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Analysis and Characterization of Anti-Caking Additives Used in Rock Dust to Mitigate Mine Explosions

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Abstract

Experiments conducted with limestone dusts and dolomitic marble dusts have indicated that when rock dust is wetted and subsequently dried, it becomes a solid, non-dispersible cake. However, in order to be effectively inert a coal dust explosion, rock dust must be able to disperse as individual particles to air. To counteract this, rock dust manufacturers created treated rock dusts that will resist caking after moisture exposure. National Institute for Occupational Safety and Health (NIOSH) researchers conducted a series of laboratory-scale experiments on four base rock dusts and their treated counterparts to assess the effectiveness of various anti-caking additives after being exposed to moisture and then dried. The dusts were exposed to moisture using humidity cabinets having a high relative humidity (99% RH) and by also exposing the rock dust bed to water through bottom wicking. The dusts were then evaluated for dispersibility after drying using the NIOSH-designed dust dispersion chamber. The anti-caking additives were different concentrations of stearic acid, oleic acid, and xylene-based surfactants. All results were compared to a reference rock dust used to conduct large-scale experiments in the Lake Lynn Experimental Mine (LLEM), Fairchance, PA. When the untreated dusts were dried after exposure to moisture for 1 day, no dispersion was measured. However, rock dusts treated with anti-caking agents were readily dispersible even after exposure to moisture for 6 months. This report details the analysis and characterization of anti-caking additives using the NIOSH-designed dispersion chamber and the 20-L explosion test chamber.

Keywords Dust dispersibility · Treated rock dust · Coal mining · Explosion prevention · Particle size

1 Introduction

In order for a coal dust explosion to propagate, five elements are required. In addition to the fire triangle in which three elements are necessary to sustain a fire—fuel (coal dust), heat (ignition source), and an oxidizer (oxygen in air)—a combustible dust explosion requires dispersion of a dust cloud (pressure wave) and confinement of a dust cloud (underground mine entry) [1]. Coal dust explosions can originate anywhere in a mine. For example, when a small volume of a flammable methane and air mixture is ignited in a closed end, the high-temperature gases rapidly expand to create a pressure wave, sometimes referred to as a "pioneer wave," that may steepen into a shock wave as it propagates away from the ignition source. The shock wave produces a wind that disperses the dust preferentially from any elevated and exposed surfaces (roof, ribs, belt structure, cribbing, etc.). Factors that are known to affect the intensity of an explosion are the dust particle size, the location of the dust within the entry, the dust dispersibility, and the volatility of the coal dust [1]. Particles less than 75 μm (< 200 mesh) in size are most reactive [2]. Dusts that are located on the roof, ribs, and elevated surfaces are more readily dispersed than dusts on the floor. Therefore, application of quality rock dust in sufficient quantities is essential to inert coal dust and to prevent continued flame propagation. Thirty CFR 75.402 and 75.403 require 80% total incombustible content be maintained within 40 ft. of the face, and 30 CFR 75.2 defines rock dust acceptable for use in underground coal mines. It also defined that "when rock dust is wetted and dried, will not form a cake".

The National Institute for Occupational Safety and Health (NIOSH) previously identified that many rock dusts used in US underground coal mines did not meet the size specifications or the caking requirements [2]. "Caking phenomenon" is typically used to describe the change of free-flowing bulk solids into agglomerated chunks. Christakis et al. summarized

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Fig. 1 Qualitative caking test using untreated rock dust. Left: dry rock dust; center: water mixed with rock dust; right: rock dust after water evaporates

the processes of powder caking due to moisture migration in five steps: (i) wetting and moisture absorption, (ii) liquid bridging, (iii) drying and moisture desorption, (iv) hardening and solid bridging, and (v) compacting and caking. When the rock dust is in contact with moisture, a saturation solution of a soluble material made up of dust particles is formed at the point of the contact. This causes the formation of liquid bridges at the particle-particle contact points [3]. Subsequently, rock dust particles cake or agglomerate, making the dust a non-dispersible cake when the moisture evaporates [4, 5].

The caking phenomenon reduces the dispersibility of a rock dust, and hence, it cannot quench or prevent a propagating explosion. Waterproofing a rock dust by using a hydrophobic additive is one way to prevent rock dust from caking. Prior work had shown that hydrophobic agents used with conventional limestone-based rock dusts lessened their ability to cake when exposed to moisture and enabled their dispersion back into the environment [6]. Some of the anti-caking additives currently used in the industry are very low concentrations (< 1%) of fatty acids such as stearic acid and oleic acid that make the rock dust hydrophobic [4]. Adding such fatty acids not only makes rock dust hydrophobic, it also acts as a fluidizing agent [7–9].

The objective of this research effort is to assess the base and treated rock dusts for their dispersibility after exposure to moisture and to explore the inerting requirements using a 20-L explosion chamber.

1.1 Experimental Procedure

In this study, four different base rock dusts and their treated counterparts are tested for moisture wicking and dispersion. These dusts will be referred to as dusts A, B, C, and D. Untreated rock dust A is also the reference rock dust, which has been widely used in NIOSH Lake Lynn Experimental Mine (LLEM) large-scale explosion testing, and is the dust used to validate the 80% total incombustible content recommendation [10–12]. Therefore, all results are compared to the untreated dust A results. The specific concentrations of the anti-caking additives are unknown and they were tested as received from the supplier. In the food and drug industry, low concentrations of oleic acid, stearic acids, and other fatty acids are used as anti-caking agents.

1.1.1 Particle Size Analysis

The particle size distributions, mean rock dust particle sizes, and specific surface areas (SSA) were obtained using a Beckman Coulter LS 13320 optical particle size analyzer (B-C). NIOSH researchers followed the procedure recommended by the analyzer manufacturer [13]. The index of refraction (RI) used was 1.8 + 0.3i for coal dust analysis and 1.68 + 0.0i for limestone rock dust analysis, where i is the imaginary component.[1] The Beckman Coulter relies on laser scattering and is capable of particle size measurements as small as 0.3 μm. The software for the B-C calculates the SSA as cm^2/cm^3 and is converted to cm^2/g by dividing by the density of the material tested (2.75 g/cm^3 for rock dust and 1.33 g/cm^3 for coal dust).

1.1.2 Qualitative Caking Test

The qualitative caking test is a pass/fail assessment of the rock dusts' ability to cake when exposed to moisture and then dried. Twenty grams (20 ml) of water is added to 20 g of rock dust in a weigh boat. The rock dust and water are then mixed with a spatula for approximately 10 s and left to dry in ambient laboratory conditions for 24 to 48 h. After the sample dries, a visual assessment of caking is made by drawing a spatula across the dried rock dust to see if the rock dust breaks or cracks (Fig. 1).

1.1.3 Moisture Wicking

Two types of wicking tests were conducted with the treated and untreated rock dusts. Bottom wicking tests were conducted to mimic the moisture seepage from the mine floor, ribs, and roof using plastic trays with holes in the bottom (Fig. 2). Tray dimensions are 14.5 cm × 19.25 cm × 2.54 cm and tray

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[1] *These were average RI values found in the Beckman Coulter manual for carbon and calcium carbonate and were not determined by a separate analysis.*

Fig. 2 A sample tray set inside a larger pan to allow moisture wicking from the bottom of the tray

specifications are presented elsewhere [14]. These plastic trays with holes were filled with rock dusts and kept inside a larger tray (having inner dimensions of 24.5 cm × 20.8 cm × 3.5 cm and a volume = 1800 cm^3) to accommodate moisture wicking from the bottom. The weight gain due to water wicking into the sample was monitored periodically. All dust trays were allowed to dry in ambient air on a bench top before conducting dispersion tests.

The moisture adsorption through the air was monitored using the humidity cabinets (Fig. 3).

Fisher Scientific desiccator cabinets were converted to humidity cabinets by placing water-filled trays in the cabinets until the relative humidity (RH) reached above 90% (measured the RH using humidity data loggers) at room temperature (25 °C). Then, the dust-filled trays were placed inside the humidity cabinets for 24-, 48-, 72-, and 96-h periods. The moisture adsorption (through weight gain) was measured on the different time intervals before conducting dispersion tests as described below. All measurements were

1.1.4 Dust Dispersion Chamber

The rock dust dispersibility was measured using a NIOSH-designed dust dispersion chamber [14]. All test samples were subjected to the same reproducible 0.3-s air pulse from a 2.8-bar (40-psi) compressed air source, providing a peak dynamic pressure of 0.3 bar (4.2 psi). An optical dust probe downwind of the pressure pulse measured the light obscuration resulting from the concentration of the dispersed dust cloud. After filling a tray with a leveled layer of dry dust, the tray was inserted into the chamber where the air nozzle supplying the pressure pulse is horizontal to the surface of the dust layer (Fig. 4). Sample dust trays were weighed before and after each air pulse to determine the mass of dust dispersed.

1.1.5 20-L Explosion Chamber

Dust inerting experiments were conducted in the US Bureau of Mines (USBM)-developed 20-L explosibility chamber. This is an ASTM standard testing apparatus (ASTM E1226) previously described in detail by Cashdollar [15, 16]. This is also the standard laboratory test chamber described in ASTM E1515 for minimum explosible dust concentration measurements. It has been used since 1982 by the former-US Bureau of Mines (USBM) and now by NIOSH to study the explosibility and inerting capability of dust mixtures. The ignition sources used for the 20-L chamber inerting tests were 5000 J electrically activated pyrotechnic igniters manufactured by Fr. Sobbe of Germany [15]. The 5000 J

Fig. 3 Humidity cabinets

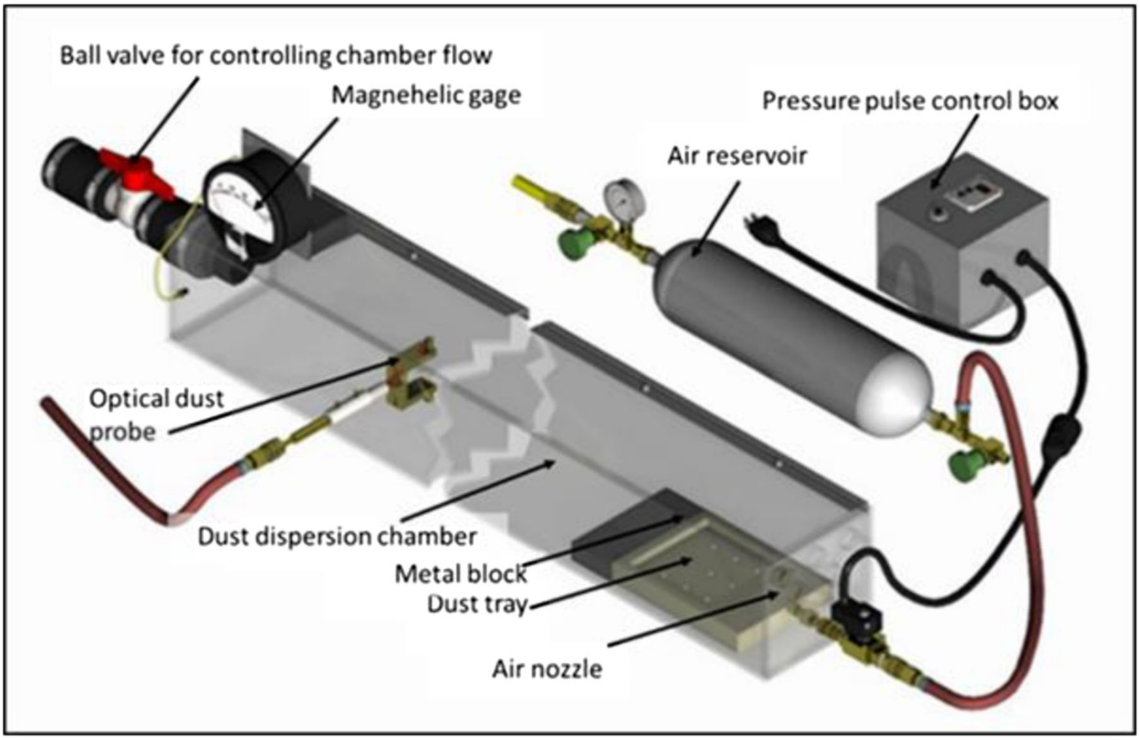


Fig. 4 Schematic of the dust dispersion chamber with the dust tray

igniter by itself produces a pressure rise of about 0.5 bar in the 20-L chamber. This ignitor pressure contribution is subtracted from the explosion pressure in determining the pressure ratio (PR = explosion pressure/initial pressure) of the ignition.

All experiments were conducted using pulverized Pittsburgh coal (PPC) at 400-g/m^3 concentration to determine the reactivity of the mixture. Tests were conducted with rock dust increments of 5%, and the final amount to inert was interpolated for values reported herein. A pressure ratio (PR) calculation is used to determine the inerting effectiveness of a particular rock dust when mixed with explosible concentrations of PPC in the 20-L chamber. The criteria to determine if an explosion has occurred is if a maximum explosion pressure ≥ 2 bar is measured.

2 Results and Discussion

All rock dusts were tested as received. The exact preparation methods of the different anti-caking rock dusts are unknown due to proprietary concerns. Some manufacturers blend the hydrophobic rock dust with the base rock dust using heavy powder mixtures. Some anti-caking rock dusts were prepared by spray treating the base rock dust with the hydrophobizing agent.

2.1 Qualitative Caking Test

Using the qualitative caking test method, the wettability differences between untreated and treated rock dust were readily

Fig. 5 Qualitative caking tests conducted with treated and untreated rock dusts. Note the cracked ring around the perimeter of the dried untreated samples

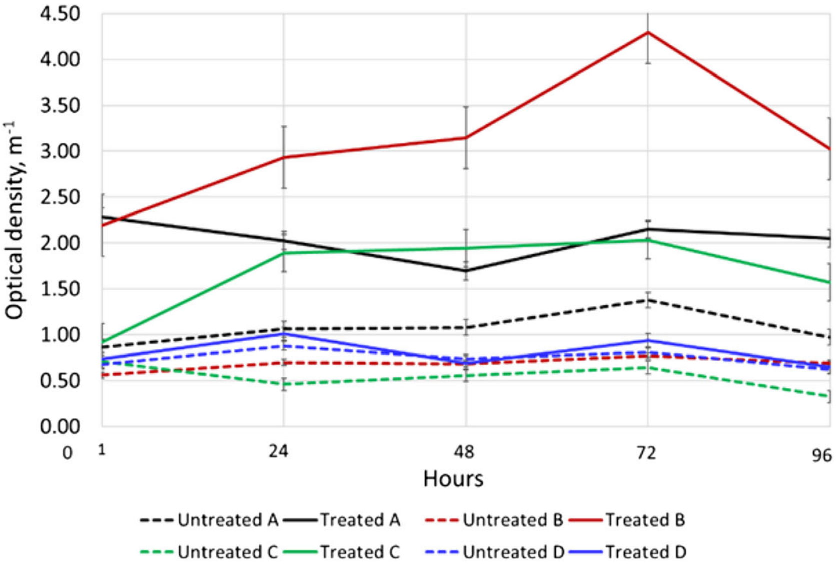


Fig. 6 Optical density of the dispersed dust vs the exposure time to moisture

and easily observed. As stated in the Experimental Procedure section, 20 ml of water is added to 20 g of rock dust, mixed with a spatula, and let dry in ambient air. The untreated rock dust rapidly wicks water and wets the particles, forming a slurry within 1 to 2 min after mixing. The treated rock dust resists wicking and the particles remain dry (Fig. 5). After 48 h of drying under laboratory conditions, the untreated rock dust forms a firm cake of agglomerated particles while the treated dust particles remain separated. The degree to which water affects the dispersibility of the treated rock dust after drying was determined by comparing the relative dispersibility in the dispersion chamber before and after exposure to water.

2.2 Dust Dispersibility after Moisture Adsorption through Air

It is commonly known that underground mine conditions can become very humid during the summer. Therefore, an effort to mimic a similar high-humidity atmosphere was made using the humidity cabinets to expose the dust to moisture in the air. Using the dust trays, treated and untreated rock dust samples were weighed and kept in humidity cabinets to monitor the moisture adsorption from the vapor phase over time.

Dispersibility was measured using the relative normalized optical density (using the reference dust) of the dust conditioned in the humidity cabinets, dried, and suspended by the air blast in the dispersion chamber. This process is depicted in Fig. 6 [14]. The normalized peak optical density (D/L) is proportional to the peak airborne concentration of the dispersed dust at the optical probe where $D = \ln I_o/I$ (I_o = signal intensity ratio before air pulse, I = signal intensity just after the air pulse) and L is the path length of the optical probe.

It is evident that treated dusts A, B, and C (particularly treated dust B) displayed relatively high dispersibility, even after exposure to high humidity, up to 96 h. Treated rock dust D shows no improvement over the untreated rock dust after moisture exposure in humidity cabinets. This may be correlated with a crust formation noted after 24 h of exposure to high humidity. As expected, all four untreated rock dusts displayed relatively low dispersibility after exposure to high humidity.

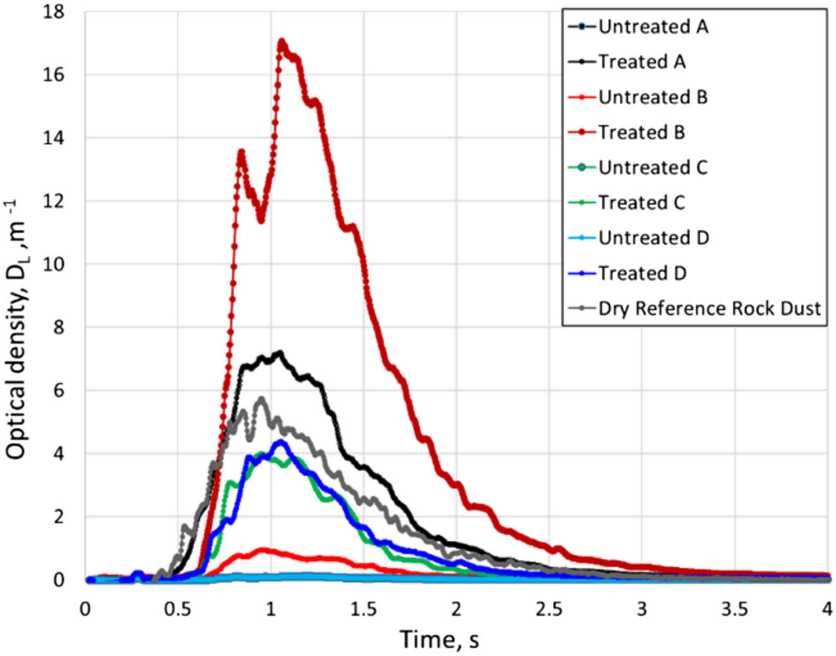
2.3 Dust Dispersibility After Moisture Wicked from the Bottom of the Trays

Similar to moisture wicking through the air, dispersibility of the rock dusts was assessed after bottom wicking. Using humidity cabinets, a high relative humidity environment was again generated. This time, the dust trays were kept inside larger water-filled trays so that the rock dust could be exposed to moisture through the holes in the bottom of the tray. After 24 h, the treated and untreated rock dust samples were taken out of the humidity cabinets and dried on the laboratory bench top in ambient air before conducting dispersion tests. Figure 7 indicates the relative dispersibility of the dusts relative to the reference dust (peak maximum optical density is 5.75 ±

Table 1 Normalized optical density of the dust samples tested

	Optical density at peak maximum, m^{-1}	Normalized optical density
Treated A	6.72	1.17
Untreated A	0.20	0.03
Treated B	14.60	2.54
Untreated B	0.80	0.14
Treated C	3.59	0.62
Untreated C	0.20	0.03
Treated D	3.40	0.59
Untreated D	0.20	0.03

Fig. 7 Relative dispersibility of the treated and untreated rock dusts



1.15 m^{-1}). Table 1 presents the normalized dispersibility of the peak maximum optical density with respect to the reference dust (peak maximum of any rock dust/peak maximum of the reference rock dust).

The relative dispersibilities of the untreated rock dusts were much less than the treated rock dusts after bottom wicking. The low relative dispersibility of the untreated rock dust A is indicated by a peak optical density, D_L, of 0.2 m^{-1}, whereas the treated dust A measured as 6.72 ± 1.21 m^{-1} or a 30- to 40-fold higher peak airborne concentration. Similarly, untreated dust B had an optical density of 0.8 m^{-1} and the treated dust B had an optical density of 14.6 ± 1.21 m^{-1} or a 20-fold higher concentration. The peak optical density of both treated dust A and treated dust B had higher peak optical densities than the dry untreated reference rock dust, which was 5.75 ± 1.8 m^{-1}. The airborne dust concentration after a day of exposure to both high humidity and the wicking of water from the bottom of the dust bed was seen to be greatest for the treated dust B, followed by the treated dust A, and finally the treated dusts C and D. The untreated latter dusts produced effectively no airborne dust from a dispersing air pulse. When the optical density was normalized against the reference dusts, similar trends followed.

As the wicking and caking tests indicate, all untreated rock dusts cake in the presence of moisture. Figure 8a–c indicates the untreated and treated rock dusts tray prepared and conditioned in a similar manner as for the moisture tests. As expected, the

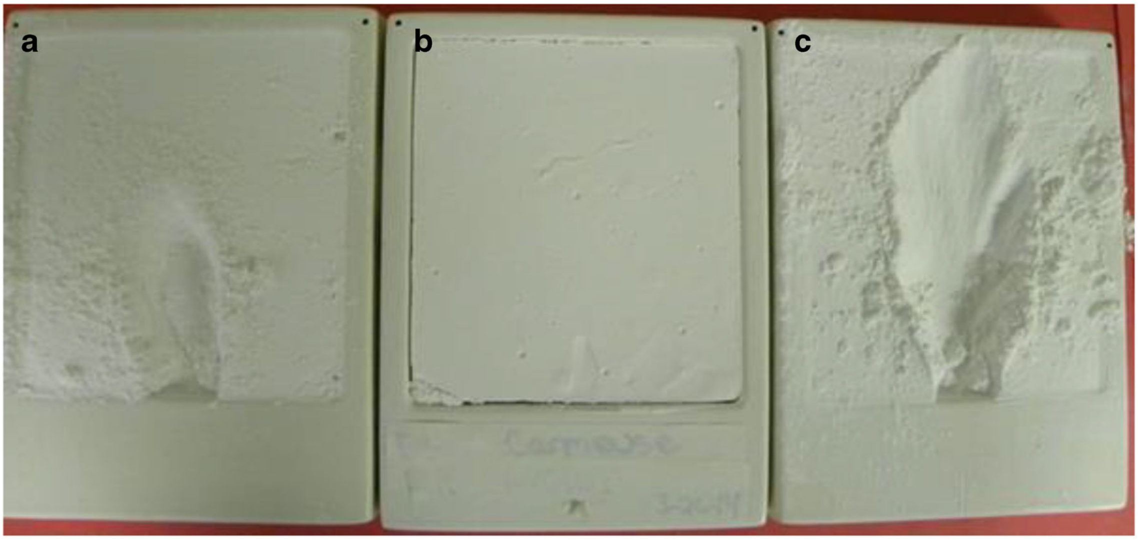


Fig. 8 Dispersion of treated and untreated rock dust. **a** Dry untreated rock dust after dispersed. **b** Untreated rock dust after exposure to water from the bottom for 24 h and then dispersed. **c** Treated rock dust after exposure to water from the bottom for 24 h and then dispersed

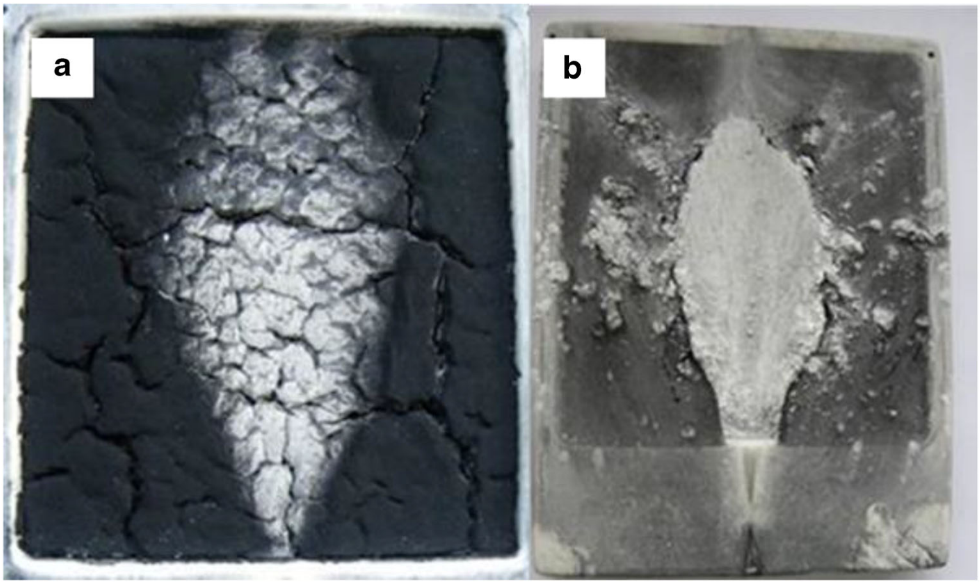


Fig. 9 **a** Float coal dust on top of caked rock dust after exposure to water from the bottom for 24 h. **b** Float coal dust with the treated rock dust after exposure to water for 24 h

untreated rock dusts rapidly wicked up moisture, saturating the rock dust beds, while the trays with the treated rock dusts did not show obvious changes after water exposure. The trays were then removed from the humidity cabinets and allowed to dry in ambient temperature to a constant weight before testing in the dispersion chamber. Dry untreated rock dust dispersed in the dispersion chamber (Fig. 8a) whereas caked untreated rock dust showed no dispersion at all (Fig. 8b). Treated rock dust dispersed similarly to the dry untreated rock dust after exposure to water from the bottom for 24 h (Fig. 8c).

Coal dust is not easily wetted and can remain dispersible in the presence of water. Figure 9 indicates the results of tests conducted in a similar manner except that 5 g of coal dust was sprinkled on top of the ~ 0.5-in-thick rock dust layer to form a thin cover layer of float coal. The tray was then placed in the humidity cabinet and exposed to the liquid and vapor water phases. Figure 9a shows that the untreated caked rock dust does not disperse with the coal dust after moisture exposure, whereas the coal dust lying on top of the caked rock dust preferentially disperses, leaving the rock dust layer mostly intact. In contrast, the same strength air pulse not only removes the surface coal dust but also a significant fraction of the underlying treated rock dust as shown in Fig. 9b. These illustrations clearly demonstrate the potential danger of float coal lying on top of untreated rock dust. Rock dust must disperse with the coal dust to be effective in preventing coal dust explosion propagation [9].

Table 2 20-L inerting limits and the SSA of the treated and untreated rock dust

Rock dust	SSA, cm^2/g	% Rock dust concentration		
		70%	75%	80%
Untreated A	2530	Explosion	Inert	N/A
Untreated B	2262	Explosion	Inert	N/A
Untreated C	3386	Explosion	Inert	N/A
Untreated D	2123	N/A	Explosion	Inert
Treated A	2828	Explosion	Inert	N/A
Treated B	3556	Explosion	Inert	N/A
Treated C	4348	Explosion	Inert	N/A
Treated D	2227	N/A	Explosion	Inert

2.4 20-L Inerting Testing Using the BOM 20-L Explosion Chamber

In addition to the wicking and dispersion tests, the explosibility of the treated and untreated rock dusts was tested using the NIOSH 20-L chamber. Previous data from NIOSH 20-L chamber tests have shown that a coal dust (400-g/m^3 coal dust concentration) and rock dust mixture must contain at least 75% limestone rock dust to inert the PPC, which contains 80% minus 200 mesh particles [2]. It is important to note that the 20-L chamber data indicate trends but cannot be directly scaled to large-scale results such as those obtained in other studies performed at the Lake Lynn Experimental Mine [10]. Based on the past NIOSH studies reporting on LLEM large-scale experiments, it has been found that one can equate a 75% inerting rock dust concentration given by 20-L tests to an 80% total incombustible content requirement for mine inerting [2]. The baselines in both the LLEM and 20-L chamber tests were established using PPC and a reference rock dust. This reference rock dust has been acquired from the same rock dust manufacturer and has historically consistent particle size distributions. The inerting limit of each treated and untreated rock dust was also determined using the same PPC concentration of 400 g/m^3 (for the 75% RD mixtures, the RD concentration was 1200 g/m^3). This PPC concentration is commonly

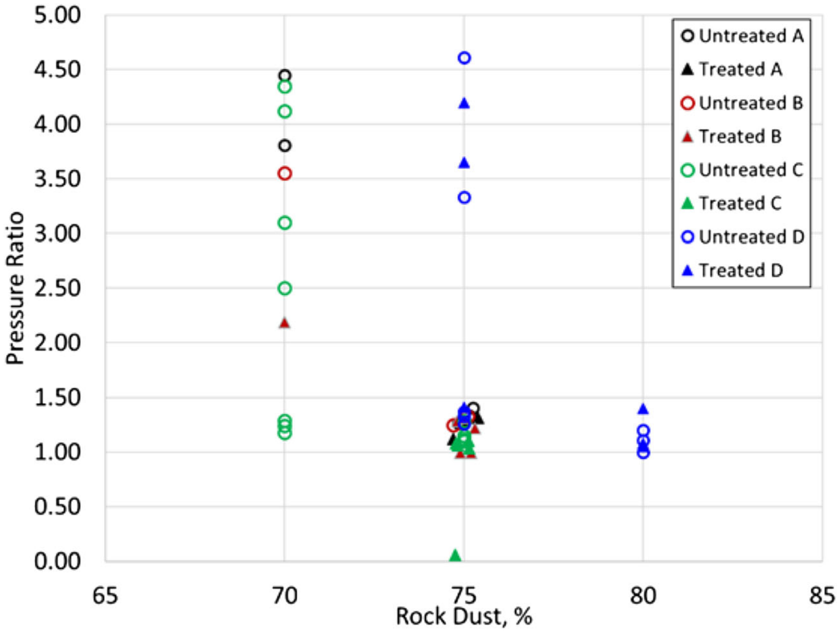


Fig. 10 20-L inerting results of treated and untreated rock dust

the “worst-case” concentration. The 20-L chamber results of rock dust mixtures are shown in Table 2.

Successful inerting or a non-explosion was defined as PR < 2 and $D_p/D_t < 1.5$ bar/s, while an explosion or non-inerting was determined when the PR ≥ 2 and a $D_p/D_t \geq 1.5$ bar/s. This boundary between explosion and non-explosion was determined within 5% rock dust concentrations. Summarized in Table 2 are the results of these measurements. Experimental results indicate that, except for the treated and untreated rock dust D, all other rock dusts inerted at 75% rock dust concentration as shown in Fig. 10.

The particle size or the SSA of both treated and untreated rock dust D was found to be relatively smaller than the other rock dusts tested in this study. This may be a reason that dust D requires 80% rock dust to inert (instead of 75%). NIOSH research has shown that rock dust is most effective for inerting propagating coal mine dust explosions if the particle size is at least 95% finer than 200 mesh or 75 μm, and more importantly has a minimum surface area of 2600 cm^2/g [17, 18]. The SSA of both treated and untreated dust D was less than 2600 cm^2/g.

3 Summary and Conclusion

Laboratory investigation of four untreated and treated rock dusts revealed that all untreated rock dusts caked. The treated rock dust indicated high relative dispersibility after moisture exposure and adsorption through air and bottom moisture wicking. Treated dust B had a very high relative dispersibility ($14.6 \pm 1.21\ m^{-1}$) compared to the other treated rock dusts. This may be due to the higher SSA or the smaller particle size of the treated rock dust B compared to the other treated dusts. Overall, the treated dusts dispersed much better in the dispersion chamber than the untreated rock dusts after exposure to moisture. NIOSH research revealed that treated rock dust disperses even after exposure to moisture for 6 months or more [14]. Currently, full-scale experiments with treated rock dust are ongoing.

Investigations using a 20-L chamber revealed that both treated and untreated dusts inerted at 75% rock dust concentration using 400 g/m^3 PPC, with the exception of treated and untreated rock dust D. This may be due to the smaller SSA of rock dust D compared to the reference rock dust A where the SSA is 2600 cm^2/g. In all cases, the anti-caking treatment did not affect the dusts’ ability to inert a coal dust explosion.

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Compliance with Ethical Standards

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

Conflict of Interest The authors declare that they have no conflict of interest.

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