A study of Particle adhesion for Cohesive Powders using a Novel Mechanical Surface Energy Tester

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Powder cohesiveness has a strong correlation with particle adhesion, which is studied using a novel mechanical surface energy tester and a method to measure Bond numbers of the powders. The mechanical surface energy tester measures particle adhesion by detaching particles adhered to a substrate surface. When the substrate is dropped from a set of heights and stopped against a stopper, the particles are subject to a detached force, which in principle is equivalent to the particle adhesion force between the detached particles and the substrate. The detached particles are collected for further particle size analysis. The Bond number of the powders is calculated as a ratio of adhesion to gravity with the particle physical properties measured such as solid density and full-size distribution.

In this study, particle adhesion forces for a wide range of sample powders were selected and investigated with powder tabletted substrates (same as the test powders), including Calcium Carbonate, Lactose, Microcrystalline Cellulose, Paracetamol, Ibuprofen and Titanium Dioxide for a wide range of material properties. Influences of substrate materials on the measurements are studied between the powder tabletted substrates and other standardised materials such as mild steel, glass, stainless steel and TIVAR. The study shows that the substrate material has little influence on the measurements of particle adhesion within a maximum variation of about 2.5%. This allows using different substrates for the measurement of Bond numbers. The adhesion forces measured are also compared to those calculated by other established methods, and some correlations have been found.

Keywords: Particle adhesion, Bond number, Cohesive powders, Surface energy tester

1 Introduction

Particle adhesive force plays a critical role in powder processes, such as powder flow of cohesive powders [1]. There are many methods widely used, such as the atomic force microscopy (AFM) method [2] and the centrifugal method [3], a comprehensive review can be found in the literature [4]. However, all methods have limitations when the measurements are correlated to the bulk behaviour of powders such as powder flow behaviour with varied particle size distributions [1]. With more particles and stresses, particle adhesion becomes more complex because of varied particle sizes, surface textures and contacts of particles. The number of particles counted for particle adhesion characterisation can be critical; otherwise, representative particles would be lost. For any traditional adhesion tests, sampling of the powder is challenging in relating to the bulk behaviours, such as a flow function test [5, 6]. Typically, in the pharmaceutical industry, content uniformity of active pharmaceutical ingredients (API) blended with excipients is key in manufacturing [7], which requires particle adhesion and flow properties at the same time. To study such information, particle adhesion

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must be measured along with known powder physical properties such as particle size distributions, non-uniformed particle shapes and true solid density.

It can be a big challenge in determining particle adhesion in conjunction with powder bulk behaviours, however the existing methods can measure particle adhesion with small quantity materials [4]. The major problems for the existing techniques are either focusing on the interaction of individual particles with the probe or limited information in conjunction with other particle properties such as particle size, shape, and density [1]. By the existing theories of adhesion [8], the particle adhesion force is a pull-off force to detach contacting particles from a contact area with specific interfacial surface energy. The adhesion force depends on surface energy of particles, minimum separation distance, and active contact area. The surface energy of particles for powders is subject to not only its chemical compound but also its physical properties [4, 8]. The contact area can vary with the size of particles and the formation of clusters (e.g. two particles). In theory, particle adhesion has a link with cohesiveness for bulk material, but it cannot represent the cohesiveness directly. For this purpose, Bond number of powders can be a helpful indicator because it gives information about particle adhesion for a specific particle size [5, 9, 10].

In this study, a novel mechanical surface energy tester developed at the Wolfson Centre [11] is used to determine particle adhesion and Bond numbers for several samples varied in a wide range of material properties. For the tester, influence of substrates on the detached force measurements is also investigated using several substrate materials such as a powdered tablet, mild steel, glass, stainless steel and TIVAR. Bond numbers of the sample powders are calculated using the adhesion forces measured from the surface energy tester. The adhesion forces measured are also compared to those calculated using the existing theories, such as calculations from surface energy of particles by well-known models.

2 Theory and apparatus

Adhesion force in powders is a complex phenomenon that depends on intrinsic material properties such as surface energy [12] and particle physical properties such as particle size, shape, and surface textures [13]. With a review of adhesion fundamentals [4, 8, 14], the particle adhesion force between particles and a surface and the measuring of the Bond number for a powder using a mechanical surface energy tester are discussed.

2.1 Adhesion force between particles and a surface

Adhesion force between particles or to a surface normally consists of van der Waals forces, capillary forces, electrostatic forces [15], which one or more of these forces existed depends on the physicochemical properties of the materials that are in contact [16]. The adhesion force can be expressed as:

$$F_{ad} = F_{vw} + F_e + F_{cf} \quad (1)$$

where F_{vw} is van der Waals force, F_e is electrostatic force, and F_{cf} is capillary force. Characterisation of individual forces is quite challenging, some theories for each force are given in the literature [4, 8, 14]. According to the theories, the individual forces are calculated based on an assumption of perfectly rigid particles in contact with uniformed particle shapes,

e.g., spheres. Also, there is a continuous debate about the separation distance between two particles for theoretical study.

By the theories, determining the adhesion forces at a bulk level for powders with or without the effect of consolidation stress is difficult because of huge variations of particle properties and uncertain contact areas between the particles, characterising these requires further investigation. One of the possibilities discussed is decoupling contributions from different particle properties on particle adhesion [1]. It has been concluded that the contribution of each particle property needs to experimentally isolated to investigate the effect of each particle property on an individual component of powder bulk behaviour [1]. In this case, the unique contribution of surface energy, surface area and particle shape on particle adhesion needs to be studied [17]. A simpler way instead is to define adhesion force to reduce the complexities involved in calculating particle adhesion forces in the existing literature.

2.2 Bond number and particle adhesion force

A cohesive granular Bond number can be helpful in representing powder cohesiveness based on the information of particle adhesion forces measured and the sizes of these particles. The bond number (B_o) is defined as a ratio of particle adhesion force, F_{ad}, to particle gravity force, F_g, for the particles as shown in Eq. 2 [10].

$$B_o = F_{ad}/F_g = F_{ad}/mg \qquad (2)$$

The Bond number of a powder can dominate powder bulk behaviours and cohesiveness [5]. For Bond numbers more than one or significant, the powder is cohesive in nature, whereas if the Bond number is less than one, the powder is free flowing. Since the adhesion force depends on the local asperity radius at the contact, the adhesion strength decreases with decreasing asperity radius [18]. Therefore, a Bond number (B_o) can represent particle adhesion force in terms of particle size such as a median size, D_{50}, as shown in Fig. 1.

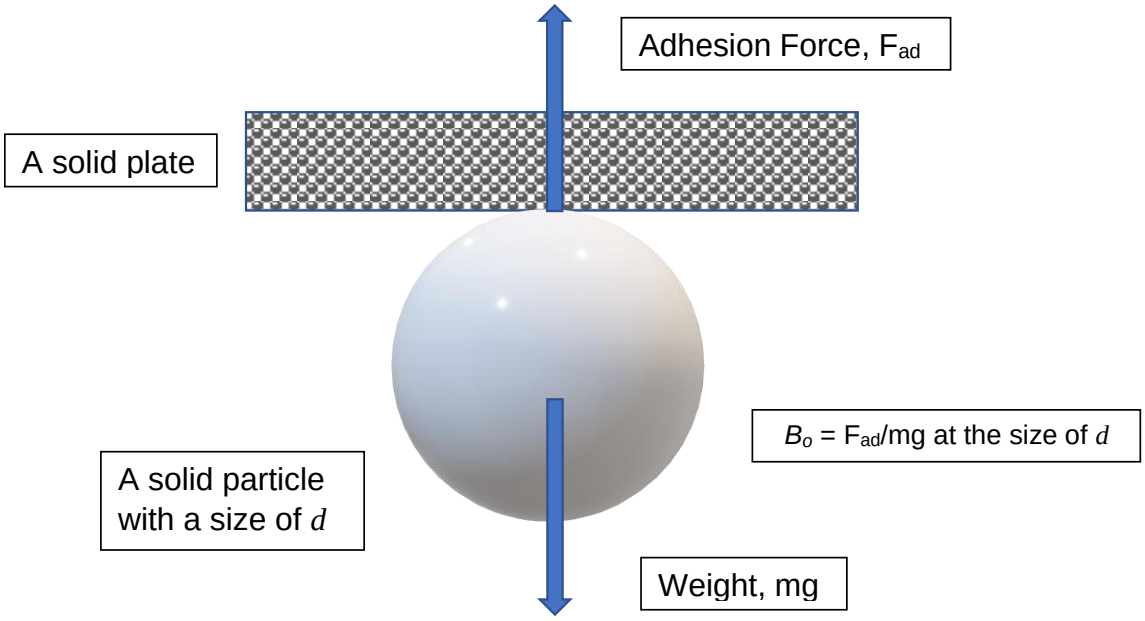

Figure 1: Principle of the 'Bond number' (B_o) defined.

As mentioned in Eq. (1), the adhesion force is a sum of several forces. If only van der Waals force is considered, the adhesion force can be expressed as [19]:

$$F_{ad} = \frac{H_a D^*}{12 z_0^2} \quad (3)$$

where z_0 is the separation distance between two surfaces, H_a is the Hamaker constant that depends on the material properties and D^* is the equivalent diameter of the particles. In the case of particles attaching to a flat surface, D^* is the diameter of the particle attached to the surface. In the adhesion theories of Johnson, Kendall and Roberts (JKR) [20] and Derjaguin, Muller and Toporov (DMT) models [21], the adhesion force is calculated as:

$$F_{ad} = 2\pi R W_A \ (DMT) \quad (4)$$

$$F_{ad} = \frac{3}{2}\pi R W_A \ (JKR) \quad (5)$$

where R is the radius of a particle in the case of a sphere–plane contact or the reduced radius of two sphere particles. W_A is the equilibrium work of adhesion for the particles.

In the Bond number detection (Fig. 1), if a particle adheres to a flat surface, the Bond number of the particle must be more than 1 because the adhesion is more dominant compared to its gravity force, making the particle attached to the surface. Therefore, for any non-cohesive powders which particle adhesion could be less than its weight, the Bond number needs to be determined by other traditional methods rather than a tester with the principle shown in Fig. 1. In the case of cohesive powders mentioned here, a surface energy tester (see Fig. 2) will have advantages to determine particle adhesion forces and Bond numbers without assuming the separation distance (z_0), Hamaker constant (H_a) of particles in Eq. (3).

2.3 Mechanical surface energy tester

A novel small-scale mechanical surface energy tester is developed and shown in Fig. 2 for measuring particle adhesion and Bond number (B_o) of a cohesive powder.

For the measurement, a powder sample is attached to a substrate of 40 mm in diameter in the sample holder (see Fig. 2). Adhesion force is assumed to be equivalent in magnitude, but opposite in direction to the deceleration force at the detachment, as seen in Eq. (6).

$$F_{ad} = -F_{de} = -ma \quad (6)$$

where a is the deceleration for the detachment and m is the mass of the detached particles.

The technique uses imaging analysis and an electronic scale to determine the status and the weight of adhered particles on the substrate surface before and after each test. The force to detach the adhered particles is believed to correlate with the force to deposit the particles on the substrate surface before the detachment tests. Therefore, in this measurement, two key aspects must be considered: i) influence of particle cluster during particle deposition; ii) influence of the substrate surface where the particles are attached.

Only 20 milligrams or less of the sample powder are used to deposit onto the substrate surface to avoid any clusters for the first point. Also, scanning electron microscope (SEM)

images of the deposited surface are randomly taken to confirm a monolayer of particle deposition is always achieved. Regarding the second point, a compacted tablet is used, which is made from either a few grams of commonly available materials or the same powder sample representing the solid surface. However, the detachment of particles is also investigated on standardised material substrates such as mild steel, glass, stainless steel, and TIVAR-88 to reduce the need for sample materials for making the substrate tablets.

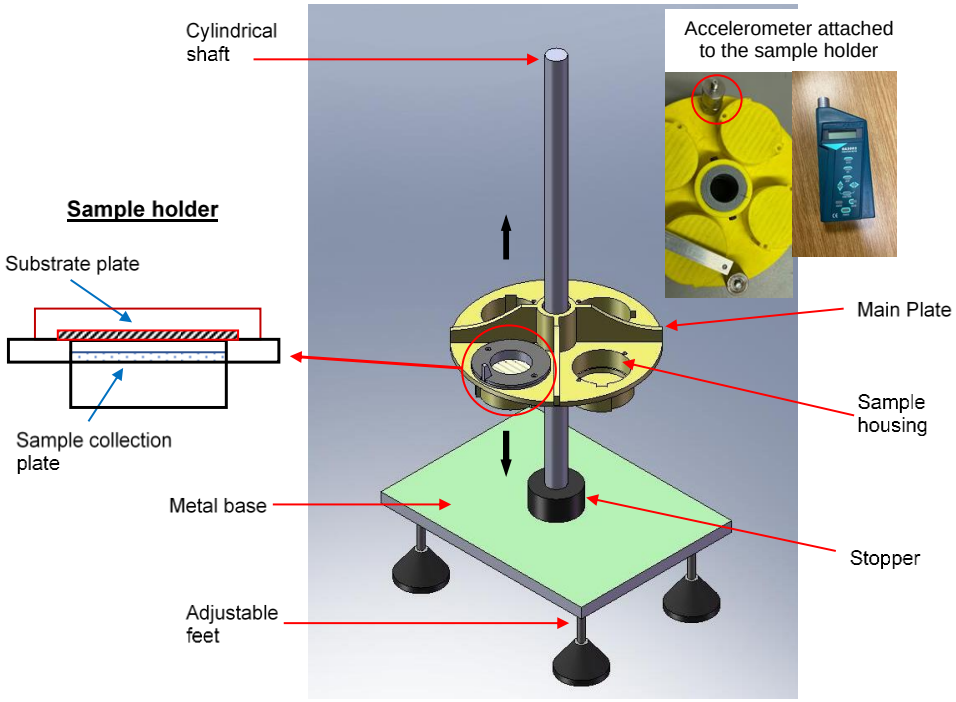

Figure 2: Principle of the novel small-scale mechanical surface energy tester [11]

Before carrying out a test, sample powder is dispersed onto a substrate using an air disperser (accessory for Malvern G3 Morphologi, Malvern Panalytical Ltd. UK) at an air pressure of 1.5 bars. After powder deposition, the substrate tablet is fitted into a sample holder (Fig. 2). Multiple sample holders in the housing plate are lifted to a certain height and then released to drop. The substrate is halted against a buffer at the stopper, so a proportion of the attached powder is detached from the surface. The deceleration value of the substrate is recorded by an accelerometer (Castle GA 2002 Vibration Meter, Castle Group Ltd, UK) attached to the main plate (Fig. 2), which gives a span of 0.1 to 100g (g, gravitational acceleration) and overall tolerance of ± 1.5dB. The detached particles are collected by a sample collection plate, measured for total mass, and examined under a Malvern G3 microscope for measuring median particle size and the nature and number of the detached particles. The particle adhesion is detected by using the deceleration value and used to calculate the Bond number.

3 Experimental methodology

An experimental study has been carried out with several different types of powders and different substrate materials regarding particle adhesion measurements.

3.1 Powder materials

A wide range of powders has been selected for this study, including calcium carbonate, lactose, microcrystalline cellulose, paracetamol, ibuprofen, and titanium dioxide giving a wide range of material properties such as particle size and particle density. The calcium carbonate is named with Eskal series grades and manufactured by KSL Staubtechnik GmbH, Germany. The lactose also has different size categories named with manufacturer series (by MEGGLE GmbH & Co. KG, Germany), and the same as the ibuprofen (provided by BASF, Germany). The rest of the materials only have one size category. Microcrystalline cellulose (MCC) is supplied by KP Snacks, UK. Paracetamol is provided by AstraZeneca, UK. Titanium dioxide is supplied by MegaChem (UK) Ltd. Specifications of the materials in the study are given in Table 1.

In Table 1. Summarised particle size percentile values (volume % measured by the Malvern Laser Diffraction method) for the materials are also shown in the table with other physical properties, including size span calculated using particle size and solid particle density measured using nitrogen pycnometer.

Table 1: List of the materials studied and material physical properties

S. No.	Materials	*Particle Size* (μm)			Size Span	Solid Density
		D_{10}	D_{50}	D_{90}	$(D_{90}-D_{10})/D_{50}$	(kg/m^3)
1	Eskal 2 (Calcium Carbonate)	1.1	2.0	34	16.47	2800
2	Eskal 4	2.0	4.0	13	2.76	2800
3	Eskal 10	5.5	10	17.7	5.54	2800
4	Eskal 15	9.6	15	26.1	9.63	2800
5	Ibuprofen	22.4	70	174.7	2.18	1118
6	Ibuprofen 25	2.6	13	33.5	2.37	1132
7	Ibuprofen 50	5.3	26	75.8	2.71	1110
8	Ibuprofen 50_Jet milled	1.3	4.0	9.0	1.92	1113
9	Lactose 140	31	150	244	1.42	1558
10	Lactose 200	48	200	330	1.41	1558
11	Lactose 230	61	230	380	1.39	1558
12	Lactose 70	19.3	90	118.6	1.10	1558
13	Microcrystalline cellulose	8.3	22.7	45.7	1.65	1590
14	Paracetamol	8.0	49	97	1.82	1280
15	Titanium Dioxide	2.0	68	84	1.21	4475

SEM images of the materials are given in Fig. 3, which shows the particles have different particle shape and agglomerations that may influence the particle adhesion. The figure shows that some powders contain more agglomerates when the particle size is smaller such as calcium carbonate and ibuprofen, extreme particle shapes e.g. paracetamol, or denser spherical particle e.g. titanium dioxide. All are believed to have strong influences on the particle adhesion forces.

3.2 Set and test procedure

The adhesion force is measured using a mechanical surface energy tester with four samples at the same time (see Fig. 2) by determining the acceleration of the detached particles from a substrate surface in a vertical direction of motion. With the acceleration measured and the mass, m of the detached particles measured using an electronic balance with an accuracy of

0.1 milligrams, the detachment force for the sample powder is calculated using Eq. (6), equivalent to an averaged adhesion force acted on the particles. All the measurements were undertaken at ambient temperature and room humidity of 40-60% related humidity.

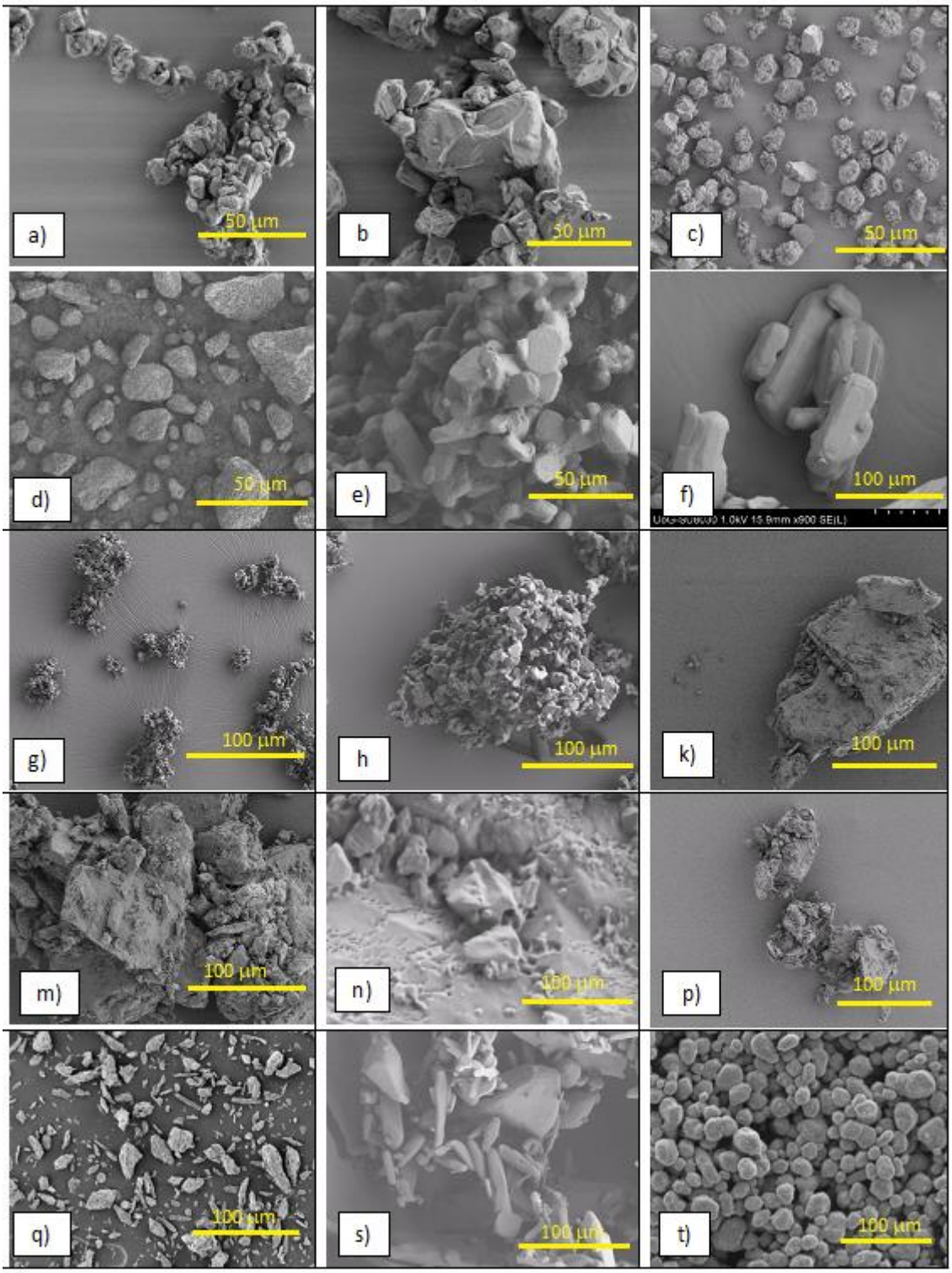

Figure 3: SEM images of the materials studied: a) Eskal 2, b) Eskal 4, c) Eskal 10, d) Eskal 15, e) Ibuprofen, f) Ibuprofen 25, g) Ibuprofen 50, h) Ibuprofen 50_Jet milled, k), Lactose 140, m) Lactose 200, n) Lactose 230, p) Lactose 70, q) Microcrystalline Cellulose, s) Paracetamol, t) Titanium dioxide.

To have a reliable result of detachment measurement, the deposition of powders onto the substrate surface and collecting detached particles are key to the study. First, preparing the substrate tablet is undertaken using either a tablet made from the same powders compacted or a standardised substrate such as glass, TIVAR, mild steel and stainless steel (see Fig. 4). Absolute surface roughness for these substrates is in a range of 0.1 μm to 0.6 μm. The surface of the powdered substrate directly represents the adhesion between the same powder materials, but the standardised substrates are used to study the detachment of particles on the different substrate surface.

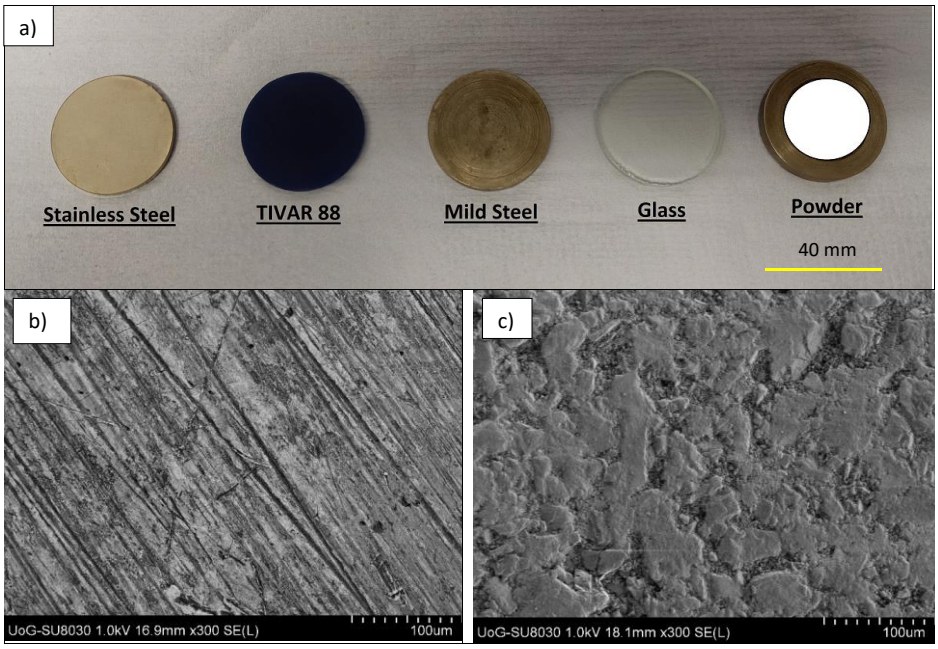

Figure 4: Photos of the substrate discs and SEM images of two typical substrates: a) Substrate discs, 40 mm in diameter, b) SEM image of a mild steel substrate, c) SEM image of a powder tabletted substrate made from lactose powder.

Dispersion of the powder sample onto the substrate using the air dispenser at 1.5 bar air pressure gives a good particle dispersion typically to avoid any overlaps of particles or possible agglomeration. However, to have a monolayer dispersion, dispersion air pressure depends on particle size and density of the powders. An example is given in Fig. 5.

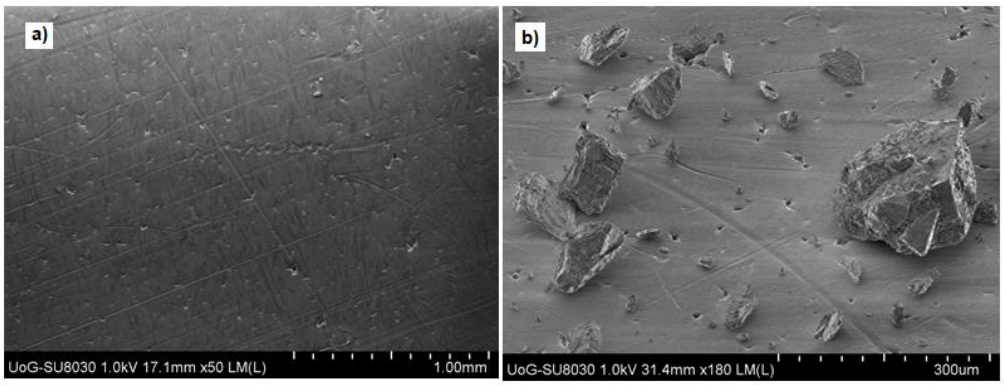

Figure 5: SEM images of a TIVAR substrate, a) before powder deposition, and b) lactose powders deposited.

As mentioned in section 2.3, the acceleration value recorded for the detached particles from the substrate is key to determining particle adhesion and the Bond number. Therefore, the detached particles carefully collected and the particle detachment from the substrate surface are examined by image analysis using a Malvern G3 microscope for particle size determination. 9 SEM images reconstructing the detachment of the particles are used to examine the particle detachment. A typical example of lactose particle detachment from a powder tabletted substrate surface is shown in Fig. 6.

All SEM images were captured on JSM-5510 Scanning Electron Microscope (make: JEOL Ltd) by applying the powders on aluminium stubs using double-sided carbon tape and coated with a 5-mm layer of gold/palladium (Au: Pd ¼ 80:20). The instrument was operated at an accelerating voltage of 1 kV and the images were taken at a magnification of 50×LM (low magnification).

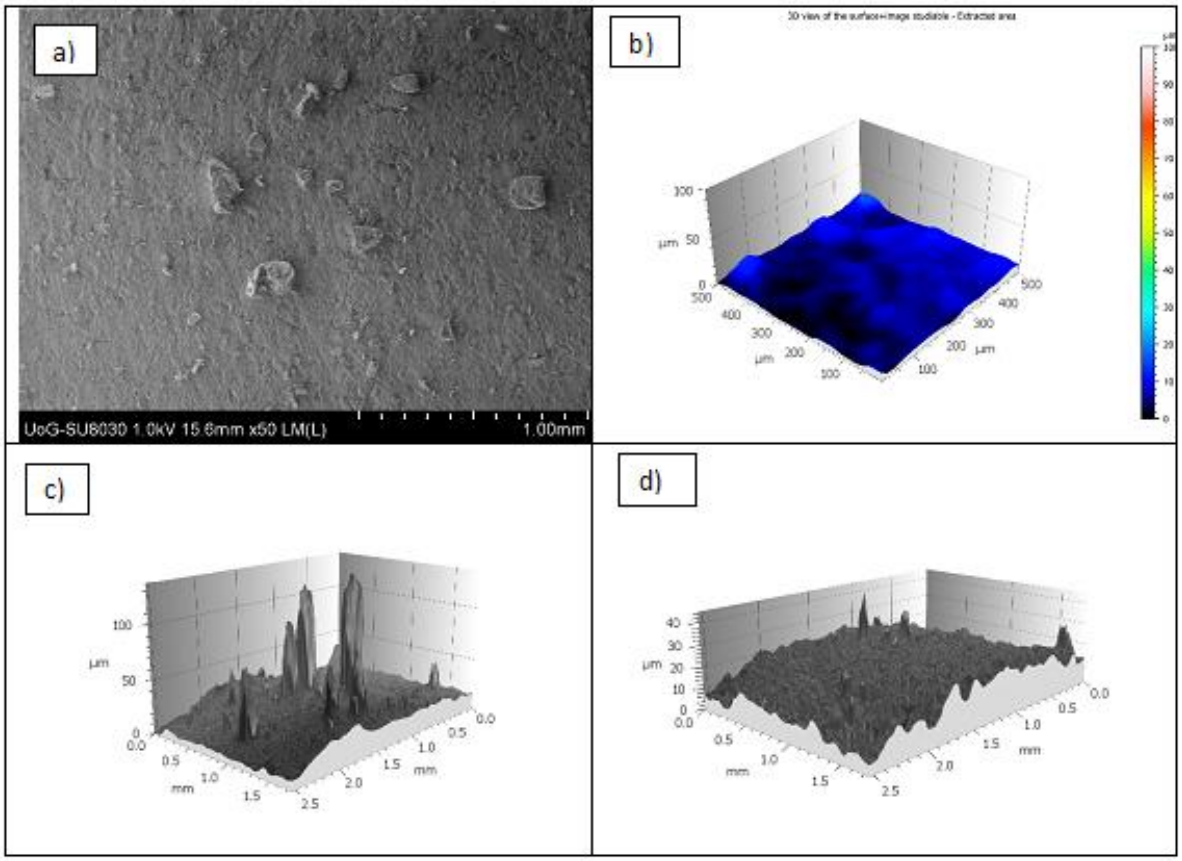

Figure 6: Images of lactose particles on a powder tabletted substrate and powder detachment: a) Powders on the substrate surface, b) The surface profile before powder deposition, c) The surface profile with particles attached, d) The surface profile after powder detached.

4 Results and discussion

Particle adhesion for 15 powder samples has been studied using a mechanical surface energy tester. Also, Bond numbers for the powders are reported and discussed here. The adhesion measured is compared to the calculation by the existing models,

4.1 Powder tabletted substrate surfaces

For particulate materials, the adhesion force between particles could be different from that between particles and a flat surface. Using the mechanical surface energy tester to measure adhesion forces in powders, the selection of the substrates would influence the measurement. To represent adhesion force between particles, a powder tabletted substrate is used, which is compacted from the same powder for particle dispersion to a tablet. The accelerations measured for all sample powders are presented in Fig. 7, which show the mass percentage of the detached material over the total material deposited versus the acceleration needed for the detachment.

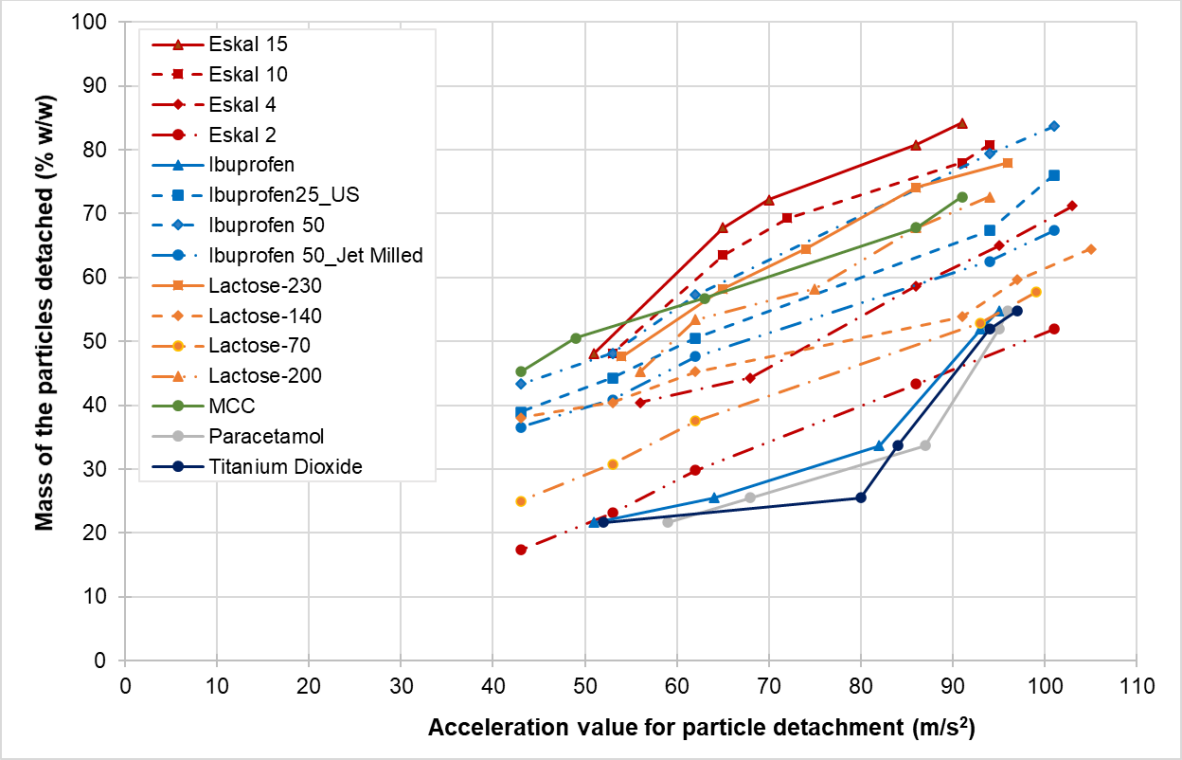

Figure 7: The mass proportions of the powders detached from a powder tabletted substrate at different decelerations for 15 sample powders.

In Fig. 7, the proportion of the material detached from the substrate surface increases almost linearly with an increased acceleration applied. Using the acceleration value measured and the mass of the detached particles, the adhesion force of the detached particles is calculated by Eq. (6). The results of particle adhesion forces calculated based on the results in Fig. 7 for the particles at the size of D50 are shown in Fig. 8, which the size is calculated using the detached particles.

4.2 Pure material substrate surfaces

The results in Fig. 7 and 8 show the adhesion force measurements between particles and a flat surface made of the same powders. To study the influences of substrate surfaces on the adhesion force measurement, a few substrate materials are selected, including mild steel, stainless steel, TIVAR, and glass. The results of accelerations measured for one sample powder (Lactose) with different particle sizes are shown in Fig. 9 for different substrate materials,

which shows mass percentages of the material detached and corresponding accelerations measured.

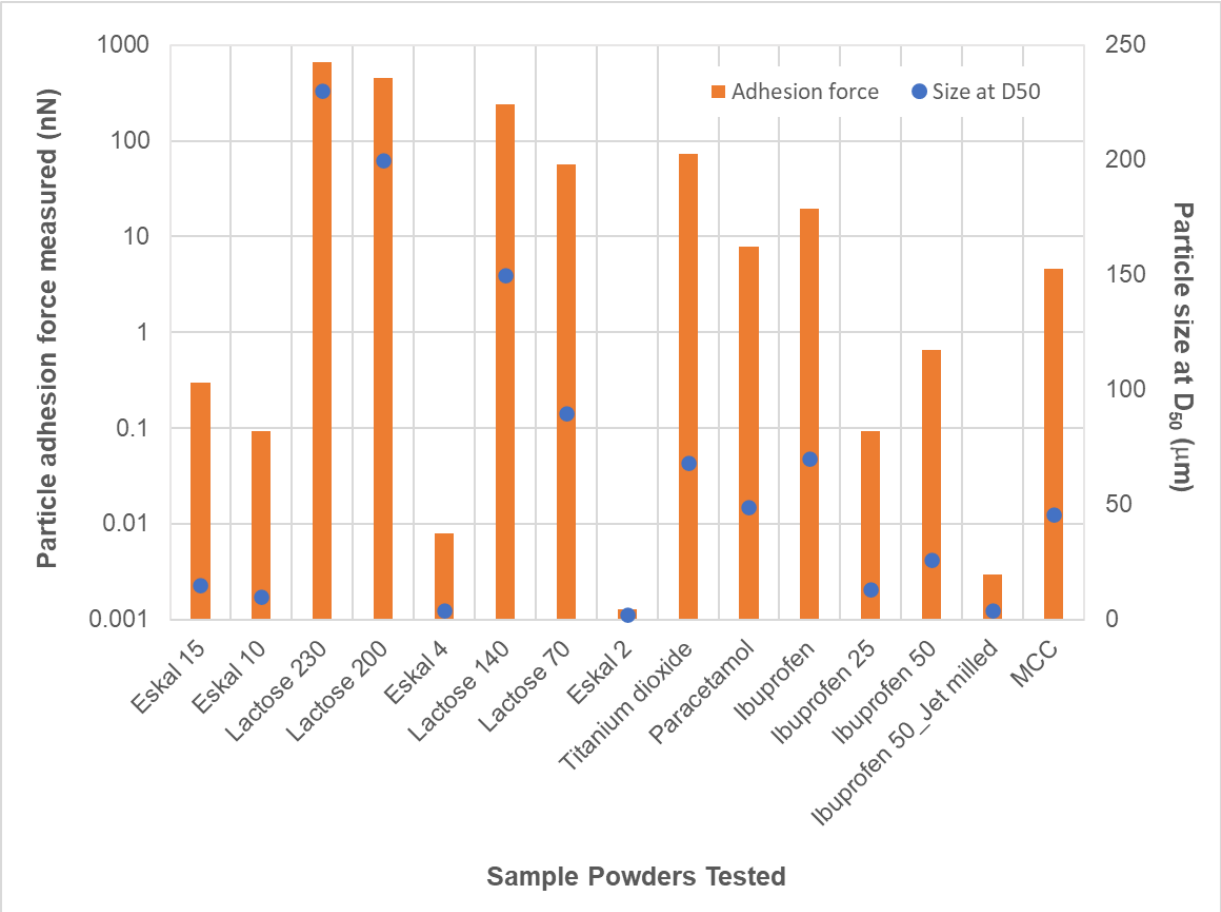

Figure 8: The particle adhesion forces measured versus particle sizes for 15 sample powders.

The results in Fig. 9 show that the particle size has a clear influence on the level of the materials detached at the identical acceleration. However, for the powder with different substrate materials, the acceleration needed to detach the same proportion of the material is almost identical. The results mean that the substrate material has little influences on the acceleration required to detach the proportion of the particles. Alternatively, the particle adhesion measurement using the mechanical surface energy tester is subject to the powders and the powder deposition method. If a consistent operation method is applied to the adhesion measurement, the results will be highly repeatable and reliable, whatever the substrate material is selected. The results of adhesion forces measured using different substrate materials, including the powder tabletted substrates, are shown in Fig. 10. Variations between the measurements are given, which shows the highest value of only about 2.5% and the rest variations are between 1.5% to 1.7%.

The measurement results prove that the substrate materials have little influence on the particle adhesion measurement from the tester. This gives an advantage to the technique by using any substrate to measure powder adhesion while it still provides a good result (2.5% errors), rather than using the sample powders for making a substrate tablet.

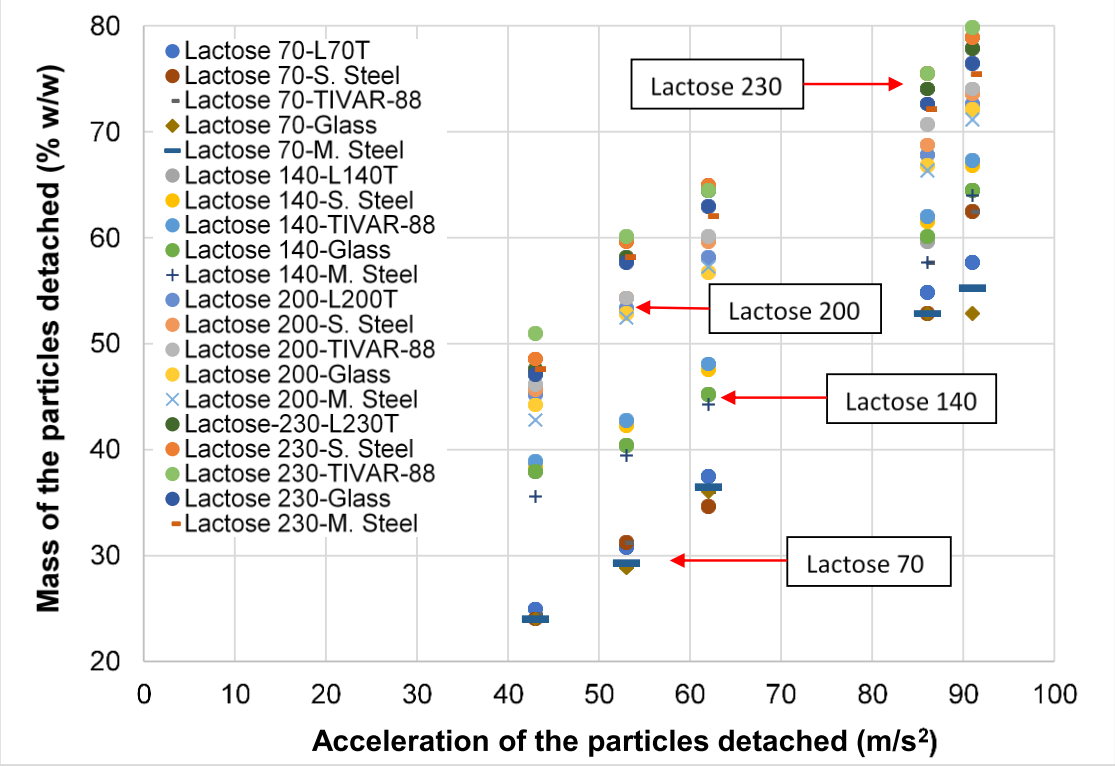

Figure 9: The mass proportions of the powders detached from pure material substrates at different decelerations for the lactose 70, 140, 200 and 230.

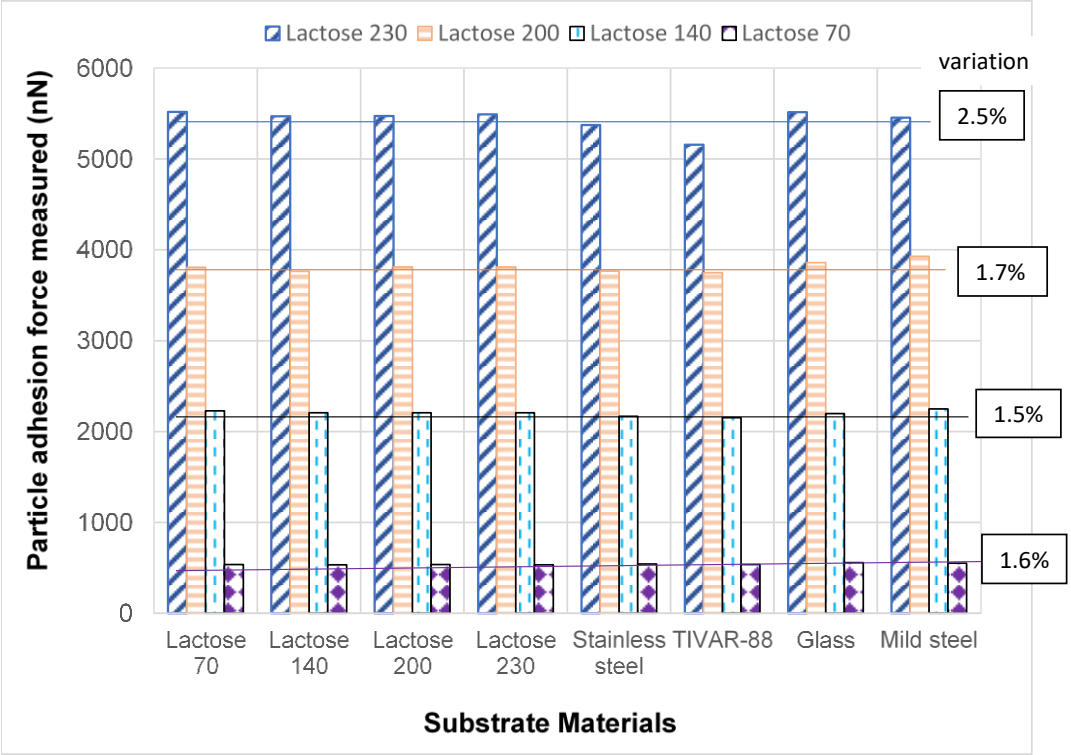

Figure 10: The particle adhesion forces measured for the powders of lactose 70, 140, 200 and 230 at an average size of D_{50} in mass detached from powder tablet and pure material substrates.

The results for different substrate materials show a possibility to measure particle adhesion using a small quantity of sample powders (milligrams of powders) on a mechanical surface energy tester, since any standardised substrate can replace the powder tabletted substrate and give a result with a maximum variation of 2.5%. These results also indicate that particle adhesion between particles and a surface is more likely subject to the particle properties and the particle deposition status on the surface, rather than the surface materials.

4.3 Particle adhesion and Bond numbers

The adhesion force for a powder can be easily detected with the acceleration measured for detaching the particles from the surface energy tester. Fig. 8 shows that a large particle tends to have a big adhesion force since the particle needs a large adhesion force against its gravity. However, it does not mean that a powder with a large particle size is more cohesive, whereby the particle size plays a significant role in the cohesiveness. Therefore, the particle adhesion measured is not suitable for assessing powder cohesiveness directly. Instead, the Bond number is more appropriate to represent powder cohesiveness because it takes a ratio of particle adhesion to particle gravity at a certain particle size.

The method for detection of a Bond number is illustrated in Fig. 11 using Ibuprofen 25 powders, which the accelerations of 50% mass detachment are selected for the adhesions of the particles against various substrate materials, including powder tablets of Ibuprofen 25, 50, and 50_Jet milled, and other standardised substrate materials including mild steel, stainless steel, glass and TIVAR_88. It shows a minimal variation in the results between different substrate materials. The Bond number of the particles at the 50% mass detachment is calculated by the adhesion force measured using the acceleration and the mass of the particles calculated from the results in Fig. 11.

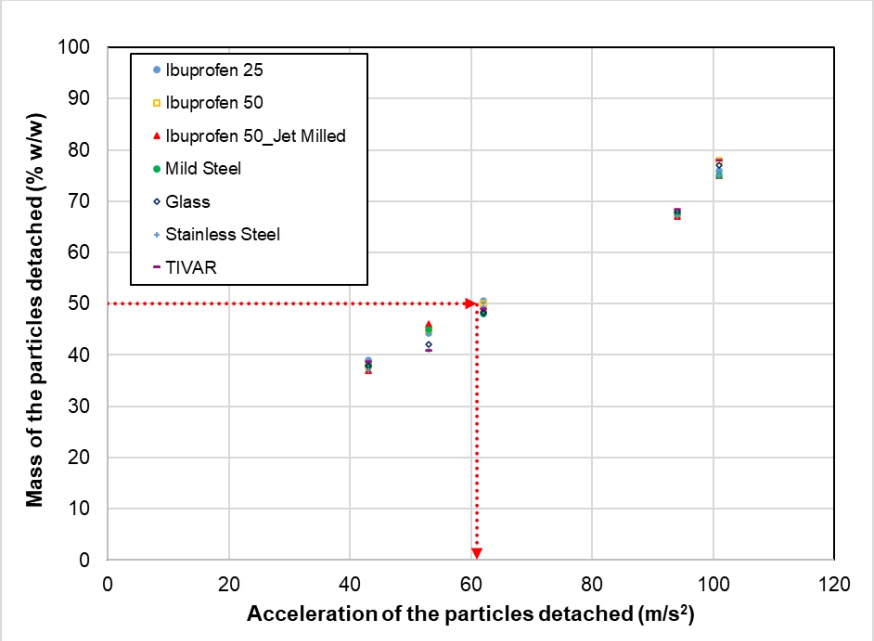

Figure 11: Acceleration values of the Ibuprofen 25 powder at 50% mass detached from varied substrate materials including powder tablets of Ibuprofen 25, 50, and 50_Jet milled, and other pure substrate materials of mild steel, stainless steel, glass and TIVAR_88.

By the method shown here, Bond numbers for the 15 samples powders are given in Table 2.

Table 2: Particle Bond numbers for the sample materials at the size of D_{50}

S. No.	Sample powders	*Particle Size, D_{50}* (µm)	Accelerations (m/s^2)	Bond Number
1	Eskal 2	2.0	98.24 ± 1.06	11.01
2	Eskal 4	4.0	75.03 ± 1.78	8.65
3	Eskal 10	10	53.13 ± 1.95	6.41
4	Eskal 15	15	50.45 ± 2.55	6.14
5	Lactose 70	90	85.23 ± 2.63	9.69
6	Lactose 140	150	76.49 ± 7.95	8.80
7	Lactose 200	200	59.98 ± 1.93	7.11
8	Lactose 230	230	56.26 ± 0.43	6.73
9	Titanium dioxide	68	89.44 ± 1.01	10.12
10	Paracetamol	49	89.46 ± 1.95	10.12
11	Ibuprofen	70	88.04 ± 1.44	9.97
12	Ibuprofen 25	13	60.62 ± 0.75	7.18
13	Ibuprofen 50	26	54.61 ± 0.47	6.57
14	Ibuprofen 50_Jet milled	4.0	70.11 ± 5.03	8.15
15	MCC	22.7	48.02 ± 0.48	5.89

With the results in Table 2, a general comparison between the particle adhesion forces measured and corresponding Bond numbers at the particle sizes of D50 for the 15 sample powders are shown in Fig. 12.

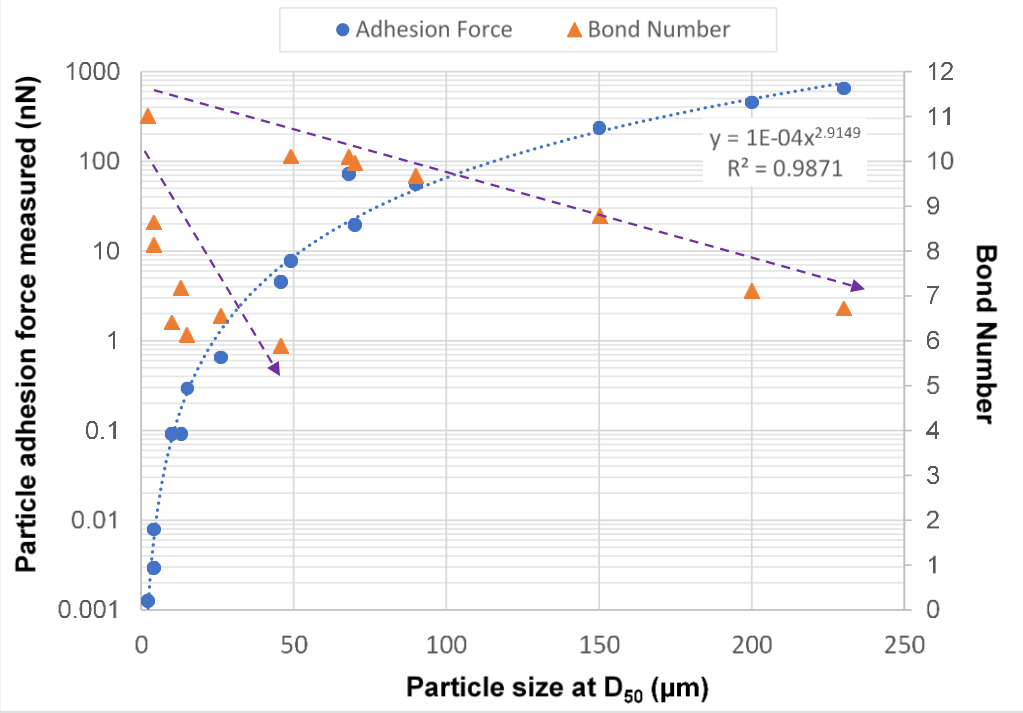

Figure 12: The particle adhesion forces measured and corresponding Bond numbers versus particle sizes of D_{50} for the 15 sample powders.

In the figure, it shows decreasing Bond numbers when the particle size is increased. Therefore, Bond number is more representative of powder cohesiveness than a direct measurement of particle adhesion force using the mechanical surface energy tester. Two trends for the Bond numbers are shown in the figure, which indicates that the surface energies of the particles cause the difference rather than the domain for the geometry of the powders. Therefore, a Bond number can represent particle adhesion at a specific size, which will be helpful for assessing powder cohesiveness at a bulk level and is also applied by other researchers [22, 23]. The results in Fig.12 also show a strong correlation between particle adhesion forces and particle sizes but cannot classify in any groups.

4.4 Comparison to the JKR and DMT models

The adhesion force measured using the surface energy tester is compared to the values calculated using the JKR model, Eq. (5) and the DMT model, Eq. (4). The surface energies for the sample powders were measured using Finite Dilution Inverse Gas Chromatography (FD-IGC) Surface Energy Analyser (Surface Measurement Systems, London, UK). For the FD-IGC measurement, about 1.2 grams of dried powder was packed into a glass tube (300 mm length and 4 mm I.D.) and covered with glass wool on the ends. The sample was tapped to remove any voids in the sample. The tube was mounted into the column oven. A series of n-alkane probes and polar probes were inserted (in dry Helium at a flow rate of 10 ml/min) to obtain the retention behaviour of the probes and the subsequent dispersive (γ_d) surface energies were calculated using the Schultz approach [24]. The surface energy values (or the equilibrium work) for the sample powders are given in Table 3. As mentioned, the surface energy for powders is regardless of particle size and only materials related, so there is only one value for the same powder materials. With the surface energy and particle size at D_{50}, particle adhesion forces are calculated using the DMT and the JKR model, as two series are shown in Fig. 13.

Table 3: Surface energies for the sample powder materials

Materials	Calcium Carbonate	Lactose	Titanium dioxide	Paracetamol	Ibuprofen	MCC
Surface Energy (mJ/m^2)	54 ± 4	44 ± 4	58 ± 2	44 ± 2	42 ± 5	42 ± 3

With the best-fitted trendlines, correlations between the measurements from the mechanical surface energy tester and the models are vital, as R^2 values are 0.993 for the DMT model and 0.997 for the JKR model. In general, the adhesion measured from the surface energy tester is a power of 3 more significant than that the models give. The reasons behind this are unknown and need further investigations.

5 Conclusions

Particle adhesions for cohesive powders have been studied using a novel mechanical surface energy tester. Experimental results show that the surface energy tester can provide a reliable result if a monolayer of particles is adequately attached to a substrate surface. The tester can be used for particle adhesion measurements between particles or particles between any

other surface by selecting representative substrate on either tabletted powder or standardised tablets. A study on different substrate materials for one powder with different particle sizes shows that the substrate has little influence on the adhesion measurement. It only gives a maximum variation of about 2.5%, and the most are about 1.5%-1.7%.

This discovery means particle adhesion using milligrams of samples on the surface energy tester can be detected with any standardised substrate rather than a powder tabletted substrate. The results also indicate that particle adhesion between particles and a surface is more likely subject to the particle properties and the particle deposition status on the surface, rather than the surface materials. The adhesion results also show that large particles have a big adhesion measured from the surface energy tester, which does not agree with powder cohesiveness. The results of Bond numbers for 15 powders show that Bond numbers decrease when the particle size is increased, which agrees with the fact that powders with large particle sizes are usually less cohesive.

The particle adhesion force measured using the surface energy tester is also compared to the values calculated using the existing models such as the JKR and DMT models. With the best-fitted trendlines, correlations between the measurements from the tester and the models are strong, as R^2 values are 0.993 for the DMT model and 0.997 for the JKR model. There is a three times difference in values. The reasons are unknown and need further studies.

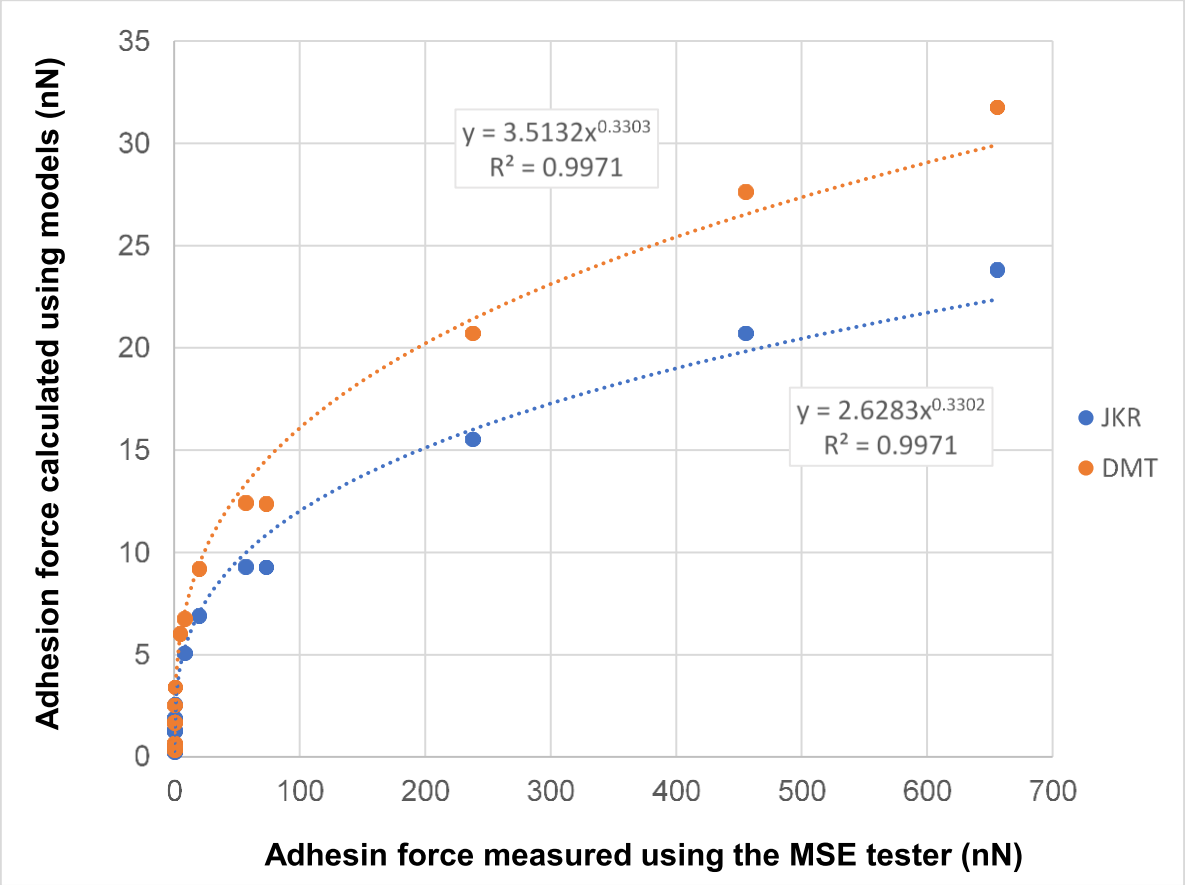

Figure 13: Comparison of particle adhesion forces measured from the mechanical surface energy tester to the calculations using JKR and DMT models for the 15 sample powders.

It is summarised that:

1) Using the mechanical surface energy tester, particle adhesion for cohesive powders can be studied in terms of particle sizes using different substrate materials.
2) Substrate materials have little influence on the measurement, including powder tabletted and standardised substrate materials.
3) A small amount of sample powders (milligrams) can be used in adhesion measurements on the surface energy tester using a standardised substrate.
4) The Bond number can be more representative of powder cohesiveness than the particle adhesion force measured on the surface energy tester.
5) The measurements of particle adhesion have strong correlations with other well-known adhesion models such as JKR and DMT model, but not in a linear relationship.

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