11/16/2016

Submitted to Advanced Powder Technology, revised version

Particle shape effects on particle size measurement for crushed waste glass

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Abstract

Recently, narrow particle size distributions, as measured by sieve analysis, of crushed waste glass were used as a replacement for Portland cement in concrete. Their chemical reactivity was successfully studied as a function of this measure of particle size. Differences between sieve analysis and laser diffraction measures of particle size prompted this current re-analysis. Extremely careful sieving was used to divide the crushed waste glass particles into 0 μm to 25 μm , 25 μm to 38 μm , and 63 μm to 75 μm sieve size ranges, but laser diffraction did not agree with these particle size cutoffs. We use these same materials to try and understand the discrepancies between particle size as measured by laser diffraction and sieve analysis by using X-ray computed tomography followed by spherical harmonic analysis to measure the three-dimensional particle shape and size, as well as the length (L), width (W), and thickness (T) of

each particle. We show how laser diffraction and X-ray CT results, along with sieve analyses, can be quantitatively related for these crushed waste glass particles in the approximate size ranges considered. In contrast to previous speculation, the particle width W does not have to correspond closely to the sieve opening – the correspondence depends on overall particle shape. In addition, we demonstrate how many particles are needed to analyze in order to achieve stable averages and distributions of the L/W , W/T , and L/T aspect ratios, which approximately define particle shape. These results have implications for how particle size is measured and interpreted in the cement and concrete and other industries.

Key words: laser diffraction; sieves; particle size; particle shape; crushed waste glass; X-ray computed tomography; spherical harmonics

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Introduction

A recent paper [1] looked at the effect of crushed glass particles (cullet) [2] on cement hydration when used as a replacement for Portland cement. Other applications of this same kind of material include fillers in composite materials [3], and in asphaltic concrete and granular base [4].

Both references [1] and [3] gave rise to a question about the meaning of particle size measurement for these materials. In [1], extremely careful sieving, including the washing away of smaller particles that tended to cling to the larger particles, was used to divide the particles

into 0 μm to 25 μm , 25 μm to 38 μm , and 63 μm to 75 μm size ranges. Laser diffraction particle size measurements were made on each size range. Laser diffraction particle size results for each size range overlapped these sieve ranges but also gave particle sizes well below and well above these size ranges. In [3], the crushed glass material was sieved between 53 μm and 63 μm , without the washing away of small particles. Laser diffraction gave particle size results between about 0.3 μm and 120 μm . Even if all the smaller particles listed by laser diffraction were due to smaller particles that had been stuck to the larger particles, this would not explain the particle diameters found that were larger than the sieve range.

In order to address this particle size question, we re-characterized some of the material from [1]. The crushed glass particles that passed the 25 μm sieve were no longer available for this study, but we have been able to use the material from the 25 μm to 38 μm and 63 μm to 75 μm sieve size ranges. Missing this lower size range is not a problem, for the following reasons. In other studies, using mineral particles that were crushed in three different ways (ball mill [5], cone crusher [6], and VSI crushing [7,8]), it was shown that particle shape statistics varied very little with particle size, as characterized by several different particle shape parameters, over a wide range of sizes, up to three orders of magnitude [6]. In this present study, we found that the shape statistics (to be defined later in the paper) for the particles that passed a 75 μm sieve and were retained on a 63 μm sieve had essentially the same shape statistics as did the material that passed a 38 μm sieve and were retained on a 25 μm sieve. In light of the current and previous findings, we present below mainly the data for the 25 μm to 38 μm sieve data, with some mention of the larger particle data, and are confident that the results would be the same for particles that passed through the 25 μm sieve.

We note that, in general, particle size measurements all vary in how the particle size is sensed and interpreted. In sieves, the size of the mesh determines what particles can pass through the mesh openings. Meshes can be made up of wires overlaid in a square pattern or can be made up of square or circular holes in a continuous sheet. The sieve only measures one dimension of a particle, the one that fits through the sieve mesh. In the laser diffraction method, the diffraction pattern from a cloud of particles is measured and fit to the diffraction pattern arising from a diameter range of assumed spherical particles. In X-ray CT, the particles are suspended in epoxy or some other medium, and the actual 3-D shape of the particle is imaged and analyzed. There is no shape assumption, but the technique takes more time and typically analyzes fewer particles out of a population than do sieves or laser diffraction instruments.

In previous work [9], we compared the results of sieve analysis, laser diffraction, and X-ray computed tomography (CT) on fine mineral powders. In this paper, we build on these results and go beyond them to do further comparative analysis among these three techniques. We also use these results to determine the minimum number of particles that need to be analyzed in order to determine accurate average shape determinations, assuming that the particles were randomly chosen from the greater particle population. Obviously, for general use of the X-ray CT technique for determining average particle shape, the fewer particles that need to be analyzed the better.

Materials and Techniques

Glass powder with a narrow particle size distribution, as measured by sieve analysis, was prepared in [1]. Six steps were followed: washing, crushing, milling, dry sieving, wet sieving, and sedimentation. The glass, collected as waste glass, was washed and dried before crushing to remove any residues. After crushing to particles that passed through a 1.18 mm sieve (ASTM no. 16), the glass particles were further processed in a laboratory ball mill. The next step was wet and dry sieving, which was judged to be the most important step for providing glass powder in different sieve size ranges. Dry sieving was performed only for the larger particle size class, 63 μm to 75 μm , because of sieve clogging for the smaller particle sizes. After dry sieving the glass was wet-sieved using isopropanol alcohol through the 63 μm and 25 μm sieves. The final step, in order to try to remove small particles that might be clinging to the larger particles and which might have survived the wet sieving step, was sedimentation based on Stokes' law. 30 g to 50 g of wet-sieved powder was poured into 200 mL of isopropanol alcohol and completely stirred so that the glass particles were suspended. After a few seconds, a visible layer of larger particles would settle to the container bottom. The alcohol containing the finer particles, still in suspension, was removed, along with the finer particles, with a pipette. This sedimentation step was repeated several times. Scanning electron microscopy of some of the particles used did not show any visible fine particles adhering to the particles in the 25 to 38 and 63 to 75 sieve size ranges, which, however, are usually seen in this kind of material [e.g., see Figure 5 in Ref. 3]. Therefore, the sedimentation step was judged to be effective in removing these finer particles stuck to larger particles.

Combining X-ray μ CT with spherical harmonic (SH) analysis for particle size and shape measurements was introduced in [7, 9-15] and has been applied to similar crushed aggregate materials in [6,7]. Use of spherical harmonic analysis requires that the particles are star-shaped. A very large class of non-convex particles, which contains most particles used in industrial processes and particles dug up from the ground, is the class of star-shaped particles. An object is star-shaped if there is a fixed point, in its interior, for which a straight line drawn from that point to any other point inside the object lies entirely within the object. The star-shaped requirement will exclude from consideration particles with overhangs on their surface, which are removed in most natural and industrial processes, including the crushing and grinding process used for the waste glass cullet studied in this paper. Particles that are star-shaped with respect to their outer surface but containing internal porosity are not star-shaped, but these internal pores are easily removed in the data analysis so that just the external shape of the particle is studied.

Sample preparation for the present X-ray CT analysis involved casting samples of the glass cullet in epoxy at about 5 % volume concentration and allowing hardening while minimizing segregation. This process takes about one hour. After hardening, the samples were scanned using a Bruker Skyscan 1172 X-ray CT instrument¹. Complete three-dimensional renderings of the digitized particle size and shape were obtained within the limitation of the voxel size used and gray-scale threshold chosen. Scan times were about 6 h per sample. The gray-scale threshold was usually the same for all the images within the scan of one sample but could vary

¹ Certain commercial equipment and/or materials are identified in this report in order to adequately specify the experimental procedure. In no case does such identification imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the equipment and/or materials used are necessarily the best available for the purpose.

randomly from sample to sample. Particles with volumes above a cutoff value of 1000 voxels were used for the analysis. This lower limit is employed since for any smaller volumes, reliable shape information cannot be obtained, since the particles' outline is not sufficiently resolved [7,11]. The image size, pixel/voxel size, smallest analyzed particle sizes, and number of analyzed particles are listed in Table 1. "Image size" means the size of one cross-sectional slice – all the slices are stacked up to give a three-dimensional data structure from which the particles are computationally extracted. The parameter VESD (volume equivalent spherical diameter) for a given particle is equal to the diameter of a sphere with the same volume, so it is a particle "size" measure.

Table 1: X-ray CT particle analysis parameters for crushed glass particles

The many X-ray CT image slices in each scan were stacked together and the 3D image was segmented. Determining the appropriate gray-scale threshold to use usually took about 30 min. Since the smallest detail was a voxel, and in the segmenting process the interface between particle and matrix was uncertain to within a voxel, it is probable that the volume of the particles in the image were a little smaller or a little larger than reality, on the order of a voxel length times the surface area, due to the choice of threshold used in the segmentation process. However, for larger particles whose volumes could be easily experimentally measured by Archimedes' principle, this technique did give an accurate value (1 % to 2 % uncertainty) of particle volume [12]. Estimating the uncertainty in any linear dimension to be about one voxel

may be somewhat high, since segmenting the surface of these irregular particles will have canceling errors, so that this one voxel uncertainty is probably an upper bound. To make the above ideas be more definite, consider a rectangular box particle, $A \times B \times C$ voxels in size. If A really equals $A \pm \Delta$, where Δ is one voxel length, then to first order in Δ the volume is $ABC \pm S\Delta/2$, where S = the surface area of the $A \times B \times C$ box. The same result is obtained for a sphere, or indeed any star-shaped particle. For a specific particle out of the 63 μm to 75 μm sieve range, the volume was $166\,000\ (\mu\text{m})^3$, the surface area was $20\,060\ (\mu\text{m})^2$, the VESD was 68 μm , and the voxel size was 1.71882 μm . Therefore, the maximum relative uncertainty in the volume was 10 %, which translates to about 3.2 % in the linear dimensions. For a specific particle taken out of the 25 μm to 38 μm sieve range, the VESD was 28.3 μm , the volume was $11\,900\ (\mu\text{m})^3$, the surface area was $3145\ (\mu\text{m})^2$, and the voxel size was 1.11218 μm , giving a maximum relative uncertainty in the volume about 15 %, which translates to about 4.7 % relative uncertainty in the linear dimensions.

Once the particles were distinguished from the background, they were computationally extracted and used to generate spherical harmonic (SH) coefficients that define the particle [10]. Using SH functions, one can compute any geometric quantity of the particle, which can be defined by integrals over the volume or over the surface or any other algorithm using points on the particle surface or within the particle volume, since the SH approach gives an analytical, differentiable mathematical form for the particle surface and volume [10,16,17]. The process of extracting particles and computing geometrical quantities took about 4 h of computer run time per 10 000 particles. There can be errors in the surface area due to “ringing” effects in the

actual SH coefficients themselves, akin to the Gibbs' phenomenon in 2-D Fourier series [16,17], but these errors for random particles are small, less than 5 % to 10 %.

To better define the particle size and to give an estimate of particle shape, the length (L), width (W), and thickness (T) can be defined (ASTM 4791 [18]). L is defined as the longest surface-surface distance on the particle, W is defined similarly, but it must be perpendicular to L , and T is the longest surface-surface distance that is perpendicular to both L and W [12]. Therefore, $T \leq W \leq L$. There are two independent aspect ratios, say L/T and L/W , which are then an estimate of the average aspect ratio of a particle, since L , W , and T define a rectangular box, of dimension $L \times W \times T$, that just encloses the particle. The uncertainty in L , W , and T were about the same as those taken for a given particle, as was discussed above, since the algorithm that computes L , W , and T has negligible error in it compared to the segmentation of the original X-ray CT images. Another shape parameter that was used but not as a measure of particle size is the curvature ratio, which is defined as twice the inverse of the integrated mean curvature (mean curvature integrated over the surface and normalized by the surface area [10,12]) divided by the VESD. This shape parameter is equal to unity for a sphere and is denoted as "curvature" later in the paper. Judging from the above and the computation algorithms, there is about 5 % to 10 % relative uncertainty in this parameter. Other shape parameters based on other particle quantities (e.g. elements of moment of inertia tensor, surface area) can easily be computed [10]. Note that the W dimension has been thought to most closely correspond to the particle size as measured by ASTM sieves [6,9,19]. This conjecture will be closely examined later in this paper.

Standard wire mesh with square opening sieves were used for the sieve analysis, in standard ASTM sizes. The sieves were not examined with a microscope to determine how much the sieve openings deviated from the nominal value, rather we relied on the ASTM standard analysis. From ASTM E-11, USA Standard Sieves Specification [20], Table 2 gives the permissible standard deviation in the sieve openings, along with the maximum sieve opening size allowed. That means that sieve openings can exist up to this maximum value and the sieve will still meet the standard. There are different levels of scrutiny for sieves, resulting in three classifications: compliance grade, inspection grade, and calibration grade. The vast majority of sieves in use are compliance grade, which have the fewest sieve openings inspected. The sieves used in this study were compliance grade, matching what is common practice.

Table 2: Sieve number, nominal opening size, permissible standard deviation in the openings, and maximum allowed sieve opening size for the four ASTM sieves used in this study

A LECOTRAC LTS-150 laser diffraction particle size analyzer was used to measure the glass particle size distribution, as was mentioned in the introduction. The glass particles' size distribution was measured by first dispersing the particles in ethanol with an ultrasonic wand for 60 seconds. The samples were then introduced into the analyzer. The particle size distribution was calculated assuming spherical particle shapes, which is the usual assumption in this method. Although the reproducibility of this method has been discussed [21], its inherent

accuracy for non-spherical particles has, to our knowledge, not been quantitatively discussed, since one can define many single values of particle “size” for a non-spherical particle [9,12].

Results: “Size” ranges for the two particle sets

The X-ray CT results, listed in Table 3, give the minimum and maximum values of VESD, L , W , and T for each sample class. Table 3 also lists the minimum and maximum laser diffraction particle “size” or diameter found in the previous experiments [1]. The laser sizes come from Fig. 1 below, which shows the experimentally measured particle size distribution from laser diffraction [1]. From Table 3, we can clearly see that both the minimum and maximum values for the laser diffraction measurements, and L , W , and T from the X-ray CT results, fall outside the nominal minimum and maximum bounds of the sieve openings. Note that the minimum and maximum laser sizes and the minimum and maximum values of L appear to match quite closely. This kind of agreement between L and laser diffraction sizes has been seen previously [9,22]. The maximum sieve opening in the #400 and #200 sieves according to ASTM and Table 2, 50 μm and 103 μm , agree fairly closely with the maximum values of VESD found for these sieves (see Table 3), 55 μm and 98 μm .

Figure 1: Experimental laser diffraction results for the two particle size ranges considered (25 μm to 38 μm , 63 μm to 75 μm).

Table 3: Various minimum and maximum dimensions for the two particle classes, as determined by laser diffraction and X-ray CT. The range of the sieve classes give the sieve-determined minimum and maximum dimensions. All dimensions are in micrometers.

Results: Particle “size” distributions for the 25 μm to 38 μm sieve size range particles

In this section, we compare the particle size distributions as measured by laser diffraction and various particle dimensions, used to characterize “size”, taken from the X-ray CT results. The measured “laser diffraction size” was used as an abscissa for all data, including the L , W , T , and VESD size distributions from the X-ray CT data. Therefore, the histograms generated from the X-ray CT data are directly comparable with the laser diffraction data, since they have the same histogram bin sizes. All ordinate values are mass fractions = the fraction of the mass that fell into a given bin. All the particle size distribution graphs given below are normalized so that the sum of the area of all bins used is unity.

We plot the X-ray CT data, which directly measured the volume of the particles, against VESD, L , W , and T , to see which size measure best compares to the laser diffraction size measure. Note that nominally, according to how the sieves measure particle size, all the particles should have “sizes” between 25 μm and 38 μm . In Figure 2, the laser diffraction data and the X-ray CT data as a function of VESD are plotted on the same graph. The laser diffraction histogram extends

significantly to either side of the 25 μm and 38 μm marks, while the X-ray CT data overlaps these boundaries by a much smaller amount. In this case, the sieve particle size and the laser diffraction particle size are clearly different, while the VESD of each particle is closer to the sieve particle size. Since the size range of the VESD data is narrower than the laser diffraction data, and the histograms are normalized, then the VESD main peak must be much taller than the laser diffraction peak.

Figure 2: The particle size distribution of the 25 μm to 38 μm sieve particles, as determined by laser diffraction and by X-ray CT, which uses the VESD parameter as a measure of particle size.

In Figure 3, we now plot the X-ray CT size distribution using the W parameter. This parameter is thought to match up most directly with sieve size [9,19]. Comparing Figs. 2 and 3, we see that using W and VESD as measures of particle size gives similar results, although the width of the histogram is slightly larger for Fig. 3 compared to Fig. 2. This is confirmed by comparing the maximum and minimum sizes for VESD and W listed in Table 3. VESD is definitely some kind of mean particle size, and since W is between L and T , perhaps it also can be considered to be a similar mean size.

Figure 3: The particle size distribution of the 25 μm to 38 μm sieve particles, as determined by laser diffraction and by X-ray CT, which uses the W parameter as a measure of particle size.

Figure 4 shows the laser diffraction particle size distribution and the X-ray CT results using the thickness T as a particle size measure. Now the X-ray CT results are clearly skewed to smaller

Figure 4: The particle size distribution of the 25 μm to 38 μm sieve particles, as determined by laser diffraction and by X-ray CT, which uses the T parameter as a measure of particle size.

sizes, since by definition, $T \leq W$. There are values of T well below the 25 μm sieve limit, although the upper limit on T is close to the 38 μm upper sieve limit.

Finally, Figure 5 shows the same laser diffraction data but now the X-ray CT data used the length L as the particle size measure. It is clear that L overestimates the laser diffraction particle size, yet it does capture larger particle “sizes” up to 120 μm , as does the laser diffraction data.

Figure 5: The particle size distribution of the 25 μm to 38 μm sieve particles, as determined by laser diffraction and by X-ray CT, which uses the L parameter as a measure of particle size.

Judging by the results of Figures 2-5, the laser diffraction particle size histogram could perhaps be accurately expressed as a weighted average of the L , W , and T X-ray CT results, since the T data captured the particle “sizes” below 25 μm , while the L data was necessary to capture the “sizes” greater than 60 μm . Since we know the mass fractions in each bin for the L , W , and T data and the laser diffraction data, we can perform a simple least squares fitting procedure.

Let f_i = the mass fraction in the i th bin for the laser diffraction data, and L_i , W_i , and T_i be the mass fraction in the i th bin using the X-ray CT data. For a total of M bins, the sum of each separate mass fraction variable over all M bins is normalized to unity. To choose the weights for the L , W , and T mass fraction histogram data, we used a linear regression process, where we simply minimize the quadratic quantity A , where α , β , and γ are unknown multipliers:

$$A = \sum_{i=1}^M (f_i - \alpha L_i - \beta W_i - \gamma T_i)^2 \quad (1)$$

Eq. (1) expresses the fact that we want to choose the three weights that will most closely approximate the laser diffraction data in a least-squares manner. If we take three partial derivatives of A with respect to α , β , and γ , and set these expressions equal to zero, we get three linear equations in the three unknown weights α , β , and γ . Using the notation

$$HK \equiv \frac{1}{M} \sum_{i=1}^M h_i k_i \quad (2)$$

which is the average, over the total M bins, of any two of the mass fractions multiplied together, the three equations become:

$$\begin{aligned} FL &= LL \alpha + WL \beta + TL \gamma \\ FW &= WL \alpha + WW \beta + TW \gamma \end{aligned} \quad (3)$$

$$FT = TL \alpha + TW \beta + TT \gamma$$

This set of equations listed in eq. (3) can be solved for α , β , and γ . Note that the solution does not guarantee that $\alpha + \beta + \gamma = 1$, though it should be close. Therefore, the results of this calculation were normalized by dividing each parameter by the sum of all three parameters, so that the sum of the weights were unity, forcing the weight under the combined curve to also remains at unity. When simultaneously solving the three equations of eq. (3), the algebra is simplest if we write the solution for γ explicitly, in terms of FL , LL , WL , TL , FW , WW , TW , FT , and TT , which are known constants, determined from the measured mass distributions, and then solve for β in terms of γ and finally solve for α in terms of both β and γ .

$$\gamma = \frac{[(FT LL - TL FL)(WW LL - WL WL) + (TL WL - TW LL)(FW LL - FL WL)]}{[(TW LL - TL WL)(WL TL - TW LL) + (TT LL - TL TL)(WW LL - WL WL)]}$$

$$\beta = \frac{FW LL - FL WL + (WL TL - TW LL)\gamma}{WW LL - WL WL} \quad (4)$$

$$\alpha = \frac{FL - WL \beta - TL \gamma}{LL}$$

Figure 6 shows a comparison of the particle size distribution fit from using a weighted average size distribution and the measured laser particle size distribution. For the 25 μm to 38 μm sieve data, it was found that the values of were $\alpha = 0.419$, $\beta = 0.445$, and $\gamma = 0.111$, with their sum equal to 0.975, within 3 % of unity. After normalization, the final values of the parameters were $\alpha = 0.428$, $\beta = 0.452$, and $\gamma = 0.120$, with a sum of unity.

Figure 6: The particle size distribution of the 25 μm to 38 μm sieve particles, as determined by laser diffraction and by a weighted sum of the L , W , and T parameters computed from the X-ray CT data.

Qualitatively, the laser diffraction data is about an equal mix between the L and W data, with a small amount of the T data mixed in to match the low size part of the laser diffraction data.

Since the laser sees all aspects of the particles, sometimes it sees the small end of a particle and sometimes the large end, so that the laser diffraction data is spread out from approximately the smallest value of T to the largest value of L , or at least those with mass fraction enough to show up on the laser diffraction particle size distribution graph. This same phenomenon has been seen in computations of the exact light diffraction results for particles with equal volume but ranging from spheres to 20:1 prolate and oblate ellipsoids of revolution [22]. The width of the computed “particle size distribution” roughly ranged from 0.05 ($1/20$) to 20.

Results: Particle shape distributions for the 25 μm to 38 μm sieve size range particles

Now we turn to examining the actual particle shapes in this size range, as determined from the X-ray CT data. For measured values of L , W , and T , there are three possible aspect ratios, L/W ,

L/T , and W/T . We show all three in Figures 7-9, but emphasize that only two are mathematically independent.

Figure 7: The L/W aspect ratio distribution for the 25 μm to 38 μm sieve particles, as determined by the X-ray CT data and spherical harmonic analysis.

Figure 8: The W/T aspect ratio distribution for the 25 μm to 38 μm sieve particles, as determined by the X-ray CT data and spherical harmonic analysis.

Figure 9: The L/T aspect ratio distribution for the 25 μm to 38 μm sieve particles, as determined by the X-ray CT data and spherical harmonic analysis.

In each of Figures 7-9, there is a single peak, which rises steeply from the sphere aspect ratio of 1, then has a broad tail towards higher values of the aspect ratio. There are also very small mass fractions at higher aspect ratio values beyond the abscissa limit.

Table 4: Maximum values of the aspect ratios for the two sieve ranges of particles studied.

The maximum values of the aspect ratios for both sieve ranges are given in Table 4. Note that the two sieve ranges have similar values. Figure 10a shows a particle with $L/W = 5.5$, $W/T = 1.4$, and $L/T = 7.6$, so its volume would not contribute to Fig. 9. If L is much larger than W , and W is similar to T , then the particle is needle-like. Figure 10b shows a very flat particle with $L/W = 1.2$ and $L/T = 4.62$. So thin needle-like shards and flat, disc-like shards, are both found in the glass particles. The digitally rough area near the center of the particles in Fig. 10a and Fig. 10b are an artifact of the visualization process. Figure 11 shows an SEM image of the 25 μm to 38 μm , and 63 μm to 75 μm , sieve size ranges. Particles can be identified in the images that correspond in shape to the particles shown in Fig. 10.

Figure 10: a) $L/W = 5.5$, $W/T = 1.4$, $L/T = 7.6$; b) $L/W = 1.2$, $L/T = 4.6$ – front view; c) side view of b) particle.

Figure 11: SEM images of crushed glass particles from a) the 25 μm to 38 μm sieve size range, and b) the 63 μm to 75 μm sieve size range.

The averages of the various aspect ratios for the 20 μm to 38 μm sieve data are: $\langle L/W \rangle = 1.467$, $\langle W/T \rangle = 1.679$, and $\langle L/T \rangle = 2.433$. Mathematically, $L/T = (L/W) (W/T)$, and it can be seen that the averages also approximately follow this relation: $2.433 \approx 1.467 \cdot 1.679 = 2.463$. These are larger values than those seen previously for various crushed rock and many Portland cements [5-8,9,12,15] and are probably due to the brittleness of glass with respect to most naturally-occurring minerals.

The number of particles needed to obtain a stable average for these aspect ratios was also examined. These are volume averages, so we computed them over different numbers of particles. The results are listed in Table 5 below. The first row of the table, for 112 169 particles, repeats the values obtained for all the particles analyzed. Even when the number of particles fell below 100, the average only deviated by about 2 % from the average over all 112 169 particles for the aspect ratios but less than 1 % for the curvature ratio. The standard deviation of these ratios (not shown), which is just a measure of the width of the distribution of values, stayed the same within 10 % for all the rows in Table 5. However, one needs a larger number of particles to generate a statistically valid distribution, which involves higher moments of the distribution. We generated the L/T distribution for the same number of particles as is listed in Table 5, using the same number of bins and bin size, and found that the L/T distribution was very similar down to 560 particles, and were estimated to be significantly different by at or somewhat greater than 224 particles.

Table 5: The average values of the three aspect ratios as a function of how many particles were used to compute the volume averages.

Figure 12 shows the 112169 and 224 particle L/T distributions plotted on the same graph, with the same axis limits (which are different from Figure 9). The 224 particle distribution is not all that much different from the full particle case, however some significant differences began to appear. Therefore, it is estimated that there should be about 300 particles or more to capture a

statistically valid aspect ratio distribution. The other two aspect ratios, L/W and W/T , behaved similarly.

Figure 12: The L/T aspect ratio distribution for the 25 μm to 38 μm sieve particles, as determined by the X-ray CT data and spherical harmonic analysis, computed for 112 169 and 224 particles.

Results: 63 μm to 75 μm sieve data

Forming the particle size distribution using L , W , or T as the particle size measure and comparing the results to the laser diffraction data gave similar findings as the smaller particle data, so these graphs are not presented here. The averages of the various aspect ratios for the 63 μm to 75 μm sieve data were: $\langle L/W \rangle = 1.432$, $\langle W/T \rangle = 1.725$, and $\langle L/T \rangle = 2.441$, which are within 1 % or 2 % of the values found for the 25 μm to 38 μm sieve data, indicating that the average shape of particles in these two size ranges was essentially the same. The number of particles necessary to get a stable average for these aspect ratios were similar to that of the smaller particles. The number of particles needed for a statistically valid aspect ratio distribution was not investigated for the 63 μm to 75 μm sieve data, but there is no reason to expect results differ from those of the 25 μm to 38 μm sieve particles.

Figure 13 shows the weighted sum of the X-ray CT data, using the analysis presented earlier, compared to the laser diffraction particle size distribution data.

Figure 13: The particle size distribution of the 63 μm to 75 μm sieve particles, as determined by laser diffraction and by a weighted sum of the L , W , and T computed from the X-ray CT data.

It was found that $\alpha = 0.203$, $\beta = 0.444$, and $\gamma = 0.353$ ($\alpha + \beta + \gamma = 1$), which is a bit different from the smaller particle size range. This is probably due to the fact that, even though all the L , W , and T ratios had similar average values for the two different particle size ranges, the distributions were a bit different (compare Figs. 6 and 12) so that the L , W , and T distribution weights would also differ somewhat. Due to the lack of data on other particle size ranges, one cannot infer if there is any systematic dependence of these weights on particle size for this crushed glass material. Before normalization, the sum of these three parameters was 1.02 ($\alpha = 0.207$, $\beta = 0.453$, and $\gamma = 0.360$), within 2 % of unity. Comparing Figures 6 and 13, the fit in Figure 13 to the laser diffraction results was not quite as good as the fit in Figure 6.

Discussion

We now return to the question of how the width, W , measured by X-ray CT, compares to the sieve openings. The width W is considered to be the “intermediate” dimension of a particle.

The idea of comparing W to a square sieve opening, d , is based on the following simplistic argument. Since L , W , and T are mutually orthogonal, imagine a particle oriented so that L is along the z -direction and the sieve, along with W and T , are in the $z = 0$, x - y plane. Take a 2-D projection of the particle at the $z=0$ plane – if this projected image of the particle fits through the sieve, the whole particle will fit through, thus making W comparable to the sieve opening d . Since W and T are both defined as to be perpendicular to L , the projection of the particle in the x - y , $z=0$ plane must have the same values of W and T . A few simple examples will show that the value of W can actually be larger than d and the particle will still pass through the sieve.

In Figure 14a, a square sieve opening of side length d is shown, oriented diagonally, while the $z = 0$ projection of the particle in question is considered to be an ellipse. The heavy black horizontal line shows the sieve opening dimension across the diagonal of the actual sieve. It is clear that the width W of the particle, equal to the major axis length, is horizontal and is larger than d , but the particle will still fit through the sieve opening. The thickness, T , of the particle is equal to the minor axis length and is vertical on the page in Fig. 14a.

Figure 14: a) $d \times d$ square sieve opening, particle projection in $z=0$ plane is ellipse, oriented as shown; b) $d \times d$ square sieve opening, particle projection in $z=0$ plane is as shown. In both cases, the width W of the particle is horizontal and is greater than d , and the thickness T is vertical.

In Fig. 14a, we can easily predict what the maximum value of T must be so that, for a given value of W , with $d < W < 2^{1/2} d$, the particle can just fit through the sieve opening. This problem has been worked out in [23] and can be easily found in [24]. For a given value of W , the relation

$$(W)^2 + (T)^2 = 4d^2 \quad (5)$$

must hold, which defines the maximum value of T that can be used with a given value of W . In Fig. 14b, the width W is clearly horizontal and is larger than d , with an upper limit of $2^{1/2}d$, but T , the vertical height of the object, can clearly be as large as W . These two simple examples show that particles with values of $W > d$ can pass through a square sieve opening that is d wide, if the particle has W/T significantly greater than unity. There are also good analyses of this problem in the stream geology literature [25-28].

We can now use this information for Fig. 14a, approximating the particles as ellipsoids, along with the measured shape statistics of the particles and the sieve tolerances (Table 2), to approximately explain the minimum and maximum values listed in Table 3 for the smaller sieve range, 25 μm to 38 μm . Note that the following discussion only holds for square sieve openings – the case of circular opening sieves would be entirely different mathematically. The maximum and minimum lengths L found cannot be directly related to the sieve openings, since a particle could pass through the sieve with the L direction normal to the sieve openings. However, the maximum and minimum values of L found agreed well with the maximum and minimum laser diffraction sizes found, since it was known that these should be in reasonable agreement [9,22].

Let $W = \alpha d$, where $1 < \alpha < 2^{1/2}$. Then using eq. (5), it is simple to show (for an ellipsoid) that

$$W/T = (2/\alpha^2 - 1)^{-1/2} \quad (6)$$

where T is the maximum value possible for this value of W so that the ellipse (projection of an ellipsoid) just passes through the square sieve opening. Now, consulting Table 2, the maximum sieve opening allowed in an ASTM 38 μm sieve is 50 μm . If we insert the maximum value of W/T found for the lower sieve range, 4.7 (see Table 4), in eq. (6) and solve for α , the result is $\alpha = 1.38$. Using $d = 50 \mu\text{m}$, then the maximum value of W that could go through this sieve is $1.38 d = 69 \mu\text{m}$, which is very close to the maximum value of W found listed in Table 3, 71 μm .

Now consider the minimum value of W in Table 3, 10 μm , listed for the smaller sieve range. The geometrical arguments implied by Fig. 14 cannot be used to explain a minimum value of W that did not pass through the 25 μm sieve when $W < 25 \mu\text{m}$. However, there are two ways that a particle with W less than the sieve opening might not pass through it. The first is that other particles blocked it, which must certainly happen when one is sieving thousands of particles at once. The second is that, since these particles are not spherical and $L > W$, a particle could have fallen sideways and not passed through the sieve opening d since $L > d$. For a particle for $W = 10 \mu\text{m}$, the value of L would have had to be at least $2^{1/2} d$. If $L = 2^{1/2} d$, for $d = 25 \mu\text{m}$, then $L/W = 3.5$, which is well under the maximum value of L/W found, 5.7 (Table 4), for this set of particles. So this scenario could well have happened, thus giving an explanation for the minimum value of W found for the lower sieve class range. Note that we cannot expect the sieving process to be ergodic in the sense that all orientations of each particle are sampled against all possible sieve openings. There may be slightly larger, within the ASTM tolerances, sieve openings that would have fit a particle but that particle never encountered them. There could be orientations of a given particle that would fit through a sieve opening but the sieving energy was not high

enough for that orientation to be found or for blocking particles to be moved sufficiently out of the way.

Next we consider the maximum and minimum values of T found for the lower size class range. The maximum value of T found was 52 μm . Given that the maximum sieve opening might have been as large as 50 μm , and that there were particles with W/T close to 1, then such a particle could have fit through such a sieve opening. The minimum value of T found was 6 μm . Consider a particle that could just be stopped (in the right orientation) by a 25 μm sieve opening, $W = 25$ μm . If this particle had the maximum value of $W/T = 4.7$, that would imply that $T = 5.3$ μm . So the actual minimum value of T found, 6 μm , could have been found for a particle that was retained on the 25 μm sieve.

Similar arguments can be marshalled for the larger sieve class range, 63 μm to 75 μm , to help explain the maximum and minimum values of W and T found. The maximum value of W found in the X-ray CT analysis was 156 μm (see Table 3). Using eq. (6), the maximum value of $W/T = 4.2$ (see Table 4), and the largest possible sieve opening listed in Table 3, one can predict that the maximum value of W should be about 142 μm , which is in reasonable agreement considering the fact that the particles were not really ellipsoids. The minimum value of W found for this large sieve range was 17 μm (see Table 3). If a particle fell sidewise across the 63 μm sieve opening, and at a minimum, $L = 2^{1/2} d$, for $d = 63$ μm , then $L/W = 5.2$, which is well under the maximum value of L/W found, 6.2 (Table 4). The maximum value of T was 101 μm (Table 3), and the maximum allowable sieve opening for the 75 μm sieve was 103 μm , so that this particle, as long as W/T was about unity, could pass through. The minimum value of T was 11 μm . Suppose that W was at least 63 μm , just large enough to be stopped by the 63 μm sieve (in

some orientation). Using the maximum value of $W/T = 4.2$ (see Table 4), implies that the minimum value of T would be $63\text{ }\mu\text{m}/4.2 = 15\text{ }\mu\text{m}$, which is close to the value measured.

One last shape aspect of both sets of particles is their surface area, relative to that of an equivalent sphere. For each particle, the ratio is formed of the actual surface area, as measured by X-ray CT and spherical harmonic analysis, to the surface area of a sphere with diameter equal to the VESD value for that particle. For the smaller sieve range, the volume-weighted average was about 1.26, and for the larger sieve range the average ratio was about 1.29. These results would imply that estimating surface area by computing the total surface area based on a particle size distribution where size was some kind of equivalent spherical diameter would underestimate the actual surface area by around 30 %.

Summary and Conclusions

We have reanalyzed, using a combination of X-ray CT and spherical harmonic (SH) analysis, two sieve range classes, $25\text{ }\mu\text{m}$ to $38\text{ }\mu\text{m}$ and $63\text{ }\mu\text{m}$ to $75\text{ }\mu\text{m}$, of crushed waste glass particles whose particle size was studied in a previous paper only by sieve analysis and laser diffraction particle size measurement [1]. Using the X-ray CT and SH methods, we computed the length (L), width (W), and thickness (T) of each particle analyzed, about 110 000 for the smaller sieve range and about 11 000 for the larger sieve range. Using knowledge of the sieves, and the tolerances under which they were certified, we were able to explain the maximum and minimum values of W and T measured, compared to the nominal sieve openings. The maximum and minimum values of the length L agreed well with the maximum and minimum particle

“diameters” found by the previous laser diffraction work, as was expected from previous results in the literature. The shape distribution of the crushed glass cullet particles, in terms of ratios of these particle dimensions, have been analyzed. The number of particles necessary to get stable values of the average shape parameters and shape distributions were also analyzed. Since X-ray CT and spherical harmonic techniques were used to explain both sieve analysis and laser diffraction particle size measurements, it is clear that while sieve analysis and/or laser diffraction particle size distribution measurement can be used as quality control parameters, the actual particle dimensions, and the particle shapes, can only be measured accurately by a combination of X-ray CT and spherical harmonic analysis.

The following conclusions and implications can be drawn from this work:

- For a given sieve size range, the number of particles that need to be analyzed to get stable values of the average shape parameters and their distributions were about 50 and 300, respectively, for these very non-spherical particles. This assumed that the particles were always sampled randomly from the greater particle population, so that the sub-sample’s size range always reflects the population from which it is drawn.
- The upper and lower limits of the laser diffraction results, which were far outside the limits of the sieve ranges, were well-explained by the upper and lower limits of the length L parameter, which was in agreement with previous literature analyses that showed that the laser diffraction method coupled with the largest and smallest dimensions of the particles, giving a far wider “particle size distribution” than the particles appearance would imply.

- The particle size distribution, as measured by laser diffraction, could be reasonably well fit by a linear combination of the three sets of X-ray CT size data computed for L , W , and T . Simple equations were given to compute this linear combination, given that the X-ray CT data was available. A draft standard is in preparation for this measurement method.
- The width W parameter was in general different from the sieve openings, but the upper and lower value of W found for each sieve size range was correlated to that size range, as shown in Table 3. The upper and lower limits that were found for W and for the thickness T , for each sieve size range, could be explained using the aspect ratio statistics, the tolerances of the ASTM sieves, some simple geometrical ideas, and reasonable ideas about how the particles, in various orientations, encounter the range of sieve openings available for each sieve. This analysis was only for square opening sieves – circular sieve openings would require a different analysis.
- An implication for industry is that the measurement of the size distribution of irregular particle is highly influenced by what is meant by shape and the assumptions used in the measurement technique. For example, it may take more information than just a laser diffraction particle size distribution to really describe how particles behave. Sieve analysis and/or laser diffraction particle size distribution measurement can be used as quality control parameters, but the actual particle dimensions, and the particle shapes, can only be measured accurately by a combination of X-ray CT and spherical harmonic analysis. Translating sieve analysis and laser diffraction results to applications that directly input the actual particle sizes can be misleading and inaccurate. This paper implies that term “particle size distribution” for non-spherical particles, when

represented in terms of a single linear dimension, is more of an artificial index rather than an actual physical distribution.

- Finally, computing surface area from particle size measurements that treat the particles as equivalent spheres could be up to 30 % too small, at least for the crushed glass particles considered here. Particles that are more spherical will of course result in less of a surface area error.

Acknowledgements

This research was funded in part by the National Science Foundation grant CMMI-1032636.

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There is only one way that a spherical particle can fit through a square sieve opening. Since X-ray computed tomography gives the complete three dimensional description of random particles, it is worth thinking how non-spherical, random shape particles can fit through square sieve openings, as is shown above for two simple but non-spherical particle shapes. Specifically, the particle dimensions, length L, width W, and thickness T, which are measured in X-ray CT combined with spherical harmonic analysis can be used to compute which kinds of particles will fit through various square sieve openings. In addition, these measured particle dimensions can be used to quantitatively correlate laser diffraction size measurement with sieve analysis. These findings have important implications for industrial particle size measurement of random-shape particles.

Figure 1: Experimental laser diffraction results for the two particle size ranges considered (25 μm to 38 μm, 63 μm to 75 μm).

Figure 2: The particle size distribution of the 25 µm to 38 µm sieve particles, as determined by laser diffraction and by X-ray CT, which uses the VESD parameter as a measure of particle size.

Figure 3: The particle size distribution of the 25 µm to 38 µm sieve particles, as determined by laser diffraction and by X-ray CT, which uses the W parameter as a measure of particle size.

Figure 4: The particle size distribution of the 25 µm to 38 µm sieve particles, as determined by laser diffraction and by X-ray CT, which uses the T parameter as a measure of particle size.

Figure 5: The particle size distribution of the 25 µm to 38 µm sieve particles, as determined by laser diffraction and by X-ray CT, which uses the L parameter as a measure of particle size.

Figure 6: The particle size distribution of the 25 μm to 38 μm sieve particles, as determined by laser diffraction and by a weighted sum of the L , W , and T parameters computed from the X-ray CT data.

Figure 7: The L/W aspect ratio distribution for the 25 μm to 38 μm sieve particles, as determined by the X-ray CT data and spherical harmonic analysis.

Figure 8: The W/T aspect ratio distribution for the 25 μm to 38 μm sieve particles, as determined by the X-ray CT data and spherical harmonic analysis.

Figure 9: The L/T aspect ratio distribution for the 25 μm to 38 μm sieve particles, as determined by the X-ray CT data and spherical harmonic analysis.

Figure 10: a) $L/W = 5.5$, $W/T = 1.4$, $L/T = 7.6$; b) $L/W = 1.2$, $L/T = 4.6$ – front view; c) side view of b) particle.

Figure11: SEM images of crushed glass particles from a) the 25 µm to 38 µm sieve size range, and b) the 63 µm to 75 µm sieve size range. The scale bars are shown on both images.

Figure 12: The L/T aspect ratio distribution for the 25 μm to 38 μm sieve particles, as determined by the X-ray CT data and spherical harmonic analysis, computed for 112 169 and 224 particles.

Figure 13: The particle size distribution of the 63 μm to 75 μm sieve particles, as determined by laser diffraction and by a weighted sum of the L , W , and T computed from the X-ray CT data.

Figure 14: a) $d \times d$ square sieve opening, particle projection in $z=0$ plane is ellipse, oriented as shown; b) $d \times d$ square sieve opening, particle projection in $z=0$ plane is as shown. In both cases, the width W of the particle is horizontal and is greater than d , and the thickness T is vertical.

Table 1: X-ray CT particle analysis parameters for crushed glass particles

Sieve size range	Image size (pixels)	Voxel size (μm)	Smallest analyzed particle volume (μm^3)/VESD (μm)	No. analyzed particles
25 μm to 38 μm	4000 x 4000	1.11218	1375/14	112 169
63 μm to 75 μm	4000 x 4000	1.71182	5016/21	10 659

Table 2: Sieve number, nominal opening size, permissible standard deviation in the openings, and maximum allowed sieve opening size for the four ASTM sieves used in this study

Sieve number	Nominal opening size (μm)	Permissible standard deviation (μm)	Maximum allowed sieve opening (μm)
500	25	3	35
400	38	3	50
230	63	4	89
200	75	5	103

Table 3: Various minimum and maximum dimensions for the two particle classes, as determined by laser diffraction and X-ray CT. The range of the sieve classes give the sieve-determined minimum and maximum dimensions. All dimensions are in micrometers.

Sieve size range	Laser min	Laser max	T min	T max	W min	W max	L min	L max	VESD min	VESD max
25 μm to 38 μm	11	124	6.0	52	10	72	17	115	14	55
63 μm to 75 μm	26	249	11	101	17	156	25	257	21	98

Table 4: Maximum values of the aspect ratios for the two sieve ranges of particles studied.

Sieve range	L/W (max)	W/T (max)	L/T (max)
25 μm to 38 μm	5.7	4.7	7.8
63 μm to 75 μm	6.2	4.2	8.1

Table 5: The average values of the three aspect ratios as a function of how many particles were used to compute the volume averages, along with the percent deviation from the average shape parameter for N = 112 169.

# particles	$\langle L/T \rangle$	Δ (%)	$\langle W/T \rangle$	Δ (%)	$\langle L/W \rangle$	Δ (%)	Curvature ratio	Δ (%)
112169	2.433	-	1.679	-	1.467	-	0.888	-
56084	2.432	0.04	1.681	0.1	1.464	-0.2	0.888	0
22433	2.435	0.08	1.678	-0.06	1.469	0.1	0.888	0
11216	2.431	-0.08	1.680	0.06	1.464	-0.2	0.888	0
5608	2.442	-0.4	1.681	0.1	1.471	0.3	0.888	0
2243	2.448	0.6	1.700	1.3	1.457	-0.7	0.888	0
1121	2.443	0.4	1.701	1.3	1.455	-0.8	0.888	0
560	2.452	0.8	1.705	1.5	1.453	-1.0	0.888	0
224	2.445	0.5	1.709	1.8	1.442	-1.7	0.890	0.2
112	2.447	0.6	1.691	0.7	1.452	-1.0	0.889	0.1
56	2.436	0.1	1.681	0.1	1.459	-0.5	0.890	0.2
28	2.480	1.9	1.724	2.7	1.458	-0.6	0.893	0.6