Breakage of Anisotropic Rod-Shaped Particles

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ABSTRACT Many products and intermediaries in industry are manufactured in the form of anisometric particles, depending on the molecular composition and finalization process (e.g. active pharmaceutical ingredients or pigments in dye industry). Some technological steps (filtration, filter washing, drying, transportation) can lead to uncontrolled changes in the Particle Size Distribution (PSD) due to particles attrition and breakage. When a randomly packed layer of anisometric particles is compressed (its weight, external force), it is of interest, both academic and practical, to find the relationship between the applied stress, compressibility and breakage.

In this experimental study, behaviour of dry granular layers of monodisperse cylindrical particles (8x1 mm) under uniaxial compression was studied. The layer compressibility and PSD were evaluated. The effect of several parameters was investigated: piston speed, layer size (diameter and height), applied pressure. Two modes of compression were tested: non-recurring and recurring. In the former mode, after the compression, the particles were taken out of the measuring cell for granulometry. In the latter mode, after granulometry, the particles returned to the cell for the next run with the same pressure.

It was found that neither the piston speed nor the layer size were relevant, within our experimental range. The resulting compressibility and PSD were affected mainly by the normal pressure and the way it was applied. In the non-recurring mode, with increasing pressure, PSD moves toward smaller sizes of broken particles always leaving some initial particles intact. In the recurring mode, the pressure had more destructive effect, resulting in larger particle fragmentation. The reason is seen in the particle re-arrangement between the successive tests.

1. INTRODUCTION

Many products and intermediaries in industry are manufactured in the form of anisometric particles, depending on the molecular composition and finalization process (e.g. crystals, active pharmaceutical ingredients or pigments in dye industry) [1-2]. Some technological steps (filtration, filter washing, drying, transportation) can lead to uncontrolled changes in grain size distribution due to attrition and breakage [3]. When a randomly packed layer of anisometric particles is compressed (its own weight, external force), it is of interest, both academic and practical, to find the relationship between the applied stress, compressibility and breakage. The aim of this study is to experimentally quantify the breakage events and estimate the effect of few relevant control parameters. Our experimental results can also be used for validation of the numerical modelling of granular beds [4].

2. EXPERIMENTAL

2.1 *Material properties*

Based on the preliminary tests, the rod-shaped pasta was used as the model granular material. The pasta was Capellini Major (Italy), made from durum wheat semolina. Using the multifunction tool DREMEL® 4200 equipped with diamond disk saw SC545, the 26 cm long pasta sticks of 1 mm diameter were cut into cylindrical particles of diameter d = 1.06±0.02 mm and length h = 8 mm, see Fig. 1a. Cutting impreciseness resulted in the initial distribution of particle sizes (PSD), see Fig. 1b. The material hardness parameters were determined by the three point rigidity test using Brookfield CT3 Texture analyser with the following result: the elastic modulus is 4.9 GPa, stiffness 292 kN/m, bending modulus 5.67 mNm and bending stress 57.9 MPa.

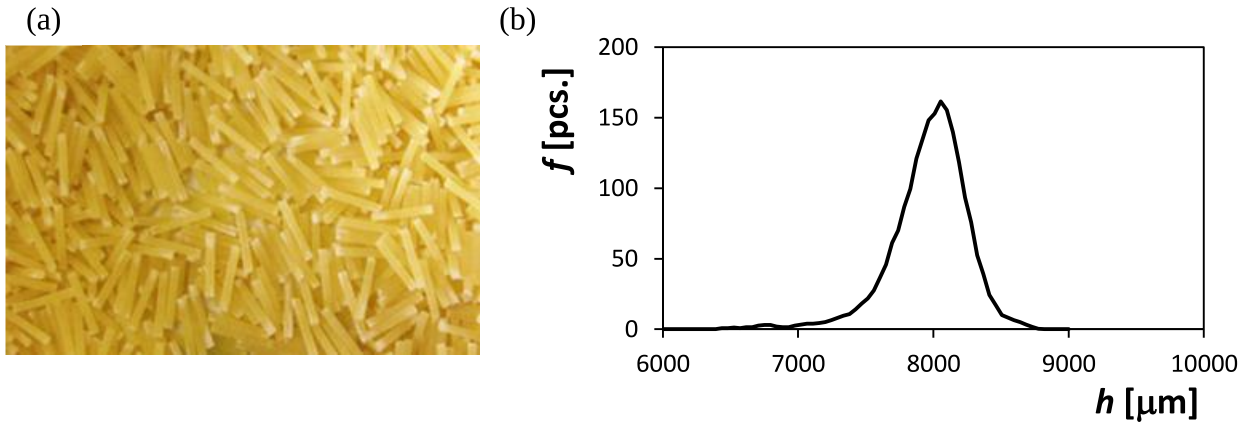

Figure 1: **R**od-like pasta material. (a) An illustrative photo. (b) Initial particle size distribution (PSD).

2.2 *Compressibility measurement*

The material was exposed to the uniaxial compression in Gamlen Tablet Press GTP-1 (maximum load 450 kg, i.e. 9 MPa in the smallest cell). Three metal (stainless steel) cylindrical measuring cells were used, with different sizes: diameters D = 25, 33, 41 mm referred to as S (small), M (medium), L (large). The nominal cells' height was 30 mm. Several distance pieces (metal disk-shaped spacers) were employed to adjust the initial height of the particles' layer to three values H = 10, 15, 20 mm. Certain amount of particles of initial volume $V_i = \pi D^2 H /4$ was put in the cell manually, by pouring through a funnel. The piston speed v and terminal load P_t was set and the measurement started. The piston moved downward at a constant speed and the resistance force of the layer was measured in time. The actual pressure P and piston position were recorded with sampling rate 200 Hz. From the piston position, the actual volume V was obtained and the compressibility calculated $C = (V_i\ -V/)/V_i$, usually shown in %. The typical reproducibility of the results from five measurements in terms of the standard deviation is 5%. The result was the dependence $C = C(P)$ shown in graphs known as compressibility curves. After reaching the terminal pressure P_t and the relating final compression C, the piston returned back to its initial position and the layer very slightly relaxed due to an elastic relieve (almost horizontal branch in the graph). Two modes of compression were tested: non-recurring and recurring. In the non-recurring mode, after the compression, the particles were taken out of the measuring cell for granulometry. In the recurring mode, after granulometry, the particles returned to the cell for the next run with the same pressure.

2.2 *Particle size distribution*

The particle size distribution (PSD) of the grains before and after the compression tests was measured by the dry granulometry device (Solids particle size analyser, CANTY, Industrial SolidSizer). An ensemble of particles (typically 2000) passed through the vibratory conveyor forming a slowly moving mono-layer. It slid along the inclined plane to ensure 2D motion of the particles in front of the black&white camera (320x240pxls, 5 fps). The particle images were analysed by the device software. Each 2D image of a particle was fitted inside enclosing rectangle whose major size was the particle length. The histograms of particle sizes were produced where the number of particles inside 200 regular bins is shown.

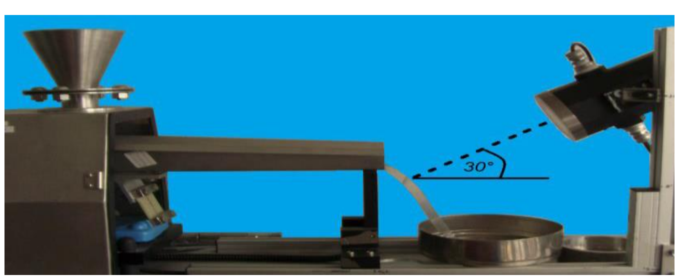

Figure 2: The arrangement of Industrial SolidSizer modified for granulometry of anisometric particles.

3. RESULTS AND DISCUSSION

3.1 *Effect of piston speed*

The effect of piston speed was studied first, to find its suitable value and fix it for the further experiments, to reduce the parameter space. The measuring cell of size M was used with the layer height H = 15 mm. The terminal pressure was P_t = 5100 kPa. Six different piston speeds were used, v = 0.1, 0.5, 1, 5, 10 and 60 mm/min, covering the range available in the device. The compressibility curves and PSD of samples were measured and the result is shown in Fig. 3. The graph of C versus P is increasing, almost linearly in the Log-Log plot. The layer relaxation is marked by the horizontal lines (pressure drop at maximum C). The distribution function is bi-modal, still having a small portion of the initial size particles (8 mm) at the end of the compression test. The population of the broken grains moves to the range of 2-4 mm, which is 1/2 and 1/4 of the original magnitude. No apparent piston speed effect can be observed, either in the graph shapes or in ordering the lines. The breaking process is independent on the piston speed. Based on this, the medium piston speed v = 5 mm/min was chosen as the reference value.

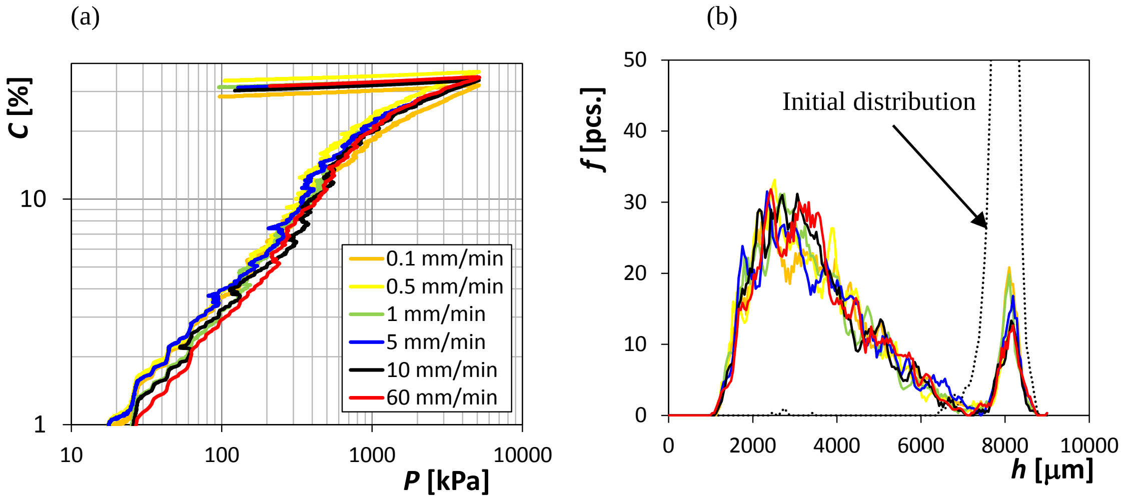

Figure 3: Effect of piston speed. (a) Compressibility curves. (b) Particle size distribution (PSD). Parameters: cell size M, layer height H = 15 mm, terminal pressure was P_t = 5100 kPa.

3.2 *Effect of cell size*

In the next step, granular layers of different dimensions were tested, three cell diameters and three heights, i.e. nine combinations. Each experiment was repeated five times and the results were averaged. The terminal pressure was P_t = 3350 kPa. The output is shown in Fig. 4. The final compressibility in cells S and M is almost identical, and it little lower in cell L. A possibility is loosing the wall effects in the large cell. The PSD is not much influenced either, beside a weak ordering trend of the lines with respect the cell sizes (L > M > S) at the initial particle length (8 mm), and, ordering conversely at smaller sizes. It was concluded that the cell size does not influence the results of the breaking experiments significantly. The medium cell size M and the middle layer height H = 15 mm were chosen as the reference ones.

(a) (b)

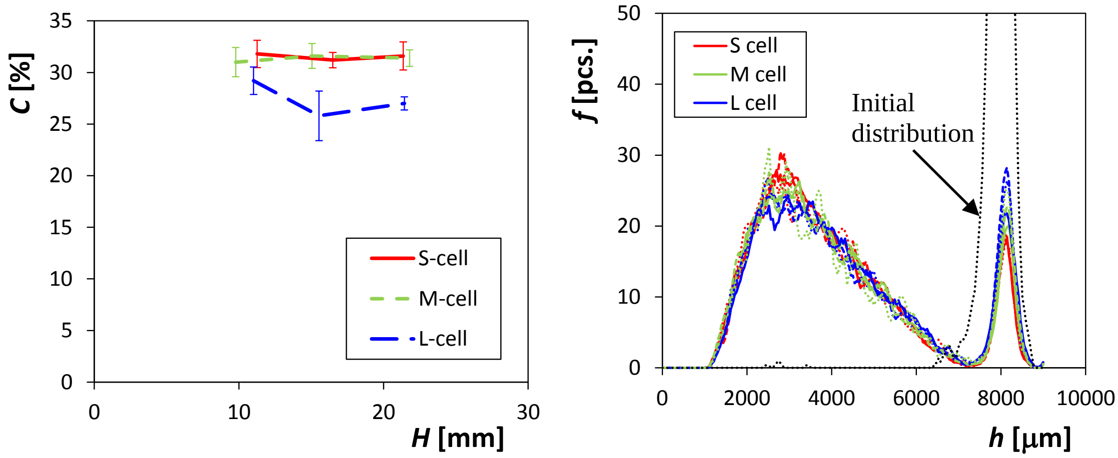

Figure 4: Effect of cell size. (a) Final compressibility. (b) Particle size distribution (PSD), dotted, dashed and full lines for H = 10, 15 and 20 mm respectively. Parameters: terminal pressure P_t = 3350 kPa.

3.3 *Effect of applied pressure: non-recurring mode*

The effect of one-time applied pressure (non-repetitive) was tested next, for eight terminal values, P_t = 100, 500, 1000, 1400, 1900, 2600, 3350, 5100 kPa. The cell size was M and the layer height was H = 15 mm. The result is shown in Fig. 5. The individual compressibility curves (5a) match the common envelope pertaining to the largest pressure. Their closeness indicates the reproducibility of the breaking process. The relaxation after unloading is larger for less compressed layers. The individual distribution curves (5b) witnesses the successive breaking of particles, when increasing the pressure above 100 kPa. At the load of 500 kPa, the particles preferable break in half (yellow peak at 4 mm). At higher loads, the particles break into smaller pieces and progressively populate the size bins within 2-4 mm. At all loads, there still remain some of the original particles unbroken (peaks at 8 mm). Typical is the zero-zone around the size of 7 mm, where almost no particles appear.

(a) (b)

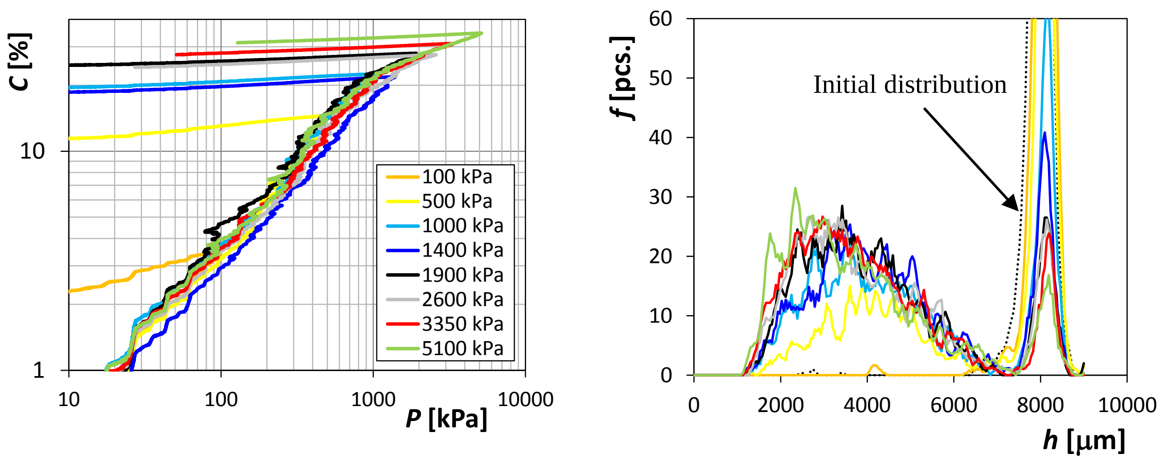

Figure 5: Effect of pressure in non-recurring compression. (a) Compressibility curves. (b) Particle size distribution (PSD). Parameters: cell size M, layer height H = 15 mm.

3.4 *Effect of applied pressure: recurring mode*

The last experiments were designed to observe the effect of the repetitive compression on the granular layer. The cell size was M and the layer height was H = 15 mm. The terminal pressure was fixed to P_t = 3350 kPa and the varying parameter was the number N = 1, 2, 3, 4, 5, of how many times the layer was consequently compressed, after emptying and re-filling the cell. The result is shown in Fig. 6. The slope of the subsequent compressibility curves progressively decreases and the observed values of C converge fast: 30, 20, 15, 13, 13 %. The PSD diagram shows an accelerated destruction of particles, as compared with that in Fig. 5. After only two runs (N = 2), the original population is almost exterminated. The set of the small-size peaks quickly shifts leftwards, ending in the interval of 1-4 mm fragments after five runs (N = 5). Thus, the repetitive breakage at a lower pressure can be more effective than the single breakage at a higher pressure. The reason is seen in the re-arrangement of the particles due to manipulation with the sample (taking out/putting in). New configuration has higher voidage and offers new breakable contacts.

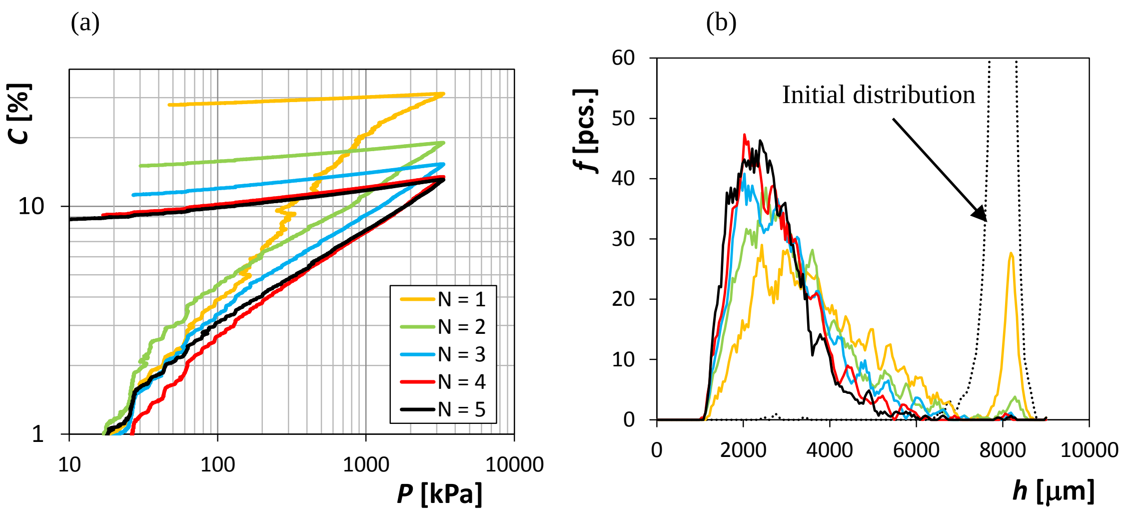

Figure 6: Effect of pressure in recurring compression. (a) Compressibility curves. (b) Particle size distribution (PSD). Parameters: cell size M, layer height H = 15 mm, terminal pressure P_t = 3350 kPa was applied N-times.

4. CONCLUSIONS

Experiments with dry granular media (model pasta cylinders) were done, where the compressibility and particle size distribution were measured. The effect of parameters were studied with these results:
- piston speed (v = 0.1, 0.5, 1, 5, 10 and 60 mm/min) did not have apparent influence,
- size of measuring cell (D = 25, 33, 41 mm, H = 10, 15, 20 mm) did not have demonstrable influence,
- one-time applied terminal pressure (P_t = 100, 500, 1000, 1400, 1900, 2600, 3350, 5100 kPa) caused majority particle breakage into 1/2 - 1/4 of the original size,
- repetitively applied terminal pressure (P_t = 3350 kPa) caused severe fragmentation of all particles into 1/2 - 1/8 of the original size.

5. ACKNOWLEDGEMENT

The support by Czech Science Foundation GACR through contract No. 15-05534S is gratefully acknowledged.

6. NOMENCLATURE

C	: compressibility [%]
d	: particle diameter [m]
D	: diameter of cell [m]
f	: frequency of particles [pcs.]
h	: particle length [m]
H	: height of layer [m]
H_i	: initial height of layer [m]
h	: length of particle [m]
N	: number of repetition [-]
P	: applied pressure [Pa]
P_t	: terminal applied pressure [Pa]
v	: piston speed [m/s]
V	: volume of material layer [m]
V_i	: initial volume of material layer [m]

7. REFERENCES

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