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MEASURING THE PHYSICAL PROPERTIES OF GRANULAR FERTILIZER IN RELATION TO FLOW

JASON D. M. COUSINS[1], MARTIN J. ROBERGE[2], LOPE G. TABIL[3]

[1] Department of Chemical and Biological Engineering, University of Saskatchewan, Saskatoon, SK, Canada
<jason.cousins@usask.ca>
[2] Design Analysis and Simulation Group, CNH Industrial, Saskatoon, SK, Canada
<martin.roberge@cnhind.com>
[3] Department of Chemical and Biological Engineering, University of Saskatchewan, Saskatoon, SK, Canada
<lope.tabil@usask.ca>

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ABSTRACT The improvement of equipment for handling, storage, and application of granular fertilizer requires a proper understanding of material properties and flow behaviour. The propensity of fertilizers to cake (i.e., agglomerate) introduces additional challenges for storage and handling. While select information can be found in the literature, a full description of material properties for granular fertilizers suitable for design and modelling is not available. Laboratory experiments were conducted to measure select physical properties for granular urea (46-0-0) fertilizer. These included: particle density, elastic modulus, angle of internal friction and cohesion, angle of repose, particle size distribution, particle shape, and coefficient of restitution. Density ($\bar{x} = 1298$ kg/m^3) and size distribution (diameter, $\bar{x} = 2.53$ mm, s = 0.44 mm) for granular urea were found to be similar to published values from the literature. Novel quantities for urea of elastic modulus ($\bar{x} = 7.04 \times 10^7$ Pa), angle of internal friction (37°) and cohesion (0.9 kPa), angle of repose ($\bar{x} = 15.4°$), and circularity ($\bar{x} = 1.05$, s = 0.05) which are absent in the literature were determined. Coefficients of restitution for several materials were also measured, including urea-urea contact, and were found to be higher than those reported in the literature. The experimental measurements obtained herein add to those available in the literature and contribute to an improved understanding of granular fertilizer flows.

Keywords: Granular fertilizer, urea, physical properties, flow properties

INTRODUCTION Many agricultural fertilizers come in the form of bulk granular materials. These materials are manufactured, handled, and used in large quantities and often behave like a continuous substance (fluidized material) but in fact consist of many small particles. Improving handling, storage, and application equipment for granular fertilizers requires engineering design and analysis. Most analysis methods, including traditional continuum mechanics and modern discrete element modelling (DEM) require material properties as input and their accuracy and robustness can be sensitive to these material properties. Unfortunately, a full description of physical properties for many granular fertilizers is absent from the published literature. This necessitates that properties be assumed, which can be inaccurate, or be measured experimentally, which can be costly and time consuming. The present study sought to determine properties for granular urea fertilizer through measurement and experimentation to supplement those properties currently found in the literature.

METHODS A total of seven tests were conducted to measure select properties of granular urea (46-0-0) fertilizer. A brief description of the operating principals and equipment used for each test is presented in the following sections. The analysis material was Terico brand urea fertilizer, manufactured in Canada. The fertilizer was in new condition and was purchased in 22.7 kg (50 lb) bags.

Particle density A gas pycnometer device was used to measure the true solid density of granular urea (not to be confused with bulk density). For the analysis of urea, an AccuPyc II 1340 gas pycnometer (Micrometrics Instrument Corp., Norcross, GA, USA) was used. The pycnometer uses changes is gas pressure to estimate the volume of gas displaced by the solid material. Combined with sample mass the true solid density is calculated. The analysis gas was helium. The automated AccuPyc device performed repeated purge and measurement cycles (10) for each sample. For details on the operating principles and procedure refer to the pycnometer’s operator manual (Micrometrics 2014).

Confined Compression Test The confined compression test was used to estimate the elastic modulus, E (Pa), of granular urea under compressive load. The procedure followed that of Coetzee and Els (2009). A sample of material was subjected to a cyclical compressive load while confined in a metal die. The applied load and resultant displacement were measured during the testing cycle from which the stress-strain curve for the confined material sample was obtained. The slope of the stress-strain curve during loading beginning with the second compression cycle (post-consolidation) was taken to be the apparent or confined elastic modulus, E' (Pa), for the material. The true elastic

Figure 1: Instron universal testing machine (left) and cylindrical die and plunger (right) used to confine sample and apply load for the confined compression test.

modulus was then calculated from the confined elastic modulus and the Poisson's ratio, ν, using the equation 1.

$$E = E'\left(\frac{(1+\nu)(1-2\nu)}{1-\nu}\right) \tag{1}$$

An Instron 3366 Universal Testing System (Instron Corp., Norwood, MA, USA) with a 10 kN load cell was used to apply the compressive load and measure the induced response. The material sample was confined in a cylindrical die constructed of stainless steel with an accompanying plunger made from the same material. The diameter of the cylinder was 57 mm and the height of the sample once loaded was 30 mm (nominal) for a diameter to height ratio of approximately 2:1. Each sample was subject to a 5 kPa preload to ensure a consistent starting position (from which the extension was zeroed) followed by 4 load-unload cycles applied at a rate of 2.0 mm/min and to a maximum compressive extension of 1.0 mm. The Instron tester, load cell, cylindrical die and plunger are seen in Figure 1.

Direct Shear Test The direct shear test measures friction and cohesion properties for soils and granular materials. The material is placed into a sample chamber (termed the shear cell) which is split in half along its horizonal mid plane (see Figure 2). A vertical load is applied to the top of the material in the shear cell to generate a normal or confining stress. During the test, the motor displaces the connected half of the shear cell causing shear failure in the material sample along a shear plane between the two halves of the shear cell. The force transducer measures the resulting shear force for a given normal stress. The material's yield locus is constructed by repeating the shear test for multiple normal stresses and plotting the resulting shear stress, T (kPa), vs. normal stress, N (kPa). The yield locus can generally be approximated by a straight line, of which the slope (φ) is used to calculate the angle of internal friction ($\theta = \tan^{-1}\varphi$) and the intercept gives the cohesion, c (kPa), of the material.

$$T = \varphi N + c \tag{2}$$

The direct shear tester used to conduct measurements for granular urea was a Humboldt 5750. This type of tester is typically used for measurement of soils and differs in some respects from the Jenike shear tester more commonly used for bulk granular materials.

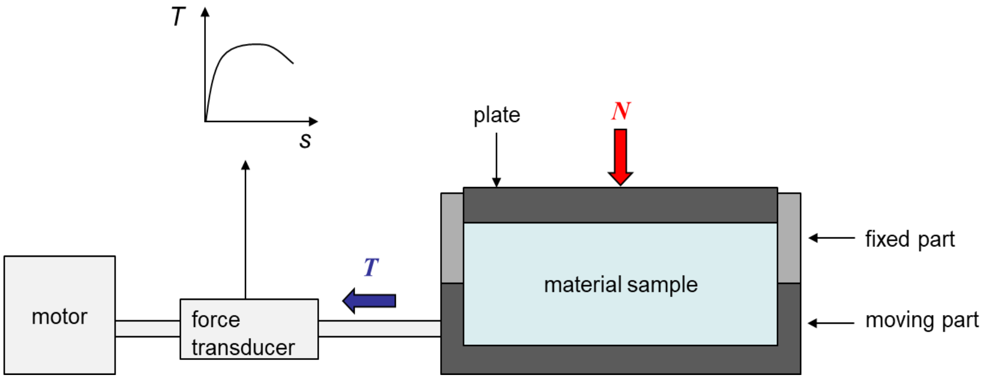

Figure 2: Diagram of the standard shear cell apparatus (reproduced from Keppler et al. 2016).

However, the operational principle is the same for all shear testers. Shear tests were conducted for granular urea using a shearing rate of 1 mm/min and normal stresses of 14.5, 27.9, and 41.8 kPa. The rectangular sample size was 60 mm x 60 mm with a nominal thickness of between 20-25 mm. An important note, shear tests typically involve a pre-shear stage which is meant to achieve a repeatable state of material consolidation prior to beginning the primary shear. However, a pre-shear stage was not conducted for the results reported here.

Slump Test A slump test was conducted to measure the static angle of repose for granular urea. The angle of repose is an extrinsic property of the material which reflects the amount of internal friction and cohesion. In the slump test, a cylindrical container forms a column of particles. When removed, the unsupported column collapses to form a pile. The angle between the slope of the pile and the horizontal ground plane is the angle of repose, i.e., the maximum slope that the unsupported material can maintain.

A slump test apparatus was constructed using a split cylinder design as seen in Figure 3. The cylinder (measuring 88.9 mm I.D. and 305.5 mm high and manufactured from acrylic) was mounted on spring loaded arms which pivot around a point above the cylinder. A pin holds the cylinder in the closed position while material is loaded. Removing the pin causes the two halves of the cylinder to pull away from the column allowing it to collapse, forming the pile. The base consisted of loose urea to minimize non-urea contact. The split cylinder design was chose because of its suitability for testing cohesive materials in the future (Grima and Wypych 2011).

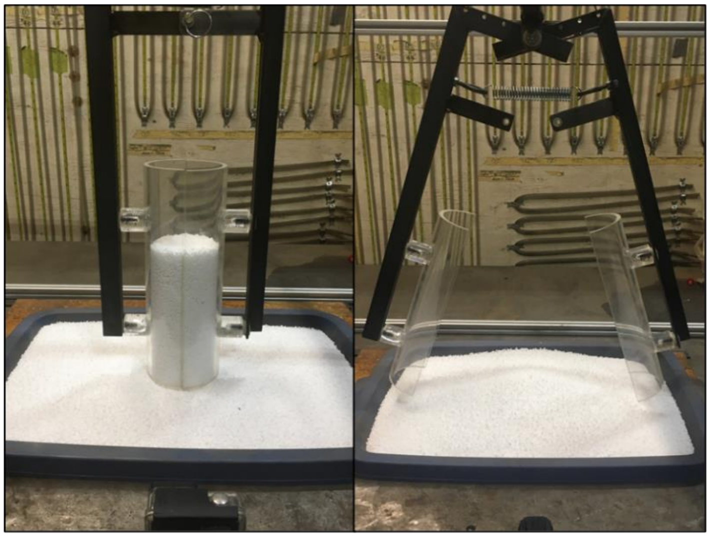

Figure 3: The slump test apparatus used for measurement of the static angle of repose in both pre-test (left) and post-test (right) states.

Particle Size Distribution The sieve size analysis is a standard method for classifying the particle size distribution of granular materials. Material is passed through a standard set of sieves/screens with progressively finer openings. Particles pass through the sieve openings until they reach a size smaller than their diameter. The distribution of sample mass retained on each size of sieve is then used to construct the particle size distribution for the material. The sieve size analysis was conducted according to ASABE Standard S319.4 (ASABE 2008) and using standard US sieve sizes. A mechanical shaker was used.

Particle Image Analysis Image processing techniques were used to quantify the shape of urea particles to permit more accurate modelling. Image analysis software identified particles and calculated several quantitative statistics. Examples of computed values include particle area, perimeter, equivalent diameter (for a circle with the same area as the particle), major and minor axes for an ellipse fitted over the particle, and circularity. Circularity is the ratio of particle perimeter to the perimeter of an equivalent area circle and gives a measure of how closely the particle resembles a circle/sphere. It is the 2-dimensional analog of sphericity.

Particle image analysis was completed using open-source libraries and custom scripting implemented in Python. To ensure good edge detection, the white urea particles were imaged against a black background to maximize contrast, as seen in Figure 4. The sample was placed directly under a light o minimize shadows. The original resolution for the input image was 3024x3024 pixels which covered an area approximately 10 cm x 10 cm. The images were cropped to remove background. A relatively basic digital camera was used to capture the images. The figure below shows samples (zoomed in) of both the original input image and processed output image for a sample of urea particles.

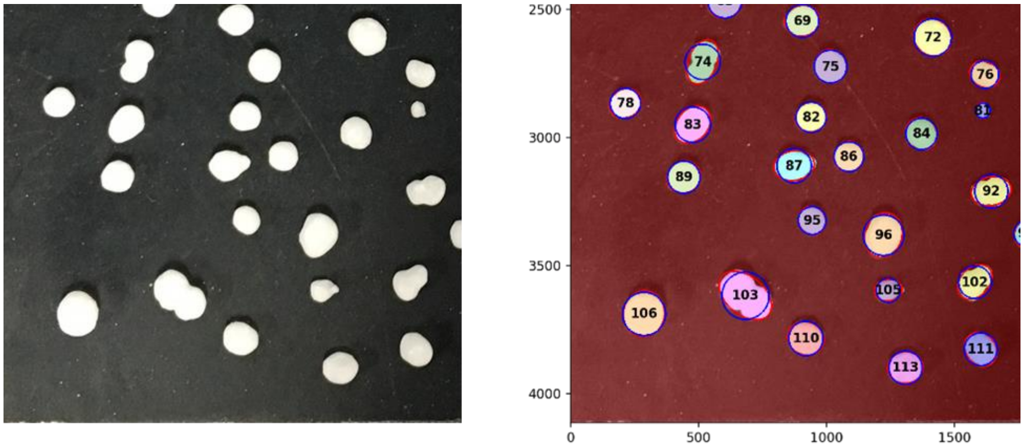

Figure 4: Particle image analysis for a sample of urea particles showing the original input image (left) and the processed output image with particle overlays (right).

To provide perspective on the image analysis results obtained for urea, image analysis was also performed on a calibration material consisting of polyethylene pellets which had a consistent shape and diameter. Due to the forming process, these pellets had a slightly elongated shape. Axis lengths for the calibration particles measured using calipers were a minor axis of $\overline{x} = 3.52$ mm and $s = 0.06$ mm, and a major axis of $\overline{x} =3.60$ mm, $s = 0.06$ mm. This was considered sufficiently spherical to act as reference point.

Particle Drop Test Results from the particle drop test were used to estimate the coefficient of restitution (CoR) for collision of urea particles with several surface materials. The coefficient of restitution is the ratio of particle velocity after collision to before collision with a surface or another particle. It indicates the degree to which the collision is elastic. The particle drop test is perhaps the simplest way to measure the CoR. A particle is dropped from a known height, h_0 (cm), onto a prepared surface. The rebound height of the particle after collision, h_f (cm), is measured. Assuming air resistance is negligible, height and velocity are proportional and the CoR is calculated using the below equation.

$$CoR = \sqrt{\frac{h_f}{h_0}} \tag{3}$$

The particle drop test was conducted inside of an acrylic tube. A ruler was fixed to the back of the tube to provide a height scale. To improve drop consistency, a sheet of paper with a pin hole was affixed to the top of the tube in which particles could be loaded and slowly pushed through the hole providing a consistent initial velocity and position. The initial drop height of the particles was 30 cm. A prepared flat surface was placed below the tube for the particles to rebound against, completing the apparatus depicted in Figure 5. The rebound of the particles was captured using a slow-motion camera to accurately record rebound height. Particles were pre-screened to remove any that were too small, too large, or too non-spherical due to the difficulty of getting vertical rebound with such specimens. Only collisions with a near vertical resultant trajectory (perhaps within 10-15 degrees determined visually) were included in the calculations.

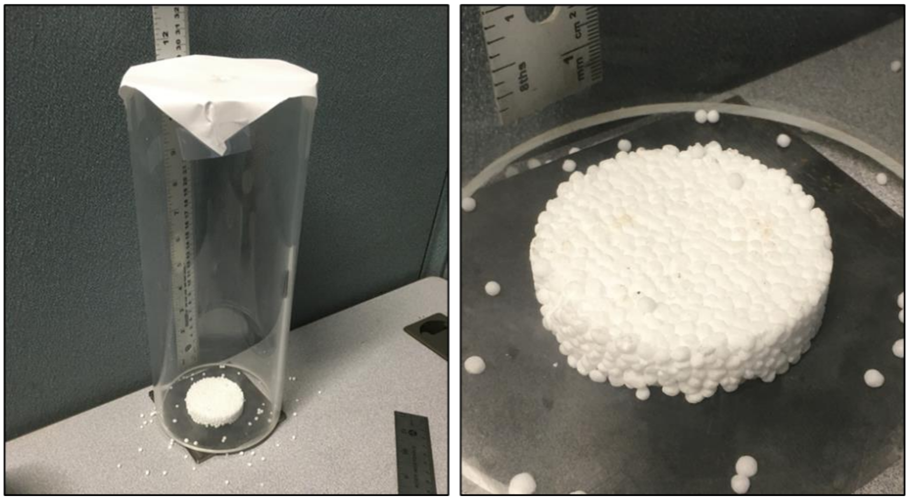

Figure 5: Experimental setup for measuring the coefficient of restitution via the particle drop method.

The CoR was measured for urea contact with four surface materials: steel, concrete, acrylic, and urea. For urea-urea contact, a flat rebound surface in the form of a urea “puck” was manufactured by compressing heated urea into the disk like shape documented in Figure 5. While this was necessary for reliable rebound, the influence of shape and surface finish changes caused by compression on the measured CoR - and how well this value reflects contact between two urea particles - has not been investigated.

RESULTS AND DISCUSSION The following sections present experimental results and measured property values for the various tests conducted as part of this study.

Particle Density Results for the solids density of granular urea as measured using the gas pycnometer are presented in Table 1. A total of 4 replications were conducted with new sample material for each. Two replications were made using the Terico brand urea in original as purchased condition while the remaining two were conducted on oven dried samples. No appreciable difference in density between the two conditions was observed. The average density for urea was measured to be 1298 kg/m^3 with a standard deviation between samples of 2.7 kg/m^3. The measured value is consistent with online sources which state the density of pure urea to be 1320 kg/m^3 (Wikipedia 2019).

Table 1: True particle density measurements made using a gas pycnometer, N = 4.

Run	Sample Mass (g)	Mean Density (kg/m^3)	Std Dev (kg/m^3)	CV (%)
1	55.432	1299.8	0.3	0.0231
2	61.477	1300.2	0.3	0.0231
3	60.576	1295.7	0.8	0.0617
4	57.635	1295.1	0.5	0.0386
Overall		1297.7	2.7	0.2059

Elastic Modulus The confined compression experiment was conducted to estimate the elastic modulus of urea. A sample of the data collected for a single specimen is presented in Figure 6. The first cycle is seen to be primarily consolidation of the material and was omitted from calculations. The slope of the 2nd, 3rd, and 4th compression cycles were used to calculate the confined elastic modulus.

The true elastic modulus for urea was calculated from the confined elastic modulus using equation 1. Since this calculation requires the Poisson's ratio for urea and no value was available in the literature, a value for the Poisson's ratio had to be assumed. The Poisson's ratio was assumed to have a value between 0.2 and 0.4. Using this range, the true elastic modulus for granular urea was estimated to be between 4.81 x 10^7 – 9.27 x 10^7 Pa. This is consistent with the values reported by Wang et al. (2013) for the shear modulus of urea, which when converted to the equivalent elastic modulus, was determined to be between 5.52 x 10^7 and 8.40 x 10^7 Pa.

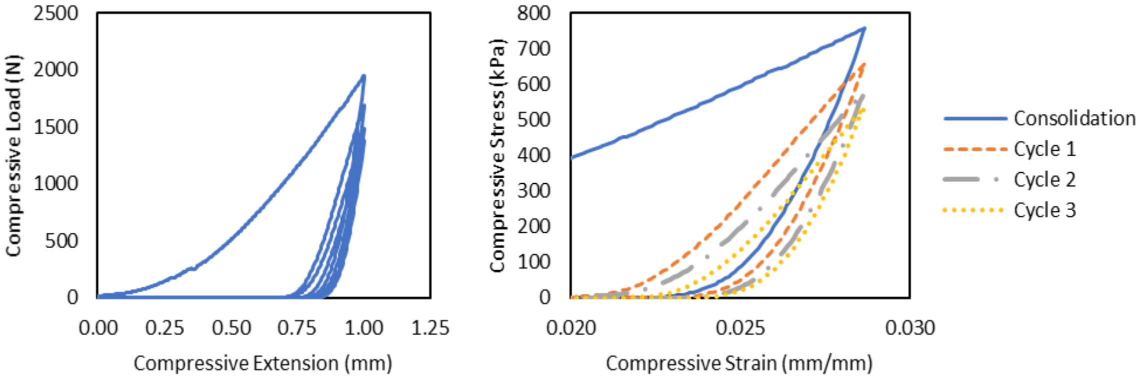

Figure 6: Sample load-displacement (left) and stress-strain (right) data for the confined compression experiment for a single urea sample.

Internal Friction and Cohesion Results for the direct shear test of granular urea are depicted in Figure 7 below. Raw results for shear force vs. shear displacement are shown in the left plot for a range of normal stresses. The peak shear force for each run was used to construct the yield locus for urea. Not all test data is shown.

The instantaneous yield locus is the relationship between maximum shear stress and normal stress for a range of normal stresses and was used to calculate friction and cohesion properties for the granular material. The yield locus for granular urea is presented in the right plot of Figure 7. Measured values for granular urea include an angle of internal friction of 37° and cohesion of 0.9 kPa. Dry granular urea in good condition was found to exhibit very low cohesion and can probably be assumed to be without cohesion for modelling purposes. No data was available for urea in the literature with which to compare results for the direct shear test.

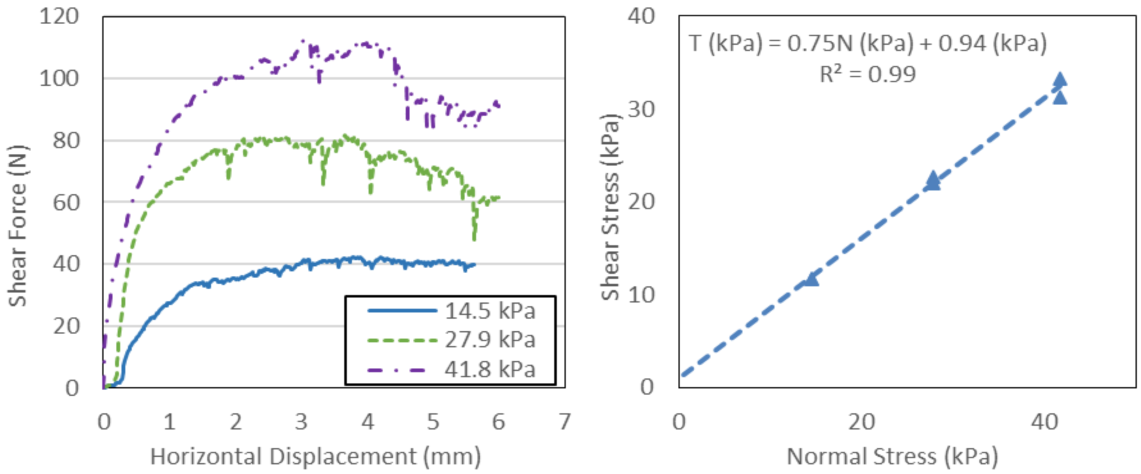

Figure 7: Shear force-displacement curves recorded during the test (left) and the instantaneous yield locus constructed from shear results (right) for granular urea.

Angle of Repose The angle of repose (AoR) was measured for granular urea using the slump test. Samples of 1000 g were placed in the custom slump test apparatus which was used to form the material pile. Grima and Wypych (2011) found that the ratio of column height to diameter did impact AoR results. In the present study the height to diameter ratio was 2.3. The static angle of repose for granular urea was measured to be 15.4°.

Table 2: Angle of repose measurements made using the slump test, N = 3.

#	Mass (g)	H_0 (mm)	H_f (mm)	H_0/D	AoR (°)
1	1000	204	40	2.3	15.3
2	1000	203	37	2.3	15.8
3	1000	203	41	2.3	15.1
Average					15.4

H0 is the original column height, Hf is the height of the pile, D is the diameter of the column, AoR is the angle of repose.

Particle Size The particle size distribution for granular urea was measured by sieving. The average particle diameter for Terico granular urea was measured to be $\overline{x} = 2.53$ mm with a standard deviation of s = 0.44 mm. This is slightly larger, but comparable to, values found in the literature of $\overline{x} = 2.16$ mm, s = 0.42 mm (Aphale et al. 2003).

Particle Size and Shape Particle size and shape distribution for granular urea were analyzed using particle image analysis. Figure 8 summarizes the image analysis results for urea and the plastic calibration material. The particle size distribution based on sieve analysis is also shown for reference. Examining first the calibration set, particles are seen to have a relatively uniform minor diameter (assuming an elliptical shape) of 3.58 mm which compares very closely with the 3.52 mm measured using calipers. Diameter measurement appears to be relatively accurate. Interestingly, the size distribution measured for urea was higher that that obtained from size analysis (which was corrected to be on a population basis rather that a mass basis). Image analysis measured the average particle diameter to be approximately 0.5 mm higher vs. the sieve analysis results. This could be due to the fines content being largely ignored in image analysis resulting in an upward bias, however, this has not been investigated in detail. Conversely, the particle diameter distribution's shape and range between minimum and maximum was comparable between imaging and sieve results.

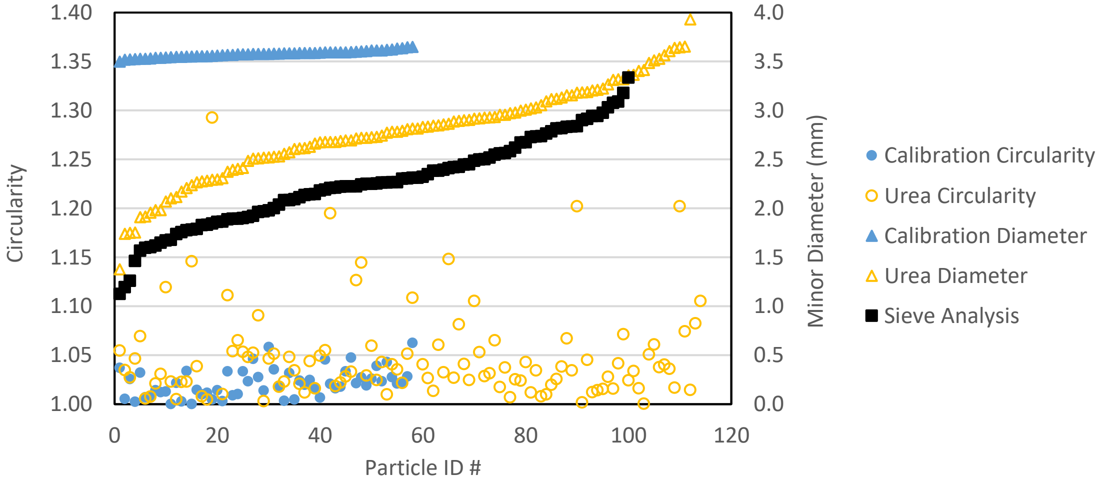

Figure 8: Particle size and shape distribution as determined by particle images analysis.

Circularity of the calibration particles forms a tight cluster between 1.00 and 1.05 reaffirming that the calibration particles are nearly spherical ($\overline{x} = 1.02$, s = 0.01). For urea, the distribution of circularity is less uniform ($\overline{x} = 1.05$, s = 0.05). While most particles were measured to have circularity below 1.05, indicating that the average urea particle is reasonably close to spherical, there were a significant number of particles distributed through higher circularity values, ranging as high as 1.29. Thus, while the average urea granule is semi-spherical, there was a sizeable portion of distorted particles which reduced the uniformity of urea particle shape.

Coefficient of Restitution The coefficient of restitution for urea particles colliding with several surface materials was measured using the particle drop test. Materials included steel, concrete, acrylic, and urea. Values for each of the materials are listed in Table 3. The lowest value was measured for urea-urea contact at 0.45. The highest measured value was for urea-acrylic contact at 0.61. Values from the literature reported by Albadarin et al. (2017) range from 0.07 (for PVC) to 0.34 (for stainless steel) which are considerably lower that the values measured in the present study.

Table 3: Coefficient of restitution measurements made using the particle drop test.

Material	N	Rebound Height (cm)		Coefficient of Restitution	
		Mean	Std Dev	Mean	Std Dev
Steel	11	6.5	1.7	0.46	0.063
Concrete	14	8.7	2.7	0.53	0.089
Acrylic	14	11.6	3.2	0.61	0.093
Urea (puck)	26	6.2	2.1	0.45	0.080

CONCLUSION A series of laboratory tests were conducted to measure selected physical properties for granular urea (46-0-0). These tests include: the gas pycnometer which measured solid density (1298 kg/m^3), the confined compression experiment which measured the elastic modulus (7.04 x 10^7 Pa), the direct shear test which measured angle of internal friction (37°) and cohesion (0.9 kPa), the slump test for measuring static angle of repose (15.4°), a sieve particle size analysis ($\bar{x} = 2.53$ mm, s = 0.44 mm), particle imaging analysis to quantify particle shape (circularity $\bar{x} = 1.05$), and the particle drop test which measured coefficient of restitution (0.46-0.61). Many of the values measured are not currently available for urea in the literature and will make additions to the published data. These material properties will assist engineers in the modelling and design of storage, handling, and application equipment for granular fertilizers.

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