



Encounter with Potential Acidic Sulphate Soil (PASS) and Bulk Emulsion Reaction in a South Africa Iron Ore Mine

D. Scott Scovira Global Manager Blasting Science and Engineering, BME USA Inc

Dirk Voogt General Manager Production, BME, a Division of Omnia Group (Pty) Ltd

Quentin Steyl Senior Chemist - Losberg Lab BME, a Division of Omnia Group (Pty) Ltd

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Reactive Ground Blast Management in a South African Iron-Ore Mine

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Abstract

A South Africa iron ore mine reported a decline in blast performance and ore and waste fragmentation. Preliminary investigation by the explosives services company found that the straight gassed bulk emulsion was breaking down and collapsing inside some loaded blast holes.

The bulk emulsion loaded at the subject site was manufactured in country and shipped to various clients throughout the region, with normal function in blasts at other sites. Historically, the bulk formulation emulsion being used at the subject mine site also performed well and without issue. This raised the question, "why is this bulk emulsion now breaking down and not firing in blast holes?".

Iron ore mines are not typically associated with reactive or acidic ground, and ground sample reactivity testing against the emulsion confirmed this. So, if not reactive ground or acidic water, then what is causing the emulsion to breakdown?

A series of tests were conducted with a mixture of the straight gassed bulk emulsion, plus mine blast hole water, plus blast hole cuttings. This testing scenario represented the total hole loading condition at the mine. This testing scenario identified that this mixture creates an environmental system in the blast hole that results in autocatalytic acid generation with subsequent over gassing and breakdown of the emulsion. A deeper investigation into the ground mineralization revealed the presence of Potential Acidic Sulphate Soils (PASS).

The investigation revealed a new mechanism for bulk emulsion breakdown driven by Potential Acidic Sulphate Soils. The PASS mechanism for over gassing of bulk emulsions had not previously been experienced by the explosives services provider and was the first observed in over 50 years of usage. An extensive search of the public body of knowledge also uncovered no previous description of PASS versus bulk emulsion reactions described by others.

Inhibition of the Potential Acidic Sulphate Soils ground reactions with the bulk emulsion was achieved by introducing an emulsified gassing agent that is mechanically homogenized into the base emulsion off the mobile manufacturing unit. Homogenization produces a very high viscosity bulk emulsion with improved gassing agent dispersion.

The finished homogenized bulk emulsion reduces interaction between the gassing agent and the Potential Acidic Sulphate Soils. These modifications to the bulk explosives' system have proven to provide a window of protection for the bulk emulsion against Potential Acidic Sulphate Soils ground conditions and enhanced resistance to product degradation by the action of dynamic water at the subject mine site.





Introduction

A South Africa iron ore mine reported a decline in blast performance and ore and waste fragmentation. Preliminary investigation by the explosives services company found that the bulk emulsion was breaking down and collapsing inside of some loaded slept blastholes.

See Figure 1 – Decomposed straight gassed bulk emulsion at the collar of blasthole.

Thebulkemulsionloadedatthesubjectsitewasmanufacturedincountryandshippedtovariousclientsthroughout the region, with normal function in blasts at other sites. Historically, the bulk formulation emulsion being used at the subject mine site also performed well and without issue. This raised the question, "why is this bulk emulsion now breaking down and not firing in blastholes?".



Figure 1 Decomposed straight gassed bulk emulsion at the collar of the blast hole.





Reactive Ground and Acidic Water Testing

Mechanisms for AN-based explosives interactions between sulphide reactive ground and acidic water conditions are well known. Iron ore mines are not typically associated with ground or acidic water conditions. In this instance, sulphide reactive ground conditions and/or acidic water were not initially ruled out as suspect mechanisms for the emulsion decomposition being experienced. Preliminary investigation tested exhaustively for AN-based explosive versus suspect ground reactivity and for evidence of acidic water and associated effects.

AN-based explosive interaction with certain sulphide reactive ground types is well documented. 45+ suspect ground samples were collected from the site and tested for AN-based explosive versus ground reactivity. None of the tests returned results that would indicate ground reactivity with the bulk emulsion used at the subject site. Accordingly, it was deduced that reactive ground conditions were not the primary driver for the bulk emulsion decomposition being experienced.

Acidic water is also known to drive over gassing of emulsions that are sensitized with sodium nitrite in water solution. Over gassing decreases the density of the emulsion to the point that bubbles coalescence, the explosive product column collapses, and the emulsion components disassociate, thus rendering the product no longer being functional. See Figure 2 – Field examples of sodium nitrite gassed bulk emulsion decomposition caused by acidic mine water.





Figure 2 Field example of sodium nitrite gassed bulk emulsion decomposition caused by acidic mine water.





At the subject site, emulsion decomposition was associated with loading straight gassed bulk emulsion in water filled holes and/or water saturated ground. In this instance, the issue often expressed itself as a mixture of decomposed bulk emulsion and water expelled out the holes and onto the bench. See Figure 3 - Mixture of decomposed emulsion and water expelled from blasthole. This pointed to the possibility that acidic water could be a suspect cause for emulsion decomposition.

Table 1 pH Measurements

Sample	рН	Temp ℃
1	8.11	18.9
2	7.42	18.9
3	7.49	18.7
4	8.35	18.8
5	8.17	18.9



Figure 3 Mixture of decomposed emulsion and water expelled from blasthole.

Water samples were collected from the subject site and screened. None of the collected water samples exhibited low pH that would be characteristic to acidic water conditions. pH measurements showed the water samples had a neutral to slightly alkaline pH. See Table 1 – pH Measurements. Accordingly, it was deduced that acidic water conditions were not the primary driver for emulsion decomposition.



These systematic eliminations and deductions continued to lead to the question, if it is not reactive ground or acidic water, then what is causing the straight gassed bulk emulsion to breakdown?





New Mechanism Identified for Emulsion Decomposition

The investigation revealed a new mechanism for bulk emulsion breakdown driven by <u>P</u>otential <u>A</u>cidic <u>Sulphate S</u>oils (PASS). The PASS mechanism for over gassing of bulk emulsions had not previously been experienced by the explosives services provider and was the first observed in over 50 years of usage. An extensive search of the public body of knowledge also uncovered no previous description of PASS versus bulk emulsion reactions described by others.

<u>A</u>cid <u>S</u>ulphate <u>S</u>oils (ASS) are soils and sediments containing aluminium and iron sulphides, The most common of which is pyrite. The soils are naturally occurring and when the identification of these is done there are two terms used namely, <u>P</u>otential <u>A</u>cid <u>Sulphate S</u>oils (PASS) and <u>A</u>ctual <u>A</u>cid <u>Sulphate S</u>oils (AASS) (Queensland Government, 2019).

Potential Acid Sulphate Soil (PASS) can become an <u>Acid Sulphate Soil</u> (ASS) in the presence of a strong oxidizer. This conversion reaction results from the presence of <u>Reduced Inorganic Sulphur</u> (RIS) in the soil. Actual Acid Sulphate Soils (AASS) are soil materials that contained RIS, such as pyrite and have undergone oxidation to produce acid. Any pH drop in the pHfox indicates the soil's potential to form sulfuric acid once disturbed and in contact with oxygen.

Potential Acid Sulphate Soils (PASS) are soil materials that contain Reduced Inorganic Sulphur (RIS), such as pyrite, that have the potential to produce sulfuric acid as they are drained or excavated. The field pH of these soils in their undisturbed state is usually more than pH 4 and is commonly neutral to alkaline (pH 7–9).

Actual Acid Sulphate Soils (AASS) are soil materials that contain RIS, such as pyrite and have undergone oxidation to produce acid. This oxidation results in low pH (that is pH less than 4) and often appears as a yellow and/or orange to red mottling (ferric iron oxides) in the soil profile. Actual Acid Sulphate Soil contains Actual Acidity and commonly contains RIS (the source of Potential Sulfuric Acidity) and Retained Acidity.

Soil samples from the subject mine site were tested for the presence of Possible Acid Sulphates Soils. (Government of Western Australia; Department of Environment Regulation, 2015). Table 3 shows the results from the in-house laboratory testing conducted in June 2021.





Site Specific Testing

Recreating the Field Environment

The emulsion decomposition mechanism seen with the straight gassed bulk emulsion and the geology at the subject mine site most frequently occurred in the presence of water. Building upon the standard reactive ground and acidic water tests, the lab conducted an investigative series of non-standard tests to recreate representative field conditions at the subject mine to understand the reaction taking place between the bulk emulsion, the site ground, and the site water.

To more fully understand the absence of ground reactivity and water acidity, two blasthole water samples were sent to an external laboratory for ionic analysis. Table 2 shows the results obtained. The results captured the concentration of cationic species in the water that was present in the blast holes. It was thought that the differences in results between the two samples might be influencing the emulsion in-hole stability. Note the higher concentration of sulfur present in Sample 2.

Table 2 Water Ionic Analysis				
	Water Sample 1	Water Sample 2		
рН	7.97	7.58		
EC μS/cm	774	1,060		
N as NH ₄ ppm	0.31	1.26		
N as NO₃ ppm	15.53	30.93		
CI ppm	41.75	28.17		
Ca ppm	63.25	115.63		
K ppm	14.59	7.15		
Mg ppm	3.63	43.11		
Mn ppm	< 0.01	0.01		
P ppm	0.02	0		
Sppm	12.37	54.36		
Al ppm	< 0.01	< 0.01		
Na ppm	27.42	26.96		
Si ppm	10.08	13.19		
Fe ppm	< 0.01	0.05		
Zn ppm	0.04	0.05		
Cu ppm	< 0.01	< 0.01		
F ppm	0.38	0.24		

A test series was conducted with a mixture of the straight gassed bulk emulsion used on site, plus mine blast hole water, plus blast hole cuttings. This testing scenario represented the total hole loading environment at the mine. This testing scenario demonstrated a repeatable reaction where there is an autocatalytic acid generation and subsequent over gassing and breakdown of the emulsion. The acid reacts with unreacted sodium nitrite in the emulsion and causes over gassing. Over gassing compromises the shear strength of the emulsion making it unable to support the sensitising air bubbles in the bulk emulsion column and the stemming on top of the explosive column.





Presence of Potential Acid Sulphate Soils

Table 3 Presence of Potential Acid Sulphate Soils

pHf conclusion for all samples: No actual acidity - common in undisturbed acid sulphate soil

Sample	рН _f	pH_{fox}	▲ рН	pH _{fox} conclusion	▲ pH conclusion
1	6.65	1.61	5.04	Strongly indicates acid sulphate soils	Potential acid sulphate
2	6.61	1.82	4.79	Strongly indicates acid sulphate soils	Potential acid sulphate
3	7.11	4.96	2.15	Possibly acid sulphate soil	Less positive
4	7.50	3.78	3.72	Possible small amount of sulphides present	Less positive
5	7.85	5.27	2.58	Possible small amount of sulphides present	Not acid/undetermined
6	7.60	5.16	2.44	Unlikely to be acid sulphide in soil	Not acid/undetermined



Soil samples submitted from the mine showed no reactivity with the straight gassed bulk emulsion in the absence of water. Some of the samples showed the potential to become reactive and show signs of break-up in the presence of a strong oxidizer.

In this case, a sodium nitrite-based gassing solution is used to sensitize the base bulk emulsion explosive. It was thus indicative that the strong oxidizing agent used in the gassing solution, when used in the presence of the soil, catalysed a reaction that negatively impacted the emulsion matrix.





Acid Sulphate Field pH Testing Methodology

The field pH tests (pH_F and pH_{FOX}) were developed for the rapid assessment of the likelihood of existing acid sulphate soils in the field. These field tests are no substitute for laboratory analysis as they are purely qualitative and do not give a quantitative measure of how much acid has been or could potentially be produced through oxidation, however they are a useful tool for the indication of potential and existing soil acidity levels.

The pH_F (pH Field) test provides an indication of the existing soil acidity level of a soil-water paste where the pH_{FOX} (pH Field Oxidized) test provides an indication of the potential soil acidity level after oxidation.

This is based on when the sulphide is exposed to oxygen from the air or an oxidizer, and when sulfuric acid is formed according to the following reaction:

Equation 1

FeS2 + 15/4 O2 + 7/2 H2O
$$\rightarrow$$
 Fe(OH)3 + 4H⁺ + 2SO42-

The identification of acid sulphate soil is done to rapidly screen the soil for the potential of acid sulphate by doing a field peroxide pH test. A saturated pH of the field soil (pH_F) is first done and followed by a pH test of the field soil after the addition of 30% hydrogen peroxide.

If sulphate is present in the soil, then the following reaction takes place:

Equation 2

FeS2 + 15/2 H2O2 $\rightarrow \frac{1}{2}$ Fe2O3 + 11/2 H2O + 2H2SO4 + HEAT

The lab reaction ranges from nil to moderate to violent, the lab reactions occur faster than they would naturally occur in the field, however, regardless of time period in the lab or in the field, the amount of acid that forms is the same.





3 Factors Considered to indicate Positive Acid Sulfide Identification

- 1. The strength of the reaction with hydrogen peroxide is used as an indicator. In other words, the reaction is rated from low to almost volcanic. There are a few gradings in literature for the strength of the reaction. This indication alone cannot be used for positive identification as there is organic matter and other soil constituents that will also show a reaction with hydrogen peroxide.
- 2. The value of the pH_{FOX} , especially if it is < 3, together with a significant reaction is a strong indication of PASS. The lower the pH drops below 3, the more positive the presence of sulphides.
- 3. Lastly the difference between the pH_{FOX} value and the pH_{F} value. The larger the difference, the more positive it is indicative of PASS. When the pH_{F} is already low to start with, the difference is not ideal.

The pH_{FOX} value is the most important and most conclusive indicator for PASS. In literature, there are various tables that can aid one in the interpretation of the three factors. The interpretation of the results as presented in Table 3 was done using the classifications reported by Aaron Kuarila (Kaurila, 1999).

See Table 4 – Acid Sulphate Classification System Using the Field Peroxide Test.

Table 4 Acid Sulphate Classification System Using the Field Peroxide Test

Classification	Reaction	pH Drop	pH _{FOX}
a. Potential Acid Sulphate**	Moderate to violent (3 - 5)	> 2	< 3
b. Potential Acid Sulphate***	Moderate to violent (3 - 5)	1 - 2	< 3
c. Actual Acid Sulphate	Nil to slight (0 - 2)	< 1	pH _f < 3.5 pH _{FOX} < 3
d. Less Positive	Moderate to violent (3 - 5)	0 - 2	3 - 4
e. Undetermined	Moderate to Violent (3 - 5)	< 1	4 - 5
f. Not Acid Sulphate	Nil to slight (0 - 2)	< 1	> 4

Agriculturally significant | *Environmentally significant

After Kaurila, 1999





Managing PASS Emulsion Decomposition

Gassing Agent Testing and Modifications:

As the soil samples indicated a possible reaction in the presence of a strong oxidizing agent, the difference in the effect of standard sodium nitrite and water gassing solution and an <u>E</u>mulsified <u>G</u>assing <u>Agent</u> (EGA) was investigated. EGA is an emulsion and is incorporated more readily into the base emulsion on mixing and prevents the contact between the gassing agent (a strong oxidizer) and the Potentially Acid Sulphate Soils.

Samples of straight bulk emulsion were tested by gassing with both sodium nitrite in water solution and EGA in the presence of mine water.

In reference to Table 5 below, the specification for the straight gassed bulk emulsion density after 60 minutes is 1.00 to 1.20 g/cc. Table 5 shows the gassing results obtained when testing the straight bulk emulsion in the presence of mine water previously positively identified to have been in contact with PASS.

Table 5 Gassing Agent Comparative Tests

Gassing Agent Test Set up	Density (g/cc) after 60 minutes
Straight emulsion gassed with Sodium Nitrate Solution ⁺	0.94
Straight emulsion gassed with Sodium Nitrate Solution+	1.03
Straight emulsion gassed with Sodium Nitrate Solution++	0.83
Straight emulsion gassed with Sodium Nitrate Solution++	0.83
Straight emulsion gassed with EGA ⁺	1.16
Straight emulsion gassed with EGA ⁺	1.15
Straight emulsion gassed with EGA ⁺⁺	1.10
Straight emulsion gassed with EGA ⁺⁺	1.09
+ No mine water	
++ with mine water	





Homogenization of EGA into Straight Bulk Emulsion Loaded in Blastholes:

A significant degree of inhibition against PASS ground reactions with the straight bulk emulsion has been achieved by the introduction of EGS mechanically homogenized into the base emulsion off the mobile manufacturing unit.

Homogenization results in a very high viscosity straight bulk emulsion with improved gassing agent dispersion. The final gassed emulsion is very thick and holds form. See Figure 4 – Homogenized EGA straight gassed bulk emulsion.



Figure 4 Homogenized EGA straight gassed bulk emulsion.

The finished homogenized straight bulk emulsion reduces contact between the gassing agent and the PASS. These modifications to the bulk explosives system have proven to provide a window of protection for the straight bulk emulsion against site ground conditions and enhanced resistance to product degradation by the action of dynamic water also present at the subject mine site.

The increased gassing agent dispersion and reduced gassing agent bubble size have also been demonstrated in field measurements to increase the velocity of detonation of the homogenized gassed bulk emulsion as compared to non-homogenized gassed bulk emulsion.





Closing Words and Future Work

The purpose of this paper has been to bring awareness to blasting professionals and the global explosives engineering community of a newly observed and documented mechanism for straight gassed bulk emulsion decomposition under specific field application conditions.

Summary of key takeaways and learnings for the reader:

- 1. The investigation revealed a new mechanism for straight gassed bulk emulsion breakdown driven by Potential Acidic Sulphate Soils (PASS).
- Methodology to manage straight gassed bulk emulsion decomposition driven by PASS reactivity
 includes demonstrated application of an EGA (emulsified gassing agent) thoroughly mixed into the
 downhole delivery stream by a homogenizer unit off the mobile manufacturing unit.
- 3. Straight gassed bulk emulsion homogenized with EGA result in an in-hole product that has a very high viscosity and resistance to PASS ground and in-hole water interaction.
- 4. The highly thickened in-hole emulsion product also has additional resistance to attack by dynamic water.
- 5. Homogenization of straight gassed bulk emulsion results in smaller gas bubbles being dispersed throughout the final product, with subsequent measured gains in detonation velocity.

As this emulsion decomposition mechanism is recently discovered it is anticipated that additional learnings and understandings can be expected in the future, with subsequent continuous improvement to the inhibition process against sulphate ground types through the chemical formulation and mechanical manipulation.





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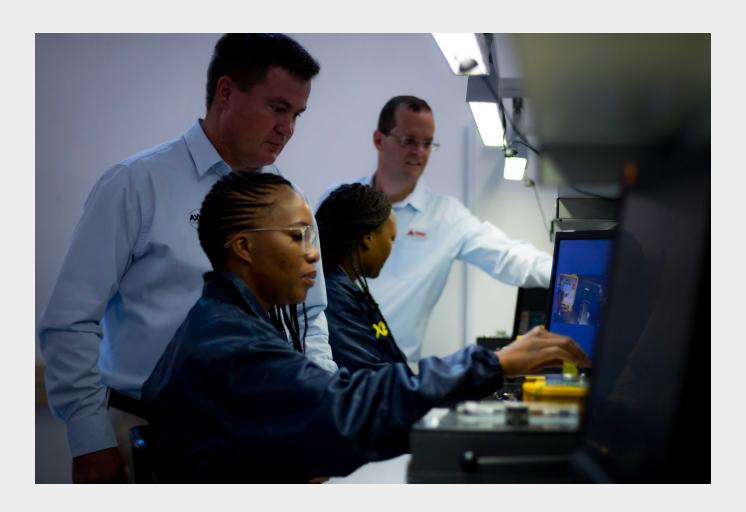




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