International Journal of Pharmaceutics, 26 (1985) 191–196
Elsevier

IJP 00869

Short Communications

Development and validation of a modified annular shear cell (MASC) to study frictional properties of lubricants

Anand R. Baichwal and Larry L. Augsburger

Department of Pharmaceutics, University of Maryland, School of Pharmacy, Baltimore, MD 21201 (U.S.A.)

(Received February 4th, 1985)
(Modified version received April 10th, 1985)
(Accepted April 23rd, 1985)

Key words: powder lubricants—friction—shear cell

Mostly, lubricants in the past have been evaluated on a tablet press. Some of the techniques used have been the force difference between the upper and lower punches (FD), the punch force ratio (R-value), the remaining force on the lower punch (REF) and the ejection force (EJF) (Holzer and Sjogren, 1981). These techniques generate parameters which effectively integrate over the whole compact-die interfacial area but preclude a point-to-point examination of the frictional conditions. The above parameters are also dependent on the stress transmission characteristics of the host material, which are not generally known. Since these parameters are usually measured on single punch presses using a filler binder excipient as the host material, it becomes almost impossible to comment decisively on the frictional properties of the lubricant under study.

Thus it was proposed to develop a basic analytical tool which would provide reproducible measurement of friction data between pure lubricants and metal wall material under a controlled set of conditions. The modified annular shear cell (MASC) (Fig. 1), similar to the annular shear cell designed to measure failure properties of powders (Carr and Walker, 1967/68; Kocova and Pilpel, 1971/72) but with a smooth polished surface on the underside of the lid instead of the recessed vanes, was designed and constructed to quantitate the amount of friction between a thin powder bed of the test lubricant and the polished steel underside of the lid. The MASC had an inner diameter of 15.24 cm and an outer wall diameter of 25.40 cm; these dimensions being chosen to give a reasonable size cell without too large a ratio

Correspondence: L.L. Augsburger, Department of Pharmaceutics, University of Maryland, School of Pharmacy, Baltimore, MD 21201, U.S.A.

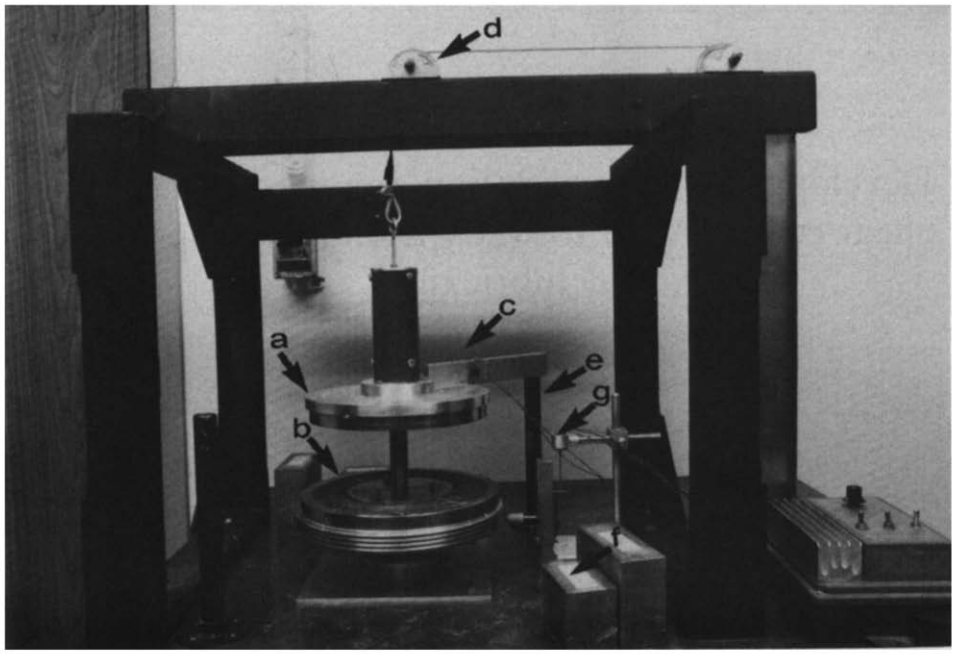

Fig. 1. Overall view of the modified annular shear cell (MASC) showing: (a) lid with underside polished steel annular surface; (b) body with sample-holder plate covered with emery cloth; (c) side arm (wing) with strain gages attached; (d) counter-weight pulley system; (e) rigid pole which restrains the side arm; (f) weights used as normal load; and (g) LVDT attachment.

of extreme strain rates (Kocova and Pilpel, 1971/72). The body and lid were made of aluminum and to the underside of the lid an annular interchangeable polished steel surface was attached. A steel sample-holder plate was bolted to the body of the cell with a corresponding annular recession having a depth of 0.0127 cm. The body of the shear cell was coupled to a gear reducer motor system through a central shaft. To avoid false shear strength readings due to possible wall effects, both the inner and outer walls of this recessed plate were machined at a 45° angle so that no powder would be entrapped between this and the upper polished steel surface while shearing. A sheet of emery cloth covered the annular trough of the sample-holder plate so that when the test material was sheared between this surface and the polished steel surface on the underside of the lid, slippage occurred predominantly at the metal powder interface. The lid was restrained during shearing by means of wings (Fig. 1) to which strain gages [1] were attached and the shearing stress recorded and calculated. The movement of the body of MASC when shear occurred was detected by means of an LVDT [2] attachment (Fig. 1). Here, a linkage to the core rod

[1] Type CEA-13-25OUT-350 Micro-Measurements, Raleigh, NC, U.S.A.

[2] DC–DC Linear Variable Displacement Transducer Series 240-000, Trans-Tek, Ellington, CT, 06029, U.S.A.

of the LVDT rode in the track of a pipe-thread machined onto the outer surface of the shear cell body. As the cell rotated, the pitch of the thread raised the core a distance proportional to the rotational displacement of the shear cell on the horizontal axis of an x–y recorder.

A test procedure similar to that described by Jenike (1961) was adopted. The lubricant to be tested was spread uniformly over the roughened (emery cloth) [3] surface of the shear cell, the lid was lowered, the smallest load placed on top, and shear force applied until the recorded force passed through a maximum and achieved a steady-state value. This was repeated with the load on the lid increasing in steps, shearing after each increment. No record was made of the shear maxima observed at this stage. The cell was then unloaded in steps, carefully removing the same amount of weight in an identical sequence each time. Shearing was induced after each load decrement by switching on the motor. When shear actually occurred, the strain gage response on the vertical axis of the recorder went through a maximum. Simultaneously the pen also deflected horizontally since the body of the shear cell had moved. At this instant shearing was stopped, the predetermined weight removed in the same sequence as before and shearing induced again to obtain the next recording for the lower normal load.

The resulting maximum shear strength value was dependent on the state of consolidation and conditions under which the previous data point was obtained. It is therefore very important in shear cell analysis to be consistent and hence the procedure was carefully executed each time, paying attention to such details as load decrement, starting and stopping the shearing action, consolidation, etc. The maximum recorded readings on the vertical axis were then converted to the shearing stress and these values were used to plot wall yield loci (Fig. 2). The yield loci were generally almost linear ($r > 0.9$) with a very small intercept on the y-axis in some cases. The slopes of these plots yielded friction coefficients for various lubricants at the powder/metal interface (Fig. 2).

To ensure that the plane of shear was at the powder/metal interface, tests and observations routinely employed by workers in this field were conducted. Firstly, the integrity of the powder bed was visually inspected after each test. It was repeatedly observed that the powder bed was continuous and intact with distinct shear marks on the surface which was in contact with the lid. To observe any change in volume of the test specimen, a displacement transducer resting on the lid of the cell (Williams and Birk, 1967) is usually utilized. Similarly, another LVDT [4] was mounted over the lid of the cell to detect any possible change in bed volume due to rearrangement of particles. There was no change in volume observed during shear thus indicating lack of movement within the powder bed. Finally, a thin sheet of polytetrafluoroethylene (about 0.08 cm), utilized as a standard material, was cut identical to the annular surface and sandwiched between the emery cloth and the polished steel lower surface

[3] Crystal Bay Emery Cloth, OH3A Coarse, 3M Company, St. Paul, MN, U.S.A.

[4] DC-DC Linear Variable Displacement Transducer Series 244-000, Trans-Tek, Ellington, CT 06029, U.S.A.

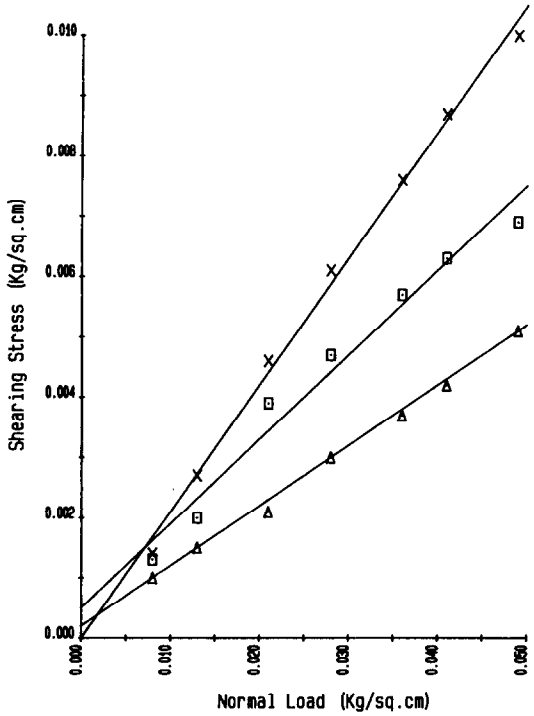

Fig. 2. Wall yield loci of some commonly used powder lubricants. Key: △———△, magnesium stearate; □———□, stearic acid; and × ——— × sodium lauryl sulfate; 10 g lubricant used in each case.

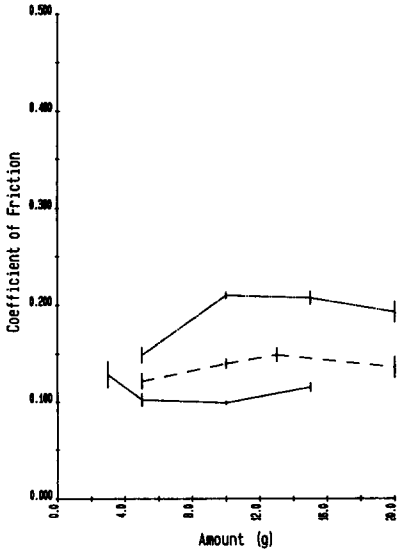

Fig. 3. The effect of amount of lubricant used on the friction coefficients of some commonly used lubricants. Key: ———, magnesium stearate; — — —, stearic acid; and ·–·–·, sodium lauryl sulfate. (Vertical lines represent S.E. bars.)

of the lid. A shear cell analysis was conducted as before to determine the friction coefficient of this material. The calculated friction coefficient for polytetrafluoroethylene on steel was around 0.03, which was close to the reported value (Bowden and Tabor, 1950) for polytetrafluoroethylene. Based on the above observations it was considered reasonable to assume that the plane of shear was at the powder/metal interface.

As with any new analytical tool, a thorough validation was undertaken as the first step in the use of MASC. Certain variables such as the amount of lubricant, the rate of shear (RPM), the maximum load, and the type of consolidation procedure were studied. Based on this a procedure was optimized which was most reproducible and sensitive to small differences between lubricants in terms of their friction coefficients.

The data for the effect of amount of lubricant on the friction coefficients (Fig. 3) indicated that an optimum amount of material was needed to cover the entire bottom surface (about 10 g). The differences between materials were evaluated at two sample sizes by two-way ANOVA. That analysis showed statistically significant differences in the friction coefficients of these lubricants at both amounts (5 and 10 g). One-way ANOVA on the effect of amount of a given lubricant showed no

Fig. 4.

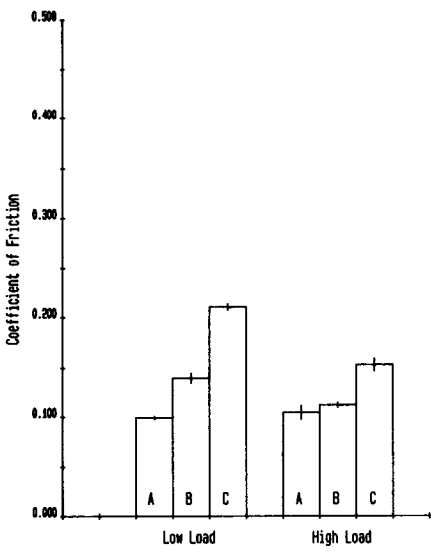

Fig. 4. The effect of maximum load on the friction coefficients of some lubricants. Key: (A) magnesium stearate, (B) stearic acid and (C) sodium lauryl sulfate.

Fig. 5.

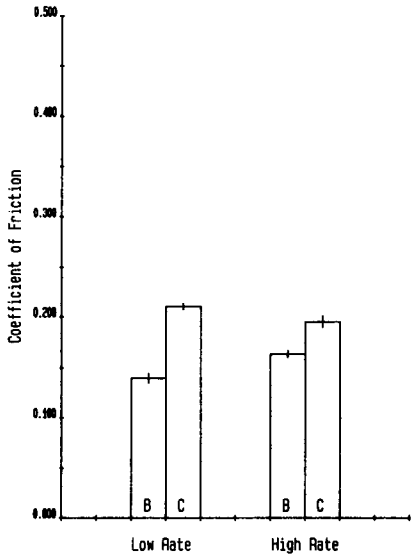

Fig. 5. Effect of rate of shearing (rpm) on the friction coefficients of some lubricants. Key: (B) stearic acid and (C) sodium lauryl sulfate.

differences in friction coefficients above 10 g; lesser amounts exhibited friction coefficients significantly different from the values generated for 10 g or more of a given lubricant. For magnesium stearate, lesser amounts exhibited higher friction coefficients indicating insufficient coverage. Less than 10 g of lubricant for stearic acid and sodium lauryl sulfate showed lower friction coefficients than for 10 g or more of these lubricants. This was due to the tendency of these materials to agglomerate rather than spread; these agglomerates supported the normal load and prevented full contact of the shearing surface with the powder bed. Larger sample sizes resolved this problem. Based on these findings, the amount of lubricant used in further studies was standardized at 10 g.

An examination of the effect of load showed (Fig. 4) that with a maximum load of about 16 kg (low load) differences between friction coefficients of lubricants were more pronounced than when compared with a maximum load of 34 kg (high load). The effect of another procedural variable, the type of consolidation, was also evaluated at these high and low maximum loads. The first procedure was as mentioned above wherein the consolidation load was added progressively. In an alternative procedure, the entire maximum consolidation load was added at once and shear induced until a steady-state value was reached. A statistical analysis revealed no differences in friction coefficients due to the type of consolidation procedure followed.

Similarly, a low rate of shear (0.0083 rev./s) as recommended for an annular type of shear cell (Kocova and Pilpel, 1971/72) proved to be more sensitive to material differences (Fig. 5) than a higher rate of shear (0.0167 rev./s).

With the development of the modified annular shear cell and a well validated experimental procedure it is possible to evaluate small quantities of pure lubricants with respect to their functionality in terms of their friction coefficients. This analytical tool should be helpful in better understanding lubricants and in the design and development of newer lubricants and lubricating systems.

Acknowledgements

This study was supported by a grant from FMC Corporation, Food and Pharmaceutical Products Division. The authors wish to thank FMC Corporation, for their support.

References

Bowden, F.P. and Tabor, D., Friction and Lubrication of Solids, Oxford University Press, London, 1950, p. 327.

Carr, J.F. and Walker, D.M., An annular shear cell for granular materials. Powder Technol., 1 (1967/68) 369–373.

Holzer, A.W. and Sjogren, J., Evaluation of some lubricants by the comparison of friction coefficients and tablet properties. Acta Pharm. Suec., 18 (1981) 139–148.

Kocova, S. and Pilpel, N., The failure properties of lactose and calcium carbonate powders. Powder Technol., 5 (1971/72) 329–343.

Jenike, A.W., Gravity flow of bulk solids, Bull. 108, Utah Engng. Expt. Stn., 1961, p. 206.

Williams, J.C. and Birks, A.H., The comparison of the failure measurements of powder with theory. Powder Technol., 1 (1967) 199–206.