Particle Size and Surface Area Effects on Explosibility Using a 20-L Chamber

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Abstract
The Mine Safety and Health Administration (MSHA) specification for rock dust used in underground coal mines, as defined by 30 CFR 75.2, requires 70% of the material to pass through a 200 mesh sieve (< 75 µm). However, in a collection of rock dusts, 47% were found to not meet the criteria. Upon further investigation, it was determined that some of the samples did meet the specification, but were inadequate to render pulverized Pittsburgh coal inert in the National Institute for Occupational Safety and Health (NIOSH) Office of Mine Safety and Health Research (OMSHR) 20-Liter chamber. This paper will examine the particle size distributions, specific surface areas (SSA), and the explosion suppression effectiveness of these rock dusts. It will also discuss related findings from other studies, including full-scale results from work performed at the Lake Lynn Experimental Mine. Further, a minimum SSA for effective rock dust will be suggested.

1.0 Introduction

Float coal dust, consisting of very fine aerosolized particles, presents a hazard that can contribute to a major underground coal mine explosion. In order to mitigate this risk, pulverized rock dust is required to be applied to the intake, return, and belt airways (entries). Federal safety regulations (30 CFR 75.402 and 30 CFR 75.403) require rock dust to be applied so that the total incombustible content of a mine dust sample is not less than 80 percent. 30 CFR 75.2 also defines rock dust and requires rock dust to be sized such that 100 percent passes through a 20 mesh (850µm) screen and 70 percent or more passes through a 200 mesh (75 µm) screen.

This current particle size specification is so broad that it may not ensure that all rock dust will inert at the 80% incombustible level when uniformly mixed with coal dust. Past work (Man and Harris 2014) suggests that rock dust particles in excess of 75 µm provide little inerting potential and, therefore, do not need to be included in the rock dust supply. A specification of 95% finer than 75 µm would ensure that the focus is on particles with the most inerting potential yet within grinding mill tolerances for rock dust manufacturers. Furthermore, members of the industrial minerals sector have indicated that such a particle size distribution (PSD) is attainable given current grinding technology. Given that the PSD of rock dust varies widely, another attribute such as specific surface area (SSA) should be considered to ensure that only the most effective dust particles are included.

2.0 Background

MSHA rock dusting regulations were initially based upon data generated within the Bruceton Experimental Mine (BEM) by the U.S. Bureau of Mines (BOM) which suggested that the largest-sized coal dust particle that participated in explosions was 850 µm (Rice et al. 1922). At that time, the authors stated that the following circumstances may prevent 20 mesh coal dust from propagating:

1. The 20 mesh dust will not mix readily and thoroughly with air due to the weight of the coarser particles,
2. The surface area of the coarse particles is less than that of the same weight of fine particles, resulting in less surface area for instantaneous oxidation, and
3. The number of the coarse particles is less than that of the same weight of fine particles making it probable that the distance between the particles will be greater and thus prevent propagation of the flame from particle to particle.

Since those early BOM tests, other laboratory and experimental mine testing methods were developed to determine which coal dust particle sizes contribute to explosion propagation and which rock dust particle sizes contribute to explosion suppression. Understanding of these relationships is critical to properly determining those characteristics of an effective rock dust for preventing coal dust explosion propagation.

One of the well-established American Society for Testing and Materials (ASTM) laboratory methods is the use of a 20-Liter (20-L) explosion chamber to test the explosibility of various coal dust and rock dust mixtures. Previous data from NIOSH 20-L chamber tests have shown that a coal dust (400 g/m^3 coal concentration) and rock dust mixture must contain at least 76% limestone rock dust to inert the pulverized Pittsburgh coal (PPC) dust which contains 80% minus 200 mesh particles (Cashdollar and Hertzberg 1989). This finding was verified at coal dust concentrations of 150–700 g/m^3. Dastidar et al. (2001) also tested PPC in a 20-L chamber and reported a slightly lower value of 74% rock dust to inert the PPC dust at a dispersed coal concentration of 500 g/m^3. In an earlier study, Dastidar et al. (1997) had published an inerting value of 77% limestone rock dust associated with a 300 g/m^3 PPC concentration. The differences were described by the authors as “due to the nature of flame propagation, which is probabilistic at limit conditions.” The latter observation reinforces the idea that multiple trials are needed to safely conclude that the mixture will remain non-explosive at all coal concentrations.

It is important to note that the 20-L chamber results indicate trends but cannot be directly scaled to full-scale results such as those obtained in another study performed at the Lake Lynn Experimental Mine (LLEM) (Sapko et al. 2000). The differences between the laboratory chamber results and the LLEM full-scale results include but are not limited to important differences between the dimensions and geometry of the mine and the laboratory chambers, differences in the ignition source (pyrotechnic ignitors in the 20-L chamber vs. an initiating methane-air explosion in the LLEM), and the manner in which the dust is introduced and dispersed. The chamber criterion for explosibility is based on the measured overpressure rise whereas the LLEM criterion is based on self-sustained flame propagation beyond the influence of the ignition source. Through previous research (Cashdollar 1996, NIOSH 2010), one can equate a 75% inerting rock dust concentration given by 20-L tests to an 80% incombustible content requirement for mine inerting (at least for Pittsburgh seam coal with a 6% ash content). The baselines in both the LLEM and 20-L chamber tests were established using PPC as the coal dust and a reference rock dust (acquired from the same rock dust manufacturer and having historically consistent PSDs).

A recent NIOSH study demonstrated that larger rock dust particles (> 75 µm) are much less effective than smaller particles at inerting coal dust as indicated by the large increase in the percentage of rock dust required to inert PPC in both 20-L chamber and 1-m^3 chamber tests (Man and Harris 2014). Results further indicated that rock dust particles between 250 and 850 µm (> 60 mesh) did not inert PPC in the 20-L chamber studies. The study also showed that when rock dust particles < 38 µm (< 400 mesh) were removed from the particle size distribution, inerting was not possible at even a 90% rock dust level. Past research showing the dependence of inerting effectiveness on rock dust PSD suggested the need to further quantify this relationship using constant volume explosibility studies in the NIOSH 20-L explosion chamber (Man and Harris 2014).

A previous NIOSH investigation of rock dust revealed significant concerns with the material used in mines based on the analysis of rock dust samples collected by the Mine Safety and Health Administration (MSHA) inspectors from U.S. coal mines in 2010. One concern was the frequency of rock dust material in mines not meeting the legal size criterion (70% by weight passing through a 200 mesh sieve). In a population of 393 rock dust samples from 278 underground coal mines, 47% of the rock dust samples failed to meet the minimum size criterion (NIOSH 2011). NIOSH tested these dusts within the 20-L chamber to verify the inadequacy of the rock dust that did not meet the definition. Most importantly, some of the rock dusts that did meet the current definition did not inert PPC in the 20-L chamber.

In light of the above findings and given the need for a more definitive characterization of rock dust that is effective for inerting a propagating coal dust explosion, NIOSH researchers undertook an investigation of the rock dust particle size effects on explosibility in a 20-L chamber. The PSDs of the rock dusts vary greatly with some having multiple peaks in the distribution and although sieving can be used to characterize the PSD of rock dusts, the most effective particles for inerting lie in the respirable size range and cannot be sieved. To better characterize such wide variations, multiple and varying sized sieves would be required and the finest size to be assessed would typically be 38 µm or possibly 20 µm (635 mesh sieve not widely available commercially). However, the respirable portion of rock dust is the most effective and cannot be assessed using sieves. Therefore, in lieu of characterizing rock dust solely on the percentage finer than 200 mesh, NIOSH investigated the use of a specific surface area (SSA) designation as means to assess inerting effectiveness. The SSA is a calculation of outer surface area based upon a spherical approximation given the particle size or width. In this paper, the term “explosibility” refers to the ability of an airborne dust cloud and/or gas mixture to explode in a confined laboratory chamber or propagate flame within an experimental mine after the dust cloud or gas mixture has been initiated by a sufficiently strong ignition source. All of the full-scale LLEM explosion tests referenced earlier utilized the same limestone rock dust which is referred to herein as the Reference rock dust. Rock dust samples collected by MSHA during a survey were tested within the 20-L chamber to demonstrate their inerting abilities. The standard PPC dust and Reference rock dust were used for both laboratory and experimental mine explosions.

3.0 Experimental

3.1 Particle Size Analyzers

For a full particle size distribution and SSA, NIOSH used a Beckman Coulter (B-C) LS 13320 laser diffraction particle size analyzer equipped with a Tornado Dry Powder air dispersion system. NIOSH researchers followed the analysis procedure recommended by the manufacturer (Beckman Coulter 2011). The laser diffraction data is analyzed by the instrument in terms of equivalent spherical scatterers using a Mie scattering algorithm. The volume fraction is determined for the various particles sizes, and a specific surface area in terms of area per unit volume (cm^2/ml) is determined. That area divided by the density of the particles then gives the specific surface area (SSA) in units of area per units of mass. The complex refractive index (RI) of 1.8 + 0.3*i* was used for the coal dust analysis and 1.68 + 0.0*i* was used for the limestone rock dusts, where *i* is the imaginary (absorptive) component. These were average RI values found in the B-C manual for carbon and calcium carbonate and were not determined by a separate analysis. Control samples of PPC and the Reference rock dust were tested every 30–50 samples to confirm proper B-C operation and to detect significant deviations from the typical measured average values and uncertainty in their SSAs. The B-C system was the system of choice to use for SSA determination. The system requires only a small sample for analysis, is easy to use, gives reproducible results, and is not subject to user variability. However, another option is the use of an air-jet sieve in conjunction with the Blaine Permeability apparatus (Blaine apparatus). The Blaine Apparatus is a simple, low-cost system that can be used as an alternative to obtain SSA results.

A comparison of SSA measurements using the B-C system and the Blaine apparatus for several rock dusts are shown in Figure 1. The Blaine air permeability of a packed bed is a standard test method based on the Kozeny-Carman equation for permeability of a packed bed of particles to determine the fineness of hydraulic cements (ASTM C 204-11; Perry and Green, 1984). Following ASTM C 204-11 procedures, the NIOSH manually-operated Blaine apparatus was calibrated using the National Institute of Standards and Technology (NIST) Standard Reference Material 114q (SRM) (NIST 2001; 2008). The effective SSAs of the samples were compared with a standard dust of known SSA (NIST SRM). Despite the small sample size, an R^2-value of 0.97 between the B-C laser diffraction system (LDS) and the Blaine apparatus (Figure 1) suggests the feasibility of using the Blaine apparatus and method as an alternative to an LDS for determining minimum SSAs of rock dusts.

3.2 Dust samples

<u>3.2.1 Pulverized Pittsburgh Coal</u>

The pulverized Pittsburgh coal (PPC) dusts used for this study were produced at NIOSH OMSHR. The coal was mined on-site from the Safety Research Coal Mine (SRCM), then ground and pulverized on-site to produce the pulverized Pittsburgh coal dust. The same SRCM coal seam was mined and processed in a similar manner for the various sized Pittsburgh coal dusts used during the LLEM explosion tests (NIOSH 2010). The cumulative and differential PSDs of the PPC as measured with the B-C are shown in Figure 2. The B-C mass-mean particle size of PPC is 61.9 µm with a median particle size of 54.6 µm. PPC has an average calculated SSA of 240 m^2/kg. Some common size fraction values determined by the B-C instrument and a commercial air-jet sieve apparatus are listed in Table 1.

The optical method of particle size determination understates the percentage < 75 µm compared to results obtained from sieving methods. The B-C measurement is approximately 10% below that of the direct air sieve measurement, or 60% < 200 mesh. This difference is due to measurement of oblong particles and the inherent differences within the methods. With an oblong particle, the B-C measures the widest dimension of the particle whereas the air-jet sieve agitates the particles until the narrowest part of the particle passes through the sieve. The percentage difference between the analyzers will be different for each sieve/mesh. Despite such differences, the air-jet results are seen to be in line with the B-C analysis.

3.2.2 Rock Dust Samples

The samples referred to as MSHA survey samples are rock dust samples collected by MSHA from 278 underground coal mines as discussed in the 2011 NIOSH Hazard ID (NIOSH 2011). These included a handful of samples that MSHA had collected but which had arrived after the Hazard ID was published. All samples were selected from a population of samples collected by MSHA inspectors during inspections. These samples were sent to the MSHA National Air and Dust Laboratory at Mt. Hope, WV, for cataloging and then sent to NIOSH for analysis. NIOSH performed a size analysis on these samples. The samples were gathered from all MSHA bituminous coal districts and are believed to be representative of a random cross-sectional snapshot of the rock dust available in the operating underground coal mines. The amount of rock dust sample collected varied substantially between mines and inspectors which, for some samples, limited the number of analyses and 20-L chamber testing that could be conducted.

3.3 Explosion test chamber

The NIOSH 20-L explosion chamber was used in this study. This chamber has been extensively used as a tool to evaluate the explosibility properties of various dusts prior to and concurrent with extensive LLEM full-scale explosion propagation experiments (ASTM E1515-07 2007; Cashdollar 1996; 2000; Cashdollar and Hertzberg 1989; Chawla et al. 1996; Dastidar et al. 2001; Sapko et al. 2000). Research has shown an ~5% difference in the rock dust content to inert PPC in the 20-L chamber compared to that required to prevent flame propagation in the LLEM using the same rock dust size distribution (NIOSH 2010)—i.e., ~73% rock dust in the 20-L chamber compared to ~78% in the LLEM.

Detailed descriptions of the 20-L chamber have been previously published (Cashdollar 1996; 2000; Going et al. 2000). For the 20-L chamber experiments in this paper, 5,000 J electrically activated pyrotechnic ignitors were used as the ignition source for testing the explosibilities of mixed dusts. A pressure rise ≥ 1 bar (pressure ratio ≥ 2) was used as the criterion for determining the occurrence of an explosion during a test. A pressure ratio designation can account for the variations in atmospheric pressure. This determination is in accordance with the ASTM test for measuring the explosibility of dust clouds (ASTM 2010). A series of three or more tests were performed to confirm a non-explosion at each coal dust concentration if sufficient quantities of a particular rock dust sample were available.

Inerting tests conducted with the MSHA survey samples were limited to a PPC concentration of 400 g/m^3 due to limited quantities of the collected rock dust samples. The 400 g/m^3 PPC concentration was chosen because this is typically the most reactive concentration. The maximum pressure and the rate of rise level off as the oxygen in the chamber is consumed (Cashdollar 1996). For PPC, this leveling or limit occurs at approximately 300 g/m^3 coal dust concentration with a corresponding maximum pressure of 6.6 bar. This maximum pressure remains at approximately 6.6 bar as the coal dust concentration increases from 300 to 800 g/m^3. Therefore, considering the limited quantities of rock dust samples available, the coal dust concentration was held constant at 400 g/m^3 to determine if the rock dust was effective in inerting the dust mixture with a concentration of 75% rock dust.

These tests were conducted at 75% rock dust for comparison with the full-scale LLEM explosion test results. If the 75% rock dust mixture was explosible, no other inerting tests were conducted. If the 75% rock dust mixture was not explosible, additional tests were conducted at the same coal concentration until there was insufficient rock dust remaining to continue testing.

4.0 Results and Discussion

4.1 Particle Size Analysis

All experimental laboratory inerting results based on calculated SSAs presented were determined using NIOSH's Beckman Coulter (B-C) model LS 13 320 single wavelength dry powder system. The measured average SSA values and standard deviations in the SSA measurements are shown in Table 2 for the Reference rock dust and PPC.

The results of the B-C particle size analyses on the MSHA rock dust survey samples are graphically shown in Figure 3 which features a comparison of the B-C-determined SSAs with the corresponding percentages of dust finer than 75 µm particle size. It is apparent from the data that, although the trending is positive, there is variability in the percentages of dust finer than 75 µm and their corresponding SSAs (correlation of 0.5, n=401). It should be noted that the SSAs determined are the geometric surface areas of the dust treated as equivalent smooth spheres.

B-C particle size analyses of a random rock dust sample revealed several maxima in the differential distribution curve (Figure 4). In addition to a main peak at a greater particle diameter, there were one or more peaks at finer particle diameters. It appears as if fine rock dust particles, such as those collected by baghouse filters from the pulverizing equipment, had been added back into the rock dust supplied to coal mines. While such fine particles would be effective in quenching an incipient coal dust explosion, it allows the dust to contain larger (75 to 850 µm), likely ineffective, inerting particles while maintaining the legal size requirement for rock dust.

4.2 Explosibility Tests

The 20-L explosibility chamber tests were conducted using homogeneous mixtures of 25% standard PPC and 75% rock dust (from available MSHA rock dust survey samples[1]). The results of the 20-L explosibility chamber testing are shown in Figure 5. It appears that the transition from explosible to non-explosible occurs when rock dusts have SSA values of approximately 230 m^2/kg. By comparison, the Reference rock dust used in full-scale explosion tests within the LLEM had an SSA value exceeding 260 m^2/kg. This dust consistently inerted explosion tests at this facility and in the 20-L chamber.

[1] The number of MSHA survey rock dust samples tested was limited to those having sufficient mass remaining after quartz analysis, particle size analysis, and wet and dry mechanical sieve analysis.

For comparison with 30 CFR 75.2, the current requirement of 70% < 200 mesh is added to an overlay on the inerting data shown in Figure 5 and displayed in Figure 6. The B-C measurement for a 200 mesh fraction is approximately 10% below that of the direct air jet sieve measurement, or 60% < 200 mesh as previously mentioned (Table 1).

Interestingly, there were some samples that met or exceeded the 30 CFR 75.2 specification requirement of 70% < 200 mesh particle size but were explosible as noted in Figure 6. These samples had SSAs < 230 m^2/kg. On the other hand, some rock dust samples not meeting the 30 CFR 75.2 specification for particle size were found to be non-explosible in the 20-L chamber due to having SSAs equal to or greater than 230 m^2/kg. These results suggest the need to include a minimum SSA as a key component of effective rock dust.

Additional experiments were conducted to further quantify the effect of rock dust SSA on explosibility within the 20-L chamber using various controlled size classifications of a local limestone rock dust supply (Reference rock dust) previously used in full-scale LLEM explosion inerting studies (NIOSH 2010). The classified size fractions had SSAs ranging from 49 to 446 m^2/kg. While the results are not conclusive due to the single PPC concentration used, they do indicate the sensitivity of inerting efficiency to the rock dust SSA.

An inerting index or limit, Z, is defined as the mass ratio of rock dust to coal dust. Figure 7 shows the relationship between Z and measured rock dust SSA. These data exhibited a good fit to the following exponential expression (R^2 = 0.98):

$$Z = 385.55 * SSA^{-0.638}$$

The inerting limit Z increases as rock dust SSA decreases. This indicates that greater quantities of rock dust are needed to inert as the average rock dust particle size increases. Rock dust with an average SSA of 446 m^2/kg (Z = 1.9) required about 65% rock dust to inert the PPC, while rock dust with an average SSA of ~49 m^2/kg required about 90% (Z = 9.0) to inert the PPC. As expected, finer rock dust particles are seen to be more effective in inerting as compared to larger particles.

Previous large-scale research results in the BEM, LLEM, and in laboratory studies using the 20-L chamber have determined an experimental uncertainty of approximately ± 3% inert content (Sapko et al. 2000; Cashdollar 1996; Richmond et al. 1975). The simplest way to illustrate this 3% uncertainty when viewing the 20-L data in Figure 7 is to assume a worst-case scenario where a nominal 75% rock dust is actually 72%. A 72% rock dust mixture (28% coal dust) corresponds to a Z-value of 2.57, yielding an SSA value of approximately 260 m^2/kg.

Another way of viewing this uncertainty and its effect on a conservative value for the minimum rock dust SSA specification is to consider the variation in the Z value arising from the fraction of rock dust in the mixture. Assuming variables *f(CD)* and *f(RD)* represent the fractions of coal dust and rock dust, respectively, the following expressions hold:

$$f(CD) = 1 - f(RD)$$

and

$$f(RD) = \frac{Z}{1+Z}$$

Approximating the uncertainty in *f(RD)* as a differential, *df*, then:

$$df(RD) = \frac{[(1+Z)dZ - ZdZ]}{(1+Z)^2}$$

and

$$dZ = (1+Z)^2 df$$

With 75% rock dust and 25% coal dust, Z=3. Given an experimental uncertainty of 3%, *df* = 0.03 and then *dZ* = 0.48. Hence, $Z - dZ$ = 3.0 − 0.48 = 2.52.

Using the expression in Figure 7 yields an approximate SSA value of 260 m^2/kg, similar to that obtained graphically in the previous discussion.

5.0 Conclusions

In this study, NIOSH adopted a specific surface area (SSA) designation (surface area per unit mass) as a means to improve uniformity of rock dust particle size distributions, in lieu of relying solely on the percentage finer than 200 mesh (75 µm). The overall data showed a good correlation between the SSA measurements and the effectiveness of the rock dusts in suppressing a coal dust explosion. The study also showed that it is critical to specify a minimum SSA to ensure an effective rock dust, since some rock dusts that met the current particle size specifications of the 30 CFR 75.2 failed to inert the coal dust in the 20-L chamber.

Combining findings from this study with those from recent NIOSH publications (Man and Harris 2014; NIOSH 2010; NIOSH 2011), the following conclusions can be drawn:

- Dust particle size has the greatest influence on the propagation (coal dust) and inhibition (rock dust) of dust explosions.
- Samples collected from the MSHA rock dust survey (as discussed in the 2011 NIOSH Hazard ID), were multi-modal, and several samples appeared to have wide variations in the amount of effective finer particles.
- Rock dust particles from 200 mesh to 60 mesh are largely ineffective in inerting coal dust explosions.
- Rock dust particles < 38 µm are more effective in inerting coal dust.
- The inerting effectiveness of rock dust is correlated to the SSA of the rock dust. Results from this study suggest the need to include a minimum SSA as a critical specification for effective rock dust.

These findings show that rock dust is most effective for inerting propagating coal mine dust explosions if the particle size is at least 95 percent finer than 200 mesh or 75 µm, and more importantly has a minimum surface area of 260 m^2/kg.

6.0 Acknowledgements

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7.0 Disclaimer

Mention of any company or product does not constitute endorsement by the National Institute for Occupational Safety and Health. The findings and conclusions in this paper are those of the authors and do not necessarily represent the views of NIOSH.

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Table 1 Common size fraction designations of PPC

Mesh Size	μm	Dry Air-jet Sieve % <	B-C % <
635	20	23.7	22.4
400	38	42.8	38.4
200	75	81.6	69.8
60	250	100.0	100.0
20	850	100.0	100.0

Table 2 SSA and particle density of PPC and Reference rock dust using the B-C system

Sample	Average SSA, m^2/kg	Std. Dev., m^2/kg	Particle Density, g/cc
PPC (n = 14)	239.4	± 15.7	1.3
Reference rock dust (n = 37)	265.1	± 11.9	2.7

Figures:

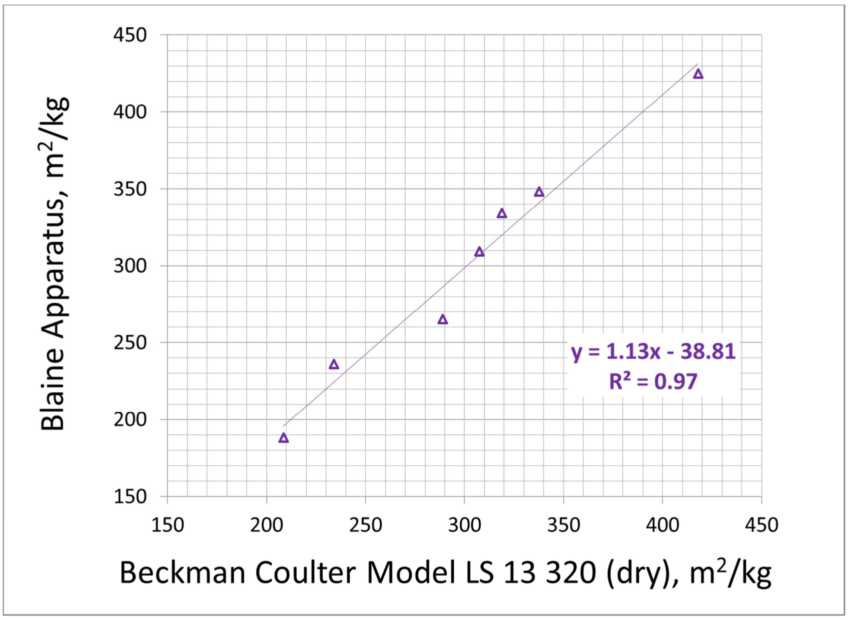


Figure 1

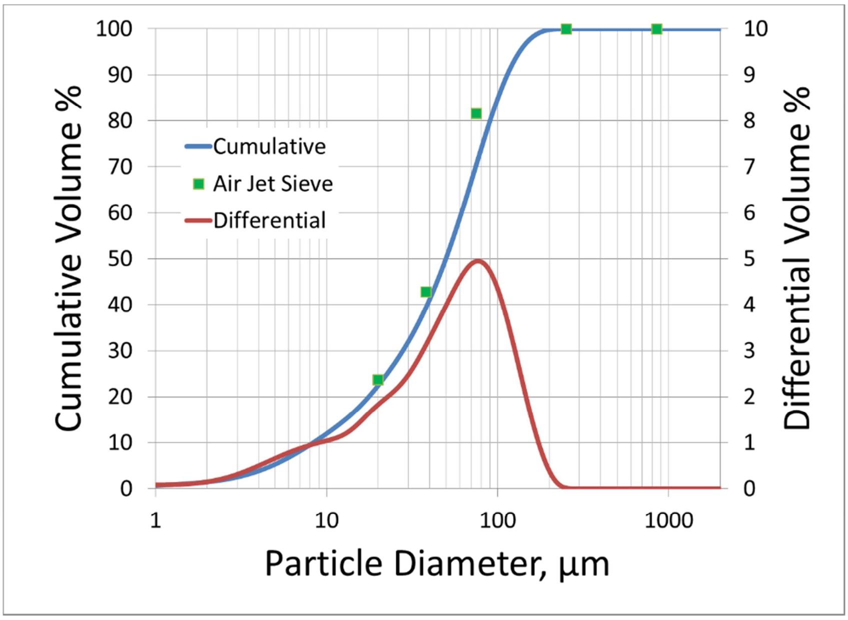


Figure 2

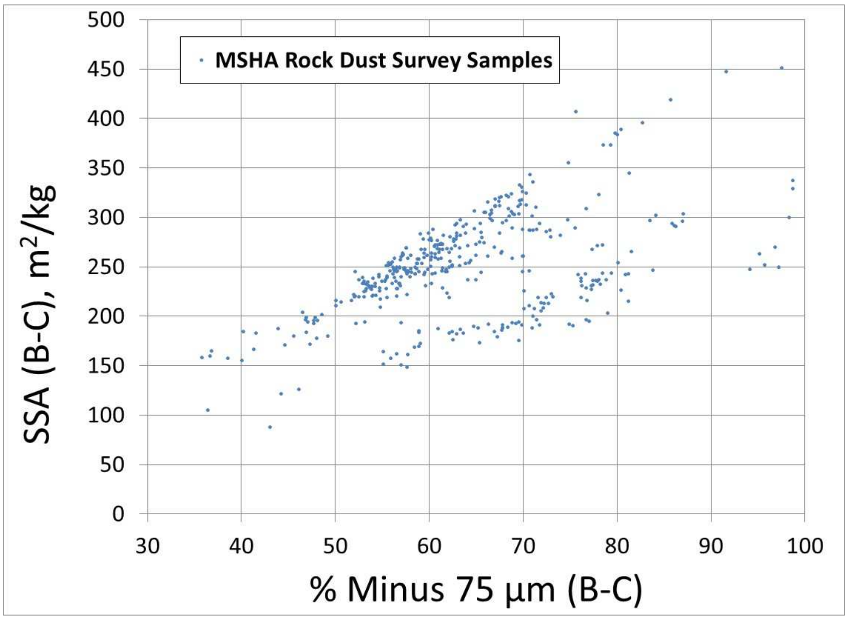


Figure 3

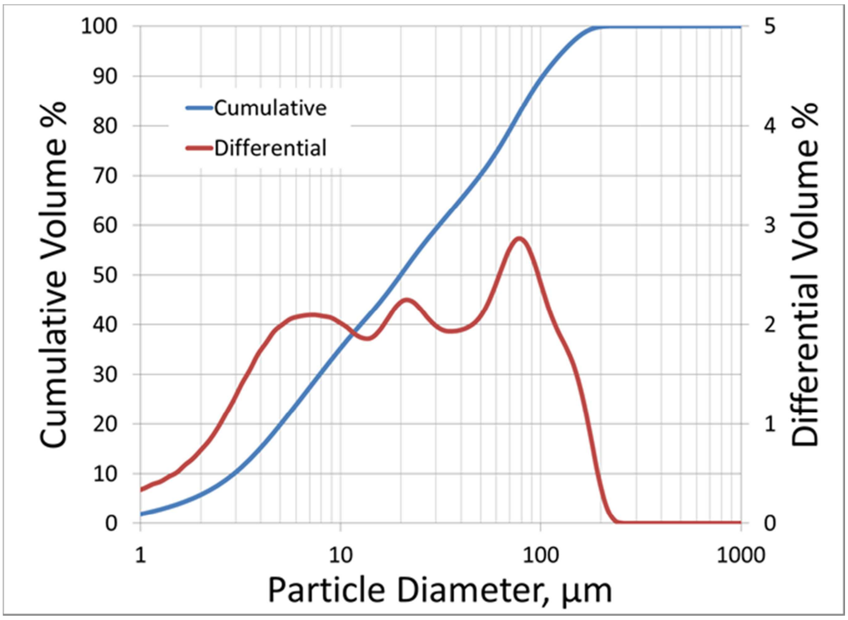


Figure 4

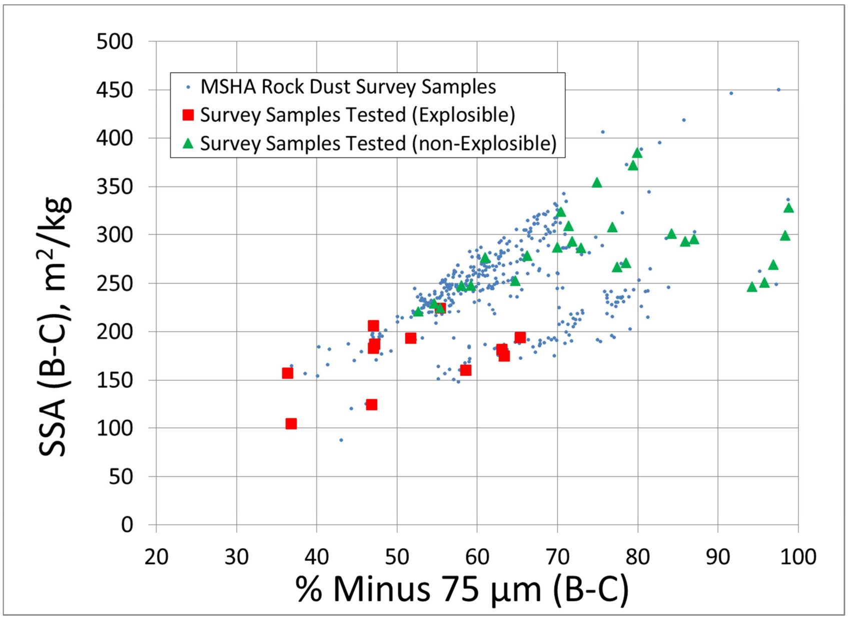


Figure 5

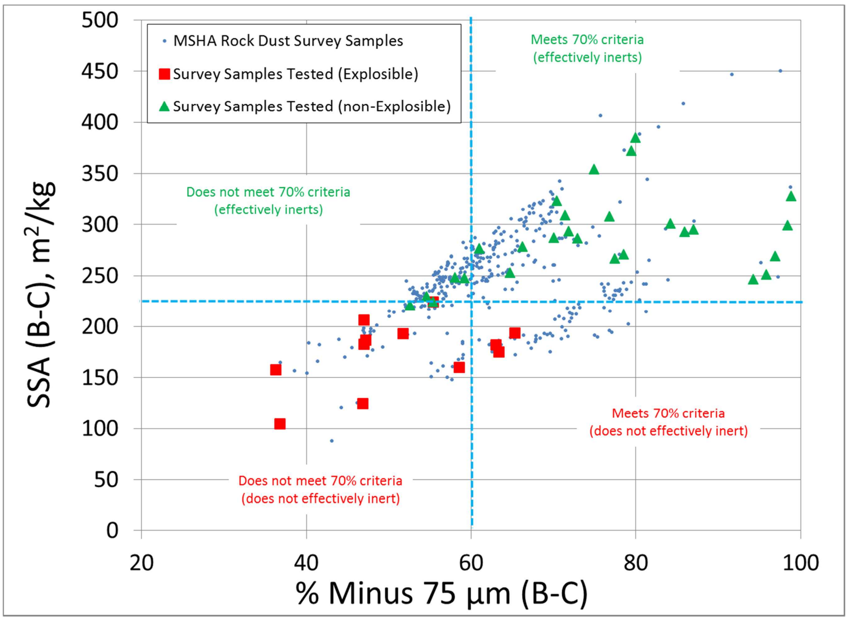
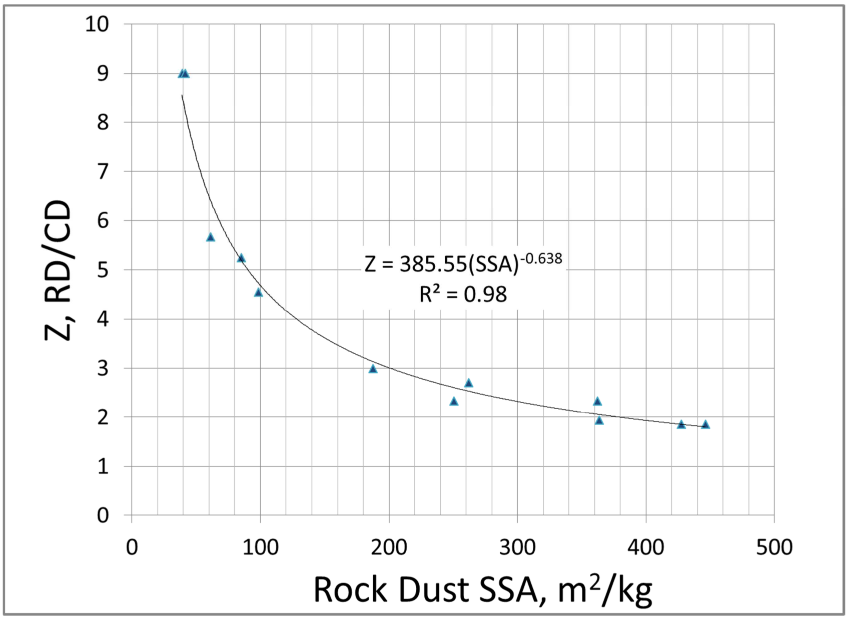


Figure 6



Z, RD/CD
Rock Dust SSA, m²/kg
Z = 385.55(SSA)^-0.638
R² = 0.98
0
1
2
3
4
5
6
7
8
9
10
0
100
200
300
400
500

Figure 7

Highlights:

- The rock dust particle size distribution widely varies as well as the associated specific surface area.
- Some rock dusts meeting the U.S. particle size requirements do not inert PPC in the 20-L chamber.
- 20-L chamber tests were conducted with several rock dusts.
- The inerting effects of specific surface area are examined.
- A specific surface area of 260 m^2/kg is a more effective inerting specification than the percentage of material passing through 200 mesh.