MINIMUM BURNING PRESSURE AS A BASIS OF SAFETY FOR EXPLOSIVES OPERATIONS

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ABSTRACT

Over the last few decades, water-based explosives (emulsion and watergels) and their precursors, despite their apparent insensitivity, have been involved in a number of deadly accidents around the world. A number of these accidents occurred when these products were pumped either for their manufacture, transport, or handling at the end-user's site.

Since seminal work carried out at C-I-L in Canada in the 1970s, it has generally been known that water-based explosives will not undergo a sustained burning below a characteristic pressure, known as the minimum burning pressure (MBP). Different explosives demonstrate different MBP values, depending on their formulation. Above the MBP, if ignited, an explosive will continue to burn. If the explosive is confined, such as in a pump, the pressure will build, causing the burning to accelerate. In extreme cases, the burning can accelerate to detonation, with potentially catastrophic results. Below the MBP, an explosive can of course be destroyed by fire, but will not burn without continuous contact with an ignition source. As a result, one potential basis of safety for explosives operations is to ensure that the system pressure remains below the MBP of the product being handled. In Canada, "Guidelines for the Pumping of Water-Based Explosives" were developed, that were endorsed by both the commercial explosives industry and the Canadian government. These guidelines stated that "using pumping pressures well below the laboratory measured minimum burning pressure (MBP) of the corresponding emulsions or watergels is a good safety practice".

However, in order to use MBP as a practical basis of safety, there needs be a solid, uncomplicated methodology to measure it. Over the last decade, a research program at the Canadian Explosives Research Laboratory (CERL) has been focused on the development of a validated protocol for realistic MBP measurements. This has required an extensive experimental program to study the many effects that can complicate the interpretation of the test results. The technology is now mature, greatly simplified compared to earlier versions and readily transferrable to other laboratories. Routine MBP measurements have been included as a requirement in standards for the classification and authorization of high explosives in Canada.

The paper provides examples of accidents involving water-based explosives, summarizes the current state of the experimental MBP methodology, and describes the effect of common formulation changes, such as water content or the presence of chemical sensitizers, on the MBP.

INTRODUCTION

While several quantitative tests have been developed to investigate thermal runaway hazards, very few tests have been put forward to quantify hazards originating from localized thermal ignitions in commercial emulsions. The closest test used on a regular basis is the Internal Ignition/DDT test in the UN TDG Manual of Tests and Criteria (United Nations Publications, 2009), in which a black powder charge is used both to ignite and pressurize a confined sample. However, this test provides no quantitative information and involves the potential detonation of 2.5 kg of explosive in a steel pipe, with all the attendant safety concerns. It is known that, following local ignition in water-based explosives, there is a minimum pressure required for combustion to propagate. The latter is usually referred to as the 'Minimum Burning Pressure' (MBP). The MBP is a very important and useful safety parameter as, if an explosive is maintained below its MBP, accidental ignition and propagation should not be possible. Above the MBP, if ignited, an explosive will continue to burn. If the explosive is confined, such as in a pump, the pressure will build, causing the burning to accelerate. In extreme cases, the burning can accelerate to detonation, with potentially catastrophic results.

Over the last few decades, water-based explosives, despite their apparent insensitivity, have been involved in a number of pumping accidents around the world. Some of these accidents have resulted in fatalities (Perlid, 1996). Two accidents were particularly significant in the Canadian context. In 1975, eight workers were killed and 19 injured during the manufacture of EGMN-sensitized watergel at the Canadian Industries Ltd. Beloeil Works; it is thought that deadhead pumping in a progressive cavity pump was the initiation source. In 1988, four people were killed and two injured at the ICI Explosives Research Laboratory in McMasterville, Quebec while using a high-pressure piston pump to process an experimental emulsion. Recognition of the hazards of pumping explosives has led to the development of pumping guidelines that were endorsed by both the commercial explosives industry and the Canadian government. These guidelines stated that "using pumping pressures well below the laboratory-measured minimum burning pressure (MBP) of the corresponding emulsions or watergels is a good safety practice" (Hanley and Shaw, 2003). However, in that document originally from 1998, there was no description of how the MBP should be measured.

While a few studies have reported on the use of various pressurized vessel tests to measure the MBP of water-based explosives, significant uncertainties about the applicability of these data arose (Wang, 1991; Hirosaki, 2000; Turcotte et al., 2005). Over the last decade, a research program at the Canadian Explosives Research Laboratory (CERL) has been focused on the development of a validated protocol for realistic MBP measurements. This has required an extensive experimental program to study the many effects that can complicate the interpretation of the test results. The present paper describes how the resulting protocol has been used to study the effect of common formulation changes on the MBP of void-sensitized, ammonium-nitrate-based emulsion explosives and the corresponding unsensitized ammonium-nitrate-based emulsions (commonly known as ANEs).

DEVELOPMENT OF THE EXPERIMENTAL TECHNIQUE

The technique to determine the MBP was pioneered for watergel explosives at Canadian Industries Ltd. in the 1970s and is a modification of the classic strand-burner technique used to measure the effect of pressure on the burn rates of propellants (Chan and Kirchnerova, 1978). In principle, MBP measurements are simple to carry out: put some explosive in a vessel, pressurize and ignite with a hot wire. If all the emulsion burns, we are above the MBP, if it doesn't, we are not. In fact, there are a number of pitfalls to avoid in order to obtain meaningful results. We have spent several years at CERL working out these details and would be happy to share them with other organizations.

The MBP for all the emulsions investigated here were determined using methodologies already described in detail elsewhere (Goldthorp et al., 2008; Turcotte et al., 2008; Turcotte et al., 2010). MBP determinations were performed in either a 4-L or 19-L pressure vessel depending on whether the emulsion contained aluminum: the 19-L vessel is equipped with a larger vent which is less easily clogged by aluminum residue upon depressurization at the end of the test. The test vessel was not vented during testing. Extensive research conducted at CERL on the mechanisms of ignition, propagation and combustion in these materials has shown that vessel size, rate of pressurization and over pressure after ignition does not play a significant role in determining their MBP values.

Numerous modifications to the original test cell design and testing methodology have been made over the years in order to improve the reliability of the results and to simplify the measurements (Goldthorp et al., 2008; Turcotte et al., 2008; Turcotte et al., 2010). A key goal for the research program has been to develop a methodology that not only produces valid results but which could also be adopted fairly readily by other organizations. The current MBP test protocol (Turcotte et al., 2010), which is greatly simplified compared to earlier versions, uses a test cell with cylindrical geometry positioned horizontally in the pressure vessel, as illustrated in Figure 1. Sample test cells are fashioned from commercially available steel nipples 7.6 cm in length with an inside diameter of 1.6 cm. About 20 g of sample is used per test. Each test cell has a 3-mm wide slit machined along the axis to allow combustion gases to escape during testing. Although the ends of the nipples are threaded to accommodate caps, the cell ends are plugged with a No. 0 neoprene stopper which has been reamed out to accommodate the copper conductor and splice connector of the hot wire ignition assembly. By using neoprene plugs, the hotwire ignition assembly is only in direct contact with emulsion available to participate in ignition and combustion.

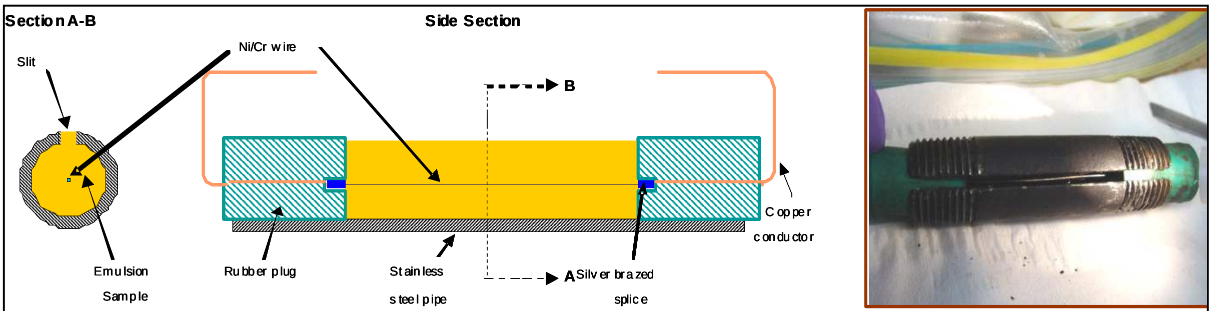

Figure 1: Horizontal testing geometry

Upon successful ignition, the combustion front propagates radially outwards from the axis of the hotwire as if it were in an infinite mass of the sample, quenching only when it reaches the walls of the cell and when no sample remains. It should be noted that the differences among the testing methodologies we have reported (Goldthorp et al., 2008; Turcotte et al., 2008; Turcotte et al., 2010) on do not significantly affect the final MBP result of the test; instead, using the current MBP methodology (Turcotte et al., 2010), the interpretation of the type of combustion event is easier with this new configuration and parameters such as burn time can be more easily quantified.

All emulsions investigated in this study were prepared in-house over a two-year period using procedures already described earlier (Goldthorp et al., 2008; Turcotte et al., 2008; Turcotte et al., 2010). The ingredients in the emulsion formulations were systematically adjusted. To closely replicate manufacturing and handling conditions of emulsion products, MBP testing was performed on freshly manufactured product.

For the purpose of illustrating the effect of common formulation changes on the MBP, the emulsions have been separated into five major classes, as summarized in Table 1. Because of the proprietary nature of emulsion formulations, the formulation table is generic and indicates only the presence or absence of chemical constituents or additives and only water content and sodium nitrate content is quantified. A variety of surfactant and oil mixtures (SURF 1- 4) in combination with wax (WAX) and diesel fuel oil (DFO) were used as oil phase constituents. Various oxidizer solutions were prepared and combinations included ammonium nitrate (AN), sodium nitrate (SN), sodium perchlorate (SP) and urea with water (WATER). Other emulsion additives included ammonium nitrate prill (AN Prill) and atomized aluminum (AL) as well as the inclusion of void sensitizers/density modifiers such as thick-walled and thin-walled, glass microspheres (GLASS), plastic microspheres (PLASTIC), and polystyrene beads (POLY).

The A, B and C-series of emulsions are packaged products with lower water content and having a stiffer rheology. All emulsions in these series contained approximately 10% SN by mass with the exception of B3. Emulsions B1-B3 also contained SP as a chemical sensitizer. The oil phase was identical in all the A, B and C formulations and was composed of a two surfactant/oil blends (SURF2 and SURF3) and included wax. The D and E-series of emulsions are typical bulk products characterized by higher water content and lower viscosities. The D-series used an oil phase based on a blend of two separate surfactant/oil blends (SURF 3 and SURF 4) with diesel fuel oil (DFO) while the E-series of emulsions used a different mixture of surfactant and oil (SURF 1). The D-series emulsions and E4 – E8 series were additive-free and the oxidizer solution was solely based on AN. In contrast, emulsions E1-E3 and E13-E18 contained approximately 9-11% and 15-20% SN by mass, respectively, in the oxidizer solution. Of the remaining E-series, E9, E10 and E11 were void sensitized/density modified with thin-walled, glass microspheres, plastic microspheres and polystyrene beads, respectively, while E13-E15, E17-E18 were void sensitized/density modified with thick-walled glass microspheres. E12 was the only formulation that contained urea.

EFFECT OF FORMULATION ON MBP

The MBP data for the A-series of emulsions is illustrated in Figure 2. This figure shows how the uncertainties in the MBP values quoted in Table 1 were obtained. Below the quoted uncertainty range, no positive events were observed. Above the quoted range, all events were positive. Within the range of uncertainties, a mixture of positive and negative events were observed. Typically, each sample is subjected to 10 – 12 experiments to determine its MBP.

Effect of Water Content

A linear regression line illustrating the relationship between the water content and the MBP for straight emulsions (i.e., those without aluminum, void sensitizers, or SP) is shown in Figure 3. From this Figure, it appears that the MBP of all-AN emulsions increases linearly with water content over the water content range studied (11 – 25%). We assume that water content controls a significant physical parameter, such as flame temperature, that itself has a strong effect on the MBP.

Effect of Sodium Nitrate

For emulsions with both AN and 9 – 11% SN by mass in the oxidizer solutions, Figure 3 illustrates that the MBP also increases approximately linearly with water content, but the slope of the regression line is much less steep than for all-AN emulsions. As a consequence, the resulting emulsions with more than about 12% water have a much reduced MBP compared to the all-AN versions.

This observed behaviour is consistent with results from small-scale closed-bomb cook-off tests on AN itself and AN/SN mixtures (Yeager, 1999). These results demonstrated that, under confined conditions (no venting), even trace amounts of SN lower the onset temperature for thermal runaway of AN. This is consistent with the lowering effect of SN on the MBP of emulsions observed in the present work.

The E13-18 emulsions contained both AN and 15 - 20% SN by mass in the oxidizer solutions. For these emulsions having both high SN and high water content (Table 1), the overall effect of high SN content on the MBP is not as obvious as described above for the 9-11% SN formulations. Their MBP values remain relatively high (Table 1), but lower than their all-AN counterparts, despite the high SN content. Presumably, the effect of SN content on the MBP may be masked by the very high water content of these emulsions.

Effect of Oil Phase

From Figure 3, plotting the MBP vs. water content for the AN-only emulsions, namely D1-D3 and E4-E8, it is evident that the MBPs of the two series of emulsions display very similar trends, despite having very different oil phases.

Effect of Substituting AN in the oxidizer with AN prills

The formulations of emulsions A2 and D2 were derived by altering the formulations of A1 and D3, respectively, so that 20% AN prill was substituted for 20% AN in the oxidizer solution. Small variations in the water content were observed as a result of water losses during mixing. In Figure 3, we observe that the MBP of A2 and D3 falls on the trend lines for all-AN or AN/SN emulsions, where the AN is in solution. Therefore, it appears that effect on the MBP of substituting 20% AN prill for 20% AN in the oxidizer solution is not significant.

Effect of Refinement

Emulsion E1a and E1b contain AN/SN oxidizer solutions and are identical formulations with the exception that, during emulsion refinement, some emulsion was removed mid way through the mixing procedure so that the role of product refinement on MBP could be assessed. The remaining base product was further refined. Inherently, as the emulsion is refined in an open container, water losses are encountered and can be significant. For the less refined product emulsion, E1b, the water content was 11.3% while the more refined product, E1a, had 9.0% water. From Figure 3 above, the MBP of emulsions E1a and E1b fall on the trend line for MBP vs. water content for emulsions with AN/SN oxidizer solutions. Thus, increasing refinement for emulsions E1a and E1b did not significantly affect the MBP.

ANWE	MBP (kPa)	AN	SN	SP	UREA	WATER	SURF1	SURF2	SURF3	SURF4	WAX	DFO	AN PRILL	AL	GLASS	PLASTIC	POLY
A1	3040 ± 160	+	+			11.2		+	+		+						
A2	2640 ± 120	+	+			8.1		+	+		+		+				
A3	1940 ± 50	+	+			11.3		+	+		+			+			
A4	1675 ± 125	+	+			10.1		+	+		+				+		
A5	1395 ± 80	+	+			7.1		+	+		+		+	+	+		
A6	2000 ± 170	+	+			7.5		+	+		+		+	+		+	
B1	940 ± 35	+	+	+		6.7		+	+		+		+	+	+		
B2	1585 ± 180	+	+	+		7.8		+	+		+		+	+		+	
B3	935 ± 40	+		+		8.1		+	+		+		+	+	+		
C1	4160 ± 90	+	+			14.7		+	+		+						
C2	2685 ± 170	+	+			10.5		+	+		+		+	+	+		
D1	8685 ± 140	+				16.6			+	+		+					
D2	5880 ± 440	+				13.8			+	+		+	+				
D3	6820 ± 175	+				14.6			+	+		+					
E1a	3290 ± 10	+	+			9.0	+										
E1b	3715 ± 25	+	+			11.3	+										
E2	4680 ± 270	+	+			17.8	+										
E3	5722 ± 38	+	+			17.1	+									+	
E4	4490 ± 780	+				11.7	+										
E5	5850 ± 620	+				14.2	+										
E6	8160 ± 240	+				17.4	+										
E7	11390 ± 96	+				20.8	+										
E8	14240 ± 50	+				24.8	+										
E9	3220 ± 100	+				16.6	+								+		
E10	9845 ± 600	+				17.8	+									+	
E11	>18000	+				15.3	+										+
E12	6050 ± 90	+			+	10.2	+										
E13	8540 ± 170	+	+			18.6	+								+		
E14	>3540	+	+			15.8	+								+		
E15	8035 ± 1025	+	+			19.8	+								+		
E16	7965 ± 285	+	+			20.6	+										
E17	>5620	+	+			15.4	+								+		
E18	9425 ± 360	+	+			17.8	+								+		

Table 1: Emulsion matrix indicating general formulations with measured MBP and water content

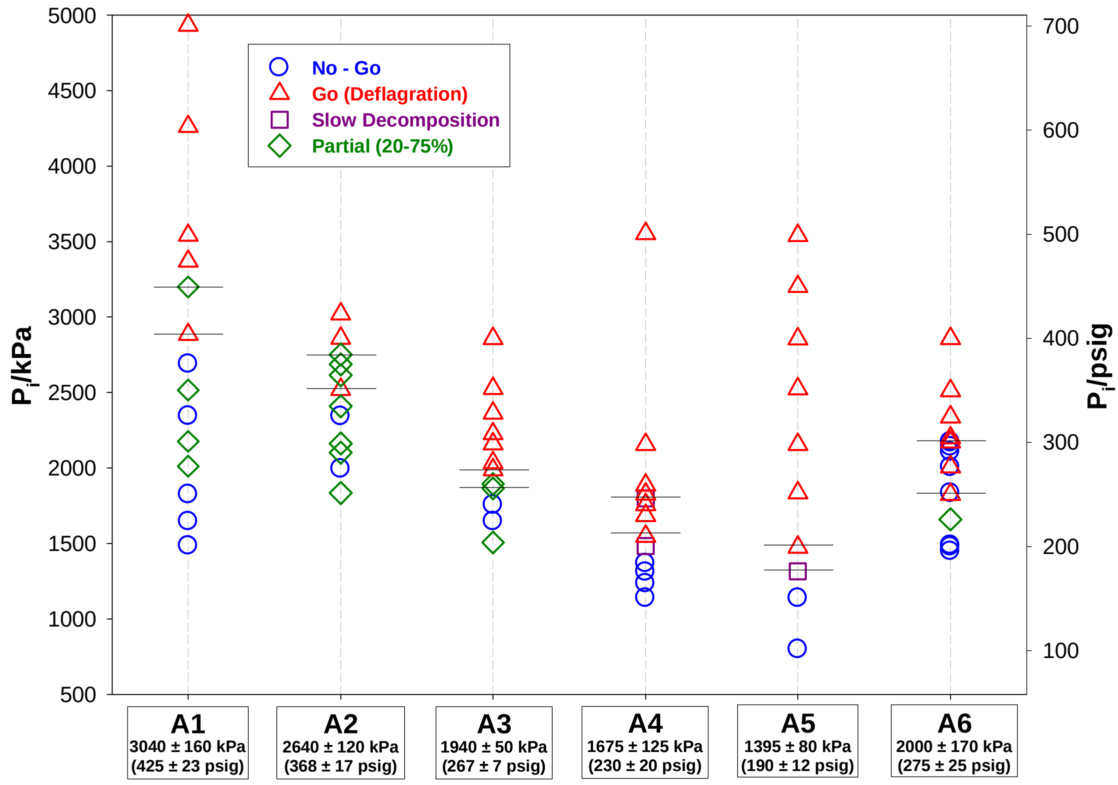

Figure 2: Summary of results for the MBP measurement of A-series emulsions

Effect of Aluminum

The MBP reported for A1 is 3040 ± 160 kPa. For emulsion A3, an equal mass percentage of the AN/SN in the oxidizer solution was substituted with aluminum. As can be seen from Table 1, A3 and A1 had identical water content and the presence of a few percent of aluminum in A3 lowers its MBP by a factor of 1.6. Therefore, it appears that addition of atomized aluminum lowers the MBP.

Effect of Density Modifiers

The effects of adding density modifiers in the form of plastic microspheres, glass microspheres or polystyrene beads, is complex and not yet fully understood. Plastic and thick-walled glass microspheres appear to increase MBP values slightly, an effect that can be understood if one considers these to be primarily inert materials that act as heat sinks.

More surprisingly, it appears that addition of thin-walled glass microspheres reduces the MBP significantly compared to the expected value of the trend line of MBP vs. water content for AN (E9) and AN/SN (A4) emulsions. The reason that thin-walled glass void sensitizer/density modifiers reduce the MBP in these formulae is not yet understood, but may be related to the entrainment of melted glass in the gas flow during combustion. More experiments are planned in the future to better understand this effect.

The single emulsion containing polystyrene beads that was tested (E11) was very difficult to ignite and did not support sustained combustion, even at pressures close to 15 MPa. Thus, adding polystyrene beads appears to either dramatically increase the MBP of this formulation or affect its ignitability. The polystyrene beads displayed a significant melting endotherm above 340℃ when we studied them by differential scanning calorimetry and this may play a role in preventing any sustained combustion.

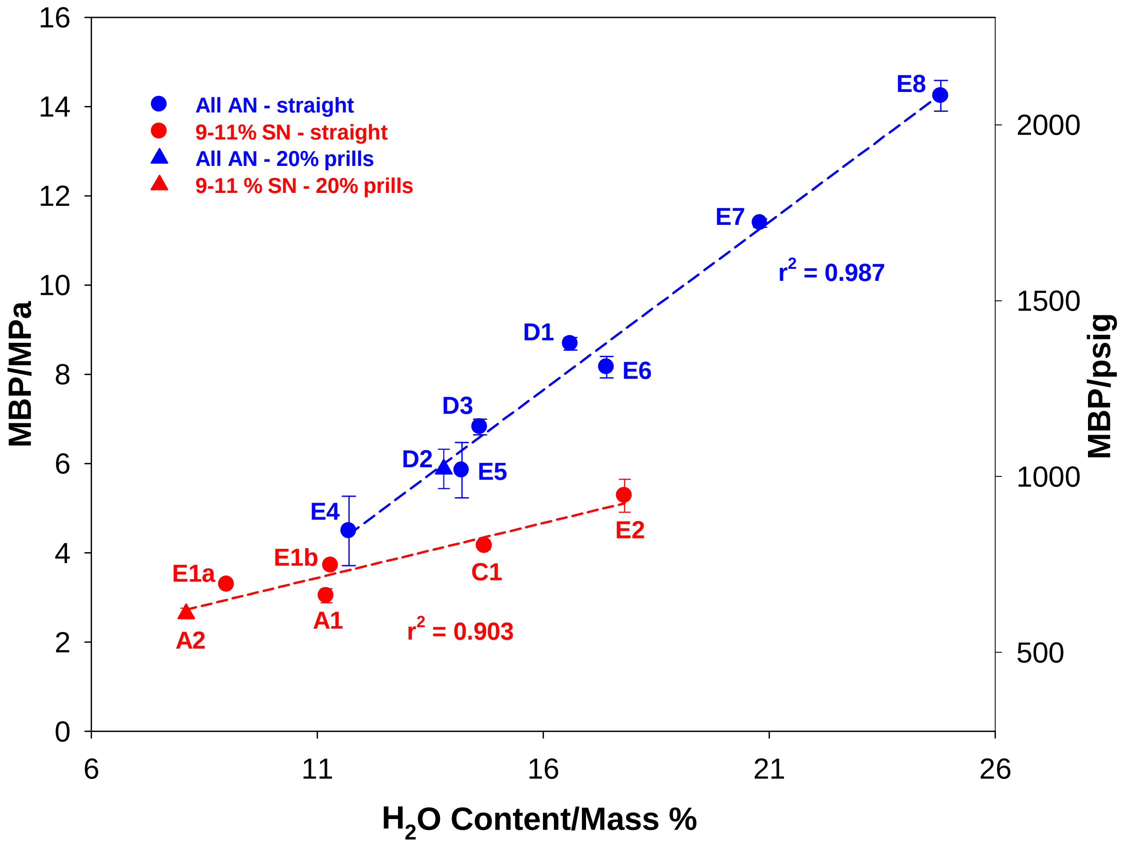

Figure 3: Comparison of the MBP vs. water content of unsensitized all-AN formulae with that of unsensitized formulae containing AN and SN.

Effect of Sodium Perchlorate

The B-series of emulsions are similar in formulation to the A-series except that sodium perchlorate is added in the oxidizer solution. From Table 1, it can be observed that this series of emulsions had the lowest MBP values, which is due to the presence of SP. This observation is consistent with previous MBP experiments that showed that the presence of SP has a lowering effect on the MBP (Goldthorp et al., 2008).

Effect of Urea

Urea has been reported previously to significantly raise the MBP (Goldthorp et al., 2008), and this observation was reaffirmed in the present series of tests. This can be demonstrated by comparing the MBP of E12 (6050 ± 90 kPa), which has some urea substituted for a portion of the AN in its oxidizer solution, with that of the unsensitized version, E4 (4490 ± 780 kPa). The MBP of the product containing urea is very significantly higher, despite having 1.5% lower water content. As for polystyrene above, urea has been shown to also display a fairly energetic endothermic behaviour in DSC experiments, which may explain its very significant effect in increasing the MBP.

MBP AS A STANDARD TEST

We believe strongly that the MBP technique has great potential to be adopted as a standard test and have put significant effort into developing and simplifying the methodology in order to facilitate its introduction into other laboratories. The apparatus can be operated by a single person and the interpretation of the measurements is quite straightforward (if the sample all burns, it is a positive result). For instance, 10 – 12 experiments have been used to obtain each MBP measurement reported in this paper. One person can perform four to six experiments per day; thus, one person performing approximately two days of experiments is required to obtain a detailed MBP measurement. However, a simple go/no-go measurement at a fixed pressure using five measurements would take one person about one day.

In Canada, the MBP test has recently been included in the national standard for blasting explosives (Natural Resources Canada, 2010). For operations involving bulk explosives, the MBP of the product now has to be at least 20% above the maximum pressure that the pumping system can develop, directly incorporating the product MBP into the basis of safety of the overall operation.

Over the last few years, we have begun to work with colleagues in other countries to help with setting up or refining MBP facilities. Progress has been steady and facilities to carry out MBP measurements are now in place in China, South Africa and Spain, as well as Canada. Inter-laboratory testing has also recently been initiated between CERL and laboratories in China and Spain. We would be most pleased to provide assistance to other organizations who wish to set up MBP measurement facilities.

While most applicable to manufacturing and process safety, MBP measurements can be useful in the area of transportation safety, not least because emulsions are regularly pumped on and off transportation vehicles. In addition, the widespread dissatisfaction with the UN TDG Series 8 test for ANEs and particularly the 8 (d) test has encouraged us to investigate whether or not the MBP test might be appropriate for inclusion in the UN Manual of Tests and Criteria. The MBP is a clear measure of the propensity of an ANE to undergo self-sustained deflagration. That being said, the application of the MBP test to transport classification is not totally straightforward, as the test evaluates the potential for deflagration following a localized thermal ignition event, rather than a deflagration as a result of an engulfing fire scenario. However, it is to be expected that materials with low MBP values will be most susceptible to deflagration-to-detonation behaviour in a fire scenario. Furthermore, the test clearly differentiates between products that are currently widely considered to be safe for transport and those that are not. For example, AN-only emulsions with more than 15% water display very high MBP values. “Bulk” ANEs typically have MBPs in the range 6 - 12 MPa, while “packaged” ANEs have MBPs in the range 0.45 – 3 MPa. As a result, we recently proposed that the MBP test be considered as a potential alternative to the 8 (d) test, with UN No. 3375 restricted to products with MBP values above a certain limit (e.g., 5.6 MPa) (United Nations Publications, 2010). The proposal was fairly well received and we plan to continue to pursue this line of investigation.

TAKE HOME MESSAGES

The MBP of a water-based explosive is a key safety parameter that can be used to as a basis of safety for explosives operations.

A validated MBP measurement protocol has been developed at CERL. The methodology has been shown to produce reliable and repeatable MBP values that are consistent with each other for a wide range of emulsion formulations. The measurements are relatively easy to carry out and can be interpreted in a straightforward fashion. Such routine measurements are now part of the standard for the classification and authorization for high explosives in Canada (Natural Resources Canada, 2010).

Clearly, the experimental data obtained from the present work strongly indicates that water content is the major ingredient controlling the MBP of the AN-based emulsions studied here. All else being equal, lower water content means lower MBP. A number of other factors that effect MBP values have been identified:

- Reduce MBP: SP, Al and SN.
- Little effect on MBP: oil phase, refinement, replacing AN in solution with AN prills
- Increase MBP: urea, polystyrene
- Variable effect: microballoons

Although a significant amount of work went into the present study, the job is not yet finished. Some of the effects that we have identified (e.g., the effect of urea) need to be confirmed with more examples before we can make definitive statements. Other effects are not yet well understood (e.g. the effect of glass microballoons) and warrant further investigation. We also plan to look at additives such as calcium nitrate that have not yet been included. Finally, essentially all of our work to date has been on emulsions, so we plan to extend it to include watergels in the future.

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