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**Effect of lubrication on the distribution of force
between spherical agglomerates during compression**

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We employ the carbon paper technique to aid understanding of in die force and spatial distributions, upon compression of approximately 1 mm sized spherical agglomerates (pellets) of microcrystalline cellulose (MCC). The aim in this study was to test for the effect of lubricant film on force and spatial distributions. Pellets of MCC were formed via granulation and extrusion/spheronisation. Investigation of pellet bed compression was performed on a materials tester. Prior to compression studies the pellets were characterised for bulk density, size and deformability. Two pellet types were investigated; MCC and MCC lubricated with magnesium stearate. The carbon paper technique relies upon carbon paper as the medium for transferring imprints from compressed pellets onto photo quality paper. The digitised images of these imprints form the basis of analysis through the use of image processing software. Using the carbon paper technique within the range of 10-30 MPa indicates that lubrication does not have a significant effect on the distribution of forces between spherical agglomerates during uniaxial compression.

Spatial analysis of the imprints revealed that the lubricated pellets exhibited a higher packing order than the unlubricated ones at low applied pressures (10 and 20 MPa), a difference that could not be observed at 30 MPa. Hence interparticle friction and/or cohesion appear to influence the initial particle rearrangement, whereas confinement is suggested to dominate at higher pressures.

Keywords: Granular materials; Compression; Force distributions; Carbon paper technique; Lubrication

1. Introduction

Any particulate system whose components are greater than $1\mu\text{m}$ in size, is referred to as belonging to a group of materials called granular matter. This definition encompasses a vast and diverse range of natural and synthetic materials. Granular matter is in terms of amount, the second most manipulated material by man [1] and due to its unique properties, which cannot be strictly classified solely according to those possessed by solids, liquids or gases, can be viewed as being an additional state of matter [2]. Most drugs and excipients can trace their origins back to a powdered state. Tablets, being one of the most widely used dosage forms, are essentially composed of compressed free flowing powder. The response of these systems under compression is dependent on factors relating to their inherent properties and process parameters. Compact formation, invariably necessitates various particulate micromechanical changes within the powder/agglomerate bed, to instigate cohesion. There is however a paradox currently inherent in tablet production, whereby, even with their longstanding and broad pharmaceutical applications, the fundamental processes involved in tablet formation are not fully understood. A major hindrance to this understanding is that, the powder particles are simply too small and numerous to investigate individually. This is an intriguing paradox that has prompted a wide arena of research. There have been various diverse approaches to understanding the in die compaction process, some involving global models attributed to Heckel [3], Kawakita [4] and Adams [5], others include in-silico modelling, such as the discrete [6] and finite element methods [7, 8], or combinations of the two [9, 10]. Often, spherical agglomerates have been used as a simple model system (in relative terms) [11-14]. The parameters developed through these approaches generally

aim to elucidate single particle properties from bulk compression events. Some workers have used imaging, as a means to elucidate the processes occurring during the compression of spheres [15], and to characterise the spherical agglomerates undergoing compression [16]. Attempts have also been made, through the use of X ray microtomography and DEM via the radial distribution function, to investigate packing of spheres [17].

The carbon paper technique has been a tool employed in physics, to highlight force distributions within granular assemblies [18-21]. Force distribution studies have not received much attention in the pharmaceutical sector. Other than a preliminary study [14], the method employed in this paper has not been previously applied to pharmaceutical tableting research. Nevertheless the carbon paper technique can equally well be used in pharmaceutical research, to study granular materials and gain greater insights into their compaction behaviour. In this work we employ approximately 1 mm sized, roughly spherical agglomerates (pellets) and carbon paper, as the mediums for investigation of force and spatial distributions upon compression.

It is innately apparent that force propagation through a bulk granular medium occurs via contact points between particles. These points are random in nature and as such, the distribution of transmitted forces through them is not intuitively apparent, and does not follow any reproducibly predetermined path. It is general knowledge now, that ensuing force chains follow certain preferential channels through the bulk collection of material (percolation sites). The distribution of these load bearing chains results in dormant

regions that do not contribute to the overall force transmission and have been aptly termed as “spectators” [22]. The locations of these chains are not predictable in nature, and can be altered by agitation. At the size ranges under investigation in this paper the effect of temperature is negligible [1, 2] and can be disregarded. It is also known, that the force registered on the base of a bed of granular material, does not reflect the true force on the system. This is due to the lateral diversion of force components onto the boundary walls of the vessel in which the granular material is contained. Although the paths of force transmission are unpredictable and random, the distribution of these forces can be rationalised when viewed through the context of probability densities. From this viewpoint, the force distribution generally takes on a slightly skewed form. Forces below the average force $\langle F \rangle$ reach a plateau at or near the mean force and those above $\langle F \rangle$ follow an approximately exponential path[18-21]. For the purposes of this investigation, 2D force and spatial distributions are considered, as the foundations of investigation are based upon imprints transferred onto a flat Cartesian plane.

2. Experimental

2.1. Materials

MCC (Avicel PH101, FMC, Ireland), magnesium stearate (Ph. Eur., Kebo, Sweden) and de-ionised water. Black carbon paper (Radex 1200, Kores, Austria) and white photo quality paper (Epson photo quality ink jet paper, S041068, Seiko Epson Corp, Japan).

2.2. Pellet preparation

Three batches of MCC pellets were produced using the same processing conditions. MCC (400g) powder was aerated in a high shear mixer (QMM-II, Donsmark Process Technology, Denmark) at ≈ 500 rpm for 5 min. The agglomeration liquid (de-ionised water, 110% mass of MCC) was manually poured into the dry mass at a rate of approximately 100 ml/min while mixing. The resulting wet mass was mixed for a further 3 min at ≈ 500 rpm and immediately extruded (NICA System AB, model E140, Sweden; holes 1.0 mm in diameter and 1.2 mm long). The extrudate was spheronised (NICA System AB, model S 320-450, Sweden) for 3 min on a 32 cm diameter friction plate with a radially designed grid at a rotation speed of ≈ 850 rpm. Spheronisation time was started once all the extrudate had been transferred into the spheroniser. The ensuing pellets were spread into a thin layer on trays and allowed to dry under ambient conditions for at least 4 days. During the drying process the pellets adhered to each other forming clumps, and de-aggregation was accomplished by gentle tapping with a spoon. The desired pellet size fraction (800-900 μm) was obtained via sieving, through the vibration (Retsch, Type RV, West Germany) of square aperture sieves for 30 min at a relative intensity of 40 (on an arbitrary scale).

2.3. Characterization

I. Powder

The apparent particle density of MCC was determined using a helium pycnometer (AccuPyc 1330, Micromeritics, USA).

II. Pellets

Texture analysis: Pellets from each batch were compressed using a texture analyser (TA HDi texture analyser, Stable Micro Systems, UK) with a 6 mm diameter stainless steel rod. The compression profiles of the pellets were analysed (n=120). The slopes of the initial linear region of the force displacement plots were used in the calculation of yield stress through the equation [23, 24]:

$$\sigma_y = \frac{4}{\pi D} \frac{dF}{dx} \quad (1)$$

where D denotes the mean pellet diameter and dF / dx is the slope of the linear region.

Density/Porosity: Pellets were weighed in a 25ml measuring cylinder. The bulk volume occupied by the pellets was estimated after applying 1000 taps on the tap density tester (Pharma Test PT-TD, Hainburg, Germany), and the ratio of weight and volume was calculated to give the tapped density (n=3). Pellet porosity ε was estimated through calculation:

$$\varepsilon = 1 - \rho^{bulk} / (\phi_c \rho^{solid}) \quad (2)$$

where ρ is density and ϕ_c is the packing fraction for random loose packing [25] with a value of 0.6284 which is within the range generally obtained for spherical particles [26].

Pellet size and shape: Pellets were spread out on a flatbed scanner (Epson Perfection 1640SU Scanner, Seiko Epson Corp., Japan) and covered by a black background. 1600 dpi images of the pellets were captured. The images were analysed via ImageJ [27], a public domain image analysis programme. The projected-area diameter was calculated through the equation:

$$D = \sqrt{4A/\pi} \quad (3)$$

where A is the projected area of the pellet. The ImageJ parameter “fit ellipse” produced the outputs “minor” and “major”, which correspond to the primary and secondary axes derived from fitting an ellipse around the pellet. The ratio minor/major was used to determine circularity. The diameter and circularity values obtained through this image analysis are an approximate estimation, and as such are presented as a guide only.

2.4. Lubrication

About a third of the MCC pellets were lubricated with 0.1% by weight of magnesium stearate via a turbula mixer (Willy A. Bachofen AG Maschinenfabrik, Basel, Switzerland) for 100 min at 67 rpm. The mixing time was based on previous experience [11], to ensure thorough and even film distribution around each pellet. Prior to their use

in the study, lubricated pellets were compressed at high pressure (200 MPa), to elucidate if the magnesium stearate film was successful in adequately reducing any in die adhesive and cohesive forces.

All pellets were conditioned in a closed chamber at 40% relative humidity and room temperature for at least 7 days prior to any tests.

2.5. Calibration

Uniaxial constrained compression analysis of single pellets was carried out via the Stable Microsystems TA HDi texture analyser (Stable Micro Systems Ltd, Surrey, UK) equipped with a 50 N load cell. Special apparatus was designed and manufactured, that would attempt to simulate conditions similar to those found at the base of a bed of pellets in a die. Fig. 1 shows a cross sectional schematic presentation of the experimental setup. Pieces of white and carbon paper of diameter 11.3 mm were cut out using a stencil tool designed for the purpose. These were placed in a double layer, with the white paper adjacent to the base and the carbon paper placed on top of it. The free moving plastic base had a cavity cut into it with the same dimensions as the teflon chamber and papers which were placed there for each compression (Fig. 1). The angular sides of the low friction teflon die allow the base to move such that the probe can be directed onto the pellet. The diameter of the die was approximately 1 mm which would act as a constraining surface for the pellet in an attempt to mimic the bulk compression environment. Each pellet was subjected to a compression rate of 0.1 mm/s, with

minimum dwell time. Forces between 2 and 50 N were applied with 10 repetitions for each increment. Due to limitations of the load cell on the texture analyser, the resulting calibration curve would require extrapolation at forces beyond 50 N. To avoid extrapolation, single pellet compression data at 100 N derived from the the Zwick Z100 materials tester (Zwick/Roell, Zwick GmbH & Co. KG, Germany) was included in the calibration (non-constrained at a compression rate of 1 mm/min). The transferred imprints were used as calibration standards.

2.6. Bulk compression

Uniaxial compression was carried out on pellet beds via the same materials tester as mentioned above. The apparatus was equipped with a mobile upper punch attached to a 100 kN load cell and a stationary lower punch (diameter 11.3 mm) and die. Prior to all compression events the die wall was lubricated with 1% w/w magnesium stearate suspension in ethanol. A weight of 1 g of pellets was manually poured into the die, covering the dual layer of white/carbon paper described above. The top of the pellet bed was also covered with the paper dual layer. Compression was carried out at a rate of 25 mm/min with minimum dwell time. Three pressure increments between 10 and 30 MPa were applied. At each increment 20 replications were made. The compact was ejected from the die immediately after completion of the compression sequence. Although tensile strengths of any coherent compacts were measured, these cannot be regarded as having any analytical value, as the compacts were much too weak and most broke apart with slight agitation during handling. As such these values are not included. Fig. 2 shows a schematic representation of the experimental setup.

2.7. Carbon paper analysis

Digital images of all imprints were captured via the flatbed scanner at a resolution of 1600 dpi. Images were processed via Photoshop Elements 5 (Adobe Systems Incorporated) prior to analysis. This was done so as to reduce file size, speed up analysis and decrease the number of separate files being produced. ImageJ was employed for this investigation. A threshold range of 1-220 was applied for all images. The ImageJ output parameters of interest were, centre of mass and area.

In calibration analysis, the known applied force and area values derived from ImageJ were correlated, and a regression function relating the two terms was derived. Imprints formed from forces of 2 N to 100 N were used in the calibration. Fig. 3 shows the calibration curve used to derive the force. The solid line is based on a third order polynomial.

Bulk compression analysis was based on an implementation of a watershed algorithm [28]. The watershed algorithm was used to clearly subdivide the relevant region of each image into a number of distinct sub-regions (basins). Each of these basins corresponded to the imprint made by a single pellet. Image smoothing (Gaussian smoothing, $s_x=s_y=10$) was employed to facilitate the application of the watershed algorithm, by reducing over segregation of the image.

2.8. Statistical analysis

The global average force $\langle F \rangle$ was calculated at each pressure increment. This was used to normalise forces within each pressure increment to give $f = F/\langle F \rangle$. Normalising the forces standardises the abscissa resulting in a clearer presentation and more intuitive interpretation of the data. The force data was binned (bin width 0.1) and the probability densities $P(f)$ and standard errors for force distributions were estimated from bin counts in a standard manner, using the R statistical programme [29]

For spatial analysis, twenty separate files were produced for each pressure increment and pellet type combination. The position of each imprint (derived from the “centre of mass” parameter of ImageJ) was used to determine the 2D pair-correlation function $g(r)$ through the R package and its SPATSTAT algorithm. A translation edge correction was applied to this. The 2D pair correlation function is defined as:

$$g(r) = \frac{1}{Nn_0\pi r} \sum_{i=1}^N \sum_{j=i+1}^N \delta(r_{ij} - r) \quad (4)$$

where N is the total number of imprints, n_0 is the average density of imprints, and r_{ij} is the distance between the centres of imprints i and j . Moreover, r may be interpreted as the distance from an arbitrarily selected particle.

3. Results and Discussion

1. Powder and pellet characteristics

Pellet characteristics are displayed in table 1. The tapped density, estimated porosity and yield stress do not deviate to a great extent from pellets produced in our group on previous occasions [12, 13]. Our pellets are approximately circular as inferred from the circularity value of 0.858. When the lubricated pellets were compressed at pressures much exceeding those employed in the analysis, they did not form coherent compacts, demonstrating that the lubrication level was adequate in reducing adhesive and cohesive forces.

II. Force transmission

Fig. 4 displays the mean transmitted force from all experimental runs for both pellet types at each pressure increment. There is a tendency of force transmission being greater through the lubricated pellet beds. This is to be expected as there is less frictional resistance to compaction. As applied pressure is increased, the amount of transmitted force as a proportion of applied pressure decreases for lubricated pellets and stays more or less constant for unlubricated pellets. It is also apparent that the transmitted forces of both pellet types begin to converge with increasing compression pressure. On average for all pressure increments there is a $\approx 83\%$ and $\approx 76\%$ force transmission for lubricated and unlubricated pellets respectively.

III. Force distribution

The probability distribution of the normal forces acting in regions of particle contact, as determined from the carbon paper imprints, may be summarised by the probability density function $P(f)$. Plots of the force distributions with their corresponding

compression pressures are shown in Fig. 5. Statistical data was gathered in a standard manner and is presented in table 2. As has been performed in previous studies, the convention for presenting force distributions is to use a semi log plot, which emphasises the smaller probabilities to a greater extent. The force distributions in this study share qualitative similarities with previous work [14, 19, 21], where it is observed that the force distribution is unimodal, with an exponential decline for forces greater than $\langle F \rangle$. The $P(f)$ profiles of the unlubricated pellets in Fig. 5 seem to have somewhat steeper exponential regions than those from work conducted previously in our group [14]. Furthermore, in comparison, the transmitted forces presented in Fig. 4 are greater at each corresponding pressure increment. These discrepancies probably have their roots in the fact that die lubrication was not employed in the aforementioned study [14].

It could be hypothesised that the lubricated pellets are by virtue of decreased friction, allowed to slide past each other and enter optimal packing arrangements, where the force is more evenly distributed between them, than is the case for the unlubricated ones.

However, the curves for the two pellet types seem to follow very similar paths and have overlapping error bars. Although values obtained through the carbon paper technique should not be considered as absolute, It seems natural to, based upon this, draw the conclusion that lubrication has no substantial effect on $P(f)$ at these pressure increments. In general the values presented in table 2 are similar for the lubricated and unlubricated pellets at each pressure increment. This similarity may be viewed as resulting from both the lubricated and unlubricated assemblies being coerced into physically constrained packing states, by virtue of increasing pressure i.e. force trumps lubrication.

Consequently the two pellet assemblies may end up sharing greater structural similarities,

and as such drive the force distributions within them toward homogeneity, effectively diminishing the role of lubrication. In line with this finding, work done by Blair et al. [21] found no significant difference when comparing the $P(f)$ profiles of their smooth and rough beads, which in this context, may be interpreted as being analogous to our lubricated and unlubricated pellets.

It is apparent that with increasing pressure, there is a tendency for a reduction in the probability of finding forces below the mean, and a narrowing and heightening of the peaks near $\langle F \rangle$. Both of these features are mirrored in work performed by Frenning and Alderborn [14]. These changes indicate that there is a tendency toward a greater concentration of forces in the vicinity of the mean with increasing compression pressure. This seems intuitive, as an increase in applied force results in greater areas and numbers of contacts between particles, which disperses the transmitted force more evenly through the bed, resulting in a global equalisation effect, whereby the system begins to enter a state of quasi-equilibrium at the mean force vicinity.

IV. Pair correlation function

The 2d pair correlation curves are displayed in Fig. 6. It is apparent that at all the pressure increments, the curves overlap and follow near identical paths both between and within increments. They all show three peaks and two troughs, with each peak being shorter, and each trough shallower, than the one preceding it. The forms of these curves follow an anticipated pattern, which is expected for spherical particles that do not interpenetrate. With progression from one coordination shell to the next, there is a reduction in the spatial density (as is evident from the reduced amplitude of the peaks) which eventually

narrows to the mean density beyond the third shell. The first coordination shell has a width of ≈ 0.9 nm and the second and third shells ≈ 0.8 nm. In general as mentioned above, the near identical paths and overlapping error bars of the curves for each pressure increment seem to suggest that there is no major effect of lubrication on the spatial distribution of pellets. However at the 10 and 20 MPa pressure increments there seems to be regions where slight differences are apparent between the lubricated and unlubricated pellets. These have a greater spread at 10 MPa and less so at 20 MPa when observing the peaks and troughs. A possible explanation for this could be that as friction is reduced, rearrangement is facilitated, resulting in greater spatial order. This trend seems to diminish with increasing pressure as the effect of applied force overcomes the frictional effects, and it seems to be non existent at the 30 MPa increment.

4. Conclusions

Using the carbon paper technique within the range of 10-30 MPa indicates that lubrication does not have a significant effect on the distribution of forces between pellets during uniaxial compression. For the investigated pellets of low porosity the force distributions were found to narrow with increasing compression pressure, in a manner independent of interparticle friction and cohesion. Spatial analysis of the imprints revealed that the lubricated pellets exhibited a higher packing order than the unlubricated ones at low applied pressures (10 and 20 MPa), a difference that could not be observed at 30 MPa. Hence interparticle friction and/or cohesion appear to influence the initial particle rearrangement, whereas confinement is suggested to dominate at higher pressures. It is hoped that the information gleaned from this and further studies can be

used to assess the applicability of this method, with the general aim of providing greater insights into the processes occurring during powder/agglomerate compression.

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Figures

Fig. 1. Schematic representation of calibration experimental setup.

Fig. 2. Schematic representation of experimental setup.

Fig. 3. Calibration curve (error bars indicate standard deviations).

Fig. 4. Average transmitted force for all compaction pressures (error bars indicate confidence intervals).

Fig. 5 Force distribution plots of unlubricated and lubricated MCC pellets (error bars indicate standard error).

Fig. 6. 2D pair correlation functions (error bars indicate standard error).

Tables

Table 1. Pellet characteristics, values in parentheses indicate standard deviations.

Characteristic	value
Apparent particle density of MCC (g/cm^3)	1.571 (0.002)
Tapped bulk density (g/cm^3)	0.877 (0.002)
Estimated porosity (-)	0.111 (0.002)
Diameter (mm)	0.919 (0.087)
Circularity (-)	0.858 (0.099)
Yield stress (MPa)	116 (11)

Table 2. Statistical measures of forces derived from carbon paper imprints.

Force moments	Unlubricated 10 MPa	Lubricated 10 MPa	Unlubricated 20 MPa	Lubricated 20 MPa	Unlubricated 30 MPa	Lubricated 30 MPa
Count	2227	2480	2499	2589	2484	2617
Mean(N)	6.993	7.627	11.972	12.222	18.411	17.040
SD (N)	3.138	3.204	4.084	3.939	5.950	5.296
Skewness	0.318	0.142	0.185	0.335	0.595	0.522
Kurtosis	0.122	-0.125	0.513	0.543	1.498	0.815