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Original Research Paper

Granule attrition by coupled particle impact and shearing ☆

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ABSTRACT

A novel device has been developed for continuous shearing and repeated impact of granules in order to simulate granule attrition and dust formation under realistic plant conditions of mechanical stresses, shear strains and strain rates. The device subjects the granules to multiple impacts at a range of velocities prevailing in typical process plants, and to shear deformations using two rollers with an adjustable gap to simulate the level of shear stresses and strains experienced during bulk motion, e.g. discharge from silos onto conveyor belts, etc. In this paper, the device operation and tests carried out to determine the settings required for attaining a desired impact velocity and shear strain rate are described. Subsequently, the extent of breakage of the granules is determined for the specified settings and the results are compared with data obtained by more established methods, e.g. annular shear cell and single particle impact tests.

1. Introduction

Qualification of newly processed bulk solids can often delay delivery and commissioning of new materials in manufacturing plants and often leads to expensive large-scale trials. Extensive work has been conducted to evaluate granule strength to resist attrition by particle impact [1–3] and shear deformation [4–6]. However, for some bulk solids such as enzyme granules, there is currently no standard approach to characterise the breakage propensity using representative manufacturing plant stresses. Ahmadian and Ghadiri [7] analysed the type and magnitude of stresses that enzyme granules experience in a typical detergent manufacturing plant and found that impact and shear deformation are dominant and occur in multiple unit operations. They evaluated test methodologies to investigate the reproducibility of the breakage behaviour of placebo enzyme granules. These included quasi-static single granule compression tests, single granule impact tests, and bulk shear strain tests. The latter included both annular shear cell test [8] and rotating drum test [9]. In spite of all the effort put into analysing granule breakage by impact and shear deformation, there is no test device that integrates both types of stresses. The importance of performing tests with a combination of impact and shear deformation was highlighted by Ahmadian [10] who noted a couple effect in enhancing attrition. Three commercial enzyme granules were tested by repeated impact, shear deformation and a combination of both. The extent of damage after repeated impacts (10 × 10 m/s) and subsequent shear straining at 20 kPa normal pressure and varying strains was different for the enzyme granules studied. Within the range and type of stresses exerted on the granules, there was a coupling effect of impact and shear for two out of the three commercial enzyme granules tested. It was proposed that during impact, the granules developed micro-cracks that could be exposed upon shearing and lead to fragmentation. With this in mind, the Particle Shear and Impact (PSI) tester has been developed in collaboration with Hosokawa Micron, Runcorn, UK, enabling testing of granules by a combination of repeated impact and shear deformation. In this paper, the operation of the PSI tester is described and results of granule breakage experiments performed on a number of enzyme granules are reported.

2. Particle Shear and Impact (PSI) tester

The Particle Shear and Impact (PSI) tester is a particle recirculation device, which subjects granules to impact and shear deformation, thus facilitating the analysis of their mechanical damage and breakage by impact erosion, sliding wear, chipping and fragmentation. A photograph of the PSI device is shown in Fig. 1. The device comprises two counter-rotating rollers with an adjustable gap to induce shearing, a pneumatic conveying section with two bends,

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Nomenclature

α	Constant for Pietsch correlation [–]	B	Roller gap size [m]
β	Constant for Beverloo's correlation [–]	D_h	Hydraulic diameter [m]
γ	Shear strain [–]	H	Shearing height in rollers [m]
$\dot{\gamma}$	Shear strain rate [s^{-1}]	r	Roller radius [m]
ω_1	Roller speed 1 [rad/s]	W	Mass flowrate [kg/s]
ω_2	Roller speed 2 [rad/s]	T	Roller thickness [m]
ρ	Bulk density [kg/m^3]		

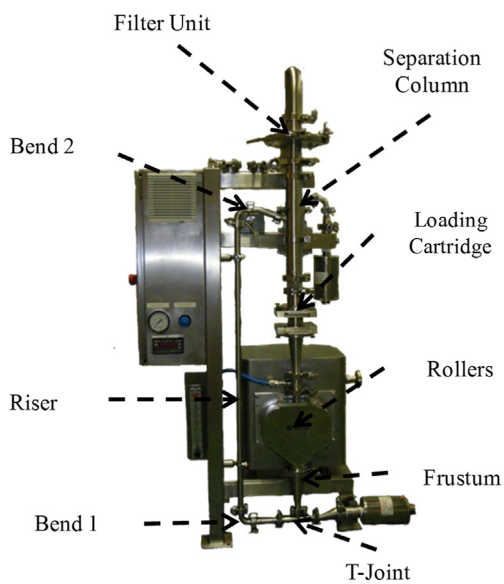


Fig. 1. PSI tester and its key features.

a granule/dust separation column, a filter unit and a loading/unloading cartridge.

The details of the individual units in the PSI tester as well as the operating range for the rollers' separation gap and rotation speeds, air velocity in the pneumatic line and separation column specification are described in Table 1. The surface of two rollers is grooved (90°) with 1 mm pitch longitudinal corrugations. The average number of particles in between the rollers should be less than 6 particles to avoid flow due to gravity [11] but larger than 2.2 particles to avoid crushing of individual particles [12]. It has previously been shown that shearing occurs in the nip region of the roller compactor. The choice of an appropriate groove size for effective shearing has previously been described by Ghadiri et al. [13], based on the work of Neil [14], who has shown that the groove size is crucial for effective gripping. Too large a groove size will provide dead volume in the grooves and will also cause grinding of particles which are in direct contact with the grooves. Too small a groove size, on the other hand, will result in unpredictable wall slippage and surface abrasion of particles in direct contact with the grooves. In both cases, bed degradation will not be solely due to particle–particle interactions. Consequently, the results cannot be taken as representative of the degradation in the shear zone. If Δz is the distance of the particle centre from the top of the groove and D_p the particle diameter, Neil [14] defined a gripping ring parameter η as η = Δz D_p/2, where 0 < η less than 1. When η = 0, half the particle is gripped by the groove, and as η → 1, very little of the particle is gripped by the groove. In order to prevent slippage at all normal stresses, an empirical value of η was suggested: $0.25 \le \eta \le 0.75$ for high normal stresses and $0.1 \le \eta \le 0.55$ for lower normal stresses, respectively. The optimum groove width is also related to the average particle diameter by groove width/$D_p \ge 1$. As the groove should accommodate particles, the groove width can be in a range to satisfy both criteria. Based on the particle size ranges used in this work a groove width and depth of 1 and 0.5 mm were chosen, respectively. In the following section, the rationale behind tuning the settings is described.

3. Experimental

The attrition tests were carried out on three commercial enzyme granules, hereinafter referred to as enzyme granules A, B and C in the enzyme laboratory of Procter and Gamble Newcastle Innovation Centre, Longbenton, Newcastle, UK. In order to avoid exposure to enzyme dust during the calibration of the device settings, placebo granules A, B and C were actually used. These granules were manufactured by the members of the Enzyme Dust Consortium according to their standard manufacturing procedures, but without incorporating the enzyme. The granules had a sieve size range of 300–850, 300–1000, and 500–850 µm and particle envelope densities of 1527, 1574 and 991 kg/m^3, respectively.

3.1. Impact velocity setting

High speed video photography (3000–4000 frames/s) for various granules was conducted to establish the impact velocities at bend 2 (see Fig. 1) at the top of the riser. The particle velocities were measured for the three placebo granules using a transparent riser, made of Perspex (0.2 m below bend 2). The air velocity in the riser pipe as well as the average measured particle velocity (10 repeats) for the three granules as a function of pump frequency are shown in Fig. 2. The particles in the riser pipe were still accelerating and never reached their terminal velocities due to the short length of the riser pipe (0.9 m). Using this approach, the correct pump frequency could be selected to provide the desired particle velocity at bend 2. By extrapolating the curves in Fig. 2 for granules A, B and C, the respective pump frequencies of 90, 83 and 70% led to an impact velocity of 10 m/s at bend 2.

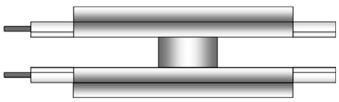
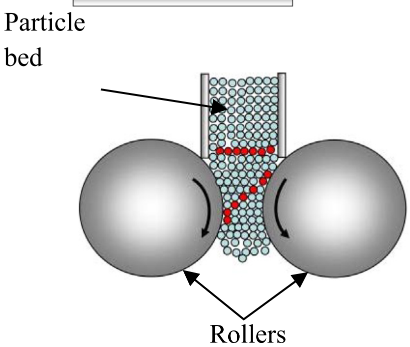
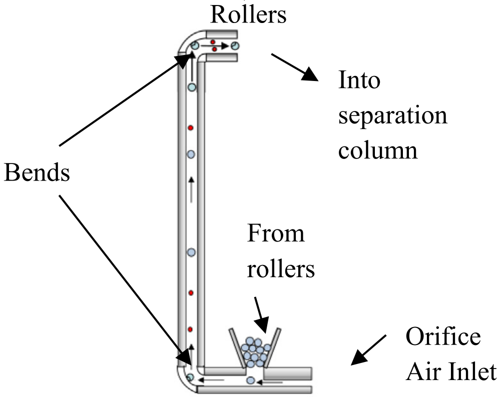
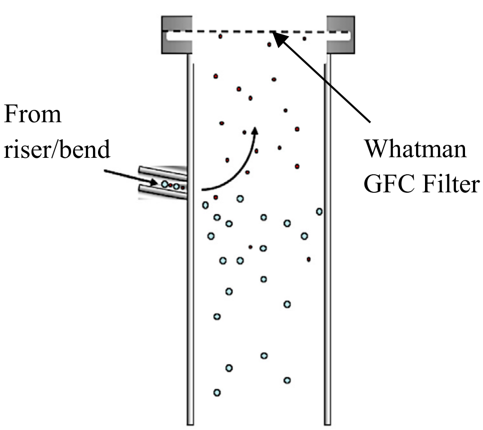
3.2. Roller shear gap size and shear rate/strain

Shearing straining occurs in the nip region (active shear region), indicated by the blue shaded rectangle in Fig. 3(a), as revealed by high speed video imaging. Therefore, the shear strain is approximately:

$$\gamma = \frac{H}{B} \tag{1}$$

where *H* is the height of the active shear region and *B* is the roller gap size. This active shear region has a height of around 10 mm. For placebo granules C, the particle motion was tracked using high speed video photography (500 frames/s) of a mixture of two-coloured granules, and the downward particle velocities were calculated for gap sizes of 3, 4 and 5 mm as displayed in Fig. 3(b). The horizontal axis gives particle position from the centreline

Table 1
Description of individual units in the PSI tester.

<table>
<tr><th>Unit</th><th>Description</th></tr>
<tr><td></td><td>Loading cartridge: Custom designed loading cartridge fabricated from stainless steel 316 with two PTFE slide valves. The slide valves are used for material loading and withdrawal from the PSI tester. The volume of the loading cartridge is 90 ml</td></tr>
<tr><td>
</td><td>Roller shearing: A dense particle bed is fed between two counter-rotating rollers with an adjustable gap, whilst the angular velocity of each roller can be set independently. The diameter and thickness of the rollers are 100 and 25.4 mm, respectively. The roller speeds can be varied from 1 to 100 rpm either in clockwise or anticlockwise direction. The roller gap can be varied from 0.2 to 6 mm</td></tr>
<tr><td>
</td><td>Orifice air inlet, impact bends and riser pipe: Particles fall from the roller shearing region into an air stream under vacuum. The air inlet is an orifice with a semi-circle cross sectional area of 49 mm^2. The pneumatic pipe air inlet has an ID of 15.29 mm (cross sectional area = 184 mm^2) and is constructed from stainless steel 316. The decrease in area in the orifice (by approx. a factor of 4) leads to high localised velocities at the air inlet and ensures that particles are entrained as they fall from the roller compactor. Particles are then pneumatically conveyed through a first bend, riser pipe (0.9 m) and second bend. Most particles will impact onto the bend walls. The average air velocities in the riser pipe can be varied from 5 to 25 m/s depending on the vacuum pump frequency</td></tr>
<tr><td>
</td><td>Separation column and filter unit: After the final bend in the pneumatic line, particles enter a separation column constructed from stainless steel 316 and with an ID of 46.7 mm. The upward air velocity in the top part of the separation column can be set as slightly higher than the terminal velocity of 100 μm dust particles. This leads to large particles falling down through the cartridge back onto the bed column above the roller compactor and fine particles to be entrained and captured onto a Whatman GFC filter (15 cm diameter). The air velocity in dust separation column will depend on the pump frequency and can be varied from 0.5 to 2.6 m/s</td></tr>
</table>

between the two rollers, normalised to the width of the nip region. The negative values refer to the particle position to the left of the centreline. The following equation is used to calculate the shear strain rate for the gap between the counter rotating rollers:

$$\dot{\gamma} = \frac{(|\omega_2| - |\omega_1|)}{B} r \tag{2}$$

where r is the roller radius and ω_1 and ω_2 are the angular velocities of the rollers. For all of the tests, the shear strain rate was kept constant at 20 s^{-1} by setting a rotational speed of 13 rpm for the first roller, while that of the second roller was adjusted based on the gap size. The granule velocity in the nip region, as shown in Fig. 3 (b), is linear for the 3 mm gap size and also to some extent for the 4 mm gap size 4, showing a clear shear straining field. However, the trend of granule velocity for the 5 mm gap size, i.e. having an almost flat profile on the right hand side of the centreline in the region affected by the faster rotating roller, suggests that the gap is too wide and granule flow is affected by both shearing and gravity.

A decrease in the gap size leads to higher stresses in the roller nip region. Measurement of stress between the rollers has not been possible due to lack of technology. However, according to Stephens and Bridgwater [15], the width of a natural shear band developing in an unbound granular medium is around six particle diameters, and this is the zone in which attrition takes place. This in fact formed the basis of the design of the Paramanathan and Bridgwater attrition shear cell [4]. Therefore, the gap size should be less than about six particle diameters to effectively shear the moving granule bed and also reduce flow due to gravity [11]. In addition, it should be larger than 2.2 particle diameters to avoid crushing of individual particles due to strong jamming [12]. In Fig. 3(b), for a D_{50} size (50% for particle size by number) of 760 μm, the

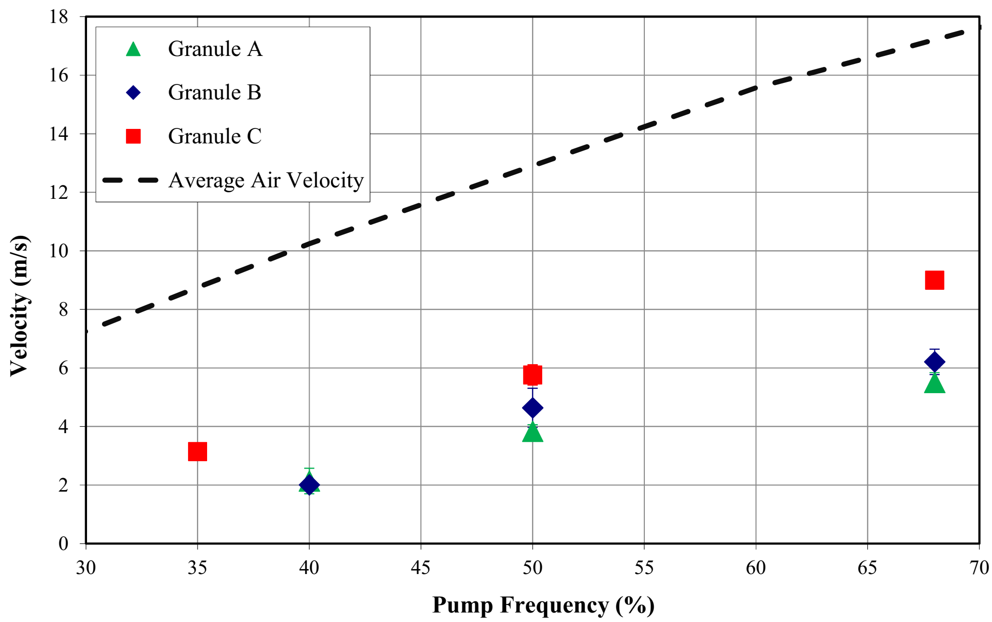


Fig. 2. Average particle and air velocities as a function of pump setting for placebo granules.

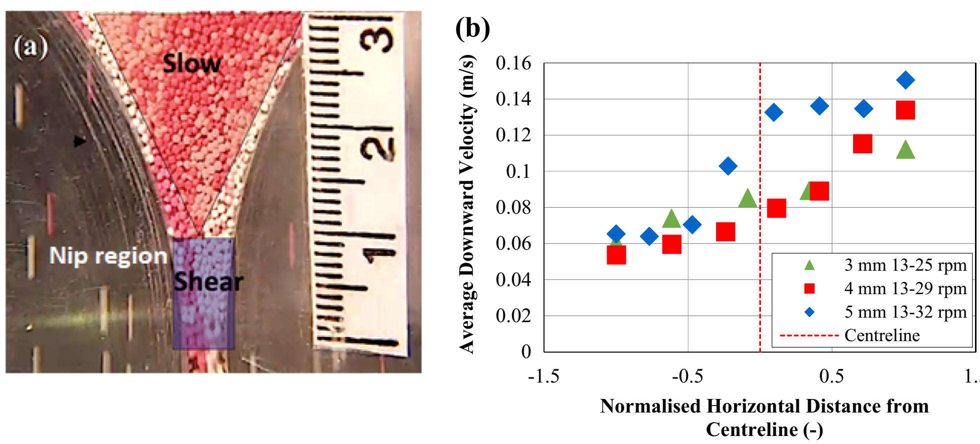


Fig. 3. (a) A frame from high speed video imaging of particles in rollers and contours of particle speeds; and (b) distribution of the downward velocity for placebo granules C in the nip region for gap sizes 3, 4 and 5 mm as a function of the horizontal distance from centreline normalised to the width of the nip region.

gap sizes 3, 4 and 5 mm encompass 4.1, 5.5 and 6.9 particle diameters, respectively. Thus, it should be possible to effectively shear a moving bed of such particles using any gap size between 2.2 and 5 particle diameters, keeping in mind that different extents of attrition are anticipated depending on the gap size. With this in mind, initially a gap size of 5 × D_{50} of the particles was deemed suitable. Accordingly, for the three commercial enzyme granules A, B and C with the respective D_{50} values of 425, 550 and 760 µm, the suitable roller gap sizes were determined. Based on these, the roller speeds required to maintain a shear rate of 20 s^{-1} were calculated using Eq. (2), as displayed in Table 2.

3.3. Time in device

The time of recirculation in the device is based on the estimate of number of impacts and shearing stages in the manufacturing plant. The time limiting step in this device is the roller shearing stage, as the particles that have gone through the gap are quickly reticulated in the pneumatic line; this results in the build-up of particles above the rollers. The recirculation time can be estimated

Table 2
Roller gap sizes and roller speeds required to ensure the formation of a shear band and a constant shear rate for the test granules.

Granule	A	B	C
D_{50} size (µm)	425	550	760
Gap = 5 × D_{50} (mm)	2.1	2.8	3.8
Shear Strain rate (s^{-1})	20	20	20
Roller 1 Speed (rpm)	13	13	13
Roller 2 Speed (rpm)	21	23.7	27.5

by measuring the particle flowrate through the rollers during rotation and by fitting the results to the following correlation which is based on the continuity equation:

$$W = \alpha \rho B T \frac{(|\omega_1| + |\omega_2|)}{2} r \quad (3)$$

where W is the mass flowrate (kg/s), ρ is the bulk density, T is the roller thickness (m) and α is an empirical constant. By opening the base of the rollers and measuring the flowrate, α is found to be 1.7, 1.2 and 1.3 for placebo granules A, B and C, respectively. Therefore,

Table 3
Recirculation times of placebo granules A, B and C in the PSI tester for impact alone and in combination with shearing.

Granule	Recirculation Time (s) for Impact alone (Eq. (4))			Recirculation Time (s) for Shear and Impact (Eq. (3))		
	11 passes	22 passes	33 passes	11 passes	22 passes	33 passes
A	16	32	48	59	117	175
B	14	27	40	48	95	143
C	24	48	71	54	107	160

for a given mass of material, the time taken for one circulation can be calculated. In cases where particles merely undergo impact, i.e. the rollers are stationary, the flowrate is calculated by the modified Beverloo equation [11]:

$$W = \beta \rho B T \sqrt{g D_h} \qquad (4)$$

where β is an empirical constant equal to 51.75, 51.75 and 38.25, for placebo granules A, B and C, respectively, and D_h is the hydraulic diameter of the roller gap. Building on this, the calculated recirculation times for 45 g of the test granules are represented in Table 3.

The device has a modular assembly structure and after testing, it is dismantled and cleaned in a dishwasher.

4. Results and discussion

The breakage profiles for the three commercial enzyme granule types A, B and C were analysed for the following conditions:

- Test impact breakage of granules with an impact velocity of 10 m/s at bend 2 for 11, 22 and 33 passes;

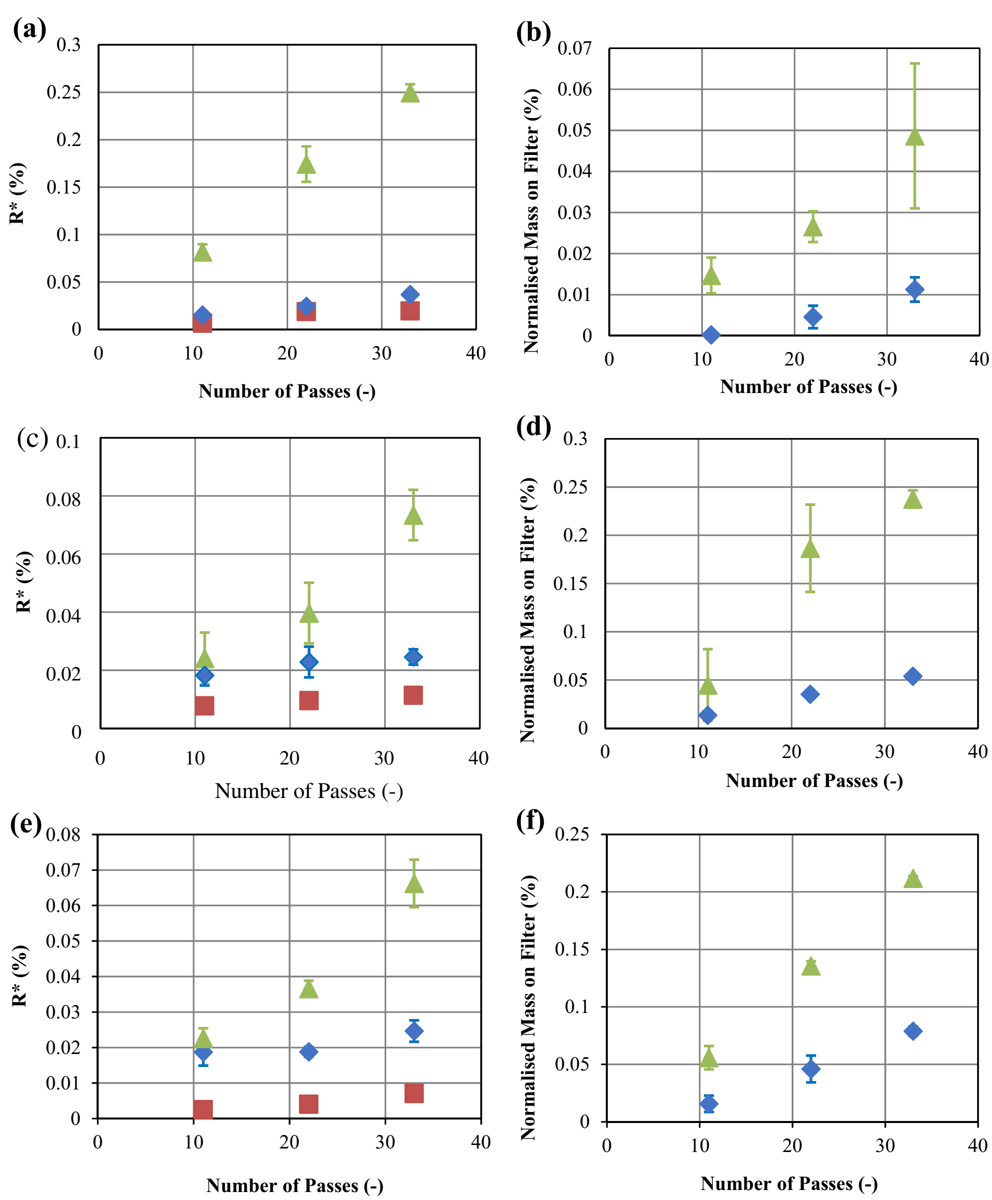


Fig. 4. Extent of breakage of granules in the PSI tester, based on the mass of debris collected from the sieve (R*) and filter paper for enzyme granules A, B and C. (a, b): enzyme granule A; (c, d): enzyme granule B; (e, f): enzyme granule C – ◆ repeated impact alone, ■ shearing alone and ▲ combination testing.

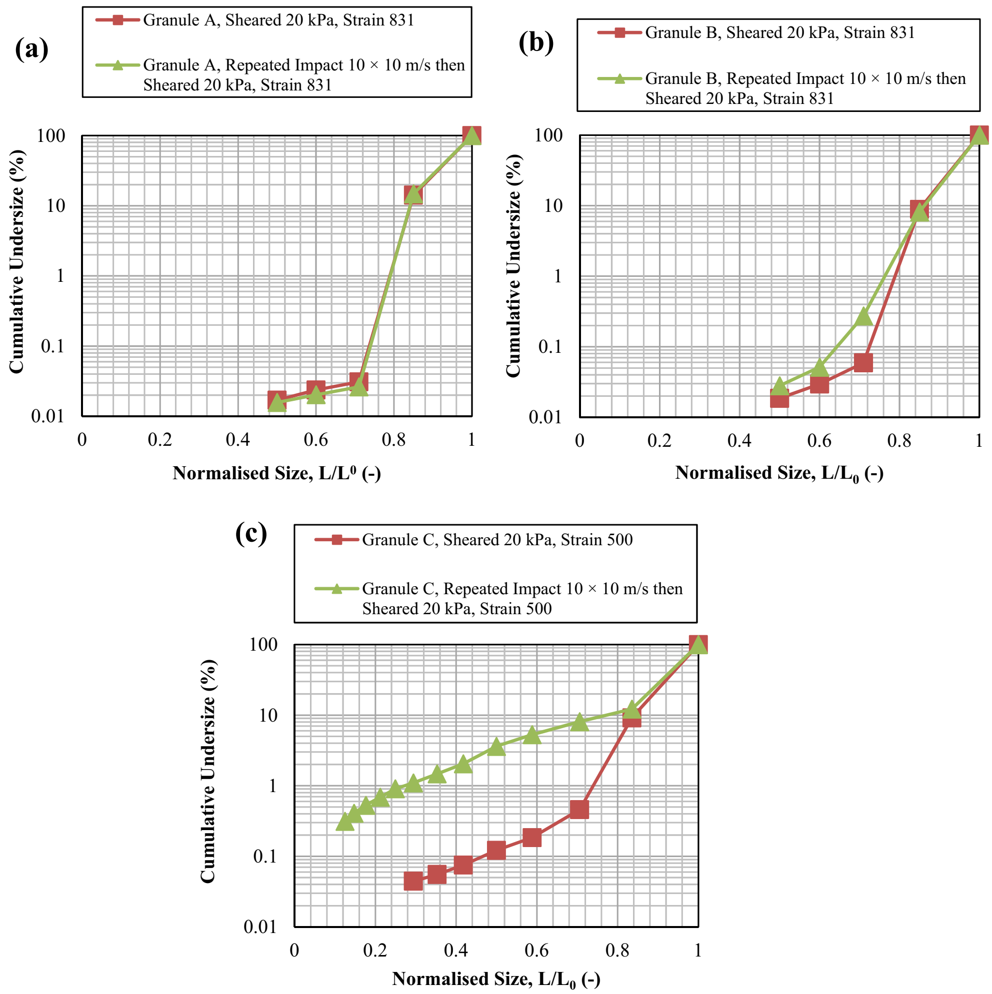


Fig. 5. Cumulative undersize as a function of normalised broken particle size for enzyme granules A (a), B (b) and C (c)– tests conducted in the Ajax shear cell with and without repeated particle impacts.

- Roller shear tests conducted at a specific gap size for 11, 22 and 33 passes;
- Test impact and shear breakage of granules with an impact velocity of 10 m/s at bend 2, and shearing with a specified gap for 11, 22 and 33 passes.

Considering the size distribution of the granules, they were sieved before the tests to ensure that they were within the size range intended. Granule breakage was quantified by two separate methods: (i) by gravimetric analysis of debris below a sieve size which could separate the debris from the mother particles, and (ii) dust collected from the filter unit. In both cases, the mass of the debris was normalised by the total mass of the feed granules and was considered as the extent of breakage, R*. The debris-characterising sieve size was chosen as 250 μm for granules A and B and 355 μm for granule C. The results are shown in Fig. 4, where a clear coupled effect is seen for all granule types; by coupled effect, the additive debris formed by repeated impact and shearing alone when tested independently, is less than when these stresses are integrated. The extent of breakage from highest to lowest is for repeated impact plus shearing, then impact, and then shearing alone. In a separate assessment, using the well-established methods of shearing in an annular shear cell [4–6] and single particle impacting [1], the three enzyme granule types were analysed by shearing under a normal load of 20 kPa for various shear strains, by impact at 10 m/s for ten times, and by a combination thereof. For the combination testing, after the granules were impacted, the surviving mother granules were separated by sieving and then were transferred to the shear cell to get sheared. Following that, all mother granules, fragments and debris were carefully collected from the shear cell for a comprehensive sieve analysis. The results are shown in Fig. 5 and may be compared with those of the PSI device given in Fig. 4. The tests have not been designed for exact quantitative comparison, but the main point here is that granules B and C exhibit a coupled effect (when comparing shearing alone vs repeated impact then shearing of surviving mother particles) in both test methods. It should be noted that granules A, B and C have completely different structures, evolved from differences in their manufacturing routes, which are unexplored due to their commercial sensitivity. Nevertheless, the methodology proposed here clearly indicates differences in their tendency for enzyme dust formation without probing into the granule structure.

5. Conclusions

The design and operation process of a new test device developed for the analysis of granule attrition propensity under simulated plant stresses are described. The device subjects granules to the prevailing mechanical stresses experienced in manufacturing plant operations, i.e. dynamic impact and quasi-static shear deformation. The test procedure is quick and the results reveal a clear coupled effect of impact and shearing, which is confirmed by another assessment that combines well-established individual tests of shearing and impact. This suggests that a realistic granule strength evaluation for mitigating attrition and dust formation should incorporate both stressing methods, especially in the case of enzyme granules where granule breakage poses a health risk to the operators. In view of this, specifying the Coefficient of Variation (CV) of enzyme dust produced in the PSI tester rises to prominence as a future step in the evaluation of the device and the minimum CV specified by Bonakdar et al. [16] for placebo enzyme granules can be used to compare the performance of the PSI tester to other test devices.

Declaration of Competing Interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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