.

Using PKB II, we expected a saturation of the magnesium stearate concentration per tablet at high spray rates. This assumption could not be verified with these data.

Further investigation will be done in the near future to explain the different gradients of the lubricant concentrations at different spray rates, especially for Lycatab C™ and Neosorb P60W™.

Nevertheless, the magnesium stearate concentration in tablets was decreased to a range of 0.032% to

0.046% using the PKB II, compared to previously added lubricant with concentrations of 0.25% to 1.5%.

Tablet Tensile Strength

In this investigation, variation of tablet tensile strength of each excipient should only depend on different lubricant spray rates. Other factors, like different die feeding and fluctuation of compaction pressure due to different punch lengths, were prevented by setting the correct die feeder speed and correct combination of punches. Therefore, tablet mass and the main compaction pressure were monitored. The mean values of mass and compaction pressure of all excipients are constant and the standard deviations (error bars), except Lycatab C, are low (Figs. 5–7). According to the relatively poor flowablity of the pregelatinized maize starch Lycytab C™, comparing the other excipients, the standard deviation of the compaction pressure, described in error bars (Fig. 8), is high. To protect punches against overloading, the compaction pressure was decreased to 251.5 MPa (16 kN) while compressing Lycatab C™.

Tablets consisted of lactose, mannitol, or the starch product and have a constant tensile strength in relation to different lubricant spray rates (Figs. 5, 7, and 8). This insensitivity to magnesium stearate has two reasons: the quantity of magnesium stearate in the tablets (compare chapter Lubricant Concentration) is extremely low and PKB II lubricated directly the tooling surfaces (Hinzpeter et al., 1997). According to external lubrication, magnesium stearate was mainly located on the surface of the tablets. Therefore, interparticular bonding of tablet mass was not disturbed by occupation free binding sites with magnesium stearate (Bolhuis et al., 1975). Tablet tensile strength is constant.

Due to the high values of tablet tensile strength of sorbitol tablets, the standard deviation described as error bars is higher (Fig. 7). Nevertheless, the mean values of tensile strength of sorbitol tablets at different lubricant spray rates are constant.

Uniformity of tablet tensile strength for all excipients at increasing spray rates is remarkable. The magnesium stearate sensitive pregelatinized maize starch Lycatab C™ especially (Bolhuis et al., 1975) shows no negative effect on higher spray rates. Lower compaction pressure, while compressing Lycatab C, caused a lower level of tensile strength in general. Press chamber coating, characterized by its low magnesium stearate insertion, obviously has less influence on tablet tensile strength. At high percentages of magnesium stearate, conventional internal lubrication causes a decrease of tablet tensile strength.

CONCLUSIONS

The PKB II was suitable for alternative lubrication, especially in eliminating problems for lubrication sensitive products such as starch products. Different spray rates of lubricant did not have a negative effect on tablets' tensile strength.

The optimum level of lubricant concentration was adjusted quickly and without problems by varying the spray rate. Insufficient lubrication was detected immediately by rapidly increasing the ejection force.

Despite the higher overall consumption of magnesium stearate using the PKB II, the range of required magnesium stearate per tablet was between 0.032% and 0.046% compared to the usual range of 0.25% to 1.5%.

Production time was decreased. The time-consuming step of previously adding and blending magnesium stearate to the raw material was replaced by the PKB II.

Another possibility is the use of the PKB II in addition to internal lubrication. Difficulties that arise from sticking during the production process can be effectively avoided by using the PKB II in addition to internal lubrication. If the magnesium stearate level for the internal lubrication is decreased about 0.02 to 0.05%, the application of the magnesium stearate from the PKBII spray would increase this concentration back to the normal level.

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