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The coefficient of restitution of some pharmaceutical tablets/compacts

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ABSTRACT

When tablets collide during manufacturing and handling operations they rebound with a force and velocity that is determined by the collision conditions and the properties of the materials. This collision-rebound behavior of solid bodies can be described using a parameter known as the "coefficient of restitution" (CoR). In this work, the CoR of a range of pharmaceutical tablets/compacts is measured using a simple "drop test", and the influences of material properties (elastic modulus, solid fraction, *etc.*) and collision conditions (substrate, energy/speed, *etc.*) are investigated. The compacted pharmaceutical materials have CoR values that range from 0.4 to 0.9, and the CoR generally increases with increasing compact solid fraction. The CoR varies with the mechanical properties of both colliding bodies and is lower for more plastic collisions and higher for elastic collisions. This behavior is consistent with theories developed for non-pharmaceutical solids, and can be predicted provided that the elasticity and yield stress of the samples are treated as porosity dependent parameters. In this case, the CoR varies with the impact velocity nearly raised to the fourth root. Having established a simple and reproducible test for the CoR of pharmaceutical compacts and tablets it should be possible to create more accurate engineering models and computer simulations of tablet manufacturing and packaging operations.

1. Introduction

While the potency, efficacy and stability of oral dosage products is the primary concern of pharmaceutical companies, the structural integrity of the tablets that deliver the active pharmaceutical ingredients (APIs) is also of great importance. During the manufacture, handling and packaging of pharmaceutical dosage forms, there are many situations where the dosage form (usually a tablet or capsule) can break due to large stresses developed during impact with neighboring tablets or with the process equipment. In order to logically design equipment for pharmaceutical manufacturing operations, or to simulate manufacturing and packaging performance (for example, using discrete element modeling approaches), it is necessary to quantify the magnitude of impact forces that occur for these types of contacts. The coefficient of restitution is an important collision parameter that is used to predict the impact forces in some granular flow models.

Recently, discrete element method (DEM) simulations of unit operations in the pharmaceutical industry have given insight into efficient material handling and processing techniques (Ketterhagen et al., 2009; Kremer and Hancock, 2006). Particle scale properties such as stresses and velocities, which are challenging to measure experimentally, can be predicted from DEM. Typically, in a DEM simulation, material properties such as the shear modulus, Poisson's ratio and density of the colliding particles along with their interaction characteristics, such as the coefficients of restitution and friction are given as inputs. These inputs are then used by a collision model (Bharadwaj et al., 2006; Di Renzo and Di Maio, 2004; Kruggel-Emden et al., 2007; Kuwabara and Kono, 1987; Stevens and Hrenya, 2005; Walton and Braun, 1986) in the simulation to calculate the post-collisional particle velocities and orientations. The success of these models depends on the accuracy of the parameters that are given as inputs to the simulation. For example, Kuo et al. (2002) performed DEM simulations of particle behavior in a V-blender and observed semi-quantitative agreement with experiments as long as reasonable inputs such as the coefficient of restitution and friction were used. Ketterhagen et al. (2008) observed that the degree of segregation during hopper filling can be affected by the coefficient of restitution between particles given as input to the simulations.

Although test procedures and data of these input parameters are available for non-pharmaceutical materials like steel, aluminum, *etc.*, there is little or no data available for typical pharmaceutical compounds. This report describes, for the first time, the test procedure used to measure one of the interaction parameters: the

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Nomenclature

e	coefficient of restitution (dimensionless)
E	Young's modulus (N/m^2)
h_1	tablet drop height (m)
h_2	tablet rebound height (m)
P_y	yield pressure (N/m^2)
V_i	impact velocity (m/s)
V_r	rebound velocity (m/s)
V_y	yield velocity (m/s)
Greek letters	
ρ	density (kg/m^3)
σ_y	yield stress (N/m^2)
ν	solid fraction (dimensionless)

coefficient of restitution and reports results for some typical pharmaceutical compacts/tablets.

The coefficient of restitution, e, is a lumped parameter that quantifies the energy losses during collision and is defined as:

$$e = \frac{-V_r}{V_i} \quad (1)$$

where V_r and V_i are the rebound and impact relative velocities of the colliding objects. The collision is said to be perfectly elastic if $e = 1$ and completely inelastic if $e = 0$. For a freely falling object under the influence of gravity, Eq. (1) simplifies to the form:

$$e = \sqrt{\frac{h_2}{h_1}} \quad (2)$$

where h_1 and h_2 are the initial drop and rebound heights of the object, respectively.

2. Background

Many testing devices and methods have been designed to measure the coefficient of restitution of spherical objects such as steel, nylon, plastic or golf balls (ASTM, 2003; Foerster et al., 1994; Goldsmith, 1960; Kharaz et al., 1999; Labous et al., 1997; Paitich, 2006; Weir and Tallon, 2005; Zeiss, 1993). Measurements of collisional properties between two spherical objects or between a spherical object and a flat substrate have been made using images captured from high speed video cameras (Labous et al., 1997), stroboscopic illumination (Foerster et al., 1994; Sondergaard et al., 1990) or by measuring signals from electrical transducers (Kharaz et al., 1999; Paitich, 2006). The devices differ in the range of impact velocities and angle of relative approach during testing, and some of the devices have also included the ability to measure the CoR for repetitive collisions (Paitich, 2006; Weir and Tallon, 2005).

Experimental measurements of the CoR for non-pharmaceutical materials are extensively described in the literature. For example, measurements of CoR for steel and Pyrex balls bouncing off Lucite plates were performed by Sondergaard et al. (1990). They observed that the CoR did not vary significantly for low impact velocities (<2 m/s), V_i, and varied as $(V_i)^{-1/4}$ for high impact velocities. A similar dependence of the CoR on the impact velocity was observed by Labous et al. (1997) for collisions between nylon spheres of different diameters. They further observed that for low impact velocities, the CoR increased with the diameter or mass of the spheres. This dependence could not be verified at large impact velocities due to the limitation in their experimental set-up. Similar trends between

Table 1
Summary of theoretical predictions of CoR by various researchers.

	Dissipation	Coefficient of restitution (e)	Applicability
Johnson (1985)	Fully plastic	$e = 1.718\left(\frac{\sigma_y^5}{E^{*4}\rho}\right)^{1/8} V_i^{-1/4}$	$V_i \gg V_y$
Thornton (1997)	Elastic–plastic	$e = 1.185\left(\frac{V_i}{V_y}\right)^{-1/4}$; $V_y = 1.56\left(\frac{\sigma_y^5}{E^{*4}\rho}\right)^{1/2}$	$V_i \gg V_y$
Labous et al. (1997)	Elastic–plastic	$e = 1.18\left(\frac{V_i}{V_y}\right)^{-1/4}$ $V_y = 0.346\left(\frac{\sigma_y^5}{E^{*4}\rho}\right)^{1/2}$	$V_i > V_y$
Kuwabara and Kono (1987)	Viscoelastic	$1 - e \propto V_i^{1/5}$	$V_i \leq V_y$

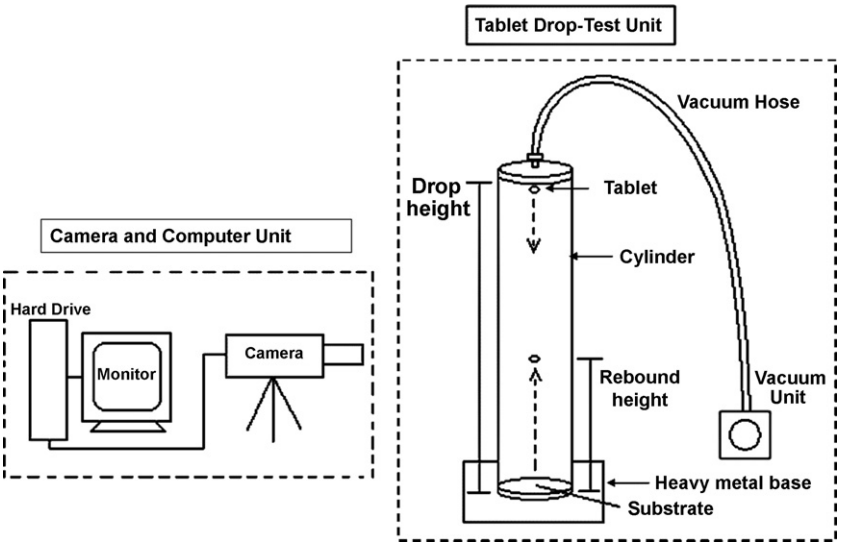

Fig. 1. Schematic of the experimental set-up of the coefficient of restitution (CoR) tester.

CoR and V_i, were also observed by Mangwandi et al. (2007) for impacts of different types of representative granules viz. melt, wet and binderless granules with a glass plate. Sondergaard et al. (1990) also observed that there is an apparent reduction in the CoR as the ratio between the sphere diameters to the plate thickness increases. The explanation for this was well captured by Zener's theory (Zener, 1941) and this apparent reduction in e was made negligible by using a plate of sufficient thickness.

Several theoretical models for the prediction of the CoR have been developed based on the different energy dissipation mechanisms that could be occurring during collisions at different impact velocities. A summary of various theoretical predictions of the CoR using quasi-static mechanics principles is given in Table 1. Kuwabara and Kono (1987) proposed a model that included viscoelastic effects based on Hertz's theory (Johnson, 1985) and found good agreement with experiments at low impact velocities (<1 m/s). Johnson (1985) proposed a model that included plastic dissipation during impact and obtained an expression for the CoR that was independent of the size of the spheres, but varied only with the impact velocity as $(V_i)^{-1/4}$. Johnson's model was verified with data obtained from experiments performed by Goldsmith (1960) using steel balls impacting different materials at high velocities. Johnson's model was applicable when the impact velocities were substantially higher than the yield velocity, V_y, of the material. In order to extend Johnson's prediction to intermediate values of impact velocities (the impact velocities are closer to the yield velocity), Labous et al. (1997) and Thornton and co-workers (Thornton, 1997; Thornton and Ning, 1998; Wu et al., 2003) proposed a model that included an elastic contribution to the total energy dissipation during the collision. In agreement with Johnson, they found that in this elastic–plastic regime, the CoR was only a function of the impact velocity as $(V_i)^{-1/4}$.

There is no existing data for the CoR of pharmaceutically relevant materials in the literature to our knowledge. The objective of the current work was to develop a procedure based on a simple drop test for measuring the CoR of tablets impacting metal or polymer substrates. CoR data for common pharmaceutical materials (excipients and blends) at different experimental conditions could then be generated to provide information needed for computer simulations of granular flow and rheological theoretical models.

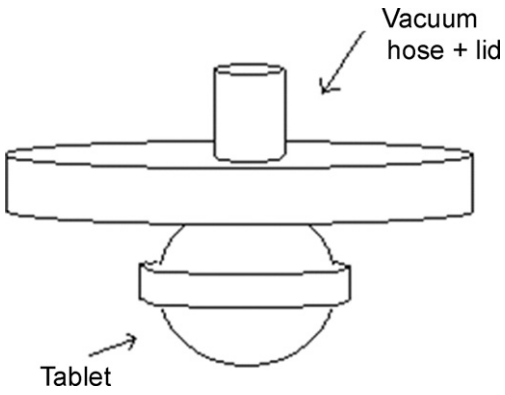

Fig. 2. Schematic of the initial tablet orientation on the lid for successful normal impact.

3. Materials and methods

The CoR tester used in this work is reliant on gravity to produce the desired velocities for the tablet impacts. The test was performed by dropping a tablet from a specified height and measuring its rebound height after impact with the substrate. Fig. 1 shows a schematic of the experimental set-up. The free-fall tester and camera unit were designed to accommodate the unique geometry of pharmaceutical tablets as their shapes are more complex than simple spheres. A graduated long hollow cylindrical tube (inner diameter = 100 mm and thickness = 12 mm) of known height (133, 310, 655 or 1000 mm) and made of clear polycarbonate (acrylic) material sits on a heavy base. At the top of the tube, there is a removable lid with a hole drilled through the center. The hole allows for a vacuum hose to be inserted, providing the necessary means to hold the tablet in place prior to free-fall. At the bottom of the tube is placed a solid cylindrical metal substrate of thickness 9 mm and diameter slightly less than the acrylic tubing. The substrate is laid on top of the base, and encompassed by the acrylic tube. This ensured that any cross-flow of air was minimized during testing.

To conduct the test, the tablet was placed under the removable lid and the vacuum was used to hold it in position. Its position was adjusted until the band is parallel with the lid (Fig. 2). This adjustment was required in order to minimize the spin of the tablet during

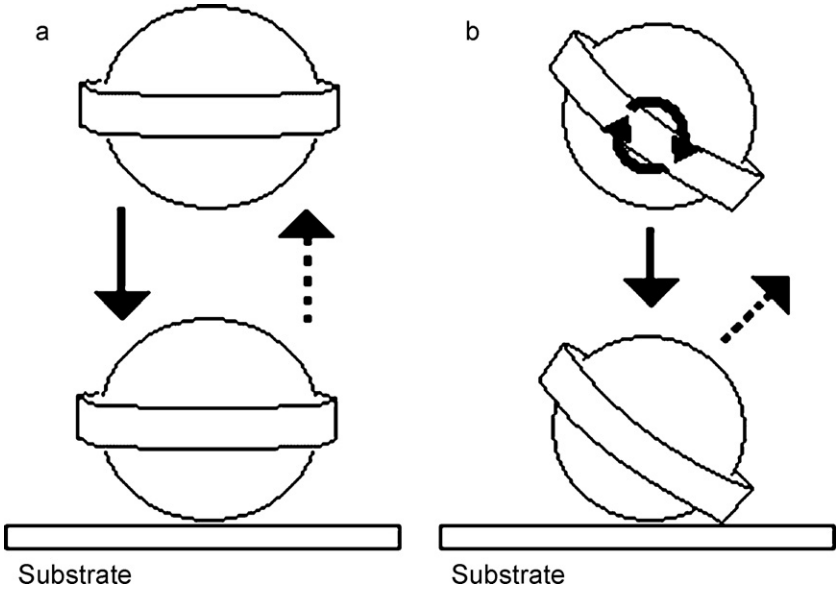

Fig. 3. (a) Successful normal impact with the substrate. (b) Unsuccessful oblique impact with the substrate.

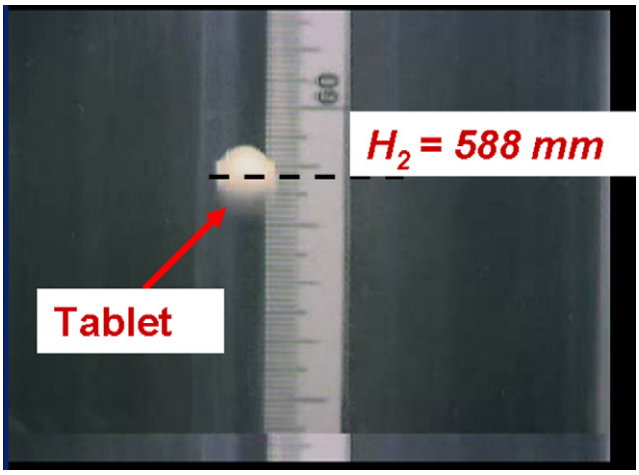

Fig. 4. Photograph of the tablet at the end of its rebound.

free fall and to achieve a successful normal impact (Fig. 3) with the substrate. The lid was then placed in position on the cylinder and after the vacuum was cut off, the tablet fell under gravity and collided with the substrate at the bottom. Oblique impacts (due to tablet rotation) with the substrate resulted in collisions with the walls of the cylinder during tablet rebound thus making measurements unsuccessful. For normal impact between the tablet and the substrate, the rebound height was noted with the aid of a tripod mounted Generic 1394 Desktop Camera, operating at 30 frames per second. The camera images were captured to a desktop computer hard drive, and viewed using simple video recording software (Microsoft Windows Movie Maker (v 1.1.2427.1)). Each frame of the video was carefully examined in sequence and the rebound height, h_2, was determined by the maximum height achieved by the center of the tablet (Fig. 4). The coefficient of restitution was then calculated using Eq. (2). The procedure was repeated three times for each tablet undergoing a successful normal impact.

Table 2 summarizes various experimental parameters used in the present study.

The tablets for the test were made at several solid fractions in a custom built pneumatic triaxial tablet press (Balog and Hiestand, 1989; Hancock et al., 2002) using a modified ball punch tooling (tip diameter and cup depth of 9.525 and 3.023 mm, respectively (TSM, 2006)). The press allowed for gradual triaxial decompression of the samples thereby minimizing the residual stresses in the tablets. A 5% suspension of magnesium sterate in methanol was applied using a cotton swab on the tool surface for lubrication. The desired solid fraction was obtained by adjusting the powder weight in the die (range: 300–500 mg), while keeping the tablet thickness constant (7.5 ± 0.5 mm). The tablets were uncoated and had no logos, bisects or scores on their surfaces. The modified ball cup depth was chosen so to keep the deviation of the shape from a sphere to a minimum thereby increasing the probability of normal impacts with the substrate during free fall. Several standard excipient materials and their mixtures were chosen for the tablet formulations. The excipients were chosen with a wide range of Young's modulus values (Bassam et al., 1990) so as to test the sensitivity of the method to the tablet elasticity. The solid fractions of the tablets typically ranged between 0.5 and 0.9. The range was narrower for bulk inorganic excipients and formulations with significant percentages of inorganic excipients as higher solid fraction tablets could not be made at normal compression forces without damaging the tooling.

The substrate materials were selected to be representative of materials commonly used for pharmaceutical processing equipment (polymethyl methacrylate or Plexiglas, steel and polytetrafluoroethylene (PTFE)). The substrates were cleaned with

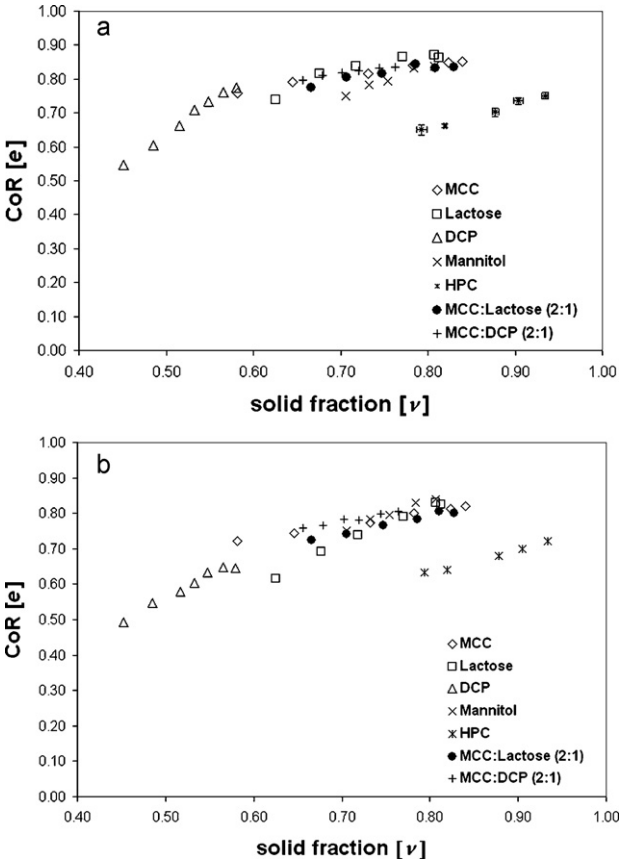

Fig. 5. The coefficient of restitution as a function of the solid fraction for various tablet formulations impacting (a) Plexiglas and (b) steel substrates at a velocity of 1.62 m/s.

alcohol wipes to remove any dust, oil, *etc.* prior to conducting the tests. The effects of tablet impact velocities on the CoR (if any) were studied by dropping the tablets from different heights: 133, 310, 655 and 1000 mm corresponding to impact velocities of 1.62, 2.46, 3.58 and 4.42 m/s.

Three tablets at each solid fraction condition were prepared and three successful trials were performed for each tablet from a particular height (impact velocity) to ensure reproducibility of data. After each trial, the tablet and the substrates were examined visually for any defects or breakage due to impact. Moreover, repeat tests on the same tablet were performed after a time elapse of ~5 min This procedure aimed to minimize the effect of tablet delayed recovery (Schwager and Pöschel, 2008); strain hardening (Mangwandi et al., 2007) and successive deformation (Weir and Tallon, 2005) that might occur due to repeated multiple impacts thus causing an apparent reduction in the CoR.

4. Results and discussion

4.1. Effect of tablet formulation on the CoR

The coefficient of restitution for various tablet formulations (Table 2) impacting Plexiglas and steel substrates at a velocity of 1.62 m/s (drop height of 133 mm) is given in Fig. 5(a) and (b), respectively. The CoR increases with the solid fraction, ν, for all the formulations. Tablets and mixtures having a larger percentage of elastic materials in their formulation (such as MCC, lactose and mannitol) had similar CoR values for the range of solid fractions investigated. Since it was difficult to prepare tablets at solid fractions >0.6 using dicalcium phosphate (DCP), it was not possible

Table 2
Summary of tablet formulations and experimental test conditions.

Tablet formulation	Tablet solid fraction range	Tablet impact velocities (m/s)		
		Plexiglas	Steel	PTFE
Microcrystalline cellulose (MCC Avicel PH 102)	0.57–0.84	1.62, 2.46, 3.58 & 4.42	1.62, 2.46, 3.58 & 4.42	1.62 & 2.46
Lactose mono hydrate (FastFlo)	0.62–0.83	1.62, 2.46, 3.58 & 4.42	1.62, 2.46, 3.58 & 4.42	1.62 & 2.46
Hydroxypropyl cellulose (Klucel EXF)	0.74–0.94	1.62 & 2.46	1.62 & 2.46	–
Mannitol (spray dried)	0.61–0.81	1.62	1.62	–
Dibasic calcium phosphate (DCP)	0.45–0.58	1.62	1.62	–
Mixture (MCC:lactose (2:1))	0.67–0.83	1.62	1.62	–
Mixture (MCC:DCP (2:1))	0.67–0.76	1.62	1.62	–

to make a direct comparison with other formulations, however, it appears that extrapolation of the data would result in similar values of CoR in comparison with the other elastic materials. Tablet formulations with hydroxypropyl cellulose (HPC) had lower CoR in comparison with the other formulations. This could be attributed to the low hardness of the compact surface in comparison with the other formulations (Mullarney and Hancock, 2006). Note that typical error bars representing the standard deviation are shown in Fig. 5 for only the HPC tablet–Plexiglas substrate combination for clarity.

4.2. Effect of substrate material on the CoR

The CoR of MCC tablet formulations impacting three different substrates at an impact velocity of 1.62 m/s is shown in Fig. 6. The CoR increases with the tablet solid fraction for impacts with both the Plexiglas and steel substrates. The CoR for MCC–Plexiglas is higher than the MCC–steel combination. For the softer polytetrafluoroethylene (PTFE) substrate, the CoR increases up to a solid fraction of 0.7 and decreases thereafter. This trend was not observed for the other substrates. Although, not visible with the naked eye, this could be attributed to some permanent deformation of the tablets after impact. Table 3 summarizes the CoR for various tablet formulations at a typical solid fraction of 0.8 impacting various substrates at velocity of 1.62 m/s. As expected (Table 1), it was evident that the more elastic substrates (Young's Modulus of PTFE < Plexiglas < steel) have higher CoR values.

4.3. Effect of impact velocity on the CoR

Fig. 7(a) plots the CoR of MCC tablets impacting a steel substrate at various velocities. The CoR decreases as the impact velocity increases for the range of solid fractions studied. Similar to previous observations for metal balls (Sondergaard et al., 1990), the CoR

Table 3
CoR of tablets at solid fraction of 0.8 impacting various substrates at a velocity of 1.62 m/s.

Tablet formulation	CoR		
	Plexiglas	Steel	PTFE
Microcrystalline cellulose (MCC Avicel PH 102)	0.84	0.80	0.79
Lactose mono hydrate (FastFlo)	0.87	0.82	0.76
Hydroxypropyl cellulose (Klucel EXF)	0.65	0.63	–
Mannitol (spray dried)	0.84	0.75	–
Mixture (MCC:lactose(2:1))	0.83	0.79	–
Mixture (MCC:DCP (2:1))	0.85	0.82	–

seems to have a power law dependence with the impact velocity for this tablet–substrate impact combination. However, there seems to be some deviation with respect to the exact relationship as observed in metal balls ($CoR \propto (V_i)^{-1/4}$). Fig. 7(b) plots the CoR

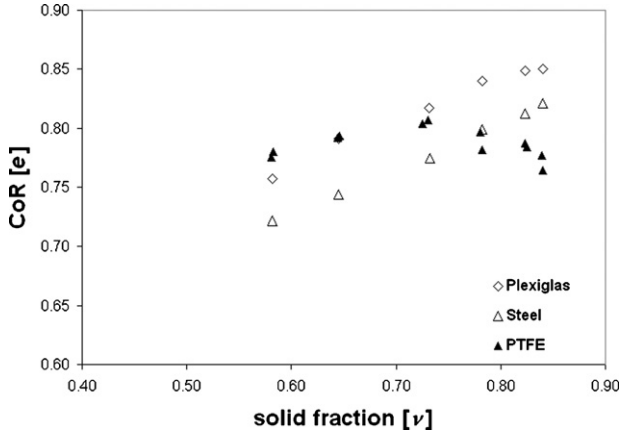

Fig. 6. The coefficient of restitution as a function of the solid fraction for MCC tablets impacting various substrates at a velocity of 1.62 m/s.

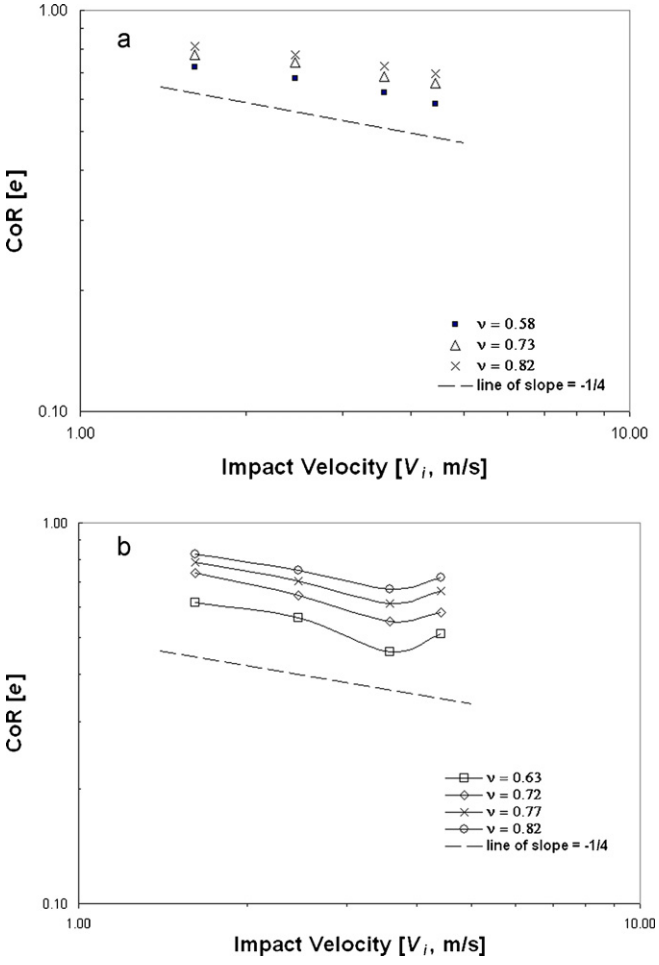

Fig. 7. The coefficient of restitution as a function of the impact velocity for (a) MCC and (b) lactose tablets of various solid fractions (□) impacting a steel substrate.

of lactose tablets impacting a steel substrate at various velocities. Although a decreasing trend with velocities was observed, there appears to be a minimum at a velocity of 3.58 m/s for the range of solid fractions studied. This is more evident at the lowest sold fraction of 0.63. Similar observations of minima at 3.58 m/s were made with MCC and lactose tablets impacting a Plexiglas substrate (data not shown). Again, these minima can be attributed to the local plastic deformation at the impact location at the high velocities. Since it was not possible to test impact velocities between 3.58 and 4.43 m/s using the current tester, more data could not be gathered to further investigate this trend. However, this could be possibly considered for further investigation in the future. The error bars representing the standard deviation are eliminated for clarity as they were <0.05% of the mean CoR values.

4.4. Comparison to theoretical predictions

Models (Johnson, 1985; Thornton, 1997; Thornton and Ning, 1998; Wu et al., 2003) (Table 1) for predicting the CoR using principles of quasi-static mechanics include properties of the materials such as the Young's modulus (E), density (ρ) and yield stress (σ_y). However, for pharmaceutical materials, properties such as the Young's modulus and yield stress are inter-dependant and are controlled by the porosity or solid fraction of the tablet. In order to make a comparison between predicted CoR and experiments for one tablet formulation viz. MCC, certain assumptions regarding the tablet properties were made which are explained below.

The Young's moduli at each solid fraction of the MCC tablets were obtained from Fig. 3 in Bassam et al. (1990). The yield pressures (P_y) corresponding to these Young's moduli were then obtained from data measured from the beam bending experiments of Roberts and Rowe in Sonnergaard (1999). It was assumed that MCC data would follow a similar linear trend as in Fig. 3 in Sonnergaard (1999). Assuming, elastic–plastic deformation during impact, the yield pressure (P_y) in the material was assumed to be ~1.1 σ_y to 3σ_y (Johnson, 1985), where σ_y is the yield stress. The predicted CoR was thus calculated at each solid fraction using the equations given in Table 1.

Fig. 8 shows a comparison between the experimental and theoretical CoR predictions for the MCC tablet–Plexiglas substrate combination. As predicted by theory, the experimental CoR data seems to follow a power law relationship with velocity (Fig. 8(a)). Previous theories seem to predict the CoR values for MCC reasonably well. However, they are sensitive to the material properties such as the Young's modulus and yield stress used in their calculations. Similar observations were made between theory and experiments for the relationship between CoR and solid fraction (Fig. 8(b)).

4.5. CoR of some commercial tablets

To compare the data for simple materials to actual pharmaceutical tablets, CoR measurements on commercial products purchased from a local pharmacy were also made using the tester. These tablets were used as-received and were similar to the tablets used in the work by Hancock et al. (2010). They included acetominophen 500 mg gel-coated tablets (Albertsons Inc., Boise, ID, lot 7ME0568), 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 sugar-coated tablets (Wyeth Consumer Healthcare, Madison, NJ, lot C12611), vitamin-C 500 mg uncoated tablets (Natures Bounty Inc., Bohemia, NY, lot 151074-01) and Tylenol PM. The tablets were biconvex in shape (Fig. 3 in Hancock et al., 2010) and multiple trials were performed to obtain successful impacts with the substrates due to frequent rotation and spin during free fall. Fig. 9 shows the

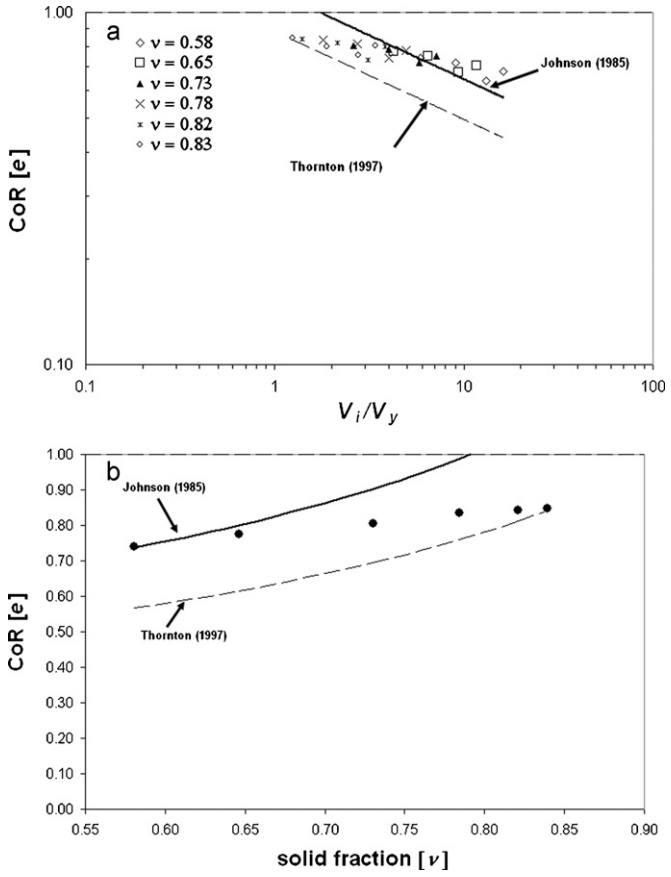

Fig. 8. The experimental and theoretical predictions of coefficient of restitution as a function of the (a) normalized impact velocity and (b) solid fraction for MCC tablets impacting a Plexiglas substrate.

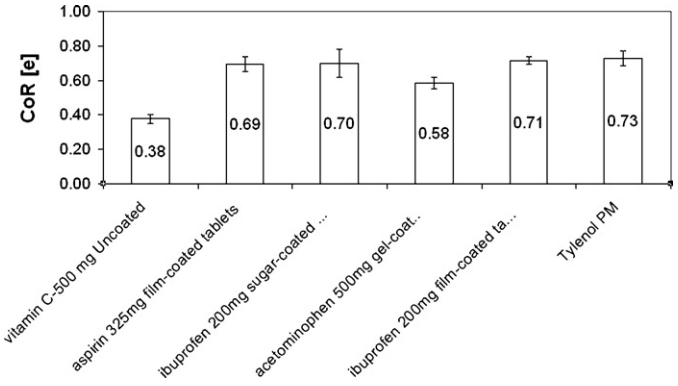

Fig. 9. CoR of commercial tablets impacting a Plexiglas substrate at a velocity of 1.62 m/s.

CoR for these commercial formulations impacting a Plexiglas substrate at a velocity of 1.62 m/s. Error bars representing one standard deviation from the mean are also shown. The data for most of the real tablets, assumed to be at a solid fraction of 0.85, was ~0.7 and were comparable to the simple formulations under similar impact conditions. The coated tablets have higher CoR values (~0.7) in comparison to the uncoated tablet.

5. Conclusions

In this work, the CoR of a range of pharmaceutical tablets/compacts has been measured, and the influences of material properties (elastic modulus, solid fraction, *etc.*) and colli-

sion conditions (substrate, energy/speed, *etc.*) were investigated. The compacted pharmaceutical materials have CoR values that range from 0.4 to 0.9, and the CoR increases with increasing compact solid fraction in most cases. The CoR varies with the mechanical properties of both colliding bodies and is lower for more plastic collisions and higher for elastic collisions. This behavior is consistent with theories developed for non-pharmaceutical solids, and can be predicted provided that the elasticity and yield stress of the materials are treated as porosity dependent parameters. In this case, the CoR varies with the impact velocity nearly raised to the fourth root.

Having established a simple and reproducible test for the CoR of pharmaceutical compacts and tablets it should be possible to create more accurate engineering models and computer simulations of tablet manufacturing and packaging operations. Collisions involving more plastic materials, such as HPC, would be expected to be 'damped' to a greater extent and result in less energetic rebound behavior. Conversely, tablets of less plastic materials or those that have been compressed to a high solid fraction would be expected to behave in a largely elastic manner and require significantly more collisions to dissipate their initial kinetic and potential energies. For tablet film coating processes using a fluidized bed system (Wurster coating), the CoR could be expected to influence the tablet fluidization and mixing patterns. Likewise, for blister and bottle packaging operations where there are a significant number of tablet collisions, the CoR could be expected to influence the overall filling efficiency and maximum filling rate.

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