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IMPACT ATTRITION OF SPRAY-DRIED BURKEITE PARTICLES

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ABSTRACT

Particle attrition in manufacturing plants handling particulate solids could cause processing as well as environmental problems, and lead to the degradation of product quality. Spray-dried powders are particularly prone to attrition because of their porous and often weak structure. Spray-dried burkeite particles are a good example and have been used as a model porous powder to investigate the effect of structure on their breakage propensity. The particles are subjected to well-defined stresses due to impact, and the change in the particle size distribution is determined by particle size analysis based on sieving. It is found that impact breakage of burkeite is affected by the structure, and some unexpected trends for breakage are observed; for a given impact velocity some smaller particle sizes break to a greater extent as compared with larger particles. This is attributed to uncontrollable variations of porosity (and hence particle envelope density) as a function of size. To study the effect of porosity on attrition of the spray-dried powders, structure visualisation and analysis have been carried out by Scanning Electron Microscope (SEM) and X-ray microtomography (XRT). SEM images show three levels of structure in a single particle of spray-dried burkeite. Based on the XRT results, the particle envelope density increases as particle size increases, and the variation of envelope density influences the impact breakage. Once the relevant values of the envelope density are taken into account, then the trend of impact breakage data becomes as expected, and the material mechanical properties of the particles can be inferred from the breakage results.

KEYWORDS: attrition; impact breakage; spray-dried powder

1. INTRODUCTION

Understanding particle breakage is a key factor in order to improve the product quality in manufacturing plants handling particulate solids. Breakage has a major impact on powder quality and on a number of process operation features, such as reliability, safety, environmental impacts as well as economic implications. Powders moving through plants can experience different types of stresses, most commonly impact and shear stresses, causing undesirable attrition. The properties of particulate solids may change as a result of attrition, such as size distribution, shape, surface area and bulk density. These could have significant influence on product attributes. Bemrose and Bridgewater [1] have studied the factors which affect the attrition of the particles such as material properties, environmental and contact conditions.

Attrition in manufacturing plants commonly occurs in pneumatic conveying lines by particles sliding on the walls and impacting at bends [2-7], inside a rotating drum, where particles may experience bulk shear deformation and impact, depending on the flow regime and drum fill [8], in fluidised beds [9, 10], and in filling and discharge from storage units and moving beds [11, 12].

There are a number of test methods to assess the breakage of the particles, whether singly or in bulk. Impact [13-18], wear [19], side crushing [20-23] and indentation [24] are some of the test methods used for single particles. According to Ghadiri et al. [12], these methods have relatively well-defined conditions and can be used to study the influence of stress field and materials properties on particle breakage; however, due to simplicity of these methods, they are not fully representative of industrial conditions.

The single particle impact test is a dynamic test method, in which the particles are accelerated and impacted onto a normal or oblique target. It has been extensively used as a test method to study the breakage behaviour of particles under impact conditions. Salman et al. [25, 26] used this method to study the breakage of fertiliser and alumina agglomerates, respectively. Arbiter et al. [27] used free fall impact to study the breakage behaviour of sand-cement agglomerates. Breakage pattern of lactose agglomerates was investigated by Ning et al. [28] and Boerefijn et al. [13]. Also materials such as sodium chloride crystals [29], sand [30], and detergents [16] have been tested in this way. Tomas et al. [31] and Khanal et al. [32] have used the approach for impact testing of large concrete balls. Ghadiri and co-workers have developed an impact tester to study the breakage of particles [15, 29].

Although there are a lot of work on the breakage behaviour of single particles and agglomerates, a full understanding of the breakage of individual agglomerates is still lacking. Subero and Ghadiri [14] found several breakage patterns of agglomerate materials: localised damage on the contact point, fragmentation by propagation of various cracks into the agglomerate body or extensive disintegration. Overall, the agglomerates can break in different patterns depending on their properties and loading conditions, leading to various failure modes. This refers strictly to the macroscopic failure presented by the whole agglomerate, rather than the failure of individual interparticle bonds [28, 33, 34].

Spray-dried powders have an agglomerate structure. Detergent washing powders could be a good example of spray-dried powders. Conversion of detergent slurries to dry powders by spray drying is an important step in the production of detergent powders [35]. In the past, spray drying method using phosphate based materials (sodium triphosphate) was a common method in production of detergent powders. However, as they were not environmentally friendly materials, they have been replaced by zeolites, sodium carbonate and sodium sulphate salts [36]. These particles are particularly prone to attrition because of their porous and weak structure. It is well known that the operating conditions of the spray drying towers such as slurry flow rate, air inlet temperature and atomizing pressure can have a significant effect on the powder mechanical strength, porosity and hence friability of the particles. The structure of the particles is also affected by operating conditions. Powders moving through the plant can experience different types of stresses, causing undesirable attrition [37]. In this work we analyse the breakage of spray-dried burkeite particles subjected to impact stresses in the velocity range typically encountered in manufacturing plants. Spray-dried burkeite is a particularly weak and friable powder which is a good model material for testing the breakage pattern of spray-dried powders. The fractional loss per impact or the shift in the particle size

distribution of impact products is used as a measure of particle breakage. The breakage data have been then related to the mechanical properties of particles, which cannot be easily measured by other methods.

2. METHODOLOGY

Spray-dried burkeite particles are used as a model material as they are a good example of a complex porous structure. They are highly porous and friable and hence prone to undergo attrition even under gentle handling. Burkeite is a co-crystal of sodium sulphate and sodium carbonate, and it has the general form $Na_4SO_4(CO_3)_t(SO_4)_{1-t}$. The particles of interest in this work have been produced by spray-drying of a slurry of mixture of the two salts. The use of TGA, Raman spectroscopy and X-ray powder diffraction show that burkeite is the only salt formed in the process [38]. The slurry is spray dried. As drying proceeds, a crust is formed and the crystals within the droplets form cluster. During the drying process, the surface of the particle becomes dry and the wet core shrinks inside the particle, and the spray-dried burkeite is formed [36, 39, 40]. A potential application of these particles is to use them as carrier/ filler of surfactant for detergent washing powders; therefore it is critical to explore the strength of the particles under different levels of stresses. Photographs obtained by Scanning Electron Microscopy (SEM) of a single particle of spray-dried burkeite and its internal structure are shown in Figure 1.

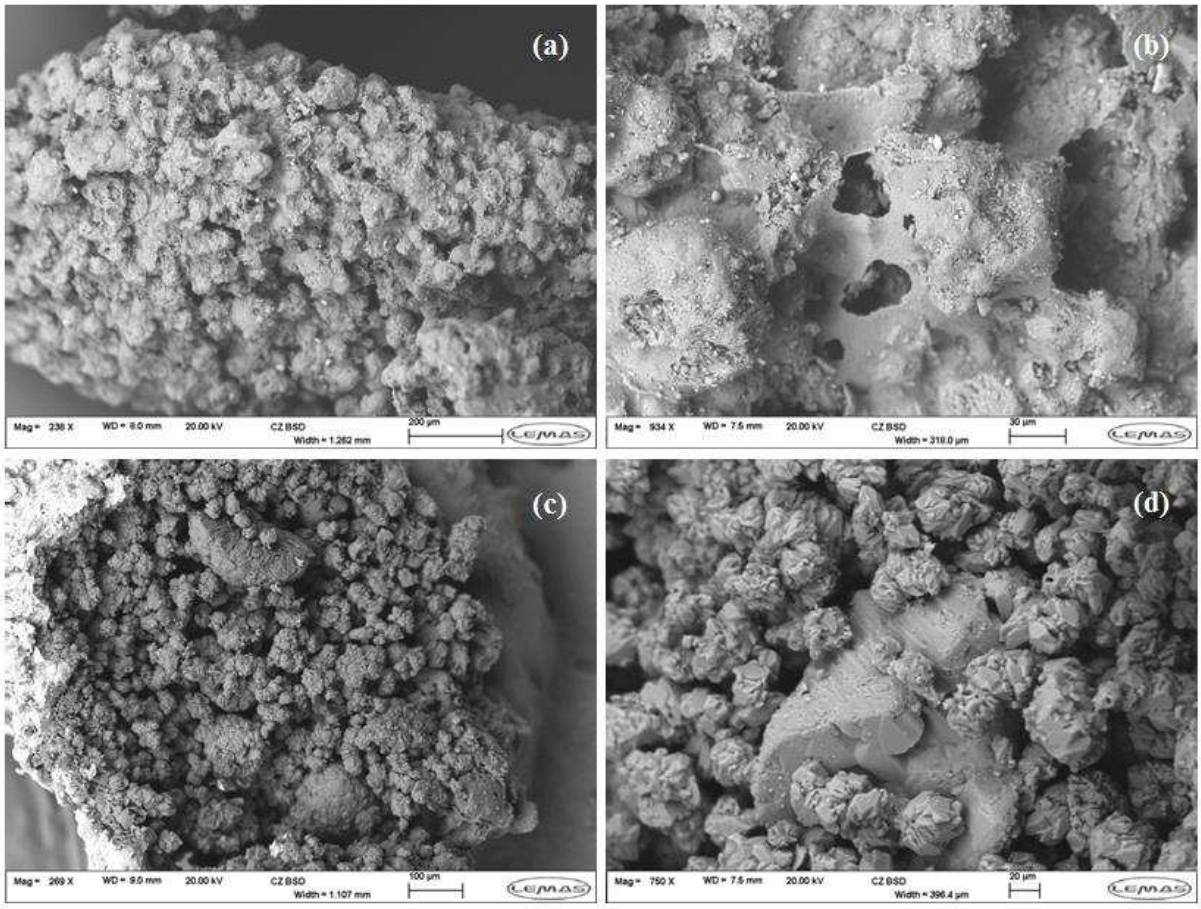

Figure 1. SEM images of spray-dried burkeite, (a) single particle of burkeite; (b) skin on the surface of the particle; (c) internal structure of one single particle; (d) clusters inside the single particle

These clearly show three different levels of structure in a single particle of spray-dried burkeite. Each individual particle has an agglomerate structure (Figure 1a) with a skin formed on the surface (Figure 1b). It is made of a number of clusters (Figure 1c), and each cluster has several crystals stuck to each other (Figure 1d). The gap between the clusters shows the porosity inside the particles.

Previous unpublished work on the impact breakage of the spray-dried burkeite particles, by using different sieve size cuts of particles, showed some anomalies which are likely to have arisen from differences in the structure of the particles between different particle sizes. It was implicitly assumed that the particle density was constant throughout the particle sizes tested. However, this may not be the case, and a more specific design of experiment taking account of possible variation of density with size is required. Therefore in order to measure the envelope density as a function of size, it is prudent to use as narrow particle size distribution as possible. For this purpose near-mesh size particles have been prepared by sieving the particles and retrieving only those caught in the mesh opening of the sieve by gentle brushing. The following sieve sizes were used, 212, 250, 500, 600, 850 and 1000 μm. The particles are then impacted on a flat rigid target at different impact velocities to explore their impact strength. Eight different impact velocities 2, 3, 4, 6, 8, 11, 14 and 18 m/s were tested. Various patterns of breakage for different impact conditions have been identified, i.g. chipping, fragmentation and disintegration. In order to study the structural differences as a function of size, the envelope density of the particles has been measured using X-ray microtomography, and used to analyse the impact breakage data.

3. EXPERIMENTAL RESULTS AND DISCUSSION

To produce statistically reliable results it is critical to use a representative sample mass [41]. Error analysis has been carried out to explore the effect of sample mass on impact breakage results. Different sample quantities, as well as different number of repeats have been used for this assessment. The standard deviation and standard error arising from different number of repeats have been determined. Considering the results of error analysis as well as the time taken for preparing the near-mesh particle sizes, it has been decided to use at least 2 g of material for small particles (212, 250, 500 and 600 μm) and 3 g for large particles (850 and 1000 μm) for each test. The single particle impact rig is shown in Figure 2. This rig used for the experiments is the modified design of the single particle impact apparatus originally developed by Yüregir et al. [42]. The particles are fed from the top of the rig, and impacted to a flat rigid target at different impact velocities by changing the vacuum pressure. The impact velocity is measured by time of flight of particles passing by two photodiodes. The time is then used by the software in the PC to calculate the particle velocity.

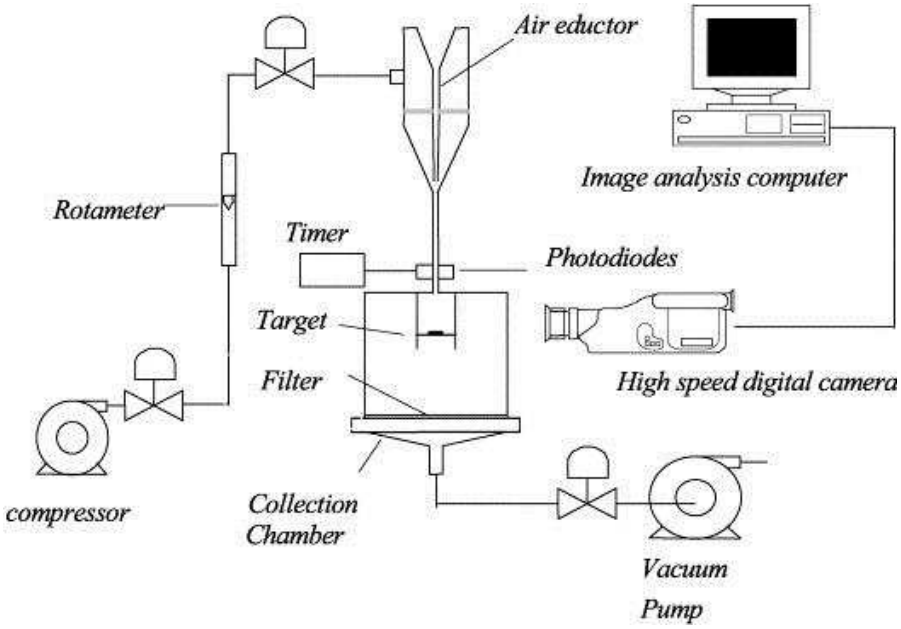

Figure 2. Single particle impact rig [15]

Particles are collected after impact testing, and sieved by two sieve sizes below the feed size. The particles passing through this sieve are categorised as debris, and the rest of the particles as mother particles. The results are expressed in terms of the extent of breakage using the equation below:

$$R^* = \frac{m_{de}}{m_m + m_{de}} \times 100\% \qquad \text{Eq. (1)}$$

where m_{de} and m_m are mass of debris and mother particles, respectively.

The extent of breakage, R^*, is plotted as a function of impact velocity for different particle sizes as shown in Figure 3. The error bars indicate a small loss of debris from the collected mass. The mother particles were sufficiently large for all to be recovered from the collection chamber.

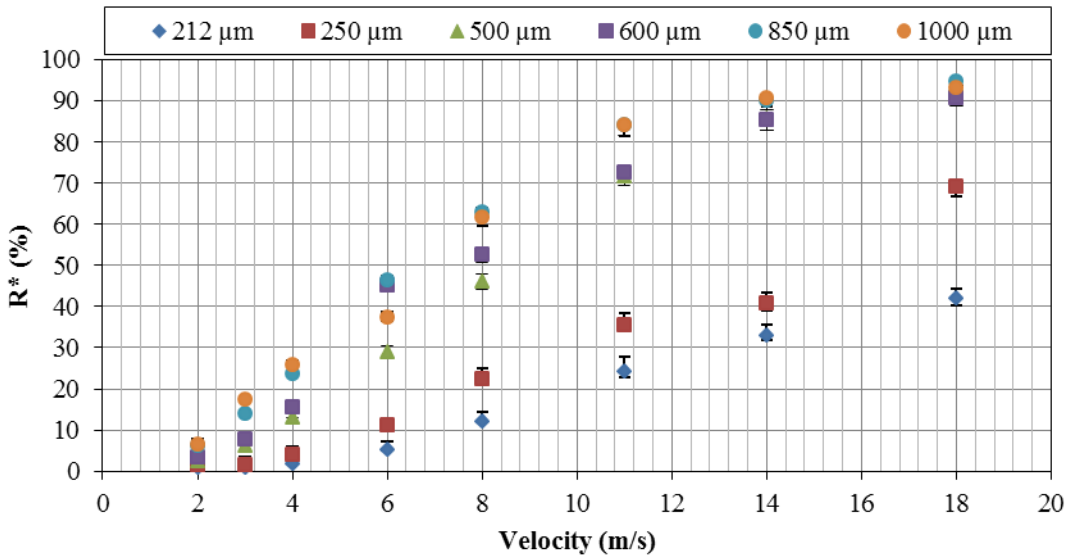

Figure 3. Extent of breakage, R^*, as a function of impact velocities for different particle sizes

The data points are very close, but nevertheless the same unexpected trend as observed in our previous unpublished work prevails here; i.e. at 6 m/s, the largest partices, 1000 μm, break less than the 600 μm and 850 μm particles. It is generally expected that the larger particles break more than the smaller ones. The anomaly in data is likely to be due to the structural differences of various particle sizes and this is explored below in this work.

In view of the above observations, it is essential to characterise the envelope density, and evaluate its variation with particle size. Mercury porosimetry can be used for this purpose, however the minimum representative sample mass required for testing is an important issue which needs to be considered. The sample quantity used for mercury porosimetry is in the sub-gram range, but this is only suitable for fine powders. For the particle size range of interest here, a much larger quantity will be required, e.g. around 2 g for 500 μm particle size. Therefore each measurement test uses insufficient material for it to be representative of the actual envelope density of the sample, particularly when there is a variation within one size of particles. Hence in this work alternatives methods for envelope density measurement have been explored, and the most reliable one is presented here.

X-ray microtomography (XRT) has been used to measure the envelope density of spray-dried burkeite particles. A bed of particles has been scanned using a Nanotom X-ray computed tomography instrument (Phoenix, Wunstorf, Germany) at a micrometre level spatial resolution.

The envelope density is then calculated using Equation (2) as given below:

$$\rho_e = \frac{\rho_b}{(1-\varepsilon)} \qquad \text{Eq. (2)}$$

where ρ_e and ρ_b are the envelope density of individual particles and bulk of the particle bed, respectively, and ε is interestitial bed voidage.

Bulk density is calculated using the mass and volume of the particle bed. The resolutions of the scans obtained for 212 μm particles (using 2 g of sample) and 1000 μm particles (using 3 g of sample) were 12 μm and 17 μm, respectively. In order to calculate the interstitial voidage, which is defined by the ratio of the air volume in between the particles to the total bed volume, the voidage inside the particles needs to be discarded. This is done in the post-processing step using Avizo Fire software by filling the internal pores. The approach has been shown in 2D in Figure 4, however the calculations are done in 3D.

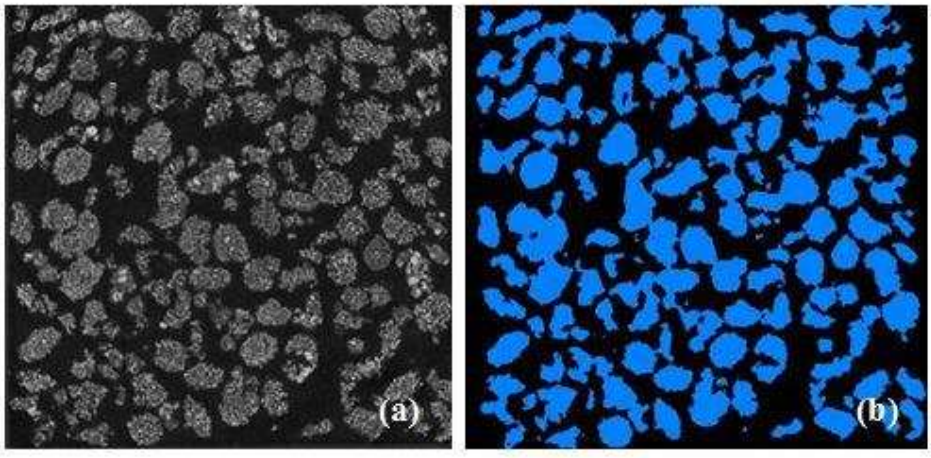

Figure 4. (a) A slice of the raw data of the bed scan for 850 µm particles; (b) A slice of the particles bed after filling the internal porosity of the particles

The black colour in Figure 4a represents air in the system, the light gray shows the particles. In Figure 4b the air inside the particles is removed (the blue colour shows the filled particles), and the interestitial voidage is calculated for the bed of the particles. Therefore, the envelope density of different sizes of the burkeite particles can be calculated using the above approach. The data are presented later along with the analysis of particle breakage.

Apart from accounting for the structural differences as a function of size, it is also critical to identify various patterns of breakage such as chipping, fragmentation and disintegration for different impact conditions. Papadopoulos [43] studied the changing trend of the size distribution curve with impact velocity based on the work of Schumann [44]. Using this approach the breakage patterns can be separated based on the trend of the curves obtained by plotting the cumulative percentage undersize as a function of normalised size. The breakage patterns are classified into chipping, fragmentation and disintegration depending on the shape of the curves as shown in Figure 5. Chipping refers to removal of a small volume of material from the surfaces of the particles by the propagation of sub-surface lateral cracks. If the particles split into several smaller fragments, formed by various types of cracks extending into the body of the particles, the process is referred to as fragmentation [45]. If the particles shatter into a large number of small fragments with wide size distribution, it is called disintegration [46].

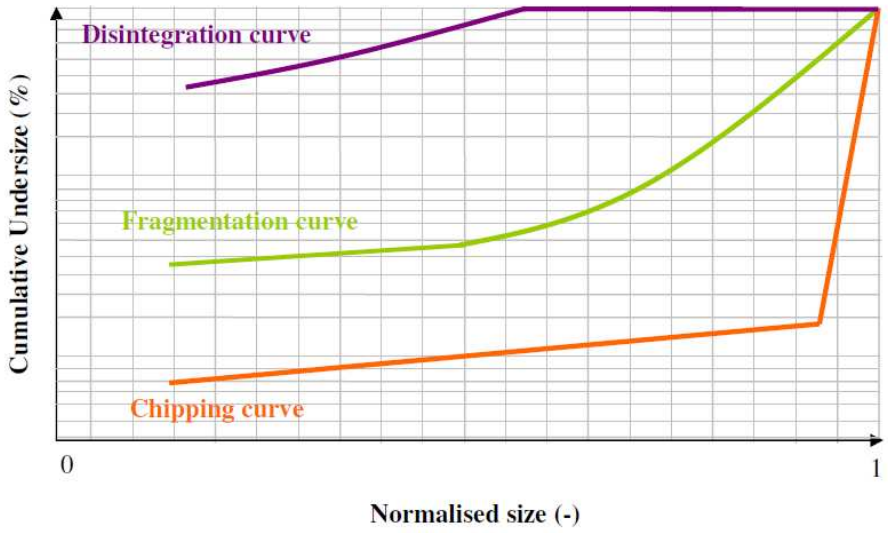

Figure 5. Breakage patterns [35]

Schumann's presentation approach has been used here to identify the impact breakage patterns of spray-dried burkeite partricles as a function of impact velocity. As an example, breakage patterns for 600 µm particles are shown in Figure 6.

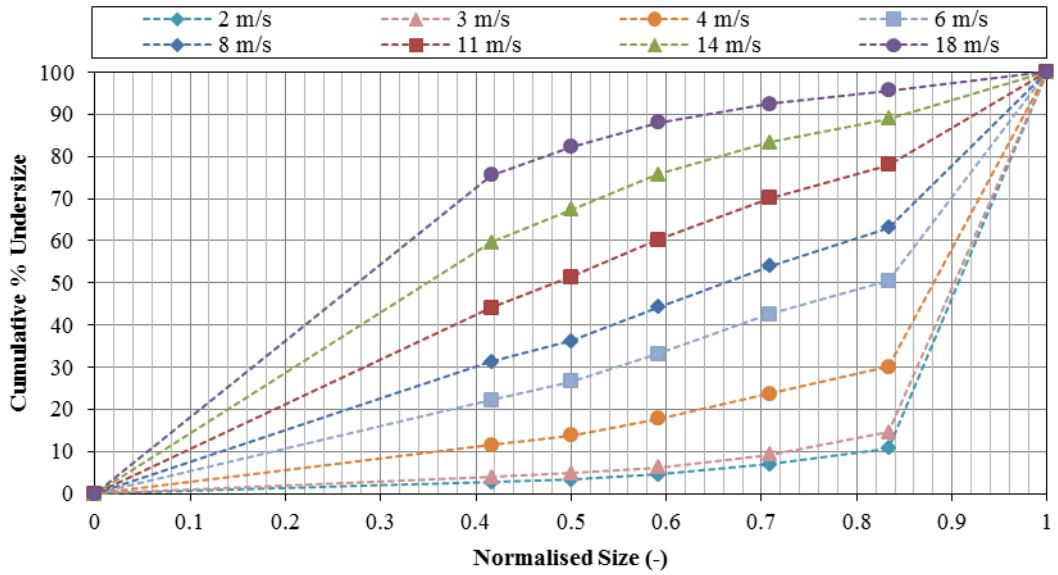

Figure 6. Different patterns of breakage for 600 µm particles based on Schumann's model

Comparison of Figure 6 with Schumann's plot (Figure 5) shows chipping regime at 2, 3 and 4 m/s, fragmentation at 6, 8 and 11 m/s and disintegration at 14 and 18 m/s as the dominant pattern of breakage for 600 µm near-mesh particles. The same approach has been applied to the rest of the near-mesh particle sizes, for which the breakage patterns have been identified. In order to analyse the trend, use could be made of various theories of impact breakage, depending on the failure mode. Due to non-uniformity of the agglomerate structure, the definitions of different modes of failure do not lend themselves well to the mechanisms of agglomerate failure. Nevertheless, the failure mode of agglomerate can macroscopically be covered by the three classical modes of failure; semi-brittle, brittle and ductile failure mode.

For the semi-brittle mode of failure, Ghadiri and Zhang [45] proposed a model for the calculation of the volume of debris based on the depth and length of the lateral cracks. The volume fraction of debris is defined as the ratio of volume of chips to the volume of original particles. The calculation of this volume fraction leads to a dimensionless group, which describes the attrition propensity due to chipping, η

$$\eta = \frac{\rho D H}{K_c^2} V^2 \qquad \text{Eq. (3)}$$

where D is a volume-based characteristic particle size, ρ is the envelope density and V is the impact velocity. H and K_c are hardness and fracture toughness, respectively.

The extent of breakage, R^*, is related to η by a proportionality factor α:

$$R^* = \alpha \frac{\rho D H}{K_c^2} V^2 = CDV^2 \qquad \text{Eq. (4)}$$

The model shows that the breakage of semi-brittle materials follows a square of velocity relationship. The parameter C represents: $\alpha\rho H/K_c^2$. The terms are material parameters that allow the extent of breakage to be related to material properties.

For the brittle mode of failure, the model of Vogel and Peukert [47] is used, which is the modified version of Weibull's equation, Eq. (5). The probability of breakage (s) is given as a function of the applied stress (σ). These fitting parameters, z, σ_s and m represent a characteristic flaw density and strength, and a parameter giving a measure of the spread of strength, respectively [48].

$$s = 1 - \exp[-z\left(\frac{\sigma}{\sigma_s}\right)^m] \quad \text{Eq. (5)}$$

In the model of Vogel and Peukert [47], Eq. (7), the parameter f_{Mat} is a fitting parameter and represents the material properties. $W_{k,min}$ is the minimum kinetic energy, which causes breakage, respectively. D is the particle size and W_k denotes single particle mass specific impact energy. f_{Mat} represents the resistance to breakage of the particle against the applied load W_k [49].

$$s = 1 - \exp\left[-f_{Mat}D\left(W_k - W_{k,min}\right)\right] \quad \text{Eq. (6)}$$

If we linearise the above model for small values of s, applicable to the chipping regime, then:

$$s = f_{Mat}D\left(V^2 - W_{k,min}\right) \quad \text{Eq. (7)}$$

Determination of $W_{k,min}$ is by experimental method, and it is subject to defining a criterion for considering particle breakage. However, for small calues of s, the method of Vogel and Peukert [47] shows the same trend in terms of dependency on particle velocity and size as that of Ghadiri and Zhang [45]. Therefore regardless of the mode of failure of burkeite particles under impact, the analysis can be carried out in terms of dependence on particle size and impact velocity. The analysis used to describe the breakage results here is based on the model of Ghadiri and Zhang [45] using the Equation 4. According to this model, the extent of breakage, R^*, for chipping regime is a function of DV^2. The chipping data for all the near-mesh particle sizes at different impact velocities have been expressed as a function of DV^2, and are shown in Figure 7.

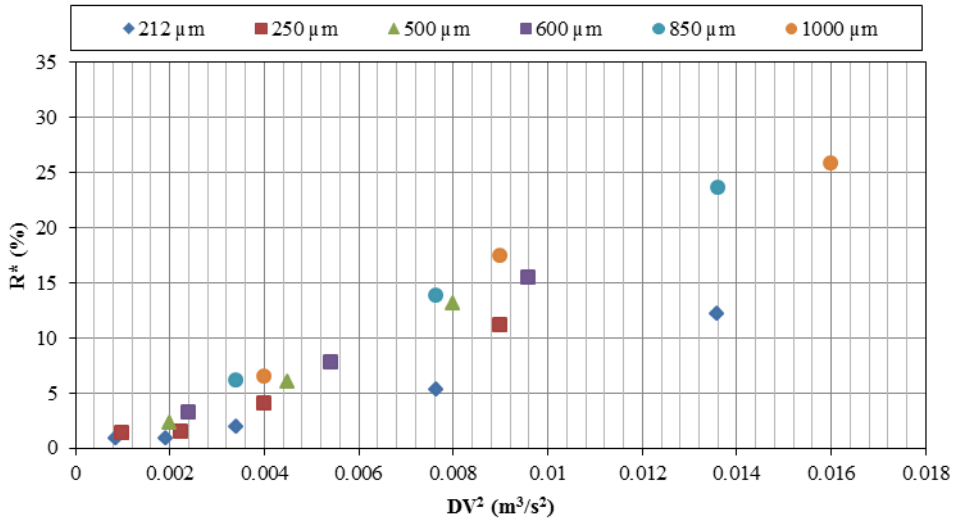

Figure 7. Extent of breakage, R^*, as a function of DV^2 for chipping regime

Clearly some unification of the extent of breakage, R^*, as a function of DV^2 is achieved as expected. However, there is scatter in the data and this is likely to have arisen from differences in the structure of the particles amongst different particle sizes, giving rise in turn to variations in the envelope density and mechanical properties, such as H and K_c according to Eq. (4).

The values of the envelope density measured by XRT are shown in Figure 8. Clearly they change with particle size, and mainly increase as the particle size is increased, except the largest particle size (1000 µm), where its value is even smaller than 850 µm particles. The 1000 µm particles are likely to have formed from the coalescence and agglomeration of smaller drying droplets, and hence the observed trend. If we consider the increasing trend of density with particle size, the difference between the expected value of density of 1000 µm particle and its actual measured value is slightly larger than the variations due to errors associated with the XRT analysis (2.4% for the latter), implying real structural differences exist amongst the particles in the size distribution. Further detailed structural analysis is needed to better quantify the role of particle growth by coalescence and agglomeration during drying. The measured envelope densities of the particles are used in the analysis of breakage data, and the extent of breakage is now expressed as a function of ρDV^2 as shown in Figure 8.

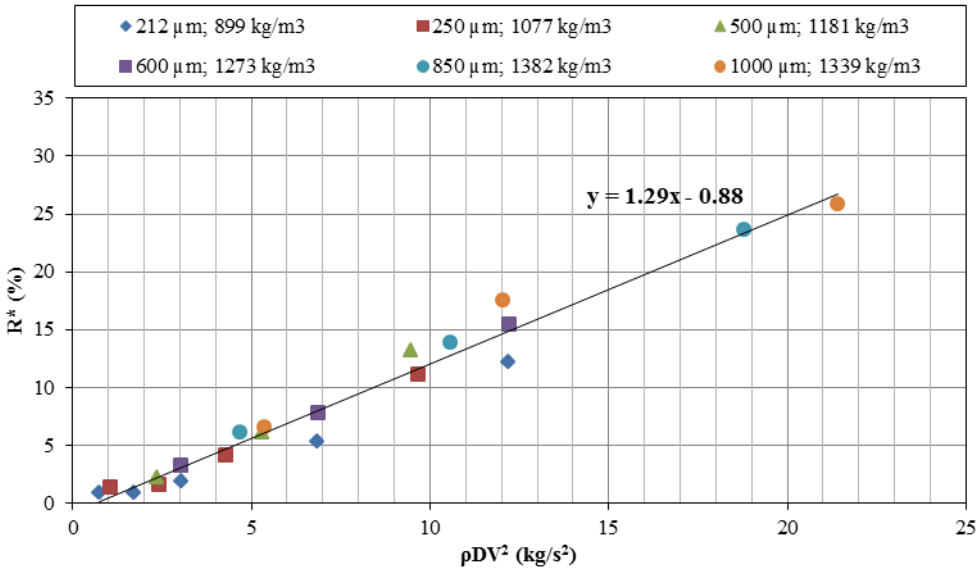

Figure 8. Extent of breakage, R*, as a function of ρDV^2 with the slope representing $\alpha H/K_c^2$, showing data unification

Clearly a much better unification than that shown in Figure 7 is obtained for a wide range of particle sizes and impact velocities by accounting for the actual envelope densities. The slope of the line is a lumped parameter representing the mechanical properties of the material, $\alpha\, {}^{H}/_{K_c^2}$. In the brittle failure mode, the strain rate has little effect on H and K_c[50], and hence their determination under quasi-static conditions would be reflecting the slope obtained in Figure 8. However, for strain rate-sensitive materials (such as semi-brittle materials), the material mechanical properties obtained by dynamic methods are different from those by quasi-static methods [43]. Therefore, the results obtained by single particle impact testing (dynamic test method) do not necessarily corroborate those from nano-indentation (quasi-static test method). Interestingly, considering the intercept of the fitted line with the abscissa, there is a minimum impact velocity for a given particle size below which there is no/ little breakage. This information is very useful for designing pneumatic conveying lines, cyclones and other items of equipment, where particles experience mechanical stress by impact.

8. CONCLUSIONS

Attrition of spray-dried burkeite particles has been studied by applying different impact stresses prevailing in a manufacturing plant. Burkeite is very weak and friable; hence it is prone to breakage even at low impact velocities. The impact tests indicated some structural differences for different particle sizes such as envelope density variation as a function of size. The envelope density of different particle sizes have been estimated using X-ray microtomography, and applied to the analysis of the breakage data. The results show that the envelope density generally increases with particle size, although the underlying mechanism given rise to these variations is unknown. It is found that a nice unification of breakage data may be obtained for the chipping regime using the measured envelope densities and extent of breakage for chipping regime. This approach can be used to estimate the mechanical properties of the material based on the model of Ghadiri and Zhang [45].

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