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An investigation into the kinetic (sliding) friction of some tablets and capsules

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ABSTRACT

The kinetic (or sliding) friction of pharmaceutical tablets and capsules influences how they will behave during the conveying, coating, and packaging operations that are used for drug product manufacturing. In order to logically design equipment for manufacturing and packaging operations, and to simulate manufacturing and packaging performance (for example, using discrete or finite element modeling approaches), it is necessary to quantify the magnitude of the kinetic friction. In this work, the coefficient of kinetic friction of a range of pharmaceutical tablets and capsules has been measured for the first time using a pin-on-disk tribometer. Binary tablet–tablet contacts and the contacts between tablets or capsules and common equipment surfaces were studied. The range of the friction coefficients was large (between 0.00 and 0.74), and the values depended strongly on the identity of both contacting materials. Tablet–tablet contacts generally exhibited lower friction coefficients than tablet–polymer or tablet–metal contacts. Polymeric surfaces were generally less frictional than metal surfaces, even those that were highly polished. Tablet coatings appeared to have a marked effect on the kinetic friction coefficient between tablets and equipment surfaces, with the hardest coatings tending to be the least frictional. The surface roughness of the tablets and contacting surfaces did not contribute to the coefficient of kinetic friction in a consistent manner. The implications of the results for the design of conveying, processing and packaging operations are discussed.

1. Introduction

During the manufacture and packaging of pharmaceutical dosage forms there are many situations where the dosage form (usually a tablet or capsule) can come into sliding contact with one or more of its neighbors. In addition, each dosage form can rub against the surfaces of the conveying, coating, or packaging equipment. In these circumstances, the kinetic friction (also known as the sliding friction) of the relevant interface will strongly influence how the dosage form responds to any applied stresses (such as gravity).

The term 'kinetic' friction is used to distinguish this interaction term from the similar terms 'static' and 'rolling' friction (Ludema, 1996; Peterson and Winer, 1980). Static friction is used when the initial movement of contacting materials is being considered, whereas rolling friction refers to the interaction between two surfaces where at least one of them is rolling without slipping, such as for the motion of a wheel. The term kinetic friction is used to describe the contact of two sliding surfaces and is quantified using the coefficient of kinetic friction (μ_K). Assuming Coulomb-type behavior, this is given simply by the ratio of the steady-state tangential force to the load applied in normal direction for two surfaces in sliding contact (Ludema, 1996; Peterson and Winer, 1980) (Fig. 1).

The coefficient of kinetic friction cannot be predicted from first principles and it must be measured experimentally for each system of interest. It usually has a value of between 0.0 and 1.0, but more extreme values are possible (for example, for sticky silicone surfaces). In theory, the value of μ_K is independent of the contact area and the sliding velocity. Factors that are reported to influence the value of μ_K are the hardness, roughness and cleanliness of the contacting surfaces, as well as the environmental conditions and the presence of lubricants (Ludema, 1996; Peterson and Winer, 1980).

In order to correctly design equipment for conveying, coating, and packaging operations, or to simulate such operations, for example, using discrete or finite element modeling approaches (Kalbag et al., 2008; Ketterhagen et al., 2009; Kremer and Hancock, 2006), it is necessary to quantify the magnitude of the coefficient of kinetic friction for common pharmaceutical systems. For example, in a blister packaging line high kinetic friction values may mean that active mechanisms (such as screw feeders) will be needed to convey the dosage forms through the equipment. In contrast, low kinetic friction values could lead to uncontrolled product flow and overloading of filling systems. This is illustrated in Fig. 2 where a single tablet in a bulk tablet bed is shown in contact with an equipment surface. This situation may occur, for example, on the feeding chute of packaging line, on the exit chute of a rotary tablet press, or within a tablet

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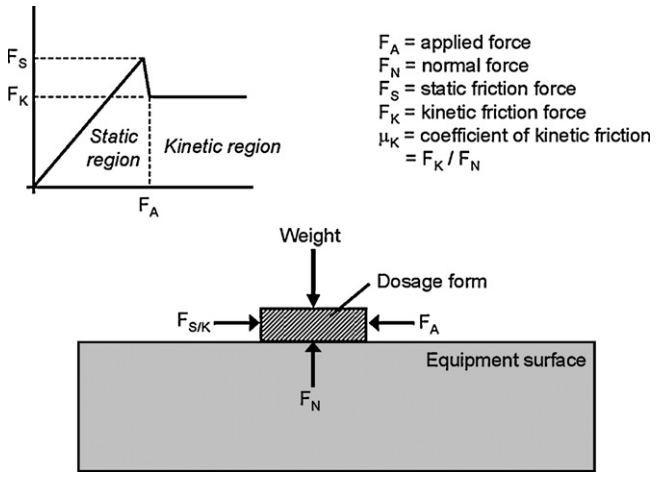

Fig. 1. Schematic defining the coefficient of kinetic friction.

coating pan. In computer simulations of tablet collision dynamics and film-coating operations reported in the literature to-date, the values of μ_K used have been arbitrarily chosen, rather than being parameterized from experimental data generated with actual pharmaceutical materials. For example, Song et al. (2006) used a value of 0.4 in their computer simulations of tablet collisions without any justification. Kalbag et al. (2008) used a kinetic friction coefficient of 0.3 for both tablet–tablet and tablet–equipment contacts in their discrete element simulations of film coating, again without any experimental data to support their choice. These authors did conduct a brief parametric study of the effect of changing the value of μ_K in their simulations and they reported a marked effect on the motion of the tablet bed in the coating pan from a 'slumping' to a 'rolling' behavior.

Up to this point, kinetic friction coefficient measurements on pharmaceutical materials have been mainly restricted to bulk powder systems. Powder 'internal friction' measurements with shear cells are commonly performed and used to predict the real-life flow behavior of pharmaceutical powders (Hiestand and Wilcox, 1968; Podczeck and Mia, 1996). So-called 'wall friction' measurements on pharmaceutical powders (also performed using shear cells) are regularly used to model the flow performance of these materials in industrial and laboratory scale hoppers and chutes (Behres et al., 1998; Haaker, 1999; Prescott et al., 1999). Such measurements of bulk friction coefficients for pharmaceutical powders have also been used to optimize the selection of lubricants to reduce powder-equipment friction (Baichwal and Augsburger, 1985, 1988). Powder friction measurements during punch-and-die compaction (conducted using instrumented tablet presses) have been widely reported (Baichwal and Augsburger, 1985, 1988; Ernst et al., 1991; Guyoncourt et al., 2000, 2001; Holzer and Sjogren, 1981a,b; Korachkin et al., 2008; Lewis and Train, 1965a,b; Strijbos, 1977), and the data generated used to identify potential issues during tablet manufacturing operations (e.g., premature tooling wear). Similar data have also been used as inputs for finite element computer simulations of tablet compaction (Cameron and Gethin, 2001; Cunningham et al., 2004; Sinka et al., 2003). On a much smaller scale, several reports have documented the measurement of kinetic friction between individual particles of pharmaceutical powders and solid surfaces (Bunker et al., 2006; Jones et al., 2004; Lee, 2007; Mullier et al., 1991). Measurements of the friction between drug and excipient particles have also been made using a specialized centrifuge technique (Podczeck et al., 1995). Unfortunately, all these different types of friction measurements on powders and single particles cannot be simply related to the friction at sliding contacts involving solid dosage forms which is the central topic of this work.

To the authors knowledge there are no previous reports of friction measurements on commercial solid dosage forms. However, two reports of this type of work with model pharmaceutical systems exist (James and Newton, 1983, 1985). In the first of these reports (James and Newton, 1983), the authors described a novel 'disk-brake' style testing apparatus and used it to measure the friction of acetyl salicylic acid and poly(tetrafluoroethylene) (PTFE) compacts against a steel surface. The kinetic friction coefficients varied from ~0.9 for the acetylsalicylic acid compacts to <0.1 for the PTFE samples, depending on the experimental conditions selected. In the second report (James and Newton, 1985), the same authors investigated the impact of the roughness of the steel surface on the

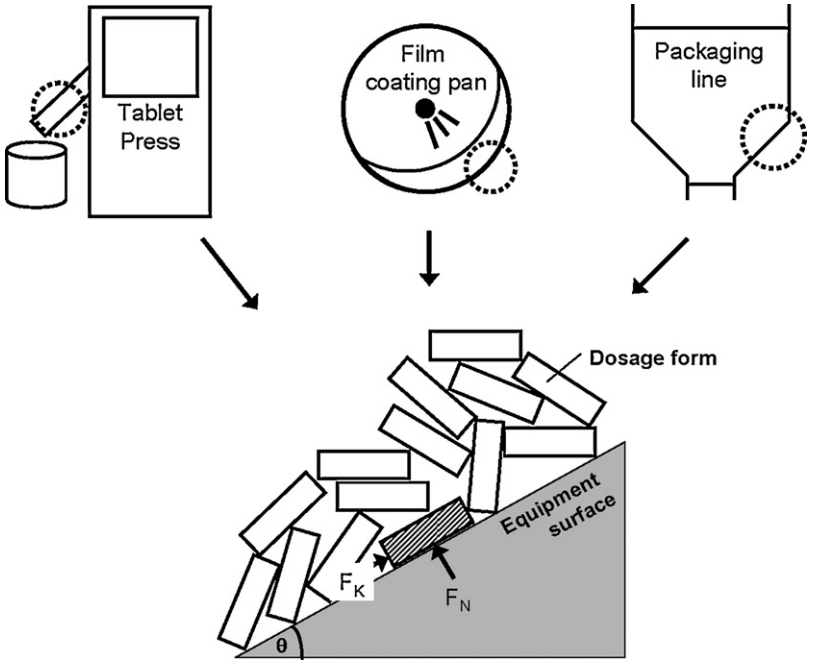

Fig. 2. Schematic of pharmaceutical processing situations where kinetic friction is important.

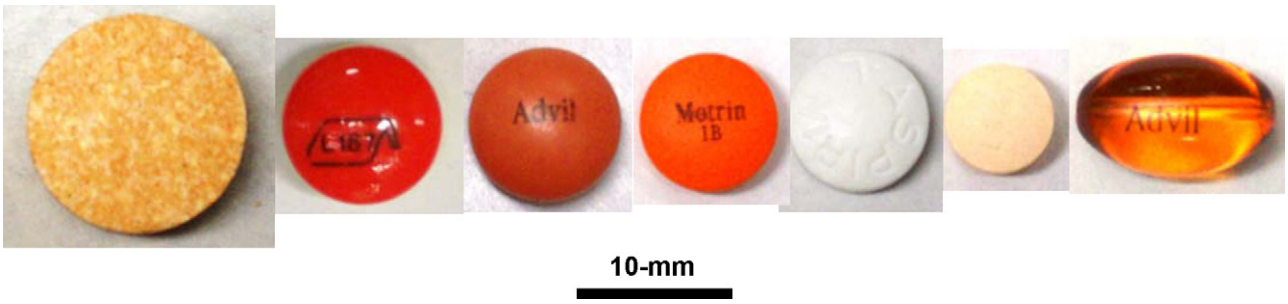

Fig. 3. Tablets and capsules evaluated in this study. Left to right: Vitamin-C, Vitamin-C 500 mg uncoated tablets, Acetaminophen 500 mg gel-coated tablets, Ibuprofen 200 mg sugar-coated tablets, Ibuprofen 200 mg film-coated tablets, Aspirin 325 mg film-coated tablets, Aspirin 81 mg chewable tablets, Ibuprofen 200 mg soft-gelatin capsules.

measured kinetic friction coefficient. They found that increasing the roughness of the steel surface resulted in a higher friction coefficient, and speculated that modifying the surface finish of processing equipment could be used to reduce processing issues caused by excessive friction.

There are numerous reports of kinetic friction measurements for macroscopic food particles, such as soy beans and wheat grains (e.g., LoCurto et al., 1997; Ross et al., 1987), and on various large objects used in industrial applications (e.g., automotive components, healthcare products) (e.g., Alcock et al., 1996; Bhushan et al., 2005; Zhou et al., 2002). The use of friction measurements in these fields is quite common, and the approaches used (ASTM, 2000) can be used to guide the development of test methods suitable for use with pharmaceutical dosage forms. Large data compilations for these non-pharmaceutical materials are also widely available (Ludema, 1996; Peterson and Winer, 1980) and used for quantitative engineering and design calculations. The effects of material properties, surface finishes, lubrication, and environmental conditions have been studied in detail for such systems and they are quite well understood. For pharmaceutical materials, it can be speculated that these types of factors will influence the frictional characteristics of solid dosage forms, but this can only be definitively ascertained through a series of carefully designed experiments with samples of this type.

The objective of the current work was to develop a simple methodology for making macroscopic kinetic friction measurements on intact solid dosage forms (tablets and capsules) and to report representative data for a range different commercial formulations and common equipment surfaces. Of specific interest are the trends (if any) in the coefficient of kinetic friction with dosage form type and surface properties so that the findings can be used to guide the design and optimization of equipment used for handling, manufacturing, and packaging bulk tablets and capsules.

2. Materials and methods

The dosage forms studied in this work were all commercial products purchased from a local pharmacy, and they were used as-received. They included acetaminophen 500 mg gel-coated tablets (Albertsons Inc., Boise, ID, lot 7ME0568), aspirin 81 mg uncoated chewable tablets (Albertsons Inc., Boise, ID, lot 5EE0517), aspirin 325 mg film-coated tablets (Albertsons Inc., Boise, ID, lot 7LE0852), ibuprofen 200 mg film-coated tablets (McNeil Inc., Fort Washington, PA, lot PHA311), ibuprofen 200 mg soft-gelatin capsules (Wyeth Consumer Healthcare, Madison, NJ, lot B32637), ibuprofen 200 mg sugar-coated tablets (Wyeth Consumer Healthcare, Madison, NJ, lot C12611) and vitamin-C 500 mg uncoated tablets (Nature's Bounty Inc., Bohemia, NY, lot 151074-01). Photographs of the tablets are presented in Fig. 3.

The other materials were selected to represent those used for product contact in pharmaceutical manufacturing, packaging and testing operations. They included two stainless steel surfaces with different surface finishes (average roughness, Ra = 1.21 and 0.04 μm) (Falex Corp., Chicago, IL), polytetrafluoroethylene (PTFE) (Ra = 1.93 μm), polycarbonate (Ra = 0.37 μm), and high-density polyethylene (HDPE) (Ra = 1.09 μm).

The kinetic friction measurements were made using a pin-on-disk tribometer (CSM Instruments Inc., Needham, MA). This type of instrument is commonly used for measuring the coefficient of kinetic friction of non-pharmaceutical materials (ASTM, 2000) and comprises a horizontally oriented disc that rotates and a vertically oriented pin that can be lowered to make contact with the disk (Fig. 4). During the measurement a controlled normal (downward) force is applied to the pin and the disk is rotated at a controlled rate. The shear force acting on the pin is measured (usually with strain gauges) and used to calculate the coefficient of kinetic friction for that specific combination of materials.

Individual tablets and capsules were rigidly mounted on aluminum rods ('pins') or disks using fast-setting acrylic glue, being

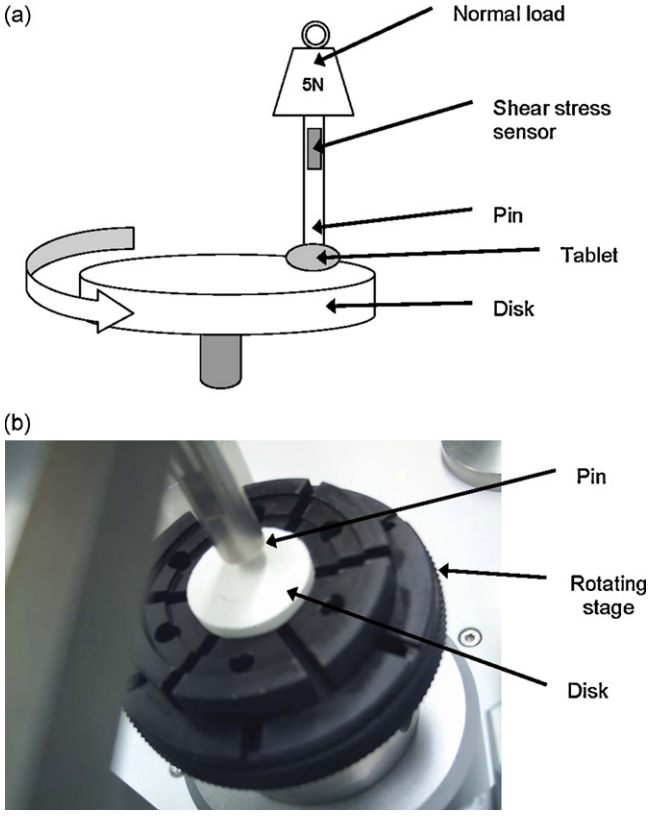

Fig. 4. Pin-on-disk tribometer (CSM Instruments Inc., Needham, MA) (a) schematic (b) photograph (viewed from above).

careful not to touch their exposed surfaces. Flat disks of 12.5 mm radius were carefully cut from each of the metals and polymers, and cleaned prior to use with a mixture of isopropyl alcohol and water (25:75) and a lint free cloth. A normal force of 5.0 N, a sliding contact linear speed of 1.0 cm s^{-1}, and a track radius of between 8 and 10 mm were used. These conditions were selected to achieve consistent friction measurements under circumstances similar to those encountered during normal tablet handling and packaging operations (Hughes and Wan, 2005; Kalbag and Wassgren, 2009). The exact stresses on a tablet in any specific situation can be calculated using standard engineering approaches, and the velocities of tablets in such situations can be determined using data from digital imaging systems (Kalbag and Wassgren, 2009).

Several preliminary trials were conducted to ensure that the test procedure was capable of providing consistent data. Samples were tested at normal forces of between 1.0 N and 10.0 N, and on multiple occasions. There was insignificant variation (~1%) in the data with time over this range of normal forces, and the experiment-to-experiment variability was typically less than 5%. At higher normal forces (>10.0 N) and after prolonged testing slight damage to the surface of the compacts was visible in some cases. At lower normal forces (<1.0 N) and higher velocities (>5 cm s^{-1}) there was significant noise due to 'skipping' at the pin–disk interface (that is, loss of contact between the two sliding surfaces). It was also noted that care was needed to securely affix the tablets and capsules to the sample holders to prevent misalignment or sample movement during testing.

The tribometer was calibrated according to the manufacturer's recommended procedures, and the temperature and humidity were "controlled ambient conditions" (20–25 °C; 20–50% RH). Shear force measurements were made at 7 Hz until steady-state conditions were reached (typically, a period of several minutes), and the kinetic friction coefficient was calculated by dividing the shear force by the normal force. Data from at least one minute of testing were used to calculate the mean friction coefficient for each test, and triplicate tests were used to calculate an overall mean value and relative standard deviation. Typical plots of the individual and mean data are shown in Fig. 5.

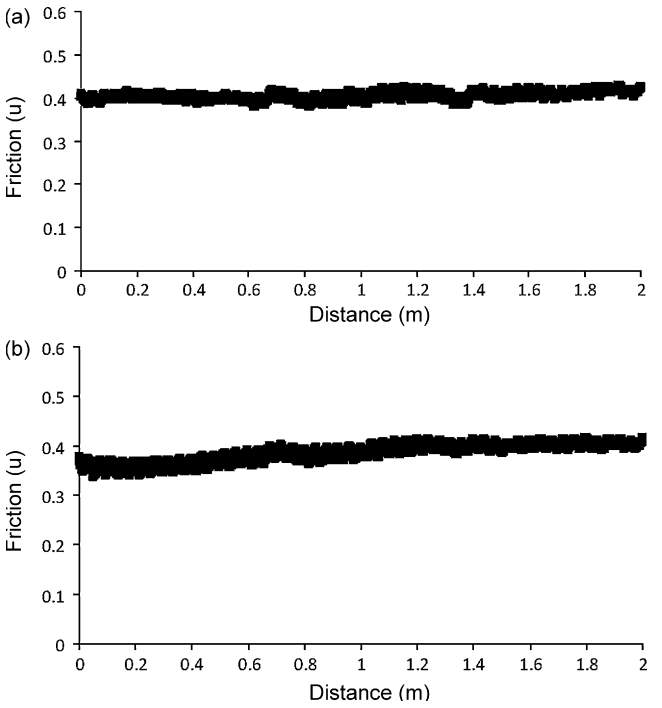

Fig. 5. Typical data from sliding friction measurement for an aspirin chewable tablet on a polycarbonate surface. (a) single experiment (b) mean of 3 replicate determinations.

Table 2
Kinetic friction coefficients measured for some pharmaceutical tablets (tablet–tablet contact).

	Sliding friction coefficient (mean and SD)
Acetaminophen 500 mg gel-coated tablets	0.00 (0.04)
Aspirin 81 mg chewable tablets	0.00 (0.05)
Ibuprofen 200 mg film-coated tablets	0.03 (0.03)
Ibuprofen 200 mg sugar-coated tablets	0.00 (0.02)

3. Results and discussion

3.1. Comparison with literature data

In order to establish that the method used for the measurement of the kinetic friction coefficients was generating accurate results, several standard materials were tested and the results compared to data for similar materials published in the literature. The kinetic friction coefficients determined in this study for steel–steel and steel–PTFE contacts were consistent with those previously reported in the literature (Table 1). In addition, it was demonstrated that the choice of material for the pin or the disk did not alter the results noticeably, as would be expected from theory (compare both sets of steel–PTFE data in Table 1). From these results it was concluded that method developed for measuring the kinetic friction of the solid dosage forms was sound.

3.2. Inter-tablet kinetic friction

Tablet–tablet contacts are common in pharmaceutical manufacturing and packaging operations (for example, during film coating), and the inter-tablet kinetic friction is therefore of great interest. Data for several representative tablet dosage forms are shown in Table 2. In all cases the inter-tablet friction was very low or zero. It appears that the 'like surfaces' were all effectively non-frictional contact partners, hence, these dosage forms would all be expected to slide against themselves very readily. This could be advantageous for gravity driven transfer in a packaging operation where high flow rates are needed. Hughes and Wan (2005), studied such a system and empirically demonstrated that tablet coatings formulations that gave high tablet flow rates from a simple hopper could be blister packaged the most rapidly. The tablets studied in this work each had different types of surface coating. The gel-coated acetaminophen tablets appeared to be very smooth, whereas the uncoated aspirin chewable tablets had a matt appear-

Table 1
Kinetic friction coefficients measured for some common materials in comparison to literature data.

Material #1 ("pin")	Material #2 ("disk")	This study (mean and SD)	Literature value[a]
Poly(tetrafluoroethylene)	Steel (stainless)	0.01 (0.00)	0.04
Steel (stainless)	Poly(tetrafluoroethylene)	0.04 (0.00)	0.04
Steel (stainless)	Steel (stainless)	0.37 (0.04)	0.42–0.57

[a] Literature data from http://www.engineershandbook.com/Tables/frictioncoefficients.htm.

Table 3
Kinetic friction coefficients measured for some tablets and capsules against common equipment surfaces (mean and SD).

	Stainless steel Type 303 (Ra = 1.21 μm)	Stainless steel Type 304 (Ra = 0.04 μm)	Polycarbonate (Ra = 0.37 μm)	HDPE (Ra = 1.09 μm)	PTFE (Ra = 1.93 μm)
Vitamin-C 500 mg uncoated tablets	0.10 (0.00)	0.21 (0.03)	0.24 (0.02)	0.22 (0.01)	0.09 (0.01)
Acetaminophen 500 mg gel-coated tablets	0.50 (0.05)	0.74 (0.02)	0.46 (0.05)	0.17 (0.05)	0.04 (0.01)
Aspirin 325 mg film-coated tablets	0.51 (0.03)	0.51 (0.06)	0.28 (0.14)	0.05 (0.02)	0.04 (0.01)
Aspirin 81 mg chewable tablets	0.25 (0.06)	0.20 (0.01)	0.38 (0.03)	0.16 (0.01)	0.05 (0.01)
Ibuprofen 200 mg film-coated tablets	0.58 (0.01)	0.52 (0.16)	0.33 (0.07)	0.06 (0.01)	0.01 (0.01)
Ibuprofen 200 mg sugar-coated tablets	0.12 (0.01)	0.15 (0.02)	0.10 (0.03)	0.08 (0.01)	0.03 (0.00)
Ibuprofen 200 mg soft-gelatin capsules	0.39 (0.01)	NA	0.23 (0.04)	NA	NA

NA = data not available.

ance. The sugar-coated and film-coated ibuprofen tablets had an intermediate surface texture as judged by subjective observation. Despite these differences in surface coating, the coefficients of kinetic friction were all very low, and, in fact, they were comparable to ultra low friction materials such as poly (tetrafluroethylene) (PTFE) (Table 1). For the coated tablets it is possible that a 'finish coating' of a low friction material such as carnauba wax was added as a final processing step (Cole et al., 1995). For the uncoated tablets the use of a lubricant during tablet compression may have contributed to the low coefficient of kinetic friction (Baichwal and Augsburger, 1988).

3.3. Kinetic friction with metals and polymers

During the normal handling and packaging of pharmaceutical dosage forms there are many sliding contacts made with metal and polymer surfaces (Fig. 2). These materials comprise the product contact surfaces of the manufacturing, packaging, conveying and testing equipment. Normally these surfaces are finished to a visually smooth appearance to reduce sticking of the product to the surface and for ease of cleaning. The kinetic friction coefficients measured for over thirty typical combinations of tablets, capsules and these types of equipment surfaces are reported in Table 3.

Amongst the different contact materials, PTFE consistently had the lowest friction with the different tablets types. This confirms the utility of this material as a non-stick surface for pharmaceutical dosage forms. The HDPE surface had the next lowest friction of the three polymeric materials and the polycarbonate surface generally exhibited the highest friction of the polymer surfaces. Amongst the various polymers the coefficient of kinetic friction generally decreased with increasing surface roughness for any given tablet sample. This is opposite to the trend reported by James and Newton (1985) for acetylsalicylic acid compacts sliding against tool steel surfaces, and is contrary to the generally held view that smoother surfaces tend to be less frictional. For non-pharmaceutical materials, an increase in kinetic friction sometimes occurs with smoother finishes, and this has been attributed to an increase in contact area and proximity for intermolecular interaction for these smoother surfaces (Ludema, 1996). This is a plausible explanation for the trends observed with the polymer surfaces in this work. It is also possible that the different chemistry of the various polymers governs the magnitude of the material interaction at the interface and this dictates the coefficient of kinetic friction for the various tablet–polymer pairs.

The polished stainless steel surfaces tended to produce higher kinetic friction coefficients than the polymeric surfaces. However, the smoother surface finish resulted in higher friction values in only some instances, and the magnitude of kinetic friction could not be simply correlated with the roughness of the equipment surface. Similar effects have been previously reported for bulk powder wall friction measurements (Haaker, 1999; Prescott et al., 1999), and have been attributed to the unique physical and chemical interactions that occur for any given pair of contacting surfaces.

Amongst the various tablet types, the film-coated tablets generally exhibited a higher coefficient of kinetic friction than the uncoated tablets with the stainless steel surfaces. The opposite

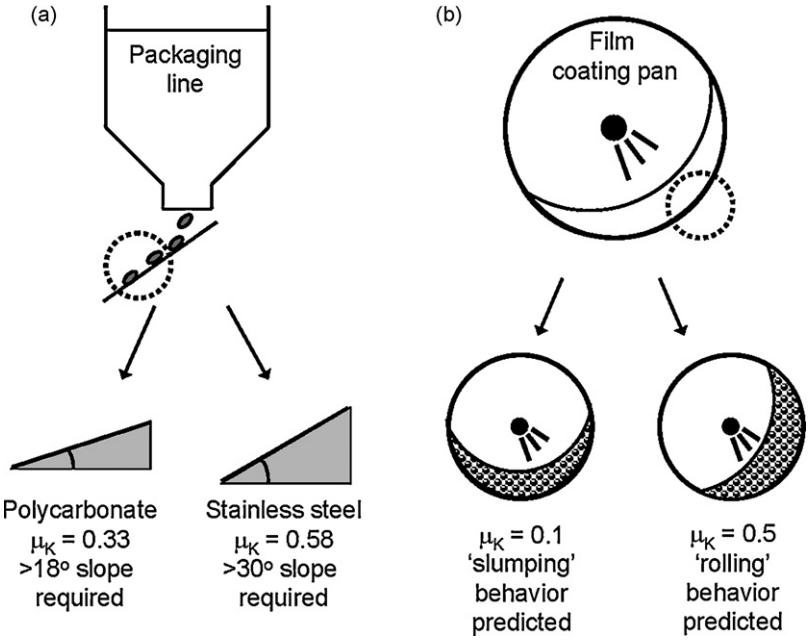

Fig. 6. Practical applications of coefficient of sliding friction data (a) for calculating chute angles (b) for estimating tablet motion in film coating pans.

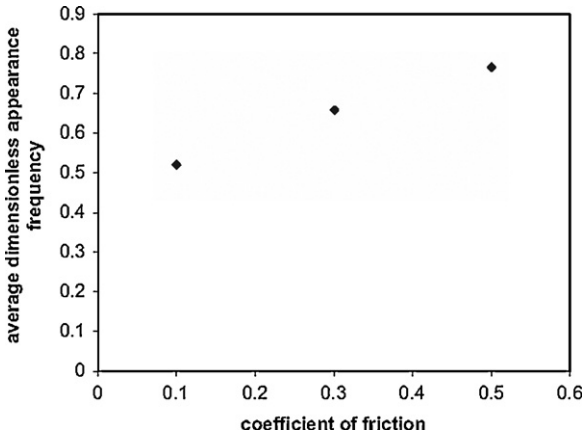

Fig. 7. Dimensionless appearance frequency plotted as function of friction coefficient for DEM simulations of tablet film coating. Reproduced from Kalbag et al., 2008 with permission of the publishers.

trend was true when considering the HDPE surface. This confirms that a combination of both tablet and surface properties controls the magnitude of the kinetic friction coefficient. The gel-coated tablets exhibited quite high friction on all surfaces, despite their smooth surface appearance. The relatively high friction for this type of coating may be due to it being a relatively soft coating that perhaps can deform under modest normal forces. This could increase the intimacy of contact between the surfaces and thus lead to a higher friction coefficient. Alternatively, this coating material may partially hydrate at ambient humidities and become somewhat 'sticky'. In contrast, the sugar-coated tablets exhibited a low friction coefficient on all the metal and polymer surfaces, perhaps because this coating was relatively hard and did not readily deform under load (Cole et al., 1995).

Lastly, it can be concluded from the data in Table 3 that there is no obvious trend in kinetic friction with the identity of the active ingredient in the dosage form, the dose strength, or the dosage form size. Also, it is apparent that the behavior of the soft gel capsules was similar to that of the tablets, although they were more difficult to fix securely to the testing apparatus and data could not always be generated for this particular type of dosage form.

3.4. Practical significance

The significance of the results in Table 3 can be better appreciated with a few practical examples of the use of such data. As shown in Fig. 6a, using the coefficient of friction data for the ibuprofen film-coated tablets, the minimum angle required for a tablet conveying chute for a packaging operation can be calculated. If the chute were to be made of polycarbonate it would need to have an angle of at least eighteen degrees from horizontal to ensure consistent tablet motion, whereas a chute made from stainless steel (303 grade) would need to be approximately twelve degrees steeper. Different chute angles might also be required for different products which exhibit different frictional behavior (see Table 3). Clearly the design of the packaging equipment needs to take into account differences in the frictional behavior of different equipment/product combinations.

The motion of a tablet bed in a film-coating pan can be modeled using discrete element method (DEM) simulations incorporating data such as that presented in Table 3 (Kalbag and Wassgren, 2009; Kalbag et al., 2008) (Fig. 6b). For a system with a coefficient of kinetic friction of 0.5, these authors reported that the tablet bed exhibited a 'rolling' behavior, whereas for a system with a coefficient of kinetic friction of 0.2 the tablet motion was described as being 'slumping'. These changes in the motion of the tablet bed are important for positioning the spray-guns during the film-coating process, and can also have a marked impact on the tablet mixing in the coating pan and on the uniformity of the coating. In this application of the coefficient of kinetic friction data, individual tablet–tablet and tablet–equipment collisions were modeled from first principles leading to a detailed prediction of tablet dynamics in the coating pan. This allowed system properties such as the duration of tablet exposure to the coating spray-zone and tablet mixing uniformity to be predicted. It is quite clear that these key performance indicators change as the coefficient of kinetic friction varies, for example, as shown in Fig. 7.

4. Conclusions

The kinetic (or sliding) friction of a range of pharmaceutical tablets and capsules against themselves and common equipment surfaces has been characterized for the first time using a pin-on-disk tribometer. The range of the friction coefficients was large (between 0.00 and 0.74), and the values depended strongly on the identity of both contacting materials. Tablet–tablet contacts generally exhibited lower friction coefficients than tablet–polymer or tablet–metal contacts. Polymeric surfaces were generally less frictional than metal surfaces, even those that were highly polished. Tablet coatings appeared to have a marked impact upon the kinetic friction coefficient between tablets and equipment surfaces, with the hardest coatings tending to be the least frictional. The surface roughness of the tablets and contacting surfaces did not contribute to the coefficient of kinetic friction in a consistent manner. Examples of the implications of the results for the design of conveying, processing and packaging operations have been highlighted. Future work should focus on understanding the effects of kinetic factors (such a sliding velocity) and the effects of material properties (such as surface roughness and surface chemistry).

Acknowledgement

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