FINAL TECHNICAL REPORT

Development of an Anti-Caking Rock Dust

in response to

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Summary

The overall objective of the project was to develop modified limestone based rock dust blend(s) capable of effectively 1) dispersing in the NIOSH dust dispersion chamber after being wetted, then dried, and 2) inerting coal dust in the NIOSH 20-L explosibility chamber. Dozens of blends of fine treated limestone with coarser untreated limestone were evaluated to determine the most desired combination. The blends were constructed upon the premise that fine treated limestone particles would fill the voids between the larger untreated limestone particles and prevent or inhibit the migration of water throughout the blend, and subsequently pass the dust dispersion chamber test.

Seven commercially available limestone products were used throughout the study (5 coarse untreated limestone products and two fine treated limestone products). The study was broken down into several phases as the optimization work focused in on specific blend components and ratios. Particle size, chemistry and stearic acid treatment levels were measured to validate that the blends were correctly proportioned. In addition, contact angle, ASTM D-698 (Proctor test) and dust dispersion chamber testing was conducted on the blends at various humidities to determine their hydrophobicity and ability to re-disperse after being wetted and dried. Finally selected blends were evaluated for their effectiveness in inerting Pittsburgh pulverized coal (PPC) dust in NIOSH's 20-L chamber.

In this study, regardless of the blend components or their ratios, all the wetted then dried rock dust blends dispersed when compared to conventional rock dusts which formed essentially a non-dispersible cake when tested in the NIOSH developed dust dispersion chamber. Initial concerns regarding the use of a treated component and the possible adverse effects of free stearic acid was investigated, and not found to be an issue at the levels tested with the base and blend samples. Blends containing greater than 12.5% of the treated limestone component offered little additional advantage when tested for 'dust dispersion' and 'inerting'. Contact angle and ASTM D-698 testing suggest blends containing <7.5% of the treated component are more susceptible to moisture pick up.

The NIOSH designed dust dispersion chamber and test protocols developed for this study were an excellent indicator of whether a rock dust would disperse into separate particles by a defined pulse of air. Contact angle measurements were beneficial for determining whether a blend demonstrated hydrophobic properties but the contact angle results did not correlate well with the dust dispersion results.

As the study progressed, several base components and their blends (Drikalite, #8 White & RO-40) were eliminated based on performance or economic factors. The remaining blends based on #10 White and Micro-White 100, all inerted the Pittsburgh pulverized coal (PPC) dust at the 75% inertant concentration within the NIOSH 20-L chamber.

Individual test results coupled with economic considerations led to the recommendation of a 20 – 25 µm untreated rock dust (Micro-White 100) blended with 10% ± 2.5% of a 3 µm treated component (Kotamite). A large 20 ton scale-up sample of this blend was subsequently

manufactured for more extensive testing within an area of NIOSH’s Safety Research Coal Mine or similar facility.

The scale-up blend sample along with an untreated rock dust (Micro-White 100) and two blends of the untreated rock dust with Kotamite (at 10% and 20%) were evaluated by NIOSH within the Bruceton Experimental Mine. The results showed that the untreated rock dust immediately wicked moisture from the mine strata and was not dispersible when wet or later when dried. All of the treated rock dust blends dispersed immediately after application and continue to disperse over time.

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Acronyms and Abbreviations

AMC	Allegheny Mineral Corporation
ASTM	American Society for Testing and Materials
AU	arbitary unit
Avg	average
BET	Brunauer, Emmett and Teller
CA	contact angle
CFR	Code of Federal Regulations
D 10%	diameter at which 10% of the material passes
D 50%	diameter at which 50% of the material passes
D 90%	diameter at which 90% of the material passes
DI	deionized
Dia	diameter
dnt	did not test
HH	high humidity
M	mesh
N	nitrogen
N/A	not applicable
ND	none detected
NIOSH	National Institute for Occupational Safety and Health
OMSHR	Office of Mine Safety and Health Research
PE	Polyethylene
PPC	Pittsburgh Pulverized Coal
PR	pressure ratio
QA	Quality Assurance
RD	rock dust
RH	relative humdity
S.A.	surface area
TGA	Thermogravimetric Analysis
XRF	Xray Fluorescence
Al_2O_3	Aluminum Oxide
CaO	Calcium Oxide
Fe_2O_3	Iron Oxide
K_2O	Potassium Oxide
K_2SO_4	Potassium Sulfate
$MgCl_2$	Magnesium Chloride
MgO	Magnesium Oxide
SiO_2	Silicon Dioxide
SrO	Strontium Oxide
LOI	Loss on Ignition

Unit of Measure Abbreviations

%	percent
~	approximate
°	degree
°C	degrees Centrigrade
°C/min	degrees Centrigrade per minute
µm	microns
cm	centimeter
ft/min	feet per minute
g	grams
g/m^3	gram per cubic meter
in	inch
J	joules
L	liter
lbs/ft^2	pounds per cubic foot
m^2/g	square meters per gram
min	minutes
ml	milliliters
ml/min	milliliters per minute
mm	millimeter
pcf	pounds per cubic foot
psi	pounds per square inch
psia	pounds per square inch absolute
s	second
wt	weight

Project Overview and Objectives

When exposed to moisture, limestone-based rock dusts have a tendency to cake and do not easily disperse when a light blast of air is applied[1]. The objective of the project was to develop economically-viable modified limestone-based rock dusts capable of effectively 1) dispersing in the NIOSH dust dispersion chamber[2] after being wetted, then dried, and 2) inerting coal dust in the NIOSH 20-L explosibility chamber.

Prior work (Vogt 2000) had shown that the use of 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. The focus of this work centered upon investigating the particle size and particle packing characteristics of the rock dusts in conjunction with varying the levels of a hydrophobic agent and evaluating these rock dusts in the dust dispersion chamber developed by NIOSH. Particle packing models (Mangulkar 2013) are based upon the concept that voids between larger particles would be filled by smaller particles thereby reducing the volume of voids or increasing the "Packing Density" (Figure 1). Packing density is defined as the ratio of the solid volume to the total volume **(Packing Density = Solid Volume / Total Volume)**. The concept of this project being that the fine treated limestone particles which are hydrophobic in nature will fill the voids between the larger untreated limestone particles and prevent or inhibit the migration or wicking of water throughout the blend. This concept is key to the success of the project.

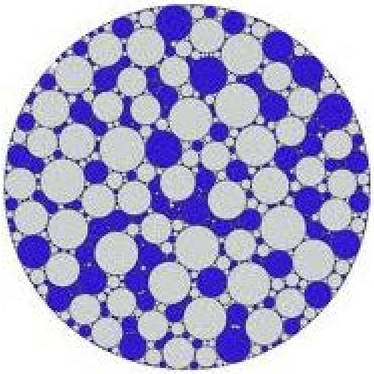

Figure 1. Illustrates the concept of particle packing

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[1] Compliant rock dust is defined in 30 CFR 75.2 as "Pulverized limestone, dolomite, gypsum, anhydrite, shale, adobe, or other inert material, preferably light colored, 100 percent of which will pass through a sieve having 20 meshes per linear inch and 70 percent or more of which will pass through a sieve having 200 meshes per linear inch; the particles of which when wetted and dried will not cohere to form a cake which will not be dispersed into separate particles by a light blast of air; and which does not contain more than 5 percent combustible matter or more than a total of 4 percent free and combined silica ($SiO2$), or, where the Secretary finds that such silica concentrations are not available, which does not contain more than 5 percent of free and combined silica."

[2] Developed by NIOSH to compare the dispersion characteristics of rock dusts.

Specifically, the project investigated blends of fine treated limestone products with coarser untreated limestone products to determine the most desired combinations that would pass the NIOSH dust dispersion chamber test and the 20-L chamber test for effectively inerting coal dust. The amount of reacted and un-reacted free stearic acid present was assessed to determine if the rock dusts ability to inert coal dust was adversely affected. A number of characterization tests were utilized to ensure that both the base and blend samples were consistent and accurately blended. Table 1 below summarizes the various test protocols used in this project and the rational for their use.

Table 1. Lists the test protocols used throughout the project

Test Protocol	**Test Rationale**
Blend Preparation	Accurate preparation of blends
Chemistry	Measurement of silica levels
Thermogravimetric Analysis (TGA)	Validation of accurate blend ratios and free stearic acid levels
Particle Size - 200 Mesh	Validation of accurate blend ratios
Particle Size - Microns	Validation of accurate blend ratios
Surface Area	Cross reference against explosibility values
Dust Dispersion Chamber Testing	Measurement of anti-caking
Contact Angle	Measure of the hydrophobic nature of the blends
ASTM D698 - Proctor Testing	Measurement of anti-caking
20-L Chamber (Explosibility)	Comparison of the explosive abatement properties of the blends

The project concentrated on evaluating blends of two commercially available treated limestone products (a 3 µm and 1.8 µm median particle size) with five commercially available untreated limestone products (ranging from a median particle size of 6 µm to ~40 µm). The project was split into several phases with the objective being to understand how the various blends would perform in the dust dispersion chamber and the 20-L chamber test for effectively inerting coal dust.

- Phase 1 - investigated the effects of increasing the amount of un-reacted stearic acid in the blend samples.
- Phase 2A - investigated the effects particle packing would have on the performance of the blends.
- Phase 2B & 2C - focused in on specific particle size products and specific blend ratios.
- Phase 3 – repeated the testing on the three most promising blend candidates.
- Pilot – a large scale up sample was manufactured for NIOSH and mine operator testing.

Table 2 on the following page tabulates the testing conducted for each of the various phases. For each phase the number of base and blend samples are noted and whether the samples were evaluated in a given test. A ‘Yes’ indicates the sample was tested, a ‘No’ indicates it was not tested. A ‘part’ designation indicated that only some of the blend samples were tested.

Table 2. Illustrates the testing conducted at each phase of the project

Phase	1	1	2A	2A	2B	2B	2C	2C	3	3	Pilot	Pilot
Base or Blend	Base	Blend	Base	Blend	Base	Blend	Base	Blend	Base	Blend	Base	Blend
# of Samples	(3)	(7)	(7)	(24)	(5)	(9)	(4)	(5)	(4)	(3)	(2)	(1)
TGA	YES	YES	YES	YES	NO	YES	NO	YES	NO	YES	YES	YES
Particle Size	YES	YES	YES	YES	NO	YES	NO	YES	NO	YES	YES	YES
Chemistry	YES	NO	YES	NO	NO	NO	NO	NO	YES	NO	YES	NO
Surface Area	NO	NO	YES	NO	NO	YES	NO	NO	NO	NO	NO	NO
Dispersion wet	YES	YES	NO	YES	NO	Part	NO	YES	NO	YES	YES	YES
Dispersion dry	YES	YES	NO	YES	NO	NO	NO	NO	NO	NO	YES	YES
Dispersion HH	NO	NO	NO	NO	NO	Part	NO	YES	NO	YES	NO	NO
CA 35% RH	YES	YES	NO	YES	NO	NO	NO	NO	NO	NO	NO	NO
CA 95% RH	YES	YES	NO	YES	NO	NO	NO	NO	NO	NO	NO	NO
CA HH	NO	NO	NO	NO	NO	YES	NO	Part	NO	YES	NO	NO
Proctor Test	NO	NO	NO	NO	NO	YES	NO	NO	NO	NO	NO	NO
Explosibility	YES	YES	NO	NO	YES	YES	NO	NO	NO	YES	YES	YES

Phase 1 Overview

Two commercially available stearic acid treated ground limestone products with median particle size values of 3 µm and 1.8 µm were utilized in the Phase 1 test work. A series of blends (see Appendix A for details on the blend procedure) were made using both the 3 µm and 1.8 µm treated products and an untreated limestone based rock dust that had a typical median particle size of 20 - 25 µm. The ratios of the blends were varied based on the amount of un-reacted stearic acid in the base treated samples. A total of 7 blends were prepared. Particle size, chemistry, stearic acid treatment levels, contact angle, and dust dispersion chamber testing were conducted on the 3 base samples and the 7 blends. In addition, the base and blend samples were evaluated for their effectiveness in inerting Pittsburgh pulverized coal (PPC) dust in NIOSH's 20-L chamber. All future reference to "inerting" in this report relates to results or activities associated with the NIOSH 20-L chamber testing.

It should be noted that Phase 1 of the project also included the construction and calibration of the NIOSH designed dust dispersion chamber. All future reference to "dispersion" in this report relates to results or activities associated with the NIOSH dust dispersion chamber. Details on the operational procedures associated with the dust dispersion chamber can be found in Appendix B, while details on the wicking protocol (sample preparation for testing wet and wetted, then dried samples) can be found in Appendix C.

The 3 commercial base samples utilized in Phase 1 were Kotamite® (3 µm treated), Supercoat® (1.8 µm treated) and Micro-White 100® (20 - 25 µm untreated). Utilizing the 3 base samples, the following 7 blends were prepared for Phase 1 testing (Table 3).

Table 3. Lists the 7 blend samples prepared for Phase 1 evaluations

Treated	Untreated
12.5% Kotamite	87.5% Micro-White 100
25.0% Kotamite	75.0% Micro-White 100
37.5% Kotamite	62.5% Micro-White 100
50.0% Kotamite	50.0% Micro-White 100
75.0% Kotamite	25.0% Micro-White 100
33.0% Supercoat	67.0% Micro-White 100
67.0% Supercoat	33.0% Micro-White 100

The 3 base samples and the 7 blend samples were subjected to a series of characterization tests to ensure that a) the 3 commercial base samples exhibited properties typical of those products in the market place and b) that the blend samples were accurately blended and contained the correct proportion of the untreated component. Once characterized, the 10 total samples were evaluated for contact angle, dust dispersion chamber testing and for their effectiveness in inerting PPC. See Table 4 for a complete list of Phase 1 tests.

Table 4. Illustrates the testing protocol used to evaluate the Phase 1 base and blend samples

1	2	3	4	5	6
Chemistry (base samples only)	Particle Size	Thermogravimetric Analysis	Dust Dispersion Chamber Test (Dry and Wet)	Contact Angle (35% and 95% RH)	20-L Explosion Testing

Phase 1 Results and Discussion

Chemistry – the 3 base samples were submitted to a third party for chemical analysis, primarily to obtain crystalline silica values to ensure the samples met the silica criteria defined in 30 CFR 75.2. Table 5 below documents the values obtained and confirms acceptable crystalline silica levels.

Table 5. Showing the chemical analysis of the 3 Phase 1 base samples

Bases	MgO	Al_2O_3	SiO_2	K_2O	CaO	Fe_2O_3	SrO	LOI	Crystalline Silica
Micro-White 100	1.40	0.29	1.28	0.08	53.30	0.18	0.02	43.40	0.80
Kotamite	1.28	0.20	0.77	0.05	54.70	0.11	0.02	42.90	0.60
Supercoat	1.33	0.18	1.07	0.05	54.10	0.11	ND	43.10	0.70

Particle Size and Thermogravimetric Analysis (TGA) analysis - was conducted to characterize the base samples to ensure that the particle size and stearic acid levels corresponded to values typically associated with their commercially available counterparts. As part of the characterization of the base samples both the free and total stearic acid levels were measured. The free stearic acid values were utilized to determine the treated and untreated blend ratios. Similarly particle size and TGA analysis was conducted on the blend samples, primarily to cross reference against the base samples to ensure that the targeted blend ratio had been successfully achieved.

It should be noted that the free stearic acid levels measured for the blends were lower than the expected levels based on the free stearic acid found on the base treated samples. It is theorized that a small amount of free stearic acid (~0.1 wt %) reacts with the limestone during the TGA measurement process. Table 6 tabulates all the particle size and TGA analysis associated with the Phase 1 base and blend samples and the results confirm that the blend samples were correctly proportioned.

Table 6. Phase 1 TGA, MicroTrac and wet sieve particle size analysis

BASES	TGA Total % S.A.	TGA % Free S.A.	MicroTrac D 90%	MicroTrac D 50%	MicroTrac D 10%	Wet Sieve % +200M	Wet Sieve % + 325M
MicroWhite 100	0%	0%	52.8 um	22.5 um	3.6 um	0.33%	7.57%
Kotamite	1.11%	0.40%	8.6 um	2.9 um	1.0 um	0.02%	0.04%
Supercoat	1.05%	0.16%	4.2 um	1.8 um	0.8 um	0%	0.02%
12.5% Kotamite / 87.5% Micro-White 100	0.15%	0%	49.2 um	17.9 um	2.2 um	0.30%	6.26%
25.0% Kotamite / 75.0% Micro-White 100	0.27%	0%	46.1 um	12.1 um	1.6 um	0.17%	5.51%
37.5% Kotamite / 62.5% Micro-White 100	0.42%	0.05%	46.1 um	8.7 um	1.4 um	0.21%	4.43%
50.0% Kotamite / 50.0% Micro-White 100	0.57%	0.12%	39.7 um	5.9 um	1.2 um	0.16%	3.40%
75.0% Kotamite / 25.0% Micro-White 100	0.82%	0.21%	23.6 um	3.7 um	1.1 um	0.08%	1.76%
33.0% Supercoat / 67.0% Micro-White 100	0.35%	0%	40.7 um	7.2 um	1.1 um	0.20%	4.28%
67.0% Supercoat / 33.0% Micro-White 100	0.71%	0.07%	30.2 um	2.5 um	0.9 um	0.12%	1.80%

Figure 2. MicroTrac X100

Figure 3. Perkin Elmer Pyris 1 TGA

Figure 2 illustrates the MicroTrac X100 that was utilized for the particle size analysis (see Appendix D for the operational procedure), while Figure 3 shows the Perkin Elmer Pyris 1 Thermogravimetric test equipment used throughout this study. The TGA procedure used to evaluate all the base and blend samples can be found in Appendix E. The following three figures (Figures 4 - 6) show examples of the TGA analysis and illustrate the differences between Micro-White 100 (base rock dust with no stearic acid), Kotamite (treated component with 0.4 wt % un-reacted and 0.71% reacted) and the 25% Kotamite / Micro-White 100 blend (blend with 0.0% un-reacted and 0.27% reacted) . The inclusion of Figures 4 - 6 serves to illustrate that the TGA analysis can be utilized to a) determine the presence of stearic acid and b) to determine the relative levels of both the reacted stearic acid (desirable) and un-reacted free stearic acid (undesirable), and in doing so was used as a tool to ensure that the blends were correctly proportioned (as in the case of the 25% Kotamite / 75% Micro-White 100 blend sample illustrated below). It should be noted that un-reacted stearic acid was only found in blends containing greater than 25% Kotamite.

Figure 4. Phase 1 Thermogravimetric analysis of Micro-White 100

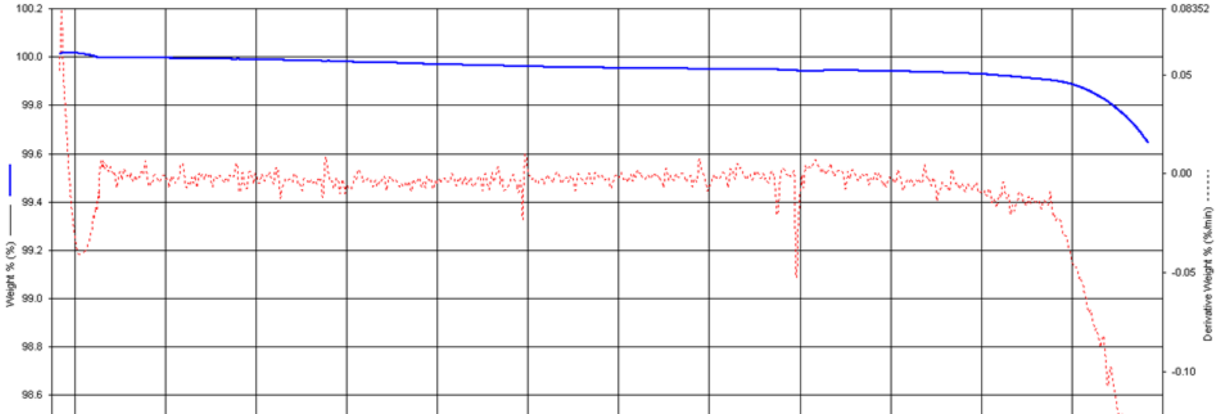


Figure 5. Phase 1 Thermogravimetric analysis of Kotamite

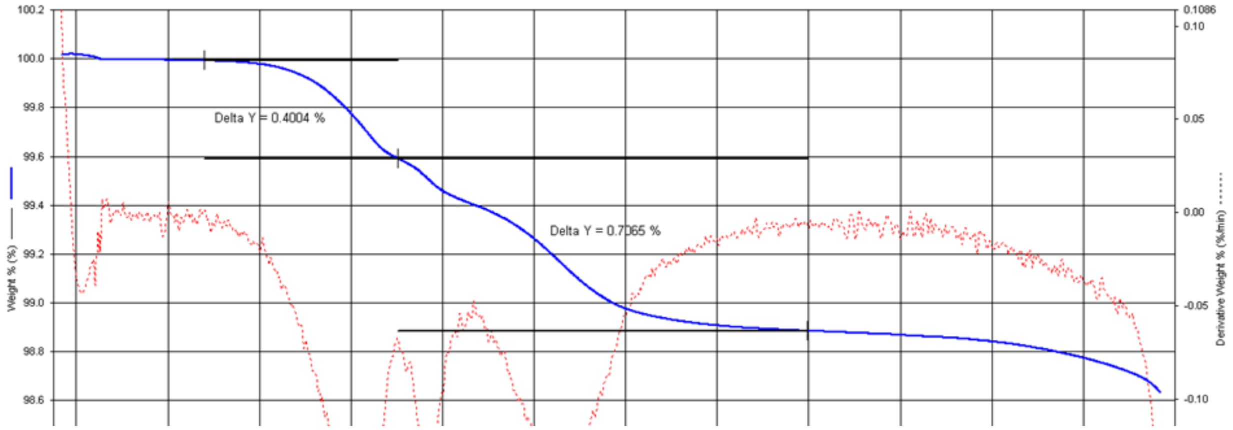
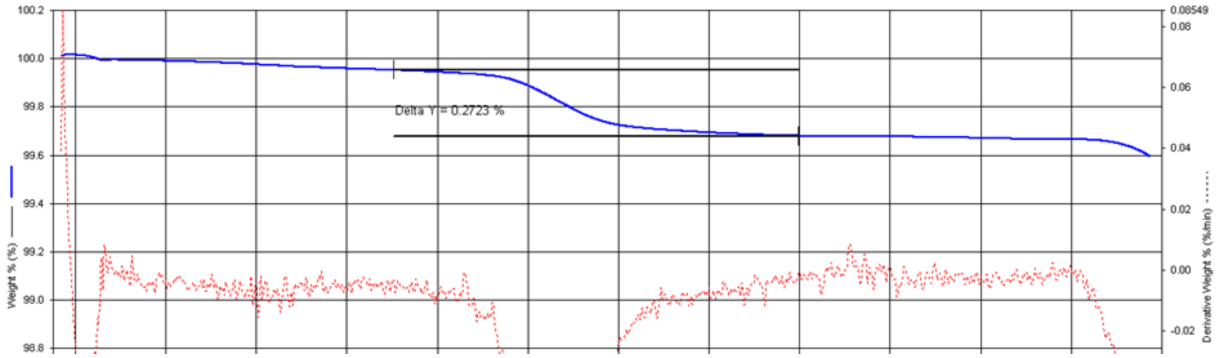


Figure 6. Phase 1 Thermogravimetric analysis of a 25% Kotamite / 75% Micro-White 100 blend



In Figures 4 – 6 above, the x axis is temperature (°C) and extends from room temperature to 650°C. The left hand y axis is % weight loss from the sample and extends from 98.0% to 102.2% and the right hand y axis is the first derivative in wt% per minute.

Dust Dispersion Chamber Testing – the Imerys dust dispersion chamber was built using the same parts and requirements as the dust dispersion chamber built by NIOSH (see Figure 7).

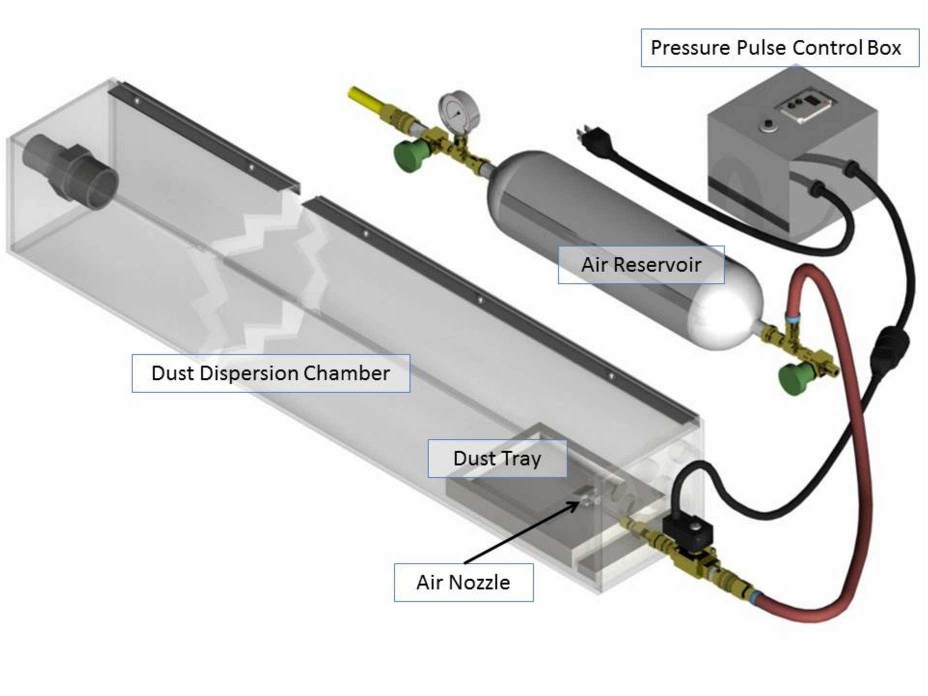


Figure 7. The Imerys dust dispersion chamber (NIOSH design)

Two rock dust samples were split and tested for dust dispersion characteristics on both the NIOSH and Imerys chambers. The same sample preparation and test procedures were used by both NIOSH and Imerys (see Figure 8 below to see an example of 'before' and 'after' the dust dispersion chamber test for a dry sample). The results (Table 7) were reviewed to determine trends. It was found that the Imerys dust dispersion chamber gave higher 'Powder Loss' values than the NIOSH chamber. It was noted that the rock dust sample with the lowest 'Powder' value with the NIOSH chamber, was also the lowest value with the Imerys chamber. It was determined that this trend of the sample values was the most important factor of the dust dispersion chamber testing, not the absolute value achieved.

Figure 8. Phase 1 dry rock dust before (left) and after (right) the dust dispersion chamber test

Table 7. Illustrates the dust dispersion chamber calibration study results

Chamber	Sample	Avg Powder Loss (g)	Avg Powder Loss (%)
NIOSH	AMC	6.23	2.85
NIOSH	Imerwhite	5.00	2.54
IMERYS	AMC	13.13	5.95
IMERYS	Imerwhite	8.83	4.23

The Phase 1 dust dispersion chamber test results (Table 8) demonstrates that under wet or damp conditions, the incorporation of a treated product improves the dispersibility of the base rock dust (Micro-White 100) versus the same base rock dust without the additive. Figure 9 below shows 'before' and 'after' the dust dispersion chamber test for a 'wet' rock dust with a treated additive. The 12.5% Kotamite / 87.5% Micro-White 100 showed the best results and closer alignment to the base Kotamite than the other blend samples. Results suggest little benefit when the treated component of the blend was utilized in excess of 25%, and as such future blends in the project were investigated at levels of 25% and below. It should be noted that the Micro-White 100 "wet" results relate to chunks and not dust being dispersed (confirming the rock dust dispersion issue).

Table 8. Dust dispersion chamber data for the Phase 1 samples

Base or Blend	Dry Avg Powder Loss (g)	Dry Avg Powder Loss (%)	Wet Avg Powder Lost (g)	Wet Avg Powder Lost (%)
Supercoat	24.18	16.62	17.26	11.84
Kotamite	30.19	17.17	20.03	11.32
Micro-White 100	21.79	11.46	2.06	1.10
12.5% Kotamite / 87.5% Micro-White 100	21.27	9.66	17.60	8.28
25.0% Kotamite / 75.0% Micro-White 100	28.05	12.93	9.96	4.63
37.5% Kotamite / 62.5% Micro-White 100	27.49	13.02	12.12	5.72
50.0% Kotamite / 50.0% Micro-White 100	28.38	13.54	15.37	7.43
75.0% Kotamite / 25.0% Micro-White 100	25.84	13.30	13.31	7.21
33.0% Supercoat / 67.0% Micro-White 100	26.98	14.53	8.72	4.64
67.0% Supercoat / 33.0% Micro-White 100	26.85	16.34	25.40	15.36

Figure 9. Phase 1 Rock dust 'before' (left) and 'after' (right) with the treated additive after being wetted and subjected to the dust dispersion chamber test

Contact Angle measurements – were utilized to assess the hydrophobic nature of the blend samples and to determine their relative performance in conjunction with the dust dispersion chamber test. The contact angle procedure requires that the rock dust blend samples be pressed into "tablets" using 18 tonne per 2 minutes in a XRF mold. The tablets were equilibrated for 60 hours at both 35% and 98% relative humidity. The droplet size used was 40 ml de-ionized water. A photograph of the contact angle test equipment is shown in Figures 10 and a close up of the contact angle test chamber is shown in Figure 11. The test procedure is outlined in Appendix F.

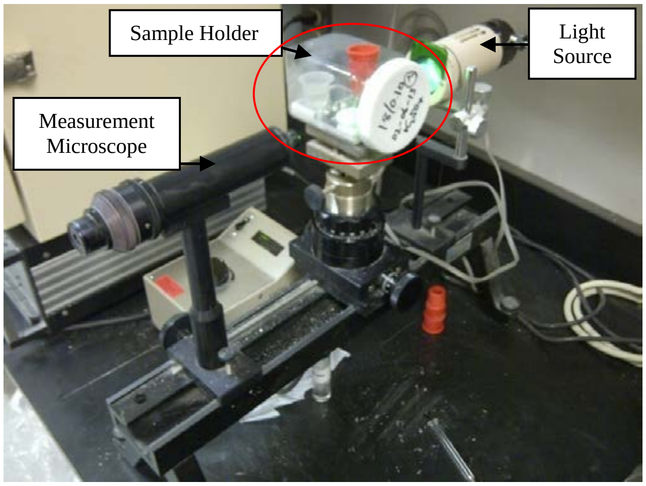


Figure 10. Contact Angle test equipment

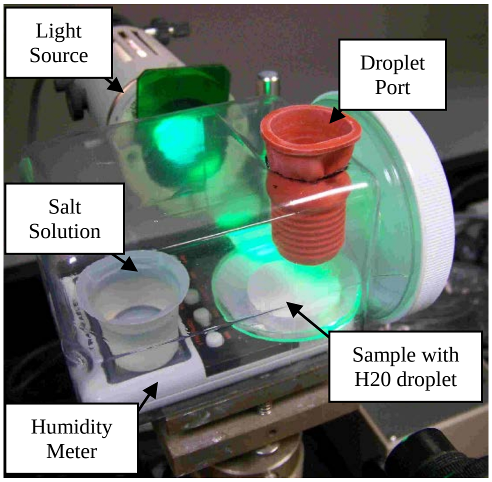


Figure 11. Contact Angle sample holder

For both the base and blend samples, contact angles were measured over time. To judge the effect of relative humidity, contact angles were also measured on polyethylene film. It was observed that the circumference of the water droplet on polyethylene film reduces over time whereas on the pressed powders it remains more or less constant (as judged from the drop diameter). It was concluded that on plastic the contact angle is not as good an indicator of droplet size as it is on powders. Figure 12 shows both contact angle and droplet diameter data for the polyethylene film. Both parameters fall with time, which indicates a reducing droplet volume due to evaporation.

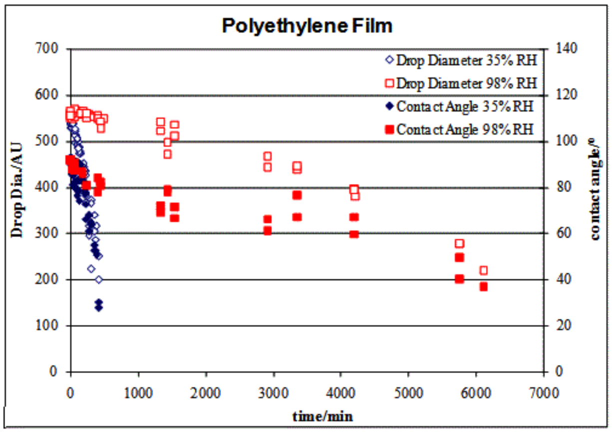


Figure 12. Phase 1 Contact angle and droplet diameter measurements on polyethylene film

In general, the pressed powders showed a constant drop diameter but a steady reduction in contact angle, so contact angle alone is a good indicator of droplet size. For the untreated Micro-White 100, the water droplet was immediately absorbed into the pellet and no contact angle measurement was possible. Figure 13 shows the contact angle of water droplets on pressed tablets of the additives Kotamite and Supercoat as a function of time at two different humidities (98% and 35%). The initial contact angles at both humidities remains high (>100°), indicating a very hydrophobic surface. At high humidity the contact angle remains high (>80°) for times up to 2,000 minutes. By contrast, at low humidity the contact angle declines much more rapidly and is <80° within 300 minutes and is <40° within 400 minutes. It is theorized at low humidities water evaporation causes the droplet size, and therefore contact angle, to decrease rapidly.

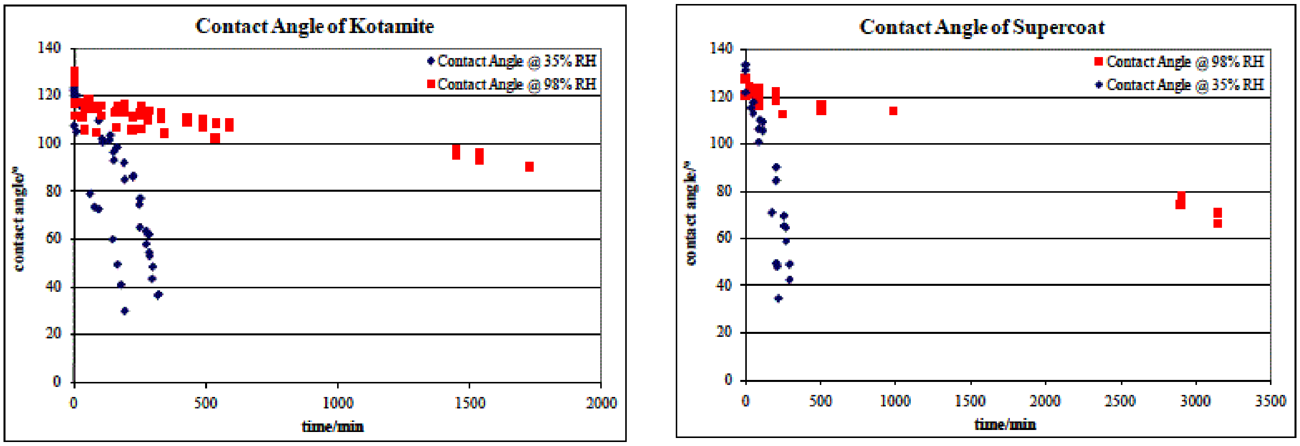


Figure 13. Phase 1 Contact angle measurements for Kotamite (left) and Supercoat (right)

Figures 14A and 14B illustrate the graphs associated with the Kotamite/Micro-White 100 blends. The results are very similar for all blends. The initial contact angles are >100° at both humidities, and remains high (>80°) to 2,000 minutes at high humidity. The 7.5% blend being the only exception where the contact angle dropped below 80° after 500 minutes. The same rapid reduction in contact angle is observed at low humidity and angles of <80° are noted within 200 minutes for all blends. In general, the Phase 1 contact angle analysis indicated that the blends with the higher percentage of Kotamite retained their contact angle slightly longer, though there was large scatter in the results.

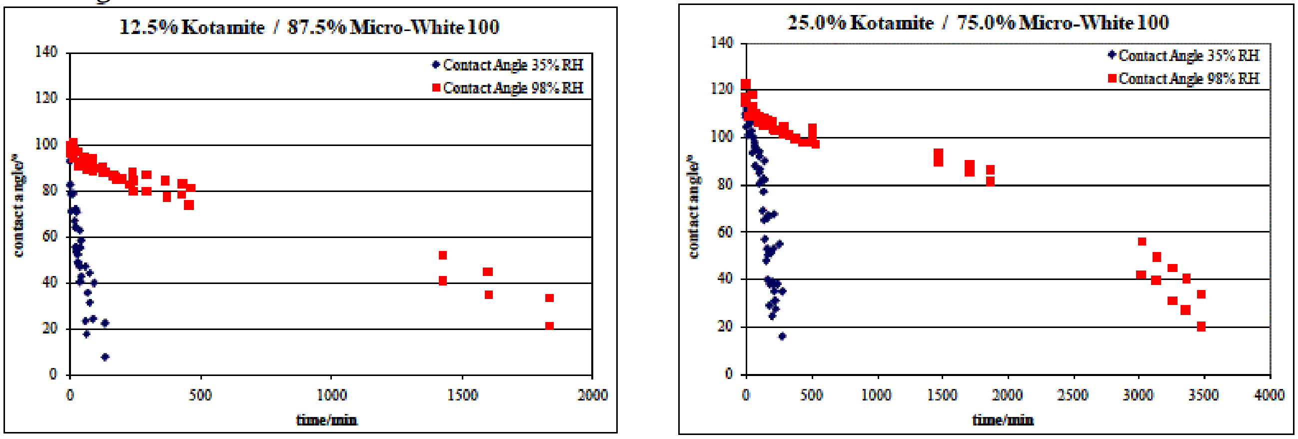


Figure 14A. Phase 1 Contact angle measurements for 2 Kotamite/Micro-White 100 blends

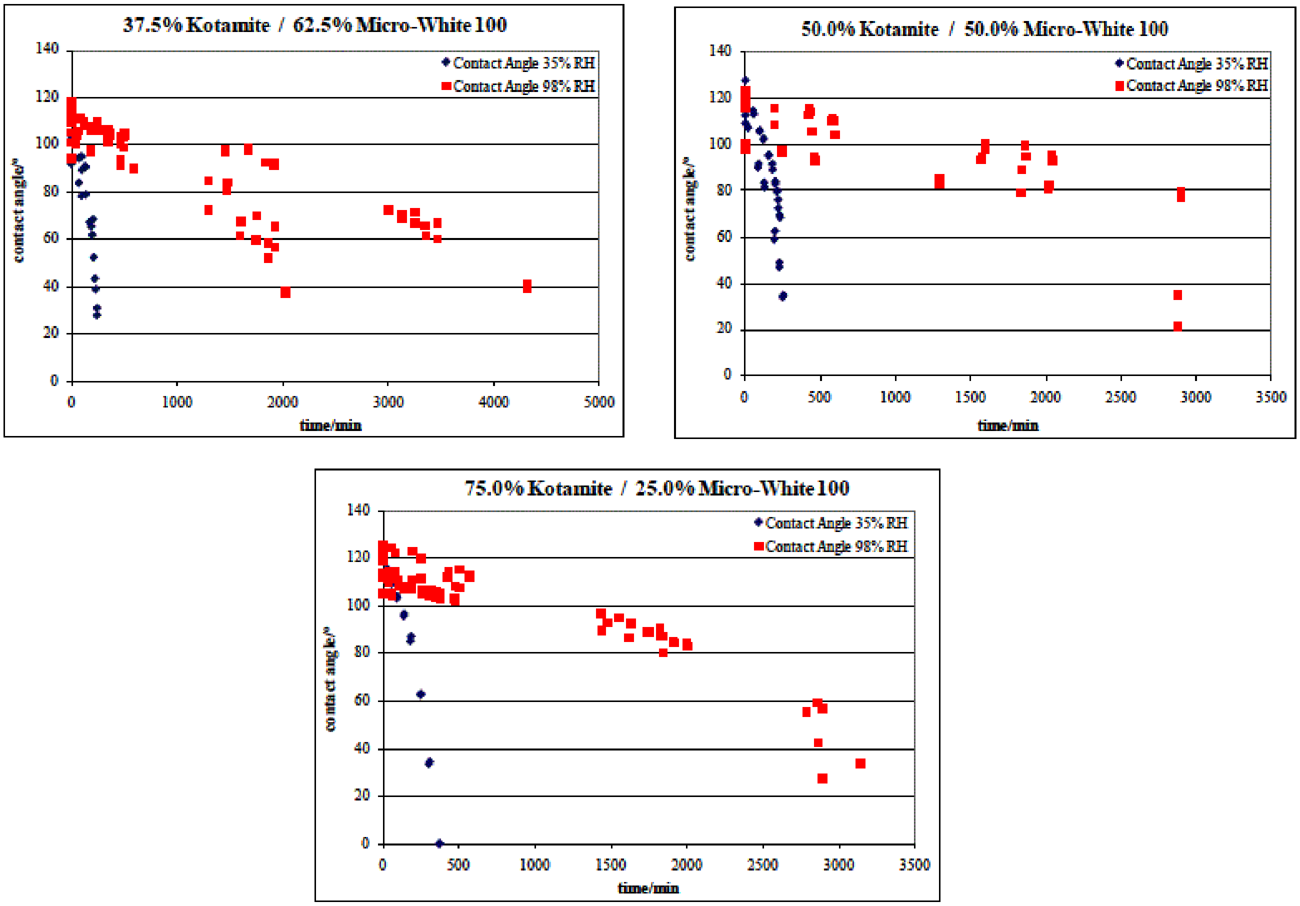


Figure 14B. Phase 1 Contact angle measurements for 3 Kotamite/Micro-White 100 blends

Figure 15 illustrate the graphs associated with the Supercoat/Micro-White 100 blends. The results are very similar to those obtained for the Kotamite blends. The 35% humidity value for the 67% Supercoat/33% Micro-White 100 blends was not measured. The initial contact angles are >100° at both humidities, and remains high (>80°) to 1,000 minutes at high humidity. The same rapid reduction in contact angle at low humidity was observed for the 33% Supercoat/67% Micro-White 100 and angles of <80° are observed within 200 minutes.

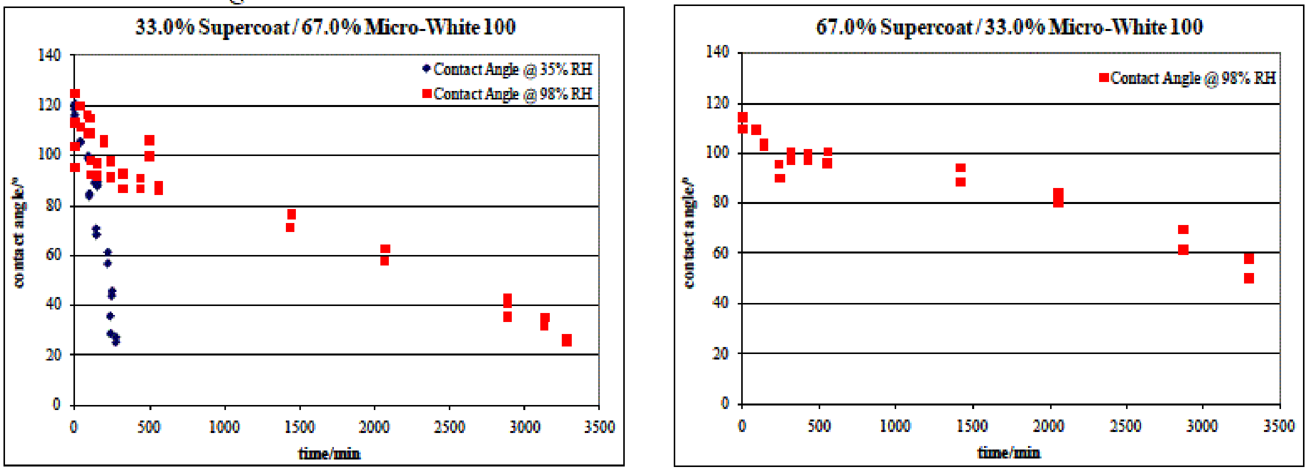


Figure 15. Phase 1 Contact angle measurements for 2 Supercoat/Micro-White 100 blends

Explosibility Testing – the 10 rock dust samples were supplied to NIOSH to determine the inerting effectiveness of the untreated and treated rock dusts through 20-L explosibility chamber testing. Three were the untreated base rock dusts and 7 were treated blends. The blends were supplied as "premixed". All samples received correlated to the base and blend samples tested in Phase 1.

The rock dust samples were tested "as-received" and the PPC was dried in a desiccator for at least 24 hr before use. PPC is a pulverized coal dust with approximately 77% < 200 mesh (< 75 µm) and 36% < 400 mesh (< 38 µm).

The NIOSH 20-L explosibility chamber (Figures 16 and 17) is an ASTM standard testing apparatus [ASTM E1515 2010]. It can be used to measure the explosibility of any dust. This chamber has been described in detail elsewhere [Cashdollar 1996, 2000].

For each experiment, a single 5,000 J electrically-activated pyrotechnic ignitor was used as the ignition source. The PPC and inerting dusts were carefully weighed and thoroughly mixed before being placed at the bottom of the 20-L chamber. Initial mixtures were typically 75% rock dust and 25% coal dust. These ratios would then be changed, generally in 5% increments, during subsequent experiments depending on whether the dust mixture exploded or not. The main objective of this research was to determine the concentration of rock dust required to inert PPC. The relative reactivity of the dust can be estimated from either the peak explosion pressure or the maximum rate of the pressure rise. Based on the past research, a pressure rise of 1 bar or greater was used as the criterion for determining an explosion (a pressure ratio of 2). The pressure ratio would account for the variations in atmospheric pressure. This is in accordance with the ASTM standard for measuring the explosibility of dust clouds [ASTM E1515 2010]. 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 PR is the final maximum chamber pressure obtained during the test divided by the initial chamber pressure just prior to ignition (atmospheric pressure or approximately 14.7 psia). For the tests with a PR < 2, the rock dust inerted the PPC; for test results with a PR > 2, the rock dust did not inert the PPC. Coal concentrations in the range 200-600 g/m^3 (in 200 g/m^3 increments) were used in this study. Non-explosions were repeated at each coal concentration to confirm a successful inertization (PR < 2; i.e., no explosion at any of the coal concentrations).

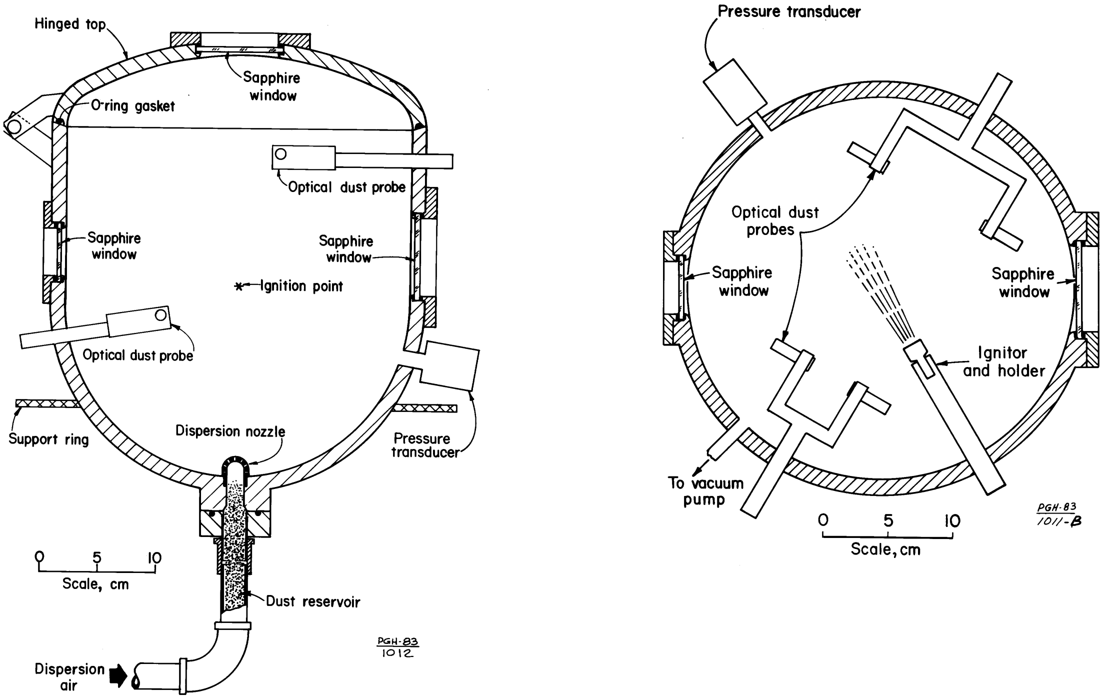


Figure 16. Vertical and horizontal cross sections of the 20-L explosibility chamber

Figure 17. Inside view of the 20-L chamber

The results at various rock dust (RD) concentrations from the 20-L chamber are summarized in Table 9. The word “go” indicates a deflagration and the word “no” indicates a non-explosion or the inhibitor was successful at preventing an explosion.

Table 9. Summary of the explosion inerting experiments in the NIOSH 20-L chamber on the Phase 1 samples

Bases and Blend	65% RD	70% RD	75% RD
Micro-White 100	dnt	go	no
Kotamite	dnt	go	no
Supercoat	go	no	dnt
12.5% Kotamite / 87.5% Micro-White 100	dnt	go	no
25.0% Kotamite / 75.0% Micro-White 100	dnt	go	no
37.5% Kotamite / 62.5% Micro-White 100	dnt	go	no
50.0% Kotamite / 50.0% Micro-White 100	go	no	dnt
75.0% Kotamite / 25.0% Micro-White 100	dnt	go	no
33.0% Supercoat / 67.0% Micro-White 100	go	no	dnt
67.0% Supercoat / 33.0% Micro-White 100	go	no	dnt

Go = explosion, No = no explosion, dnt = did not test.

The results show the rock dust samples to be favorable as they all appear to be as effective at inerting PPC as the regular limestone rock dust which is used as the standard inhibitor at NIOSH for both laboratory-scale testing and full-scale experimental explosion testing. Cashdollar [2000] showed that about 75% of regular limestone rock dust is required to inert PPC. This has been confirmed more recently by Man and Harris [2013].

The results also show that the Supercoat rock dust is a slightly better inhibitor as it only requires 70% to inert PPC compared with 75% for both the Micro-White 100 and Kotamite. This may be attributed to the Supercoat's smaller particle size. The D50 values of Micro-White 100 and Kotamite are 22.5 µm and 2.9 µm respectively but for Supercoat it is only 1.8 µm.

As expected, most of the blends of Micro-White 100 and Kotamite behaved the same as the individual blend components. However, the 50:50 blend was shown to be slightly more effective and only required 70% to inert PPC. The blends of Micro-White 100 and Supercoat appeared to perform about the same as pure Supercoat.

Overall Observations from Phase 1

The characterization (chemical analysis, TGA, and particle size analysis) of the 3 commercially available base samples verified the values of the samples to be consistent with those typically seen in the market place. Seven blend samples were prepared (5 Kotamite/Micro-White 100 samples and 2 Supercoat/Micro-White 100 samples) based on the free stearic acid values obtained from the base sample characterization.

The characterization (TGA and particle size analysis) of the blend samples confirmed that the samples were accurately blended. However, it was observed that the free stearic acid levels measured for the blends were lower than the expected levels based on the free stearic acid found on the base treated samples. It is theorized that a small amount of free stearic acid (~0.1 wt%) reacts with the limestone during the TGA measurement process. It should also be noted that the total stearic acid levels for the blends are in agreement with the projected total stearic acid levels (from the values obtained from the base samples).

Contact angle measurements of the 3 base samples were conducted at two different relative humidity (RH) values (35% and 98%). Results for the 35% RH tests seemed to be affected by evaporation of the water droplet. No contact angle measurements could be made on the base rock dust (Micro-White 100) as the water droplet was instantly absorbed by the Micro-White 100. In general, the contact angle analysis indicated that the blends with the higher percentage treated products retained their contact angle longer, though there was large scatter in the results. At 98% RH, regardless of the type or amount of treated additive used, all the blends demonstrated hydrophobicity. However, no obvious correlation could be derived when comparing the contact angle results to those from the dust chamber dispersion test.

Results obtained from the dust dispersion chamber calibration analysis showed the Imerys chamber values to be higher than those obtained from NIOSH for the same samples. This was primarily due to variations in the air nozzle pressure and the angle of the nozzle in relation to the dust tray. However, absolute agreement was not necessary as the dust dispersion chamber was used to show the relative dispersibility of dry, wet, and wetted then dried treated and untreated rock dusts. In all cases, the treated products resulted in a larger powder loss from the tray as the additives enhanced dispersion. The dust dispersion chamber test results demonstrate that under wet or damp conditions, the incorporation of a treated product improves the dispersibility of the base rock dust (Micro-White 100) versus the same base rock dust without the additive. Results suggest little benefit when the treated component of the blend is in excess of 25%. The 12.5% Kotamite/87.5% Micro-White 100 showed the best results and closer alignment to the base Kotamite than the other blend samples.

At the highest level of free stearic acid (0.4 wt%) found on the treated blend component (Kotamite), the treated blend component alone abated the explosive nature of coal dusted as tested in NIOSH’s 20-L chamber (at 75 wt% rock dust). Each of the base and blended rock dusts when mixed with the Pittsburgh pulverized coal dust (at coal concentrations of 200, 400, and 600 g/m^3) inerted the coal dust within the 20-L chamber at dust mixtures of 75% or less rock dust.

Phase 2A Results and Discussion

Based on the results from Phase 1, a second series of blends were made where various untreated limestone products with different median particle sizes (6 µm to ~40 µm) were blended with the two treated products described previously. The median particle sizes of the coarse untreated products were chosen based on their potential to particle pack with the median particle size of the specific treated product to be used in that blend.

In Phase 2A the five commercial base samples utilized were RO-40™ (30 - 40 µm untreated), #8 White™ (25 - 30 µm untreated), Micro-White 100® (20 - 25 µm untreated), #10 White™ (~15 µm untreated) and Drikalte® (~8 µm untreated). As in Phase 1 the treated additives were Kotamite® (3 µm treated) and Supercoat® (1.8 µm treated). A total of 24 blends were prepared. Based on the results of the dust dispersion chamber testing in Phase 1 where no significant advantage was seen with treated additive levels above 25% it was decided to narrow the Phase 2 testing to 7.5% - 25% treated additives. As in Phase 1, particle size, stearic acid treatment levels (TGA), contact angle, and dust dispersion chamber testing were conducted on all 24 blends. Table 10 lists the 24 blends investigated in Phase 2A.

Table 10. Lists the 24 blend samples prepared for Phase 2A evaluations

Treated	Untreated
7.50% Kotamite	92.5% RO-40
12.5% Kotamite	87.5% RO-40
25.0% Kotamite	75.0% RO-40
7.50% Kotamite	92.5% #8 White
12.5% Kotamite	87.5% #8 White
25.0% Kotamite	75.0% #8 White
7.50% Kotamite	92.5% Micro-White 100
12.5% Kotamite	87.5% Micro-White 100
25.0% Kotamite	75.0% Micro-White 100
7.50% Supercoat	92.5% RO-40
12.5% Supercoat	87.5% RO-40
25.0% Supercoat	75.0% RO-40

Treated	Untreated
7.50% Supercoat	92.5% #8 White
12.5% Supercoat	87.5% #8 White
25.0% Supercoat	75.0% #8 White
7.50% Supercoat	92.5% Micro-White 100
12.5% Supercoat	87.5% Micro-White 100
25.0% Supercoat	75.0% Micro-White 100
7.5% Supercoat	92.5% #10 White
12.5% Supercoat	87.5% #10 White
25.0% Supercoat	75.0% #10 White
7.5% Supercoat	92.5% Drikalite
12.5% Supercoat	87.5% Drikalite
25.0% Supercoat	75.0% Drikalite

Chemistry – the 7 base samples were submitted to a third party for chemical analysis, primarily to obtain crystalline silica values to ensure the samples met the silica criteria defined in 30 CFR 75.2. Table 11 below documents the values obtained and confirms acceptable crystalline silica levels.

Table 11. Chemical analysis of the 7 Phase 2A base samples

Base or Blend	MgO	Al_2O_3	SiO_2	K_2O	CaO	Fe_2O_3	SrO	LOI	Crystalline Silica
Kotamite	1.28	0.20	0.77	0.05	54.70	0.11	0.02	42.90	0.60
Supercoat	1.33	0.18	1.07	0.05	54.10	0.11	ND	43.10	0.70
RO-40	1.49	0.23	1.36	ND	54.10	0.15	0.12	42.40	0.20
#8 White	1.34	0.37	1.37	0.09	53.60	0.16	0.02	43.00	0.70
Micro-White 100	1.40	0.29	1.28	0.08	53.30	0.18	0.02	43.40	0.80
#10 White	1.14	0.30	1.15	0.06	54.10	0.14	0.02	43.10	0.80
Drikalite	1.45	0.41	1.84	0.10	53.40	0.16	ND	42.60	1.00

Particle Size and Thermogravimetric Analysis (TGA) analysis - Table 12 shows the TGA and MicroTrac particle size test results for the base rock dusts and blends that were developed for the Phase 2A evaluation. There were no measureable free stearic acid levels for the blends samples. This observation was in agreement with those observations in Phase 1 whereby at the lower treated blend levels (<25%) no free stearic acid was detected. The TGA and MicroTrac particle results demonstrate that the proper blend ratios were obtained and that the blends have been adequately mixed.

Table 12. Phase 2A TGA, MicroTrac and wet sieve particle size analysis

Base or Blends	TGA % Total S.A.	TGA % Free S.A.	MicroTrac D 90%	MicroTrac D 50%	MicroTrac D 10%	Wet Sieve % +200M	Wet Sieve % + 325M
Kotamite	1.11%	0.40%	8.6 um	2.9 um	1.0 um	0.02%	0.04%
Supercoat	1.05%	0.16%	4.2 um	1.8 um	0.8 um	0	0.02%
RO-40	0	0	135.8 um	37.3 um	4.0 um	29.99%	50.09%
#8 White	0	0	65.2 um	27.0 um	4.3 um	2.84%	21.82%
MicroWhite 100	0	0	52.8 um	22.5 um	3.6 um	0.33%	7.57%
#10 White	0	0	37.4 um	17.0 um	3.1 um	0.02%	0.66%
Drikalite	0	0	29.2 um	9.8 um	1.6 um	Tr	0.25%
7.50% Kotamite / 92.5% RO-40	0.09%	0	136.1 um	35.2 um	2.8 um	27.26%	45.91%
12.5% Kotamite / 87.5% RO-40	0.14%	0	123.0 um	29.5 um	2.3 um	25.95%	43.25%
25.0% Kotamite / 75.0% RO-40	0.21%	0	117.1 um	18.3 um	1.7 um	22.21%	37.24%
7.50% Kotamite / 92.5% #8 White	0.07%	0	69.1 um	25.2 um	3.1 um	2.74%	18.81%
12.5% Kotamite / 87.5% #8 White	0.11%	0	66.0 um	22.1 um	2.5 um	2.66%	18.38%
25.0% Kotamite / 75.0% #8 White	0.20%	0	64.5 um	16.8 um	1.9 um	2.27%	15.36%
7.50% Kotamite / 92.5% Micro-White 100	0.08%	0	53.0 um	20.9 um	2.8 um	0.34%	7.81%
12.5% Kotamite / 87.5% Micro-White 100	0.12%	0	50.9 um	18.3 um	2.3 um	0.34%	7.41%
25.0% Kotamite / 75.0% Micro-White 100	0.20%	0	45.3 um	12.8 um	1.7 um	0.24%	6.12%
7.50% Supercoat / 92.5% RO-40	0.10%	0	131.7 um	34.9 um	2.3 um	27.47%	45.58%
12.5% Supercoat / 87.5% RO-40	0.15%	0	124.6 um	28.1 um	1.7 um	26.35%	43.69%
25.0% Supercoat / 75.0% RO-40	0.26%	0	90.1 um	13.9 um	1.2 um	22.61%	37.34%
7.50% Supercoat / 92.5% #8 White	0.09%	0	68.0 um	25.4 um	2.5 um	2.73%	18.55%
12.5% Supercoat / 87.5% #8 White	0.14%	0	64.8 um	22.6 um	1.8 um	2.64%	18.46%
25.0% Supercoat / 75.0% #8 White	0.26%	0	59.2 um	14.3 um	1.2 um	2.35%	15.64%
7.50% Supercoat / 92.5% Micro-White 100	0.09%	0	52.8 um	21.6 um	2.4 um	0.32%	7.62%
12.5% Supercoat / 87.5% Micro-White 100	0.14%	0	52.5 um	18.9 um	1.7 um	0.32%	7.10%
25.0% Supercoat / 75.0% Micro-White 100	0.26%	0	50.6 um	13.6 um	1.3 um	0.22%	5.81%
7.50% Supercoat / 92.5% #10 White	0.10%	0	37.2 um	16.0 um	2.1 um	0.01%	0.19%
12.5% Supercoat / 87.5% #10 White	0.14%	0	37.1 um	14.6 um	1.7 um	0.01%	0.19%
25.0% Supercoat / 75.0% #10 White	0.26%	0	34.9 um	10.2 um	1.3 um	0.01%	0.21%
7.5% Supercoat / 92.5% Drikalite	0.10%	0	28.0 um	8.0 um	1.3 um	Tr	Tr
12.5% Supercoat / 87.5% Drikalite	0.17%	0	27.2 um	7.2 um	1.2 um	Tr	Tr
25.0% Supercoat / 75.0% Drikalite	0.26%	0	26.9 um	5.5 um	1.1 um	Tr	Tr

Dust Dispersion Chamber Testing - Table 13 shows the dust dispersion chamber test results for the blends of the three base rock dusts blended with the “Kotamite” treated product, and five base rock dusts blended with the “Supercoat” treated product, a total of eight blend sets. Each blend set consisted of three different blend ratios containing 7.5%, 12.5% and 25.0% of the treated product by weight. The results demonstrated that under wet or damp conditions, all of the blends dispersed. The results for each blend did vary and were found within the range of 2.3% to 8.1% for the average mass lost of the “Wet” dispersion test. Results for the “Wet” dust chamber dispersion test suggest little benefit when the treated component of the blend is in excess of 12.5%.

Table 13. Dust dispersion chamber data for the Phase 2A samples

Blends	Dry Avg Powder Loss (g)	Dry Avg Powder Loss (%)	Wet Avg Powder Lost (g)	Wet Avg Powder Lost (%)
7.50% Kotamite / 92.5% RO-40	20.36	8.08	5.84	2.37
12.5% Kotamite / 87.5% RO-40	21.97	8.85	8.05	3.21
25.0% Kotamite / 75.0% RO-40	21.11	8.59	8.39	3.38
7.50% Kotamite / 92.5% #8 White	26.98	11.87	14.67	6.58
12.5% Kotamite / 87.5% #8 White	24.87	11.25	12.38	5.59
25.0% Kotamite / 75.0% #8 White	26.93	11.97	8.88	3.91
7.50% Kotamite / 92.5% Micro-White 100	25.98	12.33	13.34	6.29
12.5% Kotamite / 87.5% Micro-White 100	26.39	12.45	12.87	6.09
25.0% Kotamite / 75.0% Micro-White 100	24.72	11.61	7.93	3.63
7.50% Supercoat / 92.5% RO-40	19.44	8.64	8.18	3.47
12.5% Supercoat / 87.5% RO-40	21.99	9.27	7.81	3.28
25.0% Supercoat / 75.0% RO-40	16.95	7.41	6.83	3.01
7.50% Supercoat / 92.5% #8 White	25.84	12.15	10.92	5.08
12.5% Supercoat / 87.5% #8 White	24.14	11.77	12.26	6.03
25.0% Supercoat / 75.0% #8 White	24.84	12.41	9.25	4.69
7.50% Supercoat / 92.5% Micro-White 100	24.06	11.87	10.74	5.29
12.5% Supercoat / 87.5% Micro-White 100	25.63	13.28	10.52	5.37
25.0% Supercoat / 75.0% Micro-White 100	25.67	13.36	8.77	4.58
7.50% Supercoat / 92.5% #10 White	25.24	13.40	11.32	6.12
12.5% Supercoat / 87.5% #10 White	23.93	13.38	14.22	8.07
25.0% Supercoat / 75.0% #10 White	26.18	14.81	9.82	5.64
7.50% Supercoat / 92.5% Drikalite	24.85	16.03	10.75	7.04
12.5% Supercoat / 87.5% Drikalite	23.24	15.60	10.18	6.83
25.0% Supercoat / 75.0% Drikalite	22.44	15.94	9.22	6.65

Contact Angle Measurements - Figure 18 illustrate the graphs associated with the contact angle of water droplets on pressed tablets of Kotamite with RO-40, #8 White and Micro-White 100 at 98% relative humidity as a function of time. Each blend series contained 25%, 12.5% and 7.5% Kotamite. The initial contact angles at 25% Kotamite for each of the three series was >110° and remains high (>80°) to a minimum of 1,200 minutes. Two Kotamite / Micro-White 100 series were evaluated due to the relative humidity chamber being knocked during measurement, possibly causing the droplets to ‘spread’. The 12.5% blends of Kotamite with RO-40, #8 White and Micro-White 100 showed more scatter with regards to contact angle. The initial contact angles was >95° for RO-40 and #8 White and remained high (>80°) for 500 and 900 minutes respectively. The 12.5% blends of Kotamite with Micro-White 100 showed an initial contact of only 60° - 90° and dropped to less than 20° with 400 minutes. The 7.5% blends of Kotamite with RO-40, #8 White and Micro-White 100 all showed very low initial contact angle (0° - 70°), and all dropped to <20° in less than 50 minutes. In general the graphs depict a ranking of 25% >12.5% >7.5% Kotamite with hydrophobicity (as measured by contact angle) dropping off between 12.5% and 7.5% Kotamite.

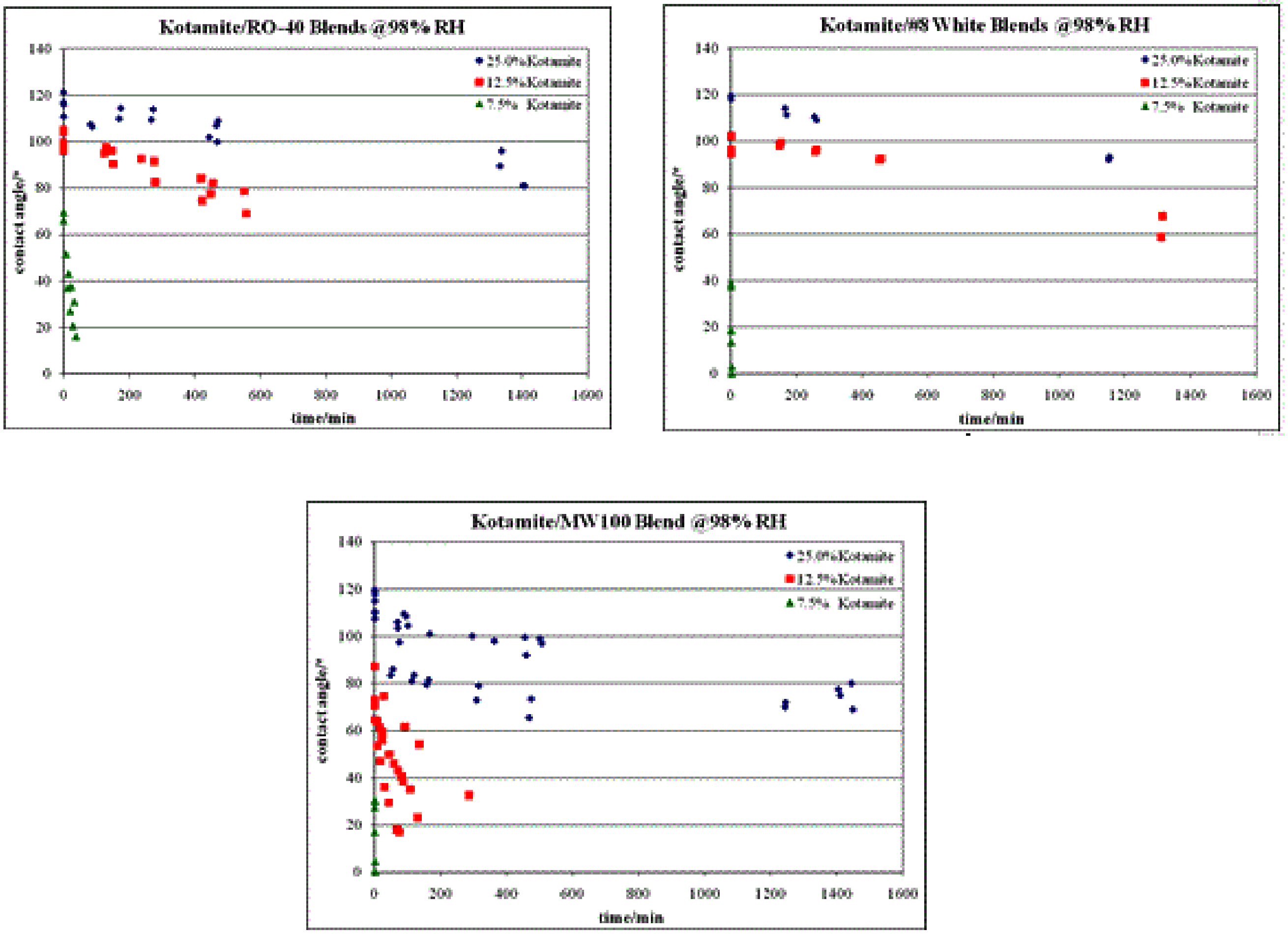


Figure 18. Phase 2A Contact angle measurements for 3 Kotamite/ rock dusts blends

Figure 19 illustrate the graphs associated with the contact angle of water droplets on pressed tablets of Supercoat with RO-40, #10 White, #8 White, Drikalite and Micro-White 100 at 98% relative humidity as a function of time. Each blend series contained 25%, 12.5% and 7.5% Supercoat. The initial contact angles at 25% Supercoat for each of the five series was >100° and remained high (>80°) ranging from 800 minutes (#10 White) to 1,500 minutes (#8 White). There was more variability at the 12.5% Supercoat loading level with initial contact angle values ranging from 100° - 120° (RO-40) to 45° (Drikalite). The 12.5% blends of Supercoat with RO-40 and #8 White maintained their contact angles the longest, >80° at 1,200 minutes. The contact angle for the remaining three series containing #10 White, Drikalite and Micro-White 100 fell quicker, <80° in 200 – 400 minutes. All the 7.5% blends of Supercoat with the five base rock dusts showed very low initial contact angles and all dropped to 0° in a few minutes. The exception being the Supercoat / RO-40 series which showed an initial contact angle of 80° - 90° which dropped to ~40° after 400 – 600 minutes. In general the graphs depict a ranking of 25% >12.5% >7.5% Supercoat with hydrophobicity (as measured by contact angle) dropping off between 12.5% and 7.5% Supercoat, and in the case of Drikalite between 25% and 12.5%.

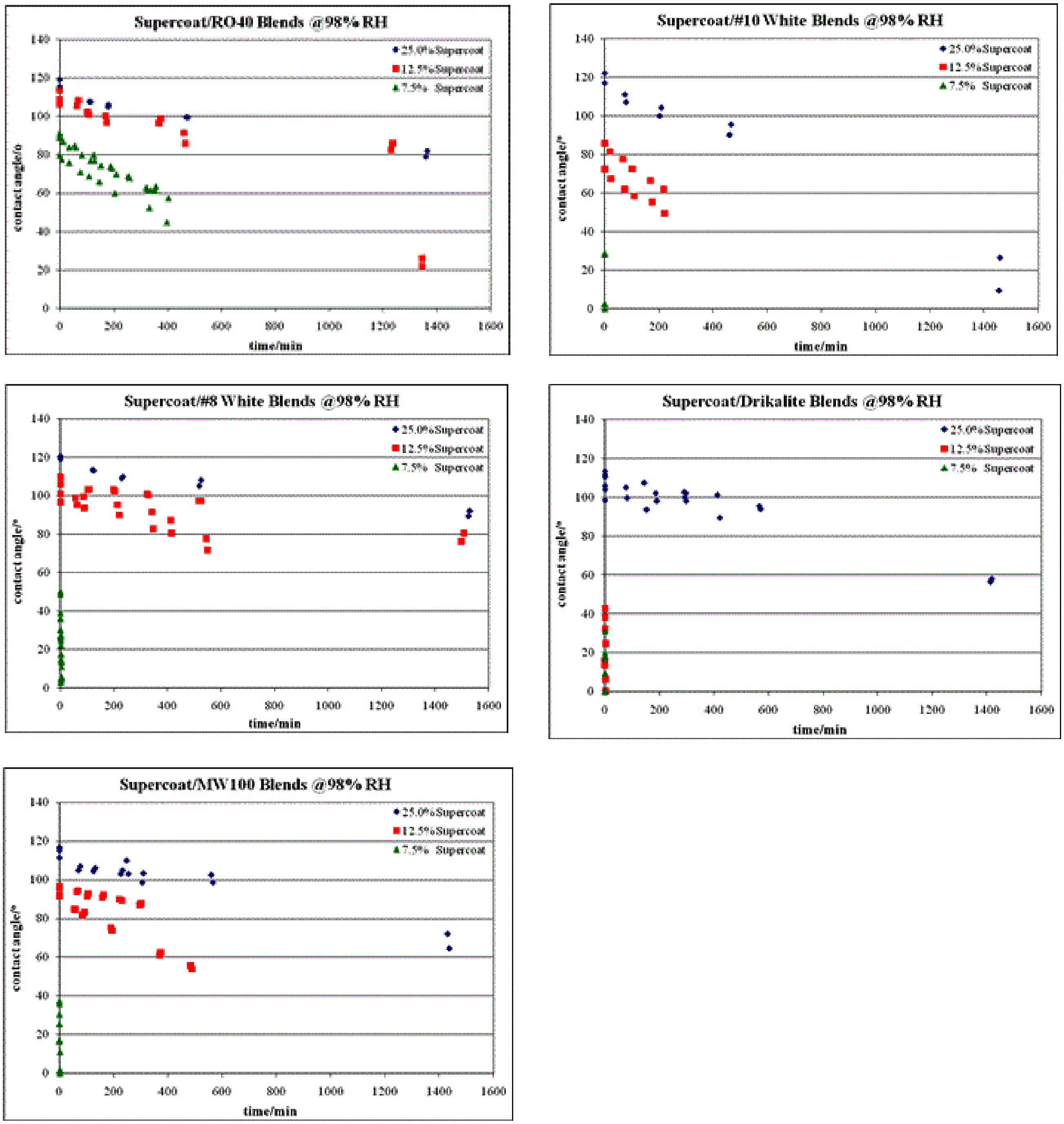


Figure 19. Phase 2A Contact angle measurements for 5 Supercoat/base rock dust blends

Observations from Phase 2A

The characterization (chemical analysis, TGA, and particle size analysis) of the 7 commercially available base samples verified the values of the samples to be consistent with those typically seen in the market place. Twenty four blend samples were prepared (3 Kotamite series and 5 Supercoat series) based on particle packing and the observations found in Phase 1 (i.e. that treated blend component levels greater than 25 wt% offered no additional dispersion benefit). The characterization (TGA and particle size analysis) of the blend samples confirmed that the samples were accurately blended.

Contact angle measurement and dust dispersion chamber testing of the base samples were not conducted as they were tested and reported upon in Phase 1. In general, the contact angle analysis (at 98% RH) indicated that the blends with the higher percentage treated products retained their contact angle longer (25% >12.5% >7.5%). As the treated blend component percentage approached 7.5%, the contact dropped dramatically. Despite the observed trend with the contact angle, no direct correlation with the dust dispersion chamber test could be ascertained.

As found in Phase 1, improved dust dispersion was obtained when the treated component was present. Overall there is some variability in the dispersion results and this can be attributed to the placement of the nozzle and the settling characteristics of the rock dust in the tray prior to initiation of the test. Results illustrate a slight advantage in dispersion characteristic with the blends containing the finer base rock dusts. This observation might be linked with particle packing and additional work would be required to understand this further. Blend ratios containing greater than 12.5 wt% treated component do not offer significant dispersion advantages. It was observed that blend ratios with lower treatment levels could be adopted without sacrificing dispersibility.

After reviewing the particle size data and discussing the data with NIOSH, it was agreed to eliminate the RO-40 series from the testing protocol (too coarse) and also the Drikalite series as it offered no performance advantage over #10 White. In an effort to further optimize the blend components and ratios, it was agreed with NIOSH to pursue a further nine blend samples that would form the basis of the Phase 2B work.

Phase 2B Results and Discussion

Based on the results from Phase 2A, 9 (nine) further blend samples were prepared for evaluations. RO-40 was eliminated as a result of being too coarse and the Drikalite was eliminated based on it being potentially too costly to the industry and did not offer any substantial benefits over #10 White. Furthermore Phase 2A dust dispersion chamber test results suggested that blends containing 12.5% or less of a treated component could be utilized without sacrificing dispersibility. Table 14 lists the nine samples that were prepared for Phase 2B. In addition to the standard characterization tests previously listed, these blends were subjected to contact angle and dust dispersion chamber testing **under high humidity for an extended time period (up to 3 weeks),** and Brunauer, Emmett and Teller (BET) surface area. In addition, these samples were tested against ASTM D698 (Proctor Test) and subjected to 20-L explosibility chamber testing.

Table 14. Lists the 9 blend samples prepared for Phase 2B evaluations

Treated	Untreated
7.50% Kotamite	92.5% #8 White
10.0% Kotamite	90.0% #8 White
12.5% Kotamite	87.5% #8 White
12.5% Kotamite	87.5% Micro-White 100

Treated	Untreated
7.50% Supercoat	92.5% #8 White
10.0% Supercoat	90.0% #8 White
12.5% Supercoat	87.5% #8 White
10.0% Supercoat	90.0% #10 White
12.5% Supercoat	87.5% #10 White

Surface Area - Table 15 provides the surface area comparison of the base rock dusts and blends being evaluated in Phase 2B. The surface area analysis was conducted using the nitrogen BET measurement method. The surface area results of the base untreated rock dusts ranged from 0.75 square meters per gram for the coarsest base rock dust to 1.15 square meters per gram for the finest base rock dust. The surface area results for the treated products ranged from 2.83 square meters per gram for the Kotamite and 4.53 square meters per gram for the Supercoat. The surface area results for the blends produced from these rock dusts and treated products ranged from 0.88 square meters per gram for the coarsest blend to 1.42 square meters per gram for the finest blend.

Table 15. N2 BET Surface area measurements of the Phase 2B base and blend samples

Bases and Blends	m2/g
Kotamite	2.83
Supercoat	4.53
#8 White	0.75
#10 White	1.15
Micro-White 100	0.87
7.50% Kotamite / 92.5% #8 White	0.88
10.0% Kotamite / 90.0% #8 White	1.10
12.5% Kotamite / 87.5% #8 White	1.02
12.5% Kotamite / 87.5% Micro-White 100	1.17
7.50% Supercoat / 92.5% #8 White	0.94
10.0% Supercoat / 90.0% #8 White	1.07
12.5% Supercoat / 87.5% #8 White	1.25
10.0% Supercoat / 90.0% #10 White	1.42
12.5% Supercoat / 87.5% #10 White	1.23

Particle Size and Thermogravimetric Analysis (TGA) analysis - Table 16 shows the TGA and MicroTrac particle size test results for the blends that were developed for the Phase 2B evaluation. The base samples used for Phase 2B were previously reported in Phase 2A. The results demonstrate that the proper blend ratios were obtained and that the blends have been adequately mixed.

Table 16. Phase 2B TGA, MicroTrac and wet sieve particle size analysis

Blends	TGA % Total S.A.	TGA % Free S.A.	MicroTrac D 90%	MicroTrac D 50%	MicroTrac D 10%	Wet Sieve % +200M	Wet Sieve % + 325M
7.5% Kotamite / 92.5% #8 White	0.08%	0	66.02	24.74	3.03	n/a	n/a
10.0% Kotamite / 90.0% #8 White	0.11%	0	64.28	22.53	2.48	n/a	n/a
12.5% Kotamite / 87.5% #8 White	0.15%	0	64.80	21.90	2.40	n/a	n/a
12.5% Kotamite / 87.5% Micro-White 100	0.14%	0	49.85	17.64	2.19	n/a	n/a
7.5% Supercoat / 92.5% #8 White	0.10%	0	67.30	24.48	2.45	n/a	n/a
10.0% Supercoat / 90.0% #8 White	0.13%	0	62.96	22.81	2.09	n/a	n/a
12.5% Supercoat / 87.5% #8 White	0.14%	0	65.64	22.35	1.85	n/a	n/a
10.0% Supercoat / 90.0% #10 White	0.13%	0	36.19	14.85	1.90	n/a	n/a
12.5% Supercoat / 87.5% #10 White	0.14%	0	35.40	12.76	1.58	n/a	n/a

ASTM D698 (Proctor Testing) - The nine blend samples were submitted to an independent laboratory (Geotechnics) for Proctor testing which is a laboratory method for experimentally determining the optimal moisture content at which a given material will become most dense and achieve its maximum dry density. The intent was to determine if the blends would cake together and quantify the force required to break the cake under prescribed conditions.

The laboratory technicians at Geotechnics had trouble testing the blend samples as the materials repelled the water and made it impossible to perform the required tests. It was determined that their standard mixing procedure utilizes a hand mixing technique whereby the sample is gradually sprayed with water and mixed to ensure uniform wetting of the sample. Figure 20 illustrates the 12.5% Kotamite/87.5% Micro-White 100 blend following the addition of 12.5% water. Under these conditions, the water failed to incorporate. This was the desirable outcome.

The laboratory then repeated the testing using increased mixing shear: 1) hand mixing using a whisk and 2) utilizing an electric mixer. In both cases, the blends failed to clump or hold together sufficiently to conduct the test.

Figure 20. Preparing a Kotamite/Micro-White 100 blend for the ASTM D698 test

In an attempt to determine where the “break point” was with regards to suitable ASTM D698 sample preparation, 1%, 3%, and 5% Kotamite/Micro-White 100 samples were prepared and submitted to the same independent laboratory for testing with a portion of each sample sent to NIOSH. The blends were analyzed using the laboratory’s standard hand mixing technique, only if unsuccessful would they utilize any mechanical mixing technique.

Using their standard mixing protocol, moisture was able to be mixed into these treated samples. An optimum moisture content and an unconfined compressive strength for each blend was obtained. The laboratory reported that all three samples essentially performed the same and any differences in values were “background noise” associated with the test. The outcome of the tests showed that these samples (Micro-White 100 with $\leq$ 5% additive) would form a cake due to an insufficient concentration of the additives. Figure 21 represents an example of the graph from the testing of the 5% Kotamite/95% Micro-White 100 blend, and shows the maximum dry density for this blend to be 108.8 lb/ft^3 at a moisture content of 15.1%.

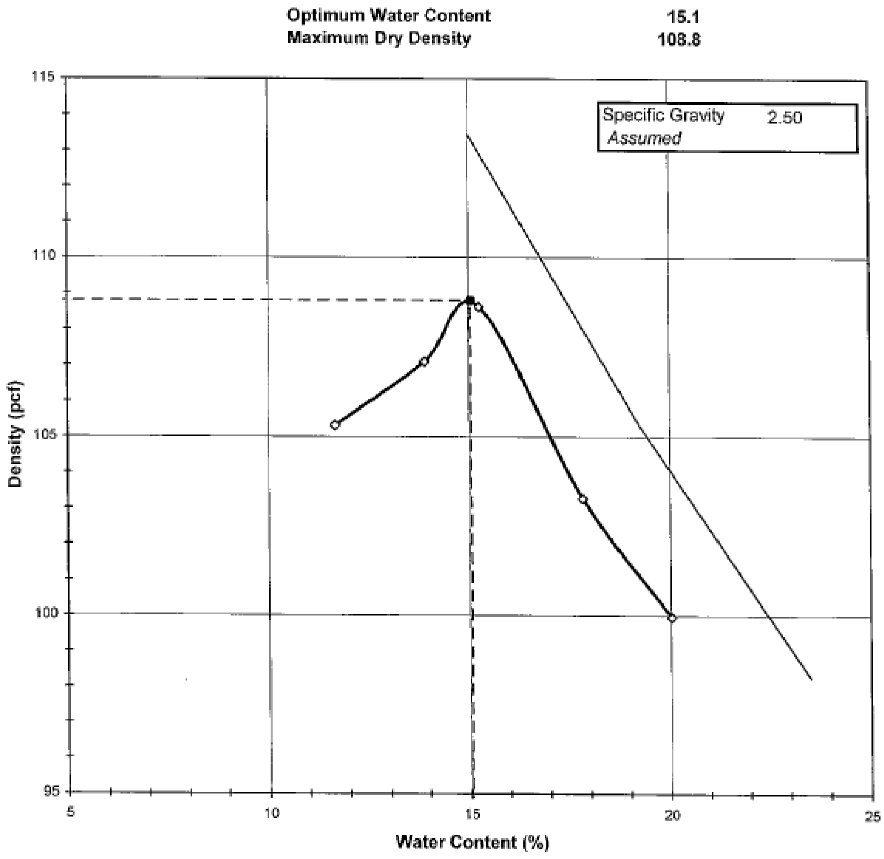


Figure 21. Illustrates a Kotamite/Micro-White 100 ASTM D698 graph

Dust Dispersion Chamber Testing - Table 17 shows the dust dispersion chamber test results for the blends that were subjected to high humidity for periods of 7, 14 and 21 days and compared against a control that was not subjected to high humidity. Six of the nine blends were tested. It was deemed unnecessary to pursue the testing of 3 of the blends based on poor explosibility results (#8 White) combined with economic considerations (the 7.5% and 10% blends performed as well as the 12.5%) that testing of some the 12.5% blends was considered redundant. The results from the remaining 6 samples demonstrated that under high humidity conditions, all the blends dispersed after 21 days. The results for each blend did vary and were found within the range of 2.8% to 5.4% for the mass lost after 21 days of being subjected to high humidity.

Table 17. Dust dispersion chamber data for the Phase 2B samples

Sample	**Days**	**H20 Pick Up (g)**	**H20 Pick Up (%)**	**Powder Loss (g)**	**Powder Loss (%)**
7.5% Kotamite / 92.5% #8 White	0	N/A	N/A	15.06	6.73
7.5% Kotamite / 92.5% #8 White	7	6.92	2.18	6.49	2.81
7.5% Kotamite / 92.5% #8 White	14	7.26	2.28	7.45	3.25
7.5% Kotamite / 92.5% #8 White	21	8.2	2.57	8.52	3.74
10.0% Kotamite / 90.0% #8 White	0	N/A	N/A	22.85	10.74
10.0% Kotamite / 90.0% #8 White	7	6.73	2.16	7.94	3.59
10.0% Kotamite / 90.0% #8 White	14	8.44	2.75	10.63	4.96
10.0% Kotamite / 90.0% #8 White	21	7.03	2.33	10.70	5.12
12.5% Kotamite / 87.5% Micro-White 100	0	N/A	N/A	21.79	10.75
12.5% Kotamite / 87.5% Micro-White 100	7	7.65	2.49	7.26	3.33
12.5% Kotamite / 87.5% Micro-White 100	14	8.17	2.79	11.36	5.78
12.5% Kotamite / 87.5% Micro-White 100	21	8.01	2.30	5.68	2.78
7.5% Supercoat / 92.5% #8 White	0	N/A	N/A	12.65	6.02
7.5% Supercoat / 92.5% #8 White	7	7.94	2.59	10.10	4.73
7.5% Supercoat / 92.5% #8 White	14	7.57	2.44	9.99	4.54
7.5% Supercoat / 92.5% #8 White	21	8.66	2.80	9.21	4.28
10.0% Supercoat / 90.0% #8 White	0	N/A	N/A	16.82	8.52
10.0% Supercoat / 90.0% #8 White	7	7.30	2.49	8.20	4.08
10.0% Supercoat / 90.0% #8 White	14	7.77	2.63	19.74	9.80
10.0% Supercoat / 90.0% #8 White	21	8.74	3.02	8.09	4.13
10.0% Supercoat / 90.0% #10 White	0	N/A	N/A	22.28	12.96
10.0% Supercoat / 90.0% #10 White	7	8.10	3.00	24.70	14.01
10.0% Supercoat / 90.0% #10 White	14	9.17	3.36	28.50	16.04
10.0% Supercoat / 90.0% #10 White	21	8.32	3.04	9.64	5.39

Contact Angle Measurements - In Phase 2B the contact angle was investigated under high humidity (wet) conditions. In one set of experiments the pressed tablets were contacted with liquid water by being placed in a dish with 2 ml de-ionized water. In the other set, the tablets were placed in a dish without water. The former are denoted ‘Wet’ and the latter are denoted ‘Dry’. Contact angles were again measured as a function of time (see Figure 22.).

The Kotamite /#8 White series contained 12.5%, 10.0% and 7.5% Kotamite. The three ‘wet’ blends and two ‘dry’ blends behaved similarly over the time period of the experiment, which was shorter than others, exhibiting initial contact angles of 100° - 120° with a very small subsequent decline to 90° - 100° after 500 minutes. The 7.5% ‘dry’ sample, by contrast, shows an initial contact angle of roughly 50°, which drops to zero within 50 minutes.

With the other three series, the general trend showed both the ‘wet’ and ‘dry’ samples exhibiting initial contact angles of ~100°, with the ‘dry’ sample showing a quicker drop off than the ‘wet’ samples. It is concluded that the imbibitions of water into the tablets in the ‘wet’ samples is impeding the absorption of the liquid droplet. In the ‘dry’ samples, of course, there is no such impediment.

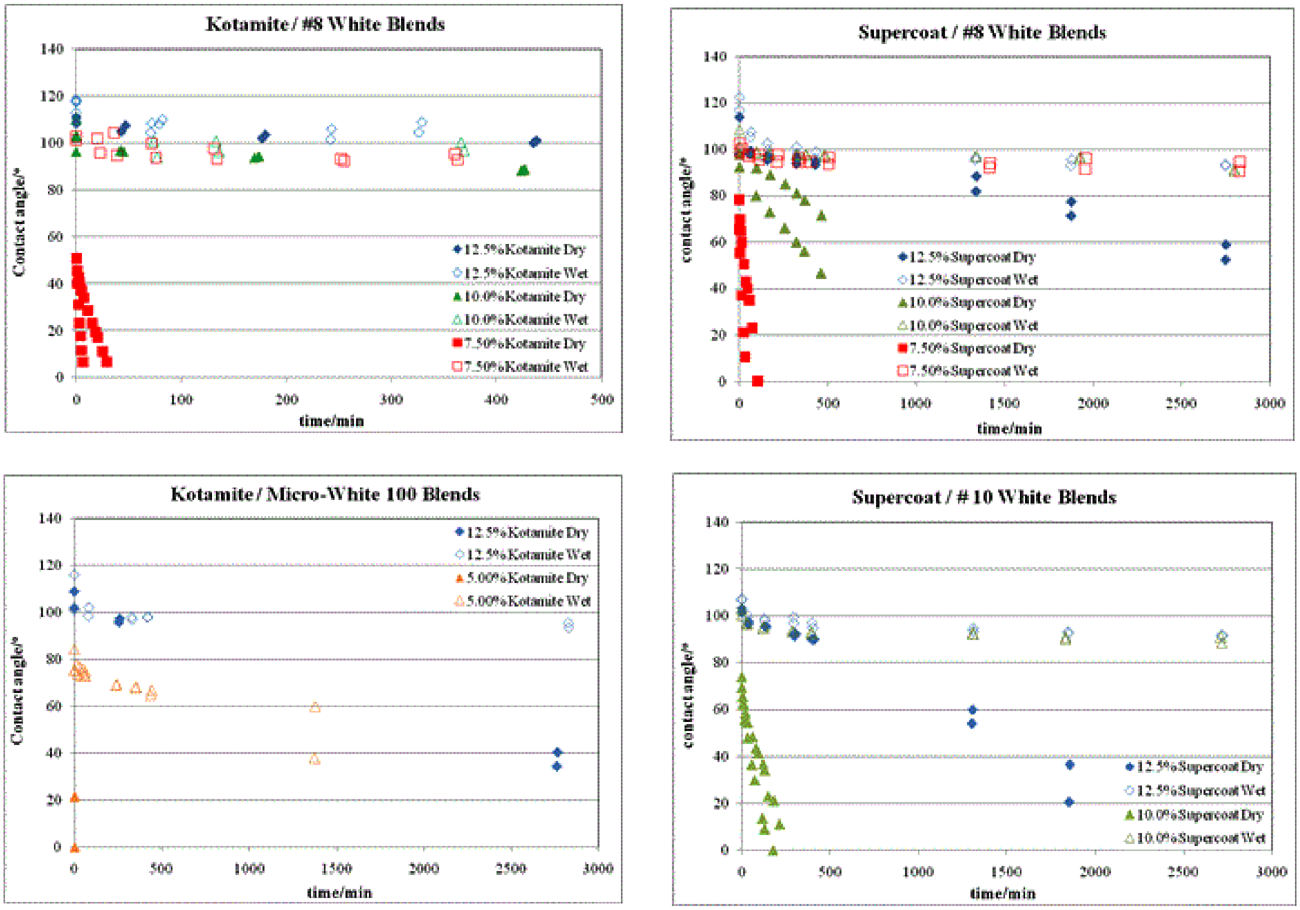


Figure 22. Phase 2B Contact angle measurements for various Kotamite & Supercoat blends.

Explosibility Testing - In Phase 2B, ten rock dust blends were evaluated using the NIOSH 20-L chamber to assess the inerting ability of each against PPC. These samples consisted of the Supercoat or Kotamite additive blended with 3 of the base rock dusts (#8 White, #10 White, and Micro-White 100). It should be noted that one additional sample, 7.5% Kotamite / 92.5% Micro-White 100 was added to the explosibility testing (see Table 18). This sample was not evaluated in any other Phase 2B tests.

Table 18. Summary of the explosion inerting experiments in the NIOSH 20-L chamber on the Phase 2 samples

20-L Chamber Coal Concentration	200 g/m^3	200 g/m^3	200 g/m^3	200 g/m^3	400 g/m^3	400 g/m^3	400 g/m^3	400 g/m^3	600 g/m^3	600 g/m^3	600 g/m^3
% Rock Dust Concentration	65	70	75	80	65	70	75	80	65	70	75
Micro-White 100	dnt	go-go	no-no	dnt	dnt	go	no-no	dnt	dnt	go	no-no
#8 White	dnt	dnt	dnt	dnt	dnt	go	no-no	dnt	dnt	dnt	dnt
#10 White	dnt	dnt	dnt	dnt	dnt	no	no-no	dnt	dnt	dnt	dnt
Kotamite	dnt	go	no-no	dnt	dnt	no-no	no-no	dnt	dnt	no-no	no-no
Supercoat	go	no-no	dnt	dnt	dnt	dnt	no-no	dnt	dnt	no	dnt
12.5% Supercoat / 87.5% #8 White	dnt	go	no-no	dnt	dnt	go	no-no	dnt	dnt	go	no-no
10.0% Supercoat / 90.0% #8 White	dnt	go-go	no-no	no-no	dnt	go	no-no	dnt	dnt	go	no-no
7.5% Supercoat / 92.5% #8 White	dnt	go	no-no	dnt	dnt	dnt	go	no-no	dnt	go	no-no
12.5% Kotamite / 87.5% #8 White	dnt	go	no-go	no-no	dnt	go	no-no	dnt	dnt	go	no-no
10.0% Kotamite / 90.0% #8 White	dnt	go-go	no-go	no-no	dnt	go	no-no	dnt	dnt	go	no-no
7.5% Kotamite / 92.5% #8 White	dnt	go	no-no	dnt	dnt	go	no-no	dnt	dnt	go	no-no
12.5% Supercoat / 87.5% #10 White	dnt	go	no-no	dnt	go	no-no	no	dnt	go	no-no	no
10.0% Supercoat / 90.0% #10 White	dnt	go	no-no	dnt	go	no-go	no	dnt	go	no-no	no
12.5% Kotamite / 87.5% Micro-White 100	dnt	go	no-no	dnt	dnt	go	no-no	dnt	go	no-no	no
7.5% Kotamite / 92.5% Micro-White 100	dnt	go	no-no	dnt	dnt	go	no-no	dnt	go	no-no	no

Shaded boxes indicate the experiments which failed the 75% rock dust requirement to inert an explosion.
Go = explosion, No = no explosion, dnt = did not test
A "no-no" designation indicates that two experiments were conducted and both tests inerted the coal dust.

In general, all of the rock dusts and the rock dust blends inerted the PPC at the 75% inertant concentration. However, there was one explosion at 75% inertant noted for the 7.5% Supercoat blended with the #8 White at the 400 g/m^3 coal concentration and two explosions at 75% inertant noted for the 12.5% and 10% Kotamite blends with the #8 White at the 200 g/m^3 coal concentration. This may be due to the coarser particle size distribution of the #8 White (31.7 µm) as compared to that of #10 White (16.6 µm) or the MicroWhite 100 (23.5 µm). The results indicated that the #10 White blends were more effective in explosion inerting in the 20-L chamber than the #8 White blends.

Observations from Phase 2B

The base components used in Phase 2B are identical to those utilized in Phase 2A and therefore no chemical, TGA, and particle size analysis was conducted. Surface area (N2 BET) analysis was conducted on the five base and nine blend samples. Results verified the values of the base samples to be consistent with those typically seen in the market place. The surface areas of the blend samples are consistent with the weighted average of the particle sizes of the individual blend components.

Nine blend samples were prepared (2 Kotamite series and 2 Supercoat series) with the treated blend component ranging from 7.5 – 12.5 wt% (prior results showed that no additional dispersion benefits were realized above 12.5 wt%). The characterization (TGA and particle size analysis) of the blend samples confirmed that the samples were accurately blended.

Contact angle measurement and dispersion testing of the base samples were not conducted based on our observations from Phase 1. In general, the contact angle analysis indicated that the blends with the higher percentage treated products retained their contact angle longer. Previously it was observed that as the treated blend component approached 7.5%, the contact angle dropped dramatically. Under high humidity (wet) conditions, the contact angle values for the 7.5 wt% blends were similar to blends with higher treated component levels. It is theorized that the higher humidity sample has imbibed water and there is no place for the droplet to go.

Geotechnics was unable to get any of the nine Phase 2B blends to clump or cake sufficiently to conduct the ASTM D698 test. In further testing, blends containing 5% or less of the treated product Kotamite did form a cake to allow for ASTM D698 to be performed. The overall value of ASTM D698 test is diminished as blends containing greater than 5% treated products cannot be tested.

After subjecting the blend samples to high humidity for 21 days, all the blend samples exhibited dust dispersion characteristics comparable to the previously tested blend samples in Phase 2A (under wet dispersion). Overall there is some variability in the dispersion results and this can be attributed to the placement of the nozzle and the settling characteristics of the rock dust in the tray prior to initiation of the test. Results continue to illustrate a slight advantage in dispersion characteristic with the blends containing the finer base rock dusts. This observation might be linked with particle packing and additional work would be required to understand this further.

After reviewing the 20-L explosion data with NIOSH, it was agreed to discontinue any further work involving #8 White blends (as these blends failed the 75% rock dust requirement to inert an explosion). To continue the optimization effort of the blend components and ratios, it was agreed with NIOSH to pursue five additional blend samples that would form the basis of the Phase 2C work.

Phase 2C Results and Discussion

As the work focused in on optimizing the blends further, 5 (five) additional blend samples were prepared. These blends represented subtle changes in the blend ratios of the treated and untreated components based on the observations from the previous phases. RO-40 being too coarse, and #8 White was eliminated from further work based on the NIOSH 20-L chamber results and the fact that it failed to prevent an explosion at the 75% inertant level. In addition to particle size and stearic acid treatment levels, the blend samples were again subjected to contact angle and dust dispersion chamber testing under high humidity for an extended time period (7, 14 and 21 days). Table 19 illustrates the blend samples prepared and tested in Phase 2C.

Table 19. Lists the 5 blend samples prepared for Phase 2C evaluations

Treated	Untreated
7.5% Supercoat	92.5% #10 White
7.5% Supercoat	92.5% Micro-White 100
10.0% Supercoat	90.0% Micro-White 100
7.50% Kotamite	92.5% Micro-White 100
10.0% Kotamite	90.0% Micro-White 100

Particle Size and Thermogravimetric Analysis (TGA) analysis - Table 20 shows the TGA and MicroTrac particle size test results for the blends that were developed for the Phase 2C evaluation. There were five blends tested; a blend of 7.5% Kotamite / 92.5% Microwhite 100, a blend of 10% Kotamite / 90% Microwhite 100, a blend of 7.5% Supercoat / 92.5% Microwhite 100, a blend of 10% Supercoat / 90% Microwhite100 and a blend of 7.5% Supercoat and 92.5% # 10 White. Again, no free stearic acid was detected. The results demonstrate that the proper blend ratios were obtained and that the blends have been adequately mixed.

Table 20. Phase 2C TGA, MicroTrac and wet sieve particle size analysis

BASES	TGA % Total S.A.	TGA % Free S.A.	MicroTrac D 90%	MicroTrac D 50%	MicroTrac D 10%	Wet Sieve % +200M	Wet Sieve % + 325M
7.5% Supercoat / 92.5% #10 White	0.12%	0	37.90	13.81	2.20	n/a	n/a
7.5% Supercoat / 92.5% Micro-White 100	0.11%	0	53.38	21.53	2.61	n/a	n/a
10.0% Supercoat / 90.0% Micro-White 100	0.12%	0	51.28	20.45	2.30	n/a	n/a
7.50% Kotamite / 92.5% Micro-White 100	0.11%	0	49.83	20.95	2.94	n/a	n/a
10.0% Kotamite / 90.0% Micro-White 100	0.13%	0	50.89	20.25	2.81	n/a	n/a

Dust Dispersion Chamber Testing - Table 21 shows the dust dispersion chamber test results for the blends that were subjected to high humidity for periods of 7, 14 and 21 days and compared against a control sample that was not subjected to high humidity. The results demonstrated that under high humidity conditions, all of the blends dispersed after 21 days. The results for each blend did vary and were found within the range of 3.0% to 4.3% for the powder lost after 21 days of being subjected to high humidity.

Table 21. Dust dispersion chamber data for the Phase 2C samples

Sample	Days	H20 Pick Up (g)	H20 Pick Up (%)	Powder Loss (g)	Powder Loss (%)
7.5% Supercoat / 92.5% #10 White	0	N/A	N/A	21.54	11.64
7.5% Supercoat / 92.5% #10 White	7	6.91	2.67	7.95	4.82
7.5% Supercoat / 92.5% #10 White	14	8.40	3.14	7.27	4.18
7.5% Supercoat / 92.5% #10 White	21	8.39	3.18	6.99	4.12
7.5% Supercoat / 92.5% Micro-White 100	0	N/A	N/A	13.68	6.89
7.5% Supercoat / 92.5% Micro-White 100	7	6.16	2.13	5.92	2.97
7.5% Supercoat / 92.5% Micro-White 100	14	7.18	2.45	6.40	3.16
7.5% Supercoat / 92.5% Micro-White 100	21	7.60	2.62	6.54	3.30
10.0% Supercoat / 90.0% Micro-White 100	0	N/A	N/A	15.79	8.34
10.0% Supercoat / 90.0% Micro-White 100	7	5.78	2.01	5.00	2.52
10.0% Supercoat / 90.0% Micro-White 100	14	6.56	2.27	7.16	3.65
10.0% Supercoat / 90.0% Micro-White 100	21	6.48	2.26	6.40	3.28
7.50% Kotamite / 92.5% Micro-White 100	0	N/A	N/A	14.24	6.92
7.50% Kotamite / 92.5% Micro-White 100	7	6.21	2.05	8.40	3.93
7.50% Kotamite / 92.5% Micro-White 100	14	7.07	2.39	4.40	2.09
7.50% Kotamite / 92.5% Micro-White 100	21	7.42	2.49	5.98	2.95
10.0% Kotamite / 90.0% Micro-White 100	0	N/A	N/A	14.44	7.09
10.0% Kotamite / 90.0% Micro-White 100	7	6.10	2.00	6.74	3.20
10.0% Kotamite / 90.0% Micro-White 100	14	6.49	2.16	10.49	5.07
10.0% Kotamite / 90.0% Micro-White 100	21	6.96	2.34	8.62	4.26

Contact Angle Measurements - In Phase 2C the pressed tablets were contacted with liquid water by being placed in a dish with 2 ml de-ionized water for periods of one, two and three weeks. These samples were designated ‘wet’. For comparative purposes, one sample was placed in a dish without water and was denoted ‘dry’. This sample was measured after 1 week. Only two blend samples were measured, the 7.5% and 10.0% Kotamite with Micro-White 100.

For the 7.5% Kotamite / 92.5% Micro-White 100 blend, the ‘wet’ samples are almost indistinguishable with initial contact angles of 90° -105°, retaining their contact angles of >80° to times of ~2,000 minutes. The exposure time (1 - 3 weeks) makes no difference to the outcome and water absorption is substantially complete within 1 week. The ‘dry’ sample, by contrast, is measured only with difficulty due to rapid droplet imbibitions, and the initial contact angle of 40° - 50° drops to zero within a few minutes.

With the 10.0% Kotamite / 90,0% Micro-White 100 blend, the contact angle measurement for all three ‘wet’ samples are very similar, initial contact angles of >100° and retain their contact angles of >80° to times of 2,000 minutes. The ‘dry’ sample exhibited an initial contact angle of ~90° and dropped to ~60° within 500 minutes. This was marginally better than the 7.5% Kotamite / 92.5% Micro-White 100 blend sample and supports the earlier observations that hydrophobicity drops away as the treated component level approaches 7.5%. Figure 23 shows the respective graphs.

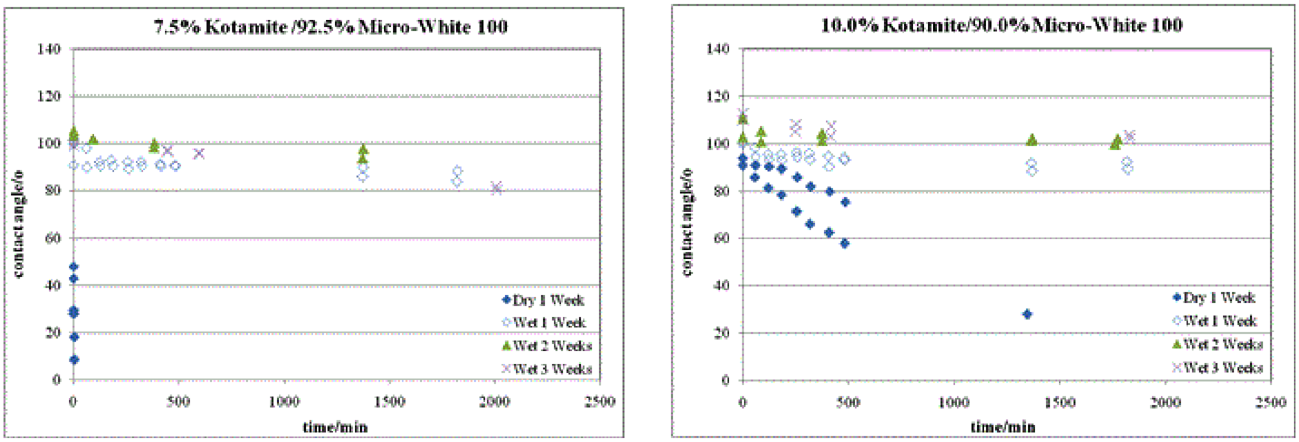


Figure 23. Phase 2C Contact angle measurements for 2 Kotamite/Micro-White 100 blends

Observations from Phase 2C

The base components used on Phase 2C are identical to those utilized in Phase 2A/2B and therefore no chemical, particle size, and TGA were conducted. Five blend samples were prepared (1 Kotamite series and 2 Supercoat series) with the treated blend component ranging from 7.5 – 10.0 wt% (as the project continues to optimize the blend components and their ratios). The characterization (TGA and particle size analysis) of the blend samples confirmed that the samples were accurately blended.

Contact angle measurement and dispersion testing of the base samples were not conducted based on our observations from Phase 1. Two of the samples were evaluated for their contact angle to ensure that prior observations were realized. This was the case.

After subjecting the blend samples to high humidity for 7, 14 and 21 days, all the blend samples exhibited dispersion characteristics comparable to the previously tested blend samples in Phase 2B. The variability found previously that was attributed to the placement of the nozzle and the settling characteristics of the rock dust in the tray prior to initiation of the test, was again observed. Results show the optimum performers to be the 7.5% Supercoat/92.5% #10 White and the 10% Kotamite/90% Micro-White 100.

As agreed upon with NIOSH, three blends were recommended for final testing and potential scale up. The blends were 12.5% Kotamite/87.5% Micro-White 100, 10.0% Supercoat/90.0% Micro-White 100 and 10.0% Supercoat /90.0% #10 White.

Phase 3 Results and Discussion

Phase 3 – As a result of the Phase 2 test work, three blends were recommended for final testing and scale-up. The three blend ratios had been previously been tested in earlier phases of the project. The intent of Phase 3 was to ensure the results associated with the earlier samples could be duplicated. A fresh set of blend samples were prepared and subjected to particle size, chemistry (base samples only), stearic acid treatment levels, contact angle and dust dispersion chamber testing (the latter two under high humidity for an extended time period). The three blend samples were sent to NIOSH for verification testing which included explosibility testing conducted by NIOSH in their 20-L chamber. The three blend samples selected for evaluation are listed in Table 22.

Table 22. Lists the 3 blend samples prepared for Phase 3 evaluations

Treated	Untreated
12.5% Kotamite	87.5% Micro-White 100
10.0% Supercoat	90.0% Micro-White 100
10.0% Supercoat	90.0% #10 White

Chemistry – the 4 base samples were submitted to a third party for chemical analysis, primarily to obtain crystalline silica values to ensure the samples met the silica criteria defined in 30 CFR 75.2 and to ensure that the Phase 3 base samples were similar in chemical composition to that of the corresponding Phase1 and 2 base samples. Table 23 below documents the values obtained and confirms acceptable crystalline silica levels.

Table 23. Chemical analysis of the 4 Phase 3 base samples

Bases and Blends	MgO	Al_2O_3	SiO_2	K_2O	CaO	Fe_2O_3	SrO	LOI	Crystalline Silica
Kotamite	1.26	0.17	1.00	0.06	52.30	0.09	0.02	44.60	0.70
Supercoat	0.72	0.13	0.59	0.05	54.30	0.13	0.02	44.00	0.60
Micro-White 100	1.00	0.37	0.78	0.08	54.20	0.09	0.03	43.40	0.50
#10 White	1.08	0.49	0.95	0.10	53.70	0.12	0.02	43.40	0.60

Particle Size and Thermogravimetric Analysis (TGA) analysis - Table 24 shows the TGA and MicroTrac particle size test results for the blends that were developed for the Phase 2C evaluation. There were three blends tested; a blend of 12.5% Kotamite / 92.5% Micro-White 100, a blend of 10% Supercoat / 90% Micro-White100 and a blend of 10% Supercoat and 90% # 10 White. The results demonstrate that the proper blend ratios were obtained and that the blends have been adequately mixed.

Table 24. Phase 3 TGA, MicroTrac and wet sieve particle size analysis

BLENDS	TGA % Total S.A.	TGA % Free S.A.	MicroTrac D 90%	MicroTrac D 50%	MicroTrac D 10%	Wet Sieve % +200	Wet Sieve % + 325
12.5% Kotamite / 87.5% Micro-White 100	0.16%	0	49.66	19.36	2.58	n/a	n/a
10.0% Supercoat / 90.0% Micro-White 100	0.12%	0	50.29	20.12	2.19	n/a	n/a
10.0% Supercoat / 90.0% #10 White	0.11%	0	37.41	15.20	2.16	n/a	n/a

Dust Dispersion Chamber Testing - Table 25 shows the dust dispersion chamber test results for the blends that were subjected to high humidity for periods of 7, 14 and 21 days and compared against three control sample that were not subjected to high humidity. The results demonstrated that under high humidity conditions, all the blends dispersed after 21 days. The results for each blend did vary and were found within the range of 3.2% to 4.8% for the powder lost after 21 days of being subjected to high humidity.

Table 25. Dust dispersion chamber data for the Phase 3 samples

Sample	Days	H20 Pick Up (g)	H20 Pick Up (%)	Powder Loss (g)	Powder Loss (%)
12.5% Kotamite / 87.5% Micro-White 100	0	N/A	N/A	14.02	6.85
12.5% Kotamite / 87.5% Micro-White 100	0	N/A	N/A	18.14	9.00
12.5% Kotamite / 87.5% Micro-White 100	0	N/A	N/A	14.55	7.23
12.5% Kotamite / 87.5% Micro-White 100	7	6.34	2.17	7.81	3.96
12.5% Kotamite / 87.5% Micro-White 100	14	6.28	2.10	6.03	2.96
12.5% Kotamite / 87.5% Micro-White 100	21	6.60	2.24	6.41	3.18
10.0% Supercoat / 90.0% Micro-White 100	0	N/A	N/A	13.47	6.96
10.0% Supercoat / 90.0% Micro-White 100	0	N/A	N/A	11.86	6.29
10.0% Supercoat / 90.0% Micro-White 100	0	N/A	N/A	14.72	7.76
10.0% Supercoat / 90.0% Micro-White 100	7	6.31	2.24	6.16	3.22
10.0% Supercoat / 90.0% Micro-White 100	14	6.52	2.31	7.52	3.96
10.0% Supercoat / 90.0% Micro-White 100	21	6.85	2.42	6.45	3.39
10.0% Supercoat / 90.0% #10 White	0	N/A	N/A	12.18	6.73
10.0% Supercoat / 90.0% #10 White	0	N/A	N/A	11.30	6.37
10.0% Supercoat / 90.0% #10 White	0	N/A	N/A	12.77	7.07
10.0% Supercoat / 90.0% #10 White	7	6.20	2.35	8.27	4.85
10.0% Supercoat / 90.0% #10 White	14	6.27	2.39	6.42	3.73
10.0% Supercoat / 90.0% #10 White	21	6.72	2.56	8.26	4.78

Contact Angle Measurements – As described in Phase 2C the pressed tablets were contacted with liquid water by being placed in a dish with 2 ml de-ionized water for periods of one, two and three weeks. These samples were designated ‘wet’. For comparative purposes, one sample was placed in a dish without water and was denoted ‘dry’. This sample was measured after 1 week. Results for the three blend samples were similar in that they showed the same trend.
All three graphs (Figure 24) show that the ‘wet’ samples exhibit initial contact angles of 100° - 120°, retaining their contact angles of ~80° or better to times of ~3,000 minutes. The contact angles for the two and three week ‘wet’ samples are very similar to that of the one week sample and suggests that water imbibition is nearly complete after 1 week. The ‘dry’ samples, by contrast, showed varying results and following an initial contact angle of between 75° - 110° dropped to away. The 12.5% Kotamite / 87.5% Micro-White 100 blend held its contact angle the longest, ~80° to 1,400 minutes, while the 10% Supercoat / 90% #10 White blend and 10% Supercoat / 90% Micro-White 100 blend recorded <40° to 500 – 1,200 minutes. The overall trend with regards to the ‘wet’ blend samples maintaining their contact angle over several weeks agrees with the prior observations and again suggests that the higher humidity samples have imbibed water and there remains nowhere for the droplet to go.

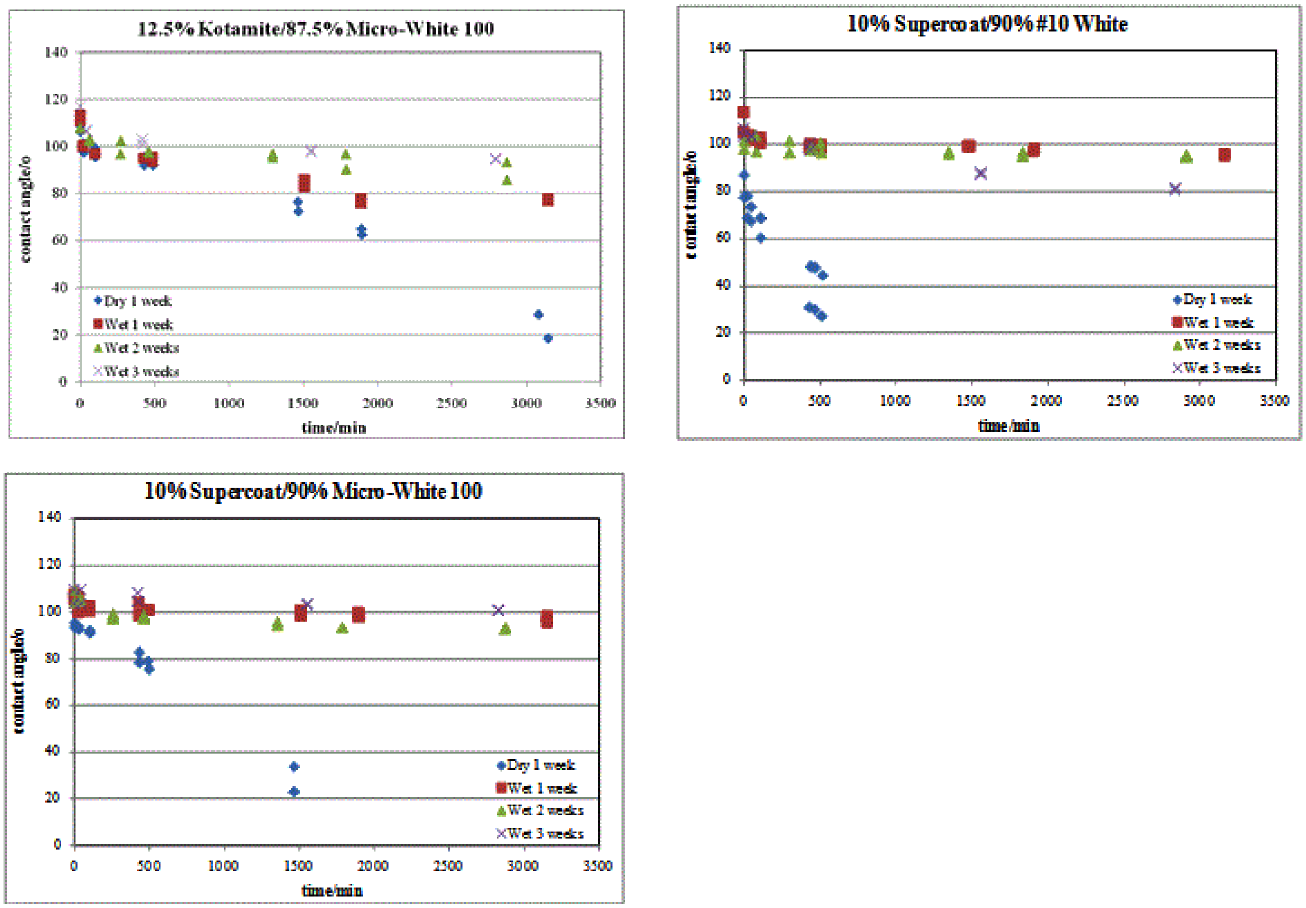


Figure 24 Phase 3 Contact angle measurements for the three most promising blends

Explosibility Testing – In Phase 3, the three most promising anti-caking rock dust samples were evaluated in the 20-L explosibility chamber to assess the inerting ability of each of the treated rock dust blends against PPC (see Table 26). These samples consisted of the Supercoat or Kotamite additive blended with one of two base rock dusts; namely, #10 White and Micro-White 100.

Table 26. Summary of the explosion inerting experiments in the NIOSH 20-L chamber on the Phase 3 samples

20-L Chamber Coal Concentration	200 g/m^3	200 g/m^3	200 g/m^3	400 g/m^3	400 g/m^3	400 g/m^3	600 g/m^3	400 g/6^3	600 g/m^3
% Rock Dust Concentration	65	70	75	65	70	75	65	70	75
Micro-White 100	dnt	go-go	no-no	dnt	go	no-no	dnt	go	no-no
#10 White	dnt	dnt	dnt	go	no-no	no-no	dnt	dnt	dnt
Kotamite	dnt	go	no-no	dnt	no-no	no-no	dnt	no-no	no-no
Supercoat	go	no-no	dnt	dnt	dnt	no-no	dnt	no	dnt
10.0% Supercoat / 90.0% #10 White	dnt	go	no-no	go	no-go	no-no	go	no-no	no
10.0% Supercoat / 90.0% Micro-White 100	dnt	go	no-no	go	no-go	no-no	go	no-no	no
12.5% Kotamite / 87.5% Micro-White 100	go	no-go	no-no	go	no-go	no-no	go	no-go	no-no

Go = explosion, No = no explosion, dnt = did not test

A "no-no" designation indicates that two experiments were conducted and both tests inerted the coal dust.

The results indicated that the #10 White and Micro-White100 blends were effective in inerting the PPC mixtures at the 75% inertant concentration within the 20-L chamber.

Observations from Phase 3

The chemistry of the base components used in Phase 3 was measured to ensure the values are similar to those obtained previously. Three blend samples were prepared (1 Kotamite series and 2 Supercoat series) with the treated blend component ranging from 10.0 – 12.5 wt% (as the project continues to optimize the blend components and their ratios). The characterization (TGA and particle size analysis) of the blend samples confirmed that the samples were accurately blended.

All three samples were evaluated for their contact angle to ensure that prior observations were realized. This was the case.

After subjecting the blend samples to high humidity for 21 days, the three blend samples exhibited dispersion characteristics similar to the previously tested blend samples in Phase 2C. The variability found previously that was attributed to the placement of the nozzle and the settling characteristics of the rock dust in the tray prior to initiation of the test, was again observed. Results show little difference in performance and all blends can be considered acceptable.

The explosibility results indicated that the #10 White and Micro-White100 blends were effective in inerting the PPC mixtures at the 75% inertant concentration within the 20-L chamber.

After reviewing all the prior data and taking into consideration the economic aspects of commercializing a blend, it was agreed with NIOSH to produce a large pilot plant scale up sample of the 12.5 wt% Kotamite/ 87.5 wt% Micro-White 100 blend.

Pilot Scale-Up Results and Discussion

Pilot Scale-Up – The most economically-viable, successful blend (12.5% Kotamite/87.5% Micro-White 100) from Phase 3 was selected for a large-scale evaluations within an area of NIOSH's Safety Research Coal Mine or similar facility and for a coal mine operator to conduct an experimental full-scale trial, with the approval of MSHA, within an area of their mine. To facilitate this, a large quantity of the blend was manufactured in a pilot scale-up trial.

On February 14, 2014, 20 tons of the blend consisting of 12.5% Kotamite/87.5% Micro-White 100 was manufactured at Sanco Inc (a custom toll processor) located in Dalton, Georgia. Sanco's personnel were responsible for the blending operation while Imerys personnel were present to observe and assist with the blending and bagging. A medium intensity 8,000 lb. horizontal powder mixer was used for the blending (Figure 25). The mixer was set up as a batch process mixing 3,500 lb of Imerys Micro-White 100 with 500 lb of the Imerys Kotamite. Both products are commercially available and were manufactured at Imerys Sylacauga facility.

Once charged with the 2 blend components, the mixer ran for a period of 20 minutes at the full speed of the mixer (medium intensity). There were 3 gangs of off-set ploughs that swept across the entire length of the mixer ensuring a nice powder flow (Figure 26). Each batch was checked for hydrophobicity by placing a sample of the blend in water and observing whether the blend floated. The 20 minutes mixing time was sufficient to ensure good blending of the two components. Each batch was bagged into 2 x 2000 lb. semi-bulk bags, placed on individual pallets and stretched wrapped. Each cycle of charging the blender, mixing and bagging took approximately 50 minutes. In total ten individual batches were prepared, with samples being taken from each batch and made into three larger trial samples for further testing. Samples from each of these three larger trial samples were taken and combined to make one large composite sample of approximately 30 pounds.

Figure 25. Horizontal Powder Mixer

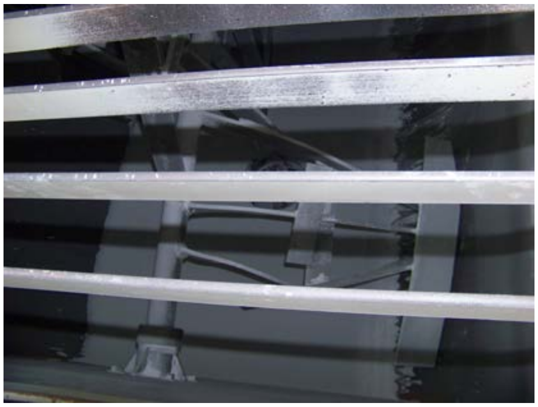


Figure 26. Horizontal Powder Mill showing inside view of mixing blades

The blend composite sample was split into a 20 lb sample for NIOSH and a 10lb sample for Imerys. The sample for NIOSH was shipped February 25, 2014 (via Fedex) for further testing. The three in-process composite samples were shipped to Imerys Marietta, Georgia laboratory where they were analyzed for chemistry (base samples only) particle size, stearic acid treatment levels, contact angle and dust dispersion chamber testing (wet and dry). The samples were also evaluated using the NIOSH 20-L chamber to assess the inerting ability against PPC.

Chemistry – Table 27 below illustrates the chemistry values obtained from the 2 base samples used to manufacture the blend and confirm the materials to consistent with their commercially available counterparts.

Table 27. Chemical analysis of the 2 Scale-up base samples

	MgO	Al_2O_3	SiO_2	K_2O	CaO	Fe_2O_3	SrO	LOI	Crystalline Silica
Kotamite	1.25	0.15	0.78	0.05	54.30	0.12	0.02	43.30	0.60
Micro-White 100	1.16	0.26	1.10	0.10	54.10	0.14	0.03	43.00	0.80

Particle Size and Thermogravimetric Analysis (TGA) analysis - Table 28 shows the TGA and MicroTrac particle size test results for the two base rock dusts and the 12.5% Kotamite/87.5% Micro-White 100 blend. As expected there was no stearic acid present in the base Micro-White 100. The total stearic acid level of the Kotamite (1.13%) was in good agreement with the total stearic acid level (1.11%) of the Kotamite utilized in Phase 1 through Phase 2C. However, there was a difference in the level of free stearic acid between the Kotamite utilized in the scale-up (0.2%) versus the Kotamite used throughout Phase 1 – 3 (0.4%). Un-reacted stearic acid levels of 0.2 – 0.4 wt% would fall within normal manufacturing levels. There was no measureable free stearic acid level for the blend samples. This observation was in agreement with those previously seen whereby at the lower treated blend levels (<25%) no free stearic acid was measured. The TGA and MicroTrac particle results demonstrate that the proper blend ratio was obtained.

Table 28. Scale-up TGA, MicroTrac and wet sieve particle size analysis

BLENDS	TGA Total % S.A.	TGA % Free S.A.	MicroTrac D 90%	MicroTrac D 50%	MicroTrac D 10%	Wet Sieve % +200M	Wet Sieve % + 325M
MicroWhite 100	0.00%	0.00%	54.96	24.22	3.86	n/a	n/a
Kotamite	1.13%	0.20%	8.84	2.99	1.02	n/a	n/a
12.5% Kotamite / 87.5% Micro-White 100	0.16%	0.00%	52.73	19.11	1.99	n/a	n/a

For completeness Figures 27 – 29 show the TGA analysis of the two base samples and the blend. The x axis is temperature (°C) and extends from room temperature to 650°C. The left hand y axis is % weight loss from the sample and extends from 98.0% to 102.2% and the right hand y axis is the first derivative in wt% per minute.

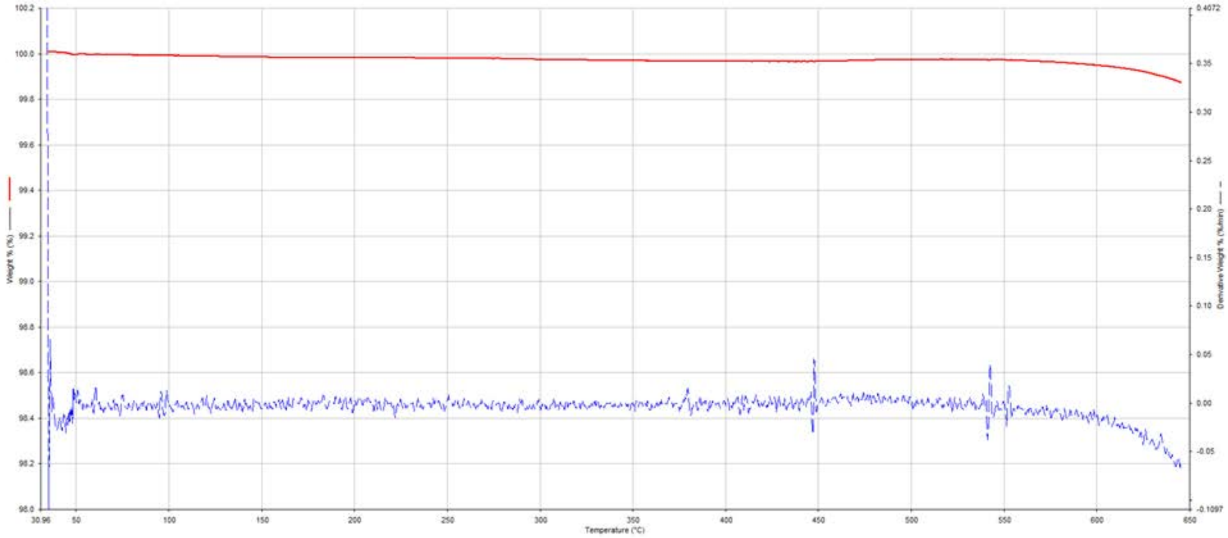


Figure 27. Scale-up Thermogravimetric analysis of Micro-White 100

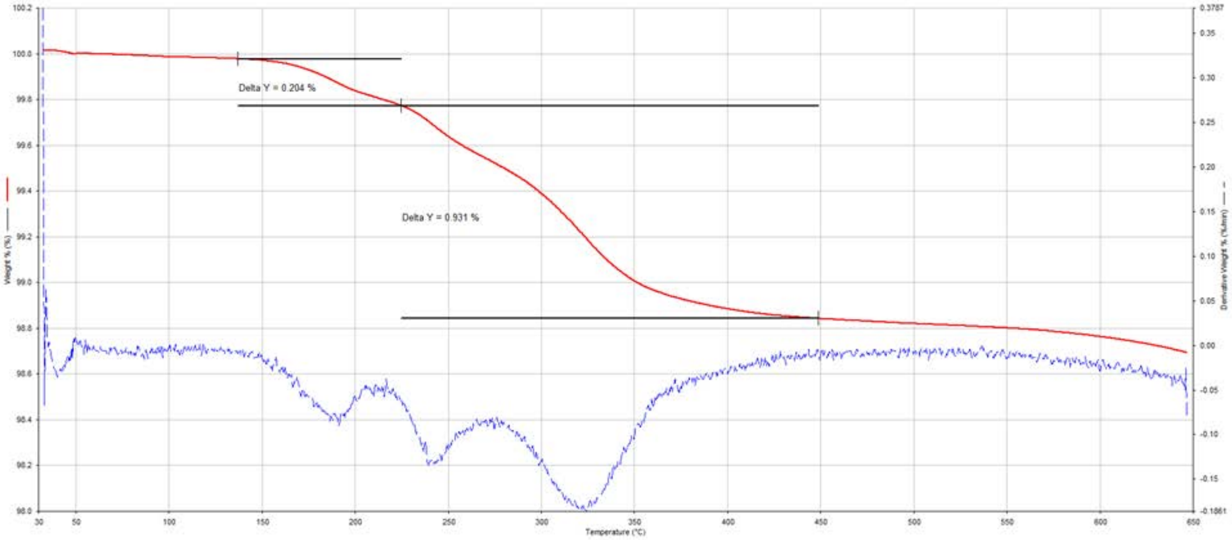


Figure 28. Scale-up Thermogravimetric analysis of Kotamite

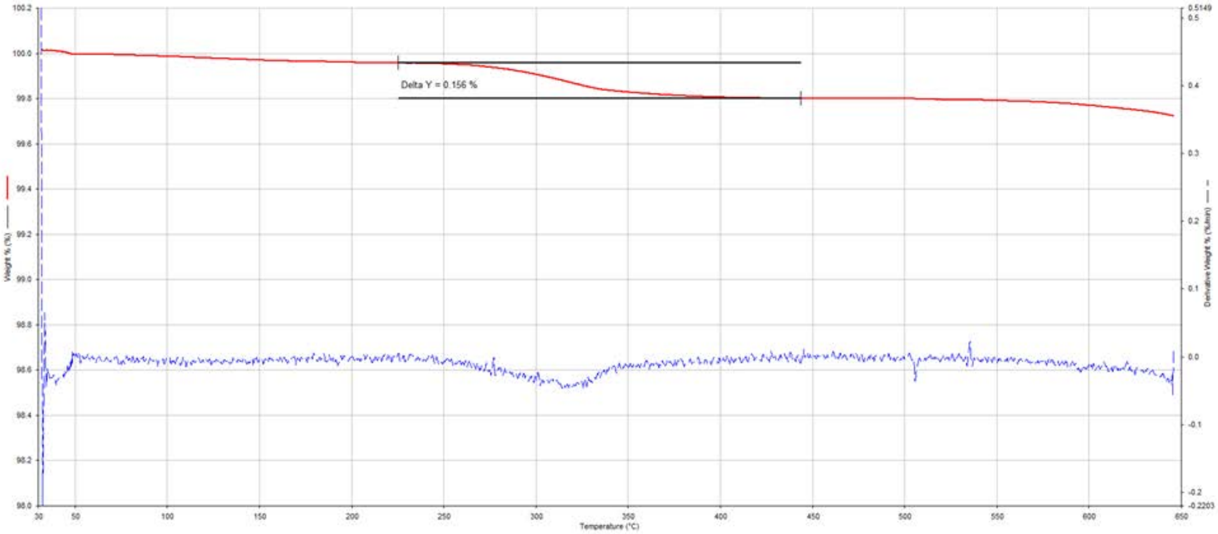


Figure 29. Scale-up Thermogravimetric analysis of 12.5% Kotamite/87.5% Micro-White 100

Dust Dispersion Chamber Testing – Table 29A shows the test results obtained from the dry dust dispersion chamber testing and the wet dust dispersion chamber testing (as described in Appendix B) for the scale-up blend samples. Four individual blend samples were tested. The test results for the various blends showed typical rock dust loss values of 8.44% to 9.12% for the dry dust dispersion chamber testing and 4.88% to 6.13% with the wet dust dispersion chamber testing. These results correlate well with values obtained in previous phases of this study.
Table 29B shows the dust dispersion chamber test results for the blend after being subjected to high humidity (as described in Appendix C) for periods of 7 and 14 days and compared against two control sample that were not subjected to high humidity. The results again demonstrate that under high humidity conditions the blend dispersed, loss values were 2.82% and 3.26% for the 7 and 14 day test respectively and agree with prior results.

Table 29A Dust dispersion chamber data for the Scale-up samples (Dry and Wet)

Base or Blend	Sample Number	Dry Powder Loss (g)	Dry Powder Loss (%)	Wet Powder Lost (g)	Wet Powder Lost (%)
12.5% Kotamite / 87.5% Micro-White 100	1	15.48	8.47	9.50	4.88
12.5% Kotamite / 87.5% Micro-White 100	2	16.60	9.12	12.09	6.13
12.5% Kotamite / 87.5% Micro-White 100	3	15.71	8.44	9.79	5.07
12.5% Kotamite / 87.5% Micro-White 100	4	15.66	8.50	11.07	5.57
12.5% Kotamite / 87.5% Micro-White 100	Average	15.86	8.63	10.61	5.41

Table 29B Dust dispersion chamber data for the Scale-up samples (High Humidity)

Sample	Days	H20 Pick Up (g)	H20 Pick Up (%)	Powder Loss (g)	Powder Loss (%)
12.5% Kotamite / 87.5% Micro-White 100	0	N/A	N/A	11.86	6.01
12.5% Kotamite / 87.5% Micro-White 100	0	N/A	N/A	16.65	8.26
12.5% Kotamite / 87.5% Micro-White 100	7	5.66	1.95	5.62	2.82
12.5% Kotamite / 87.5% Micro-White 100	14	6.89	2.38	6.54	3.26

Contact Angle - The contact angle analysis of the 20 ton scale-up blend is shown below in Figure 30. Powders were pressed as before and all samples were placed in a 98% relative humidity chamber for 1 and 2 weeks. In one case the tablet was placed in a dry dish and was not exposed to liquid water and in two other cases the tablet was placed in filter paper-lined petri dish wetted with a few ml of de-ionized water. The dry sample shows slight hydrophobicity, supporting an initial droplet with contact angle of 60-70° but declining to zero within 60 minutes. The wetted samples support a droplet to over 3,000 minutes with very high contact angles >90°. There is no difference between wetted samples exposed for 1 and 2 weeks.

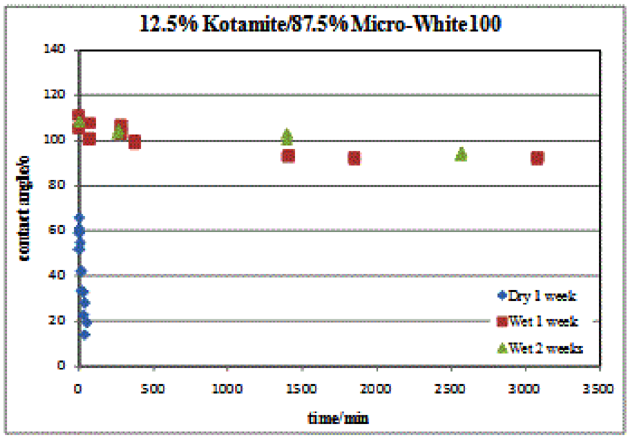


Figure 30. Pilot Scale-up Contact angle measurements the 12.5% Kotamite/87.5% Micro-White 100

Explosibility Testing – The results in Table 30 below indicate that the treated rock dust blend produced from the pilot scale-up inerted the PPC in the 20-L chamber at the 75% inertant concentration where it was as effective at inerting PPC as the an untreated limestone rock dust which is used as a standard inhibitor at OMSHR. Cashdollar [2000] showed that about 75% of this standard limestone rock dust is required to inert PPC in the 20-L chamber which is ~5% lower than the rock dust content required to prevent flame propagation in the full-scale Lake Lynn Experimental Mine explosion tests. This has been confirmed more recently by Man and Harris [2013]. A plot of the 20-L chamber results for the 12.5% Kotamite with Micro-White 100 rock dust blend is depicted in Figure 31.

Table 30. Summary of the explosion inerting experiments in the NIOSH 20-L chamber on the Pilot Scale-up sample

20-L Chamber Coal Concentration	200 g/m³	200 g/m³	400 g/m³	400 g/m³	600 g/m³	600 g/m³
% Rock Dust Concentration	70	75	70	75	70	75
Micro-White 100	go-go	no-no	go	no-no	go	no-no
Kotamite	go	no-no	no-no	no-no	no-no	no-no
12.5% Kotamite / 87.5% Micro-White 100	go	no-no	go	no-no	go	no-no

Go = explosion, No = no explosion
A "no-no" designation indicates that two experiments were conducted and both tests inerted the coal dust.

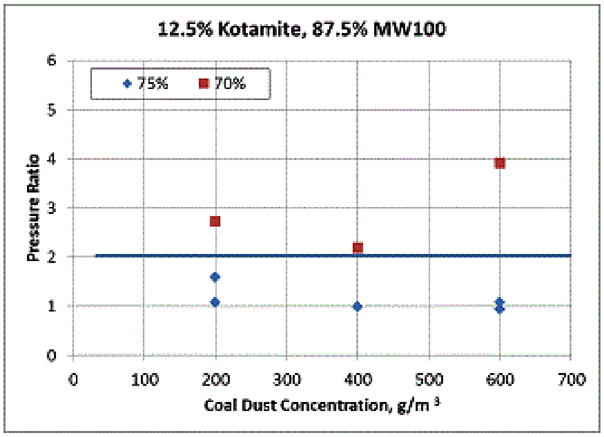


Figure 31. 20-L chamber inerting results with PPC and 12.5% Kotamite/87.5% Micro-White 100

Observations from Pilot Scale-Up

The 20 ton scale-up manufacturing trial held on February 14, 2014 was deemed successful. A blend consisting of 12.5% Kotamite/87.5% Micro-White 100 was manufactured using an 8,000 lb horizontal powder mixer with medium intensity. The mixer was set up as a batch process mixing 3,500 lb of Imerys Micro-White 100 with 500 lb of the Imerys Kotamite. Samples from the trial was collected and analyzed.

The chemistry of the two base components used in the trial was measured to confirm that the values were similar to those obtained previously. TGA and MicroTrac particle size analysis confirmed that the samples were accurately blended.

A composite sample representative of the scale up trial was evaluated for contact angle, dust dispersion testing and 20-L explosibility testing to ensure that prior observations were realized. This was the case.

Following the scale-up trial, NIOSH evaluated within the Bruceton Experimental Mine an untreated rock dust (Micro-White 100) and two blends of the untreated rock dust with Kotamite (at 10% and 20%) along with the scale-up blend (12.5% Kotamite/87.5% Micro-White 100). The results showed that the untreated rock dust immediately wicked moisture from the mine strata and was not dispersible when wet or later when dried. All of the treated rock dust blends dispersed immediately after application and continue to disperse over time (one since July 2012).

Summary and Recommendations

The study has shown that the use of a small quantity of stearic acid treated limestone as an additive improves the dispersion capabilities of traditional rock dust after it has been wetted, dried and exposed to a defined pulse of air. Regardless of the blend components or their ratios, all the rock dust blends dispersed when compared to conventional rock dusts which formed essentially a non-dispersible cake when tested in the NIOSH developed dust dispersion chamber. Additives levels as low as 7.5% were found to be effective in aiding dispersion. There was no dispersion performance advantage seen in treated additive levels above 12.5%.

Results have shown that the presence of free stearic acid in the tested rock dust blends does not cause an explosion as evidenced by the fact that two commercially available stearic acid treated limestone's (Kotamite and Supercoat) were themselves effective at inerting PPC at the 75% rock dust level within the NIOSH 20-L explosibility chamber. However, it is recommended that any additional investigation in this area should always consider the free stearic acid component. The use of Thermogravimetric analysis was found to be a useful tool in the validation of the correct blend proportions, and at blend levels above 25% proved effective at identifying the presence of free stearic acid for the products in this study. At blend levels of 25% or lower, free stearic acid was not detected and it theorized that the small amount of free stearic acid (0.1 wt%) reacts with the limestone during the TGA measurement process. Throughout the process of preparing and testing the blends there has been a confidence developed concerning validation of the correct blend proportion based upon the particle size results (of the base materials and blends) and the TGA results for the blends. It is uncertain whether this confidence would hold true for other limestones particle size distributions. This would need to be studied further.

The NIOSH designed dust dispersion chamber and test protocols developed for this study are an excellent qualitative indicator of whether a rock dust will disperse into separate particles by a defined pulse of air. Results obtained from the dust dispersion chamber calibration study showed the Imerys dust dispersion chamber values to be higher than those obtained from NIOSH for the same samples. This was primarily due to variations in the air nozzle pressure and the angle of the nozzle in relation to the dust tray. However, absolute agreement was not necessary as the dust dispersion chamber was used to show the relative dispersibility of dry, wet, and wetted then dried treated and untreated rock dusts. In all cases, the treated products resulted in a larger powder loss from the tray as the additives enhanced dispersion.

From a quantitative standpoint it was difficult to discern any trends between the various blend series. As the project progressed it became apparent that dust dispersion chamber results were affected by some chamber design inadequacies. It was noticed that the powder would settle between the time the trays were initially filled and when they were actually tested in the dust dispersion chamber. This settling allowed the pulse of air to pass over the material without contacting all the powder. This settling increased with samples that were subjected to moisture and then allowed to dry before testing. It is recommended that a tray be used with bottom support but minimal side support. The sample preparation could be a variant of the original "rock dust anti-caking" protocol provided by NIOSH in Appendixes B and C. It is believed that a more relevant quantitative dispersion test could be developed with some modification of the original NIOSH protocols.

Contact angle measurements were beneficial for determining whether a blend demonstrated hydrophobic properties. While contact angle measurements were conducted at two different relative humidity values (35% and 98%), results for the 35% relative humidity tests seemed to be affected by evaporation of the water droplet. No contact angle measurements could be made on the untreated base rock dusts as the water droplet was instantly absorbed by the rock dusts. In general, the contact angle analysis indicated that the blends with the higher percentage treated products retained their contact angle longer, though there was large scatter in the results. As the treated blend component approached 7.5%, the contact angle dropped dramatically. Under high humidity (wet) conditions, the contact angle values for the 7.5 wt% blends were similar to blends with higher treated component levels. It was theorized that the higher humidity 7.5% sample had imbibed water and there is no place for the droplet to go.

Contact angle results did not correlation well with the dust chamber dispersion results. As mentioned above the various 7.5% blends in Phase 2 showed poor hydrophobicity. The wet dust chamber dispersion results for these blend samples with the 7.5% stearic acid treated products content exhibited dispersion as good as the samples containing 12.5%. The drop off in performance that would have been expected from the contact angle work was not seen with the wet dust chamber dispersion test results.

Since particle packing was taken into consideration in the blend compositions it was not obvious how large a contribution particle packing was in the overall study especially when looking at the dust dispersion chamber and contact angle results. The study did not investigate blends that were not particle packed and this could be the focus of a future study. However, as the project continued RO-40 and #8 White blend series were eliminated for being either too coarse or failing to inert the Pittsburgh pulverized coal (PPC) dust at the 75% inertant concentration within the NIOSH 20-L chamber. In addition Drikalite was excluded based on it being potentially too costly to the industry (a fine 8 µm product) and it did not offer any substantial benefits over #10 White or Micro-White 100, the remaining two base rock dusts.

The use of ASTM D698, the Proctor Test, to quantify the caking characteristic of the various blends was only partially successful as blends containing >7.5% of a treated product did not form a cake to allow for ASTM D698 to be performed. A modified Proctor Test could be used as a qualitative pass/fail test to validate that final blends contain >7.5% of a treated product.

The dust dispersion chamber test results coupled with explosibility testing offers compelling evidence that the use of a small quantity of stearic acid treated limestone as an additive improves the dispersion capabilities of a typical rock dust. Individual test results coupled with economic considerations led to the recommendation of a 20 – 25 µm untreated rock dust (Micro-White 100) blended with 10% ± 2.5% of a 3 µm treated component (Kotamite) for more extensive testing within an area of NIOSH's Safety Research Coal Mine or similar facility.

NIOSH evaluated within the Bruceton Experimental Mine an untreated rock dust (Micro-White 100) and two blends of the untreated rock dust with Kotamite (at 10% and 20%) along with the scale-up blend (12.5% Kotamite/87.5% Micro-White 100). The results showed that the untreated rock dust immediately wicked moisture from the mine strata and was not dispersible when wet or later when dried. All of the treated rock dust blends dispersed immediately after application and continue to disperse over time (one since July 2012).

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Appendix A:

Rock Dust Blending: Welex Blender

- Purpose: To produce homogeneous mixture of stearic acid coated Calcium Carbonate and Calcium Carbonate with no coating
- Scope: This method describes a procedure to blend stearic acid coated Calcium Carbonate and Calcium Carbonate with no coating

- Responsibilities: Pilot Plant
- Work Instructions:
  - Apparatus:
  - Welex Blender
  - Balance
  - Timer
  - 2 & 5 gallon buckets
- Sample Weighing and Blending:
- Determine the weight of each material to mix
- Weigh each material on balance
- Pour the materials into Welex
- Turn on the blender
- Blend for 10 minutes
- Transfer the blend from Welex to a container
- Clean the blender

Figure A-1. Welex Blender

Figure A-2. Showing the inside of the Welex

Appendix B:

Dust Dispersion Chamber and Testing Protocols

Dust dispersion chamber

A laboratory dust dispersion chamber (Figure B-1) was developed by NIOSH to provide a reproducible air pulse across the surface of the sample to compare the relative dispersibility of rock dust samples. In particular, the objective was to compare dry and wetted then dried treated and untreated rock dusts. The air nozzle and tray arrangement of the dust dispersion chamber is shown in Figure B-2. All test samples were subjected to the same 0.3-sec air pulse from a 40-psi compressed air source to give a peak dynamic pressure of ~4.2 psi. Sample dust trays were weighed before and after each air pulse to determine the amount of dust dispersed. While this apparatus and test procedure cannot hope to duplicate the dust dispersal mechanism in advance of a propagating mine explosion, it does provide a repeatable dynamic pressure source and quantitative measure of the relative mass loss and dispersibility of dusts under controlled dispersion conditions.

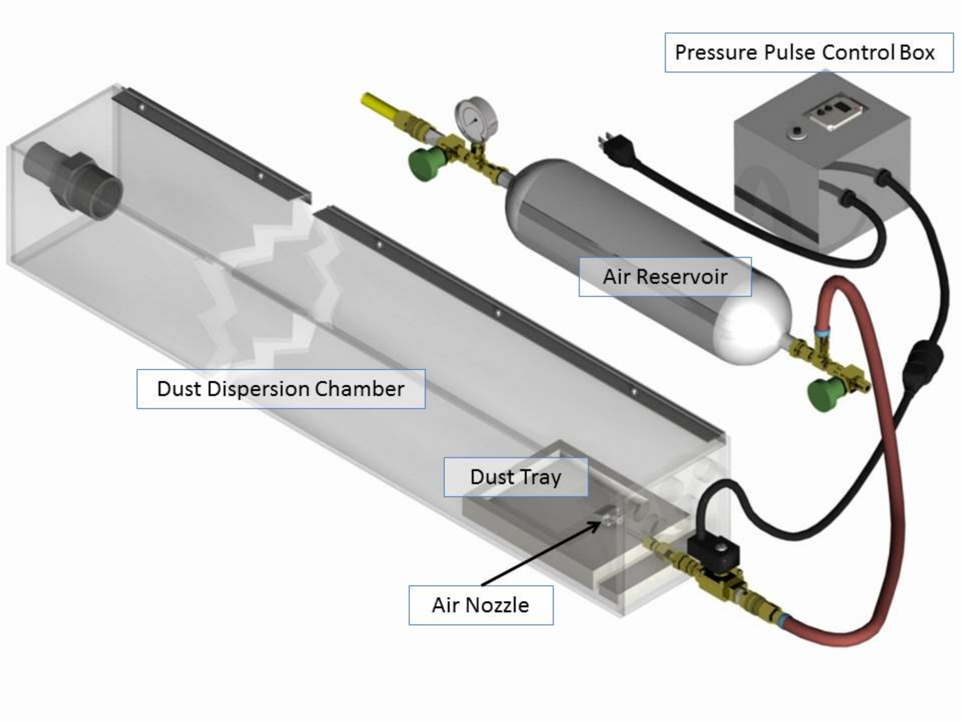

Figure B-1. The NIOSH prototype dust dispersion chamber

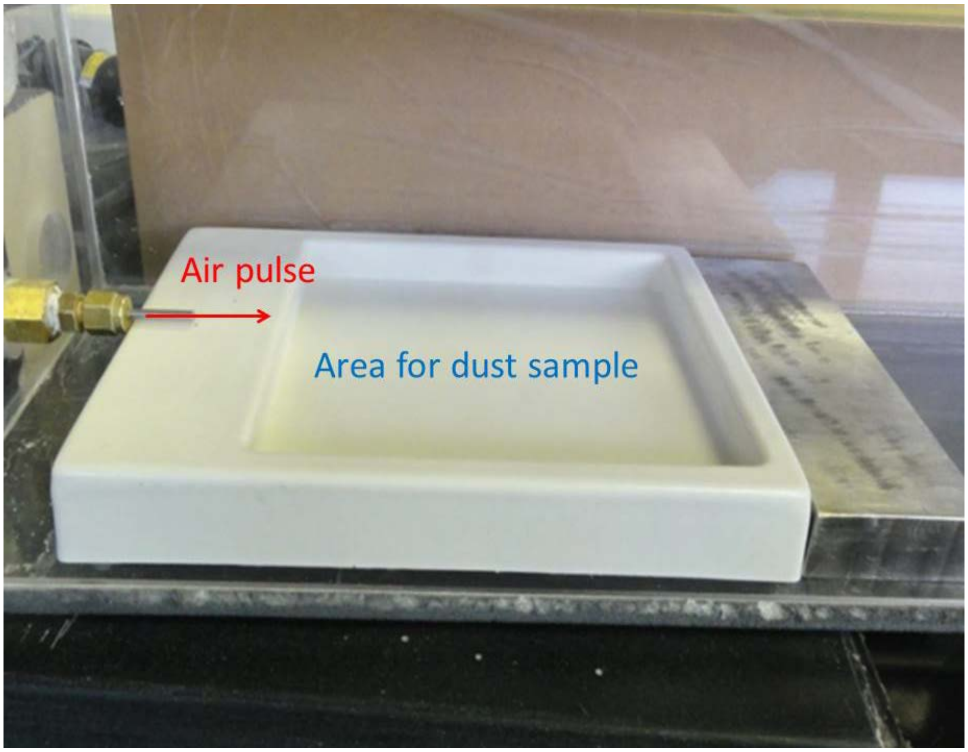

Figure B-2. The air nozzle and dust tray arrangement within the dust dispersion chamber

Dust dispersion chamber dry dust testing protocol

<u>Pretest set-up</u>

- Slide empty tray in chamber under the nozzle so that the side is parallel to and in contact with the back wall of the chamber and the front of the tray is against the nozzle support fitting. Place stainless steel buttress weight against the back of the tray to hold it in place. Close the Plexiglas front door.
- Set the gage to 40 psi. Make pressure adjustments using the 2 valves.
- Make sure that the pulse is at 030 (0.3 s) setting on the time delay relay.
- Turn on the vacuum cleaner.
- Adjust valve to maintain a differential gage pressure throughout a series of tests. Best range from 0.2 to 0.4 in WG to maintain flow within the chamber. Flows greater than 300 ft/min (~0.5 in WG) may cause the rock dust sample to erode before the pulse of air is applied.

<u>Test Set-up</u>

- Weigh empty plastic tray. Make sure that the weight of the tray is centered and evenly distributed on the balance pan. Record weight.
- Place approximately 350 g of dust in a 1 gallon plastic bag. Seal the bag (leave air in bag) and shake vigorously for 1 minute.

- Place tray on a larger pan and fill with dust. Spooning the dust into the tray works best. Overfill the tray.
- Starting at the front of the tray (large ledge) using an 8-in drywall knife at an approximate 20º angle, scrape off the excess dust so that the dust is level with the top of the tray. Repeat twice in the same direction.
- Reweigh and record the weight of the filled tray.
- Slide the filled tray in the chamber under the nozzle so that the side is parallel to and in contact with the back wall of the chamber and the front of the tray is against the nozzle support fitting. Place stainless steel buttress weight against the back of the tray to hold it in place. Close the Plexiglas front door.
- Verify that the gage is set to 40 psi. Make pressure adjustments using the 2 valves.
- Recheck that the pulse is set at 030 (0.3 s) on the time-delay relay.
- Turn on the vacuum cleaner.
- Push the button on the time delay relay to apply the controlled pulse of air across the dust surface.
- After dust has cleared, turn off vacuum and open up chamber door. Take out tray and reweigh. Record the weight.

Dust dispersion chamber wet dust testing protocol

After the dry candidate rock dust samples were evaluated as per the protocol listed above, the untreated and treated rock dust samples were wetted and dried then tested again in the dust dispersion chamber to determine the effectiveness of the anti-caking additives blended with the base rock dusts. The testing protocol was modified as follows:

- Fill a level 9 in by 13 in tray with water to a depth of ½ in.
- Set the dust-filled plastic tray in the water-filled tray for 3 minutes.
- Remove the dust-filled tray from the water and allow it to dry for 24-hr in a laboratory atmosphere with minimum exposure to dust, moisture, and vibration.
- At the end of the 24-hr drying period, reweigh the dust-filled tray and record the weight.
- Slide tray in dust dispersion chamber and follow the dry dust testing protocol listed above.

Appendix C:

Wicking Protocol for Testing Wet Dust and Wetted, Then Dried Dust in the Dust Dispersion Chamber

Wicking Protocol

- Weigh the clean tray for the initial weight.
- Insert the filter paper (Whatman # 4 filter) into the plastic dust dispersion tray and weigh the tray.
- Fill the tray with the rock dust sample (with or without anti-caking additives) as per the general sample preparation procedures.
    - Place approximately 400 g of dust in a 3.8 L (1 gallon) plastic bag. Seal the bag and shake vigorously for 1 minute.
    - Place the tray on a larger pan and, with a spoon, fill it with dust. Overfill the tray.
    - Starting at the front of the tray (large ledge) using a 20-cm (8-in) wide drywall knife at an approximate 20° angle, scrape off the excess dust so that the dust is level with the top of the tray. Repeat this step for the second time in the same direction. Carefully remove any dust from the rims and ledge.
- Weigh the tray filled with the leveled rock dust sample.
- Place each plastic (9.7 cm by 8.3 cm by 1.5 cm) or foil (20 cm by 20 cm) water tray inside the desiccator. Fill the bottom-most desiccator tray with distilled water to ensure high humidity within the desiccator cabinet.
- Place each dust tray inside a water tray. Then place each tray inside the desiccator cabinet. Carefully pour 625 ml of distilled water in the foil tray or 800 ml (do not overfill) of distilled water into each plastic water tray. The 800 ml of added water should reach nearly to the top of the sample trays.

Place compact temperature/humidity data loggers in the desiccator cabinet to monitor the temperature and relative humidity (RH). The humidity should be at least 95% RH after a day of storage.

Leave the tray samples in the desiccator cabinet for the desired storage time (7 days, 14 days, 21 days, 1 month and 3 months).

The initial protocol did not involve any substantial drying before conducting the testing in the dust dispersion chamber. This protocol involved the following steps:

- After the storage time is complete, remove each dust tray from the water tray.
- Allow the dust tray to drain on a paper towel for 1 minute and wipe the water off the sides of the plastic tray.
- Follow the general dust dispersion chamber test protocol listed in Appendix B.

The protocol was subsequently updated to allow the dust samples to thoroughly dry before testing within the dust dispersion chamber. The final protocol involved the final steps:

- After the storage time is complete, remove each dust tray from the water tray.

- Wipe clean the bottom of the dust tray with a paper towel, and let it air dry on a bench top until all the wicked water evaporates (24 -48 hours or until the pre-wicking weight is almost equal to the post- wicking weight).
- Follow the general dispersion test procedure for the dispersion chamber tests (Appendix B).

Appendix D:

Particle Size; Microtrac X100

- Purpose: To Determine the Particle Distribution and Related Properties of Calcium Carbonate Products
- Scope: This Method Describes a Procedure to Determine Particle Size Properties of Calcium Carbonate Products Using the Principle of Laser Light Scattering
- Responsibilities: QA Department
- Work Instructions:
  - Apparatus;
    Microtrac Particle Size Analyzer – X-100
    Computer with peripherals and printer
    Reagent - Isopropyl Alcohol
- Sample Preparation and Analysis:
  - Turn the Flow Switch "On"
  - From Microtrac Menu select "OK." This will take you to the "Main Menu"
  - From the Main Menu press "Select." ***Measure X-100*** will appear
  - Enter sample Identification Information – press OK
  - Press the Icon with 4 arrows, this is your auto-sequence.
  - When completed, "Sample Loading Screen" will appear
  - Status - Add Sample will appear
  - After Sample has been added, the X-100 will run 2 particle size curves.
- Record information

Appendix E:

Thermal Gravimetric Analysis

- Purpose: To determine the levels of unreacted and reacted fatty acid on the surface of calcium carbonate products
- Scope: This method describes a procedure to determine the fatty acid coating level, including both reacted and free forms, on calcium carbonate using thermal gravimetric analysis (TGA)
- Responsibilities: QA Department
- Work Instructions:
  - Apparatus
    - Perkin Elmer Pyris 1 TGA
    - Computer with peripherals
    - Compressed air flow at 30ml/min
- Sample preparation and analysis
  - Empty and clean sample pan, raise furnace and record zero weight
  - Lower furnace, load sample into sample pan (20-50mg), raise furnace and record sample weight
  - Initiate heating program (25-550^{o}C at 20^{o}C/min)
  - At end of run, identify free (150-220^{o}C) and reacted (280-400^{o}C) fatty acid components, and determine their levels from the Calculate, Delta Y function

Appendix F:

Contact Angle Analysis - Experimental Method

Experiments on pressed tablets - no direct contact with liquid water

- The powder blends (5 g) were pressed in a 31mm dia. XRF press using a plastic XRF cup at 18 tonnes for 2 minutes.
- The tablets were removed from the press and placed in a clear plastic chamber with a rubber septum inserted through a hole cut in the top.
- The plastic chamber contained a small relative humidity (RH) meter and a plastic vessel containing the appropriate salt solution.
- The chosen solutions were either $MgCl2$ (35% RH) or $K2SO4$ (98% RH).
- The tablets were placed in the chamber below the rubber septum.
- The chambers were sealed and allowed to stand for 48 hr by which time the RH had stabilized.
- De-ionized water droplets (40 ml) were carefully introduced by syringe onto the surface of the pressed tablet.
- Using a Rame-Hart goniophotometer the contact of angle of the droplet was measured.
- In most cases, two droplets were placed on the tablet surface and the contact angle on each side of each droplet was recorded to give four numbers, which were averaged.

Experiments on pressed tablets - direct contact with liquid water

This experiment differed from the first series in the following manner:

- The tablet of powder (10 g) was pressed under the same conditions but without a plastic XRF cup.
- The tablet was placed in shallow-walled plastic petri dish, lined with filter paper, to which was added DI water (1.0 ml).
- The tablet was placed in the chamber in the same way as above and allowed to condition for 1, 2, and 3 weeks.
- The chambers were inspected every few days and the water in the filter paper-lined dish was replenished by addition using a syringe. The contact angle measurement was exactly as described above:
  - The tablet of powder (10 g) was pressed under the same conditions but without a plastic XRF cup.
  - The tablet was placed in shallow-walled plastic petri dish, lined with filter paper, to which was added DI water (1.0 ml).
  - The tablet was placed in the chamber in the same way as above and allowed to condition for 1, 2, and 3 weeks.
  - The chambers were inspected every few days and the water in the filter paper-lined dish was replenished by addition using a syringe.

The contact angle measurement was exactly as described above.