Video Article

Minimum Burning Pressures of Water-based Emulsion Explosives

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URL: https://www.jove.com/video/56167
DOI: doi:10.3791/56167

Keywords: Chemistry, Issue 128, Ammonium Nitrate, Commercial Explosives, Hot-Wire Ignition, Minimum Burning Pressure, Water-Based Explosives, Emulsion Explosives, Combustion, Explosives Hazard

Date Published: 10/31/2017

Citation: Turcotte, R., Badeen, C.M., Goldthorp, S. Minimum Burning Pressures of Water-based Emulsion Explosives. *J. Vis. Exp.* (128), e56167, doi:10.3791/56167 (2017).

Abstract

This manuscript describes a protocol to measure the minimum pressure required for sustained burning of water-based emulsion explosives. Pumping water-based emulsion explosives for blasting applications can be very hazardous, as demonstrated by a number of pump accidents around the globe in the last decades, including some that resulted in fatalities. In Canada, the recognition of this hazard has led to the development of pumping guidelines that were endorsed by both the explosives industry and the Explosive Regulatory Division of the Canadian government. In these guidelines, it was noted that the minimum burning pressures (MBP) measured in a laboratory would provide a good guide to characterize the behaviour of these products in pumping systems. The same guidelines also call for the design of pump systems that prevent, whenever possible, pressures from exceeding the MBP of the product being pumped. At the time of publication of these guidelines, a methodology existed for measuring such MBP values but it had never been validated to measure the MBP of ammonium nitrate water-based emulsions (AWEs). AWEs are now used much more widely than any other water-based explosives and precursors in on-site bulk loading operations.

The Canadian Explosives Research Laboratory (CanmetCERL) has been conducting research over the last ten years to develop a validated testing protocol to measure and interpret representative MBP values for AWEs. The test, as it is performed today, will be described and the critical components will be justified by reference to recent published data. Results of MBP measurements, for a range of AWE products, will be presented. Inclusion of the MBP test in the test standards for the authorization of high explosives in Canada will also be discussed.

Video Link

The video component of this article can be found at https://www.jove.com/video/56167/

Introduction

The ammonium nitrate water-based emulsion (AWE) explosive was invented in 1961. It consists of microscopic droplets of a liquid oxidizer solution surrounded by a continuous oil phase. The first stable and practically useful emulsion blasting explosive was developed by Harold F. Bluhm in the USA (1969) [1,2]. However, the successful commercialization of this type of explosive did not really happen before the beginning of the 1980s.

With the large scale of modern mining operations and the advent of fast bulk explosive loading methodology, very large volumes of AWE explosives have to be manufactured and transported. One tanker load typically transports 20 tons of AWE and many such truck loads are usually necessary to load only one blast. Accidental initiation of such large quantities of explosives would be particularly disastrous and, therefore, a good knowledge of their hazardous properties is required to design corresponding safe handling systems. While it is well known that emulsions are relatively insensitive to mechanical events (*i.e.* impact and friction events), accidental explosions have still been reported [3] while handling this type of explosive, particularly in pumping applications.

It has been known since the 1970's [4] that a minimum ambient pressure is required for self-sustained combustion to take place into water-based explosives. This latter value has usually been termed the "Minimum Burning Pressure" (MBP). From a safety point of view, knowledge of this threshold could allow manufacturers to better estimate safe operating pressures for various handling equipment.

The Department of Natural Resources of the Government of Canada has published "Guidelines for the Pumping of Water-Based explosives" [5], which state that using pumping pressures well below the MBP of the emulsions or watergels is a good safety practice. It should be noticed that these guidelines were designed with the collaboration of most commercial manufacturers and that, in the USA, the Institute of Makers of Explosives (IME) has also published very similar guidelines [6]. However, in these documents, there was no description or prescription on how the MBP should be measured.

In the last decades, only a few studies related to MBP measurements have been reported. Chan *et al.* [4] reported the results of MBP measurements for watergel explosives, which are also ammonium nitrate and water-based. They have concluded that the MBP can have a strong dependency on several formulation factors such as water content, presence of chemical sensitizers or metallic powders. In another study,

Wang [7] described a 2.5 L pressure vessel pressurized with N_2 and used a Bruceton up-and-down method to determine the MBP for basic AWEs. With this system, MBP values of the order of 15 MPa were measured for a basic emulsion having a water content of 16 mass %.

Using a similar pressurized vessel test, Hirosaki *et al.* [8] have reported the results of some MBP measurements for AWE explosives. They have noted that the nature (*i.e.* glass or resin) of the micro-spheres being used to sensitize the explosives also has a strong influence on the results. More recently, Turcotte *et al.* [9] have developed a system similar to that of Wang and Hirosaki et al. and have attempted to use it to measure the MBP of some AWEs. However, they have found many possible problems that may lead to erroneous MBP determinations. In particular, it was noted that the ignition source geometry (nichrome wire coil) had never been properly validated for AWEs. In 2008, Turcotte *et al.* [10] and Chan *et al.* [11], have developed both an apparatus based on a calibrated ignition wire system and an associated methodology to measure the MBP of AWEs. They have also used the facility to study the ignition characteristics of typical AWEs, measured the energy requirements to obtain reliable ignitions [12] and studied the influence of physical characteristics and ingredients on the MBP of a wide variety of AWE explosives [13,14]. This MBP measurement technique is presently being proposed as a standard test within the United Nation Transport of Dangerous Goods (UN TDG) Tests and Criteria for the classification for transport of AWEs [15].

Protocol

NOTE: The materials and equipment used here are listed in the Table of Materials.

1. Preparation of Ignition Wire Assemblies

NOTE: Wearing nitrile gloves is recommended for this operation.

1. Measure a pre-determined length of nichrome (NiCr) wire and cut using a wire cutter. Cut 85 mm lengths for 76.2 mm (3") long test cells.
2. Using needle nose pliers, bend the NiCr wire to make a small loop at each end. With a proper crimping tool, splice each one into 50 cm length of 14 American Wire Gauge (AWG) solid core bare copper wire using uninsulated butt-end splice connectors.
3. Repeat Step 1.1-1.2 to make as many assemblies as required.
   NOTE: It is recommended to prepare several assemblies in advance as each MBP measurement will take 10 to 15 assemblies). A completed wire assembly is shown in **Figure 1**. If all wires are correctly spliced and crimped to the connector, the resistance measured across the wire assembly (as measured on the copper conductors on either side of the NiCr wire) must be less than 0.5 Ω.

2. Sample and Test Cell Preparation

NOTE: Wearing nitrile gloves is recommended for this operation.

1. Prepare test cells each consisting of a small cylindrical steel pipe with a length of 7.6 cm and an internal diameter of at least 1.6 cm. Ensure that each test cell has a 3 mm-wide slit machined along the axis to allow combustion gases to escape during the tests.
   NOTE: AWEs are electrically conductive.
2. To ensure that all the ignition current will go through the ignition wire, paint the interior of each cell with two coats of high-temperature non-conductive paint. Prepare several test cells in advance.
3. Prepare several No. 0 neoprene stoppers by reaming the inside face to accommodate the splice connectors and the copper conductors of the ignition wire assembly.
4. Use the horizontal slit on the test cell to insert the ignition wire into the middle of the cell. Carefully slide a prepared neoprene stopper along each copper wire and insert them into each end of the cell, ensuring that the ignition wire is not compressed or twisted.
5. Pull the ignition wire taut and bend the copper leg-wires vertically to secure them (**Figure 2**, **Figure 3**).
6. Introduce the sample into the cell through the 3 mm-wide slit with caution to avoid causing crystallization of the sample and introducing air voids in the sample. Use a spatula to tamp down the emulsion to remove air voids if necessary. Quickly tap the cell repeatedly on a countertop to ensure that the emulsion settles into voids. Repeat filling, tamping, and tapping until the emulsion no longer settles any further.

3. Loading Sample in Pressure Vessel

1. **Prepare a pressure vessel with the following characteristics to load the sample cell: an operating pressure resistance of 20.7 MPa (or 3,000 psig) (see the Table of Materials), made of stainless steel to avoid long-term corrosion damage from the gaseous reaction products, equipped with two insulated rigid feedthrough electrodes capable of carrying an electric current up to 20 A, and sealed to have a pressure rating equivalent to that of the vessel itself.**
   1. For safety reasons, install the vessel in a protected test room equipped with a rupture disc assembly (see the **Table of Materials**) designed to vent the vessel at a pressure slightly lower than its maximum operating pressure.
   2. In order to vent the vessel after a test, equip the gas outlet with a high-pressure valve that can be operated remotely.
      NOTE: This can be achieved in various ways. For example, it can be achieved using a solenoid valve/air operated valve combination. The inlet of the vessel must be connected to a gas manifold system operated from a nearby protected room capable of remotely pressurizing the pressure vessel to a chosen initial pressure using a pressurized cylinder of argon (nitrogen may be an alternative but may not be as inert). This manifold would be typically custom made out of high-pressure stainless-steel tubing, and high-pressure compression fittings and valves. It is recommended that the vessel also be equipped with a 0-20.7 MPa (0-3,000 psig) pressure transducer.
2. Introduce a test cell with sample (prepared in section 2) into the pressure vessel. Position its long axis horizontally with the slit on the top (**Figure 2**). Connect the bare copper wires to the electrodes inside the vessel. Ensure the former are not touching the body of the vessel. Close and seal the pressure vessel.
3. Using the multimeter ensure there is no electrical contact between each electrode and the body of the pressure vessel.

NOTE: If any contact is detected between an electrode and the body of the vessel, its cause(s) must be determined and actions must be taken to eliminate it before testing can proceed.

4. Performing a Test

1. In the protected room, connect the signal from the pressure transducer to the data acquisition (see the Table of Materials) or available oscilloscope. Also, connect the voltage across the high precision shunt resistor to the data acquisition (or oscilloscope). Ensure that this shunt resistor is also connected in series with a constant current source. Connect the series to the electrodes on the pressure vessel to supply a constant current through the NiCr wire.
   NOTE: Knowing its resistance, the voltage across this shunt resistor provides a measurement of the ignition current.
2. Start the PC-based data acquisition system (or available oscilloscope).
3. **Remotely close the vessel's outlet valve (see section 3.1 Note). Using a pressurized argon cylinder in the instrument room and the gas manifold (described in section 3), start pressurizing the vessel to the required initial pressure for the test.**
   NOTE: Depending on the formulation of the AWE, this pressure can vary anywhere from 0.3 to 19.3 MPa (50 to 2500 psig). If this is the first test with a given AWE product, make an educated guess of the MBP, based on the formulation of the sample, to decide at what pressure this first test must be performed.
   1. Once pressurization is achieved, close the vessel's inlet valve and leave the vessel pressurized for 5 to 10 min to check that the system has no significant leaks. Once this is established, re-open the inlet valve, adjust the pressure to the chosen initial value, and re-close the inlet valve. If a significant leak rate is detected, postpone testing until required maintenance has been performed.
4. Turn on the constant current source and allow a 10.5 A current to flow through the ignition wire. Keep the current on until the sample ignites and melts the ignition wire, stopping the flow of current; this is expected to take a few s. Turn the power supply off after ignition has taken place and the pressure has started to increase.
   NOTE: The pressure may go through one or two minima and maxima and should start to decrease continuously. When this has happened, wait for an extra 10 min before doing anything.
5. Once the test is completed, remotely open the outlet valve and vent all combustion gases to an appropriate exhaust system. Slowly purge with argon for a few min to remove all toxic gas species before opening the vessel. Ensure the vessel is back to ambient pressure before re-entering the test room.
6. **Lock out the constant current power supply (either using a lock out key or unplugging it from the AC power) and walk to the pressure vessel room. Wearing a face mask with appropriate general-purpose cartridge, open the vessel. Recuperate the test cell by undoing the copper conductors from the electrodes and note down all visual observations.**
   1. By removing the neoprene stoppers try to observe how much of the sample has burned. Further document these observations by taking photographs. Once finished, clean the vessel thoroughly (see section 6).
      NOTE: From these observations, if the sample has burned completely (combustion front reached the wall of the test cell; small amount of sample can be left on the neoprene stoppers), the result is deemed to be a 'go'. Decrease the pressure for the next test. Otherwise the result is deemed to be a 'no-go' and the pressure must be increased for the next test (see typical observations in **Figure 4A**). The pressure record from the transducer can also be used as evidence of sustained combustion or not (**Figure 4B**).
7. **Use the steps outlined in section 5 below to analyse the acquired current and pressure data. Repeat steps 4.1 to 4.6 while gradually decreasing the pressure increments (or decrements) until the MBP has been determined to the desired degree of precision (see typical examples in Figure 5).**
   1. Perform a minimum of 10 to 12 tests using this 'up-and-down' methodology.
      NOTE: The quoted MBP must be the mean between the initial pressure of the highest 'no-go' event ($P_{n,max}$) and that of the lowest 'go' event ($P_{g,min}$) (**Figure 5**). The error bar on the measured MBP must be specified as:
      $$\Delta MBP = abs\left[P_{g,min} - P_{n,max}\right]/2$$

5. Data Analysis

NOTE: See **Figure 6** for an example of a graph showing an analyzed MBP experiment.

1. First determine the time, t_0, when the ignition wire was turned on (current suddenly increases to 10.5 A). Determine the time when the ignition wire burned out (current suddenly returns to 0), t_b. Record the difference $\Delta t_w = t_b - t_0$ as the "wire lasted" time.
2. Determine the average ignition wire current, I_{iw}; this is the average of all the data points of the current record between t_0 and t_b. Determine the time when the pressure trace first deviates from the initial baseline, t_{p0}. Record the difference $\Delta t_p = t_{p0} - t_0$ as the "Time to Pressure Rise".
3. Determine the average initial pressure, P_i; this is the average of all the data points of the pressure record between t_0 and t_{p0}. Determine the maximum pressure, P_{max}; this is the maximum value of the pressure record.
   NOTE: The pressure trace may contain several minima and maxima.
4. Locate the last maximum (just before the pressure starts to continuously decrease when burning is complete); this is the burn stop time (t_s). Calculate the difference $\Delta t_b = t_s - t_{p0}$ and enter it as the "Burn Time".

6. Cleaning up

1. Clean and reuse the test cells as much as possible. Dispose of a test cell whenever it is found that solid residues are very difficult to clean out. Clean the cells with water, ethanol and paper towel. If the non-conductive paint is damaged, repaint the cell before re-using it.
   NOTE: Using soap or any detergent to clean the cells is not recommended because detergent residues may destabilize the surfactant in some emulsion formulations.
2. Clean the vessel after each run.

NOTE: Wearing a face mask with appropriate general-purpose cartridges is recommended for the person cleaning the vessel. Certain formulations, especially those that contain chemical sensitizers, may create more irritating residues than others.

3. Remove dirt and moisture from the pressure vessel using paper towels, applying water or ethanol as required. Ensure that the electrodes are cleaned in a similar manner, including the washers and nuts.
4. At the end of the day, return all unused sample material and wastes to an appropriate storage location (usually an explosives magazine).
5. Turn off the data acquisition system and computer (or storage oscilloscope).
6. Close the main valve on the argon (or nitrogen) cylinder and bleed the argon (or nitrogen) lines.

Representative Results

Typical raw signals from a test resulting in a fully propagated event (*i.e.* "go") are shown in **Figure 6**. The ignition current (blue curve) is seen to come on at t_0 = 0 and to stay on until the NiCr wire burns at t_b = 19.1 s. The computed average ignition current (*i.e.* average of all data points between t_0 and t_b is I_{hw} = 10.59 A. On the pressure record (red curve), the first sign of clear departure from the initial baseline is observed to occur at t_{p0} = 17.3 s. The computed average initial pressure (*i.e.* average of all data points between t_0 and t_{p0}) is P_i = 4.924 MPa (700 psig). From t_{p0}, the pressure is seen to rapidly increase to a maximum of P_{max} = 6.095 MPa (870 psig) at t_s = 33.7s. At this point the burning front has reached the internal wall of the cell and the pressure quickly decreases as combustion ceases.

The MBP measurement protocol presented here has been developed through a careful study of the many physical effects that can influence the outcome of the measurements. Through the publication of several documents, MBP data on a very wide variety of AWE formulations have been presented, thus establishing the usefulness and reproducibility [16] of the proposed measurement protocol.

In particular, the preponderant effect of the water content on the MBP of AWE formulations has been clearly demonstrated. This can be seen in **Figure 5** showing the MBP data for five AWE formulations with water content varying between 11.7 and 24.8 mass percent (%). For these five emulsions, the oxidizer solution consisted of only ammonium nitrate and water while the oil phase (oil + surfactant) amount and composition was kept fixed. It can be observed that, for each measurement, a series of 12 to 16 tests were performed. For each measurement, the two short horizontal bars indicate the pressure interval between of the highest "no-go (or partial)" event and the lowest "go" event, as specified in the above protocol. This illustrates well the strong dependence of the MBP of these particular formulae on the water content. From **Figure 5**, it can also be observed that the scatter in the MBP data is much higher for the two formulae with lowest water content (EM4 and EM5). Since these formulae contained only ammonium nitrate in their oxidizer solution (no other salts), they have relatively high crystallization temperatures and, as such, may be more prone to crystallization upon manipulation. This could induce a certain degree of non-uniformity in the samples and, therefore, a more important scatter in the data.

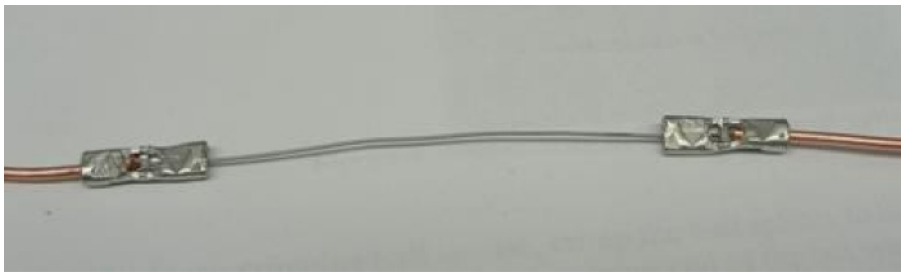

Figure 1: Complete ignition wire assembly. Please click here to view a larger version of this figure.

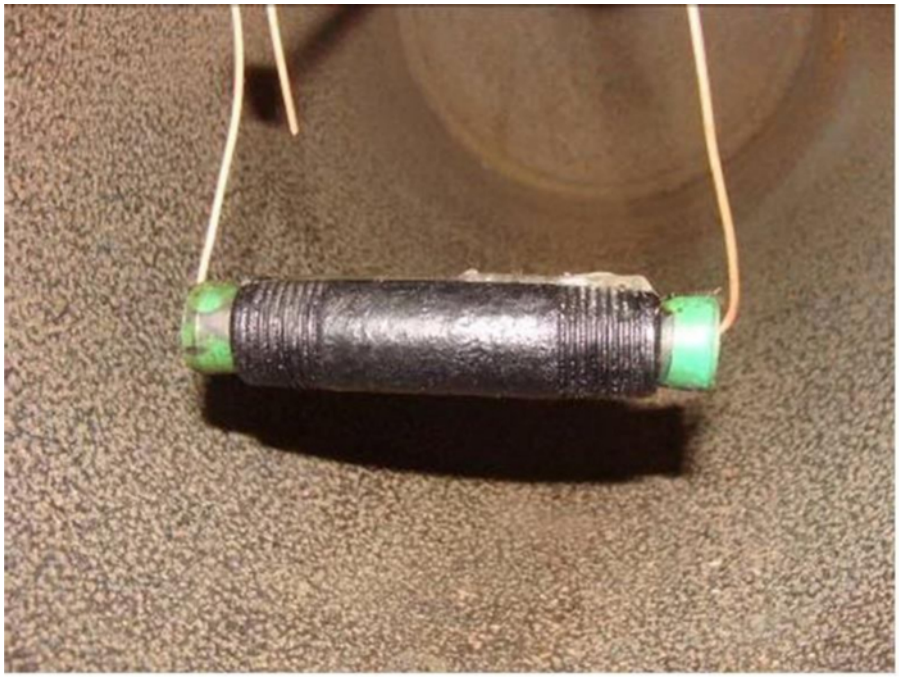

Figure 2: Typical MBP test cell with installed ignition assembly and emulsion sample. Please click here to view a larger version of this figure.

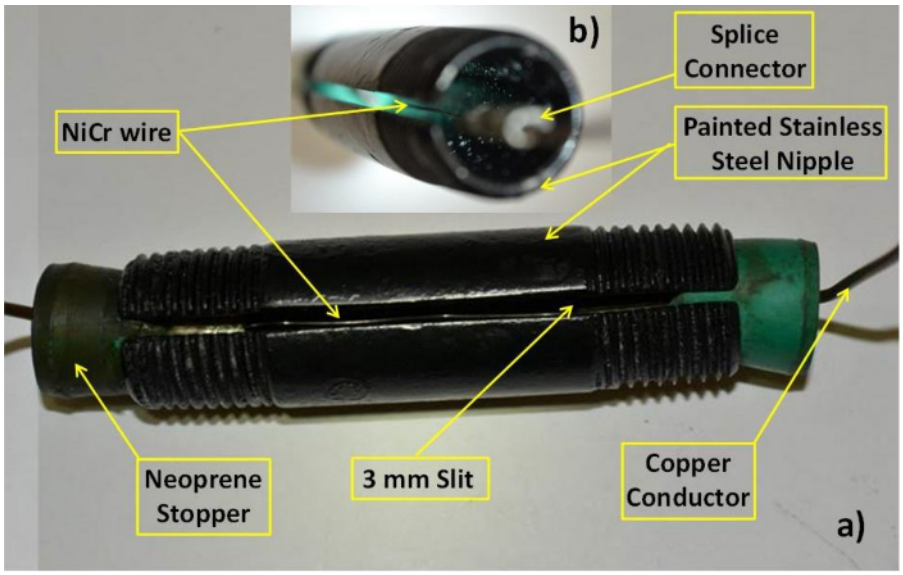

Figure 3: Test cell. (**A**) Assembled test cell just before introduction of emulsion sample through the slit. (**B**) View of test cell from one open end with neoprene stopper removed, showing the details of the NiCr wire running along the axis of the stainless-steel cell. Please click here to view a larger version of this figure.

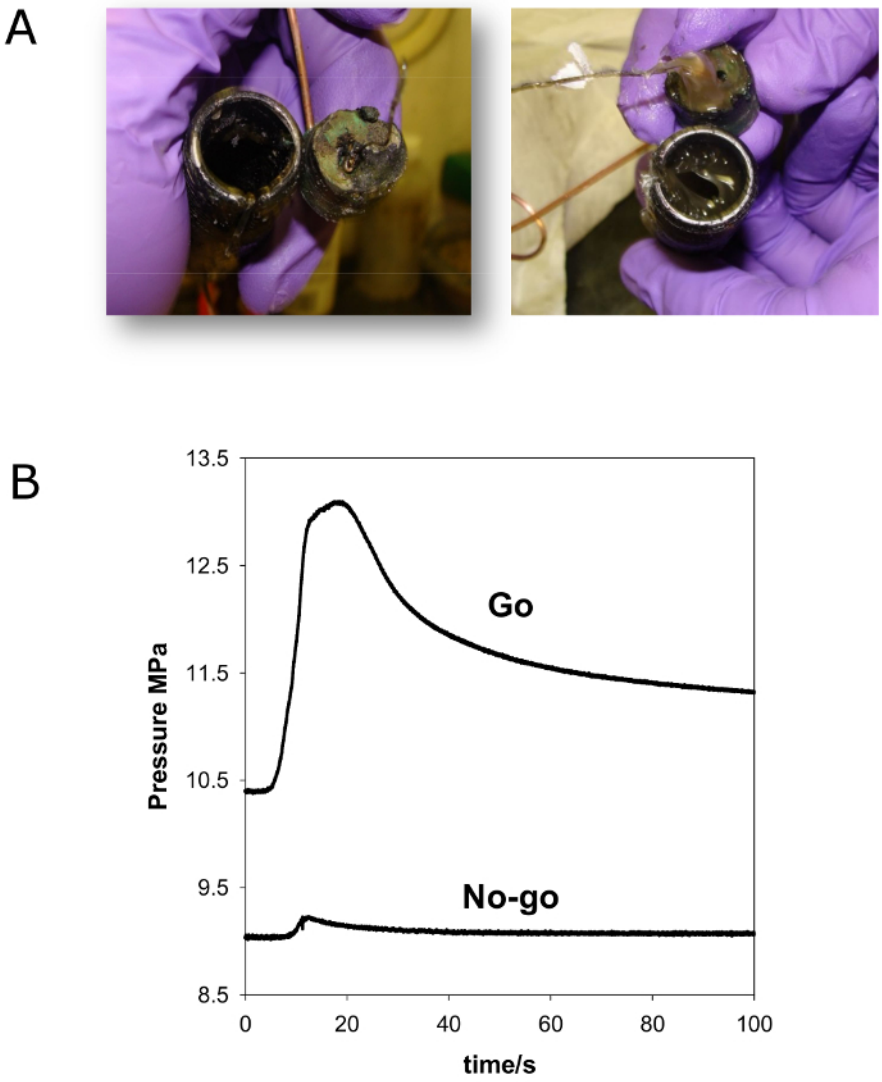

Figure 4: Visual observations and pressure recordings. (**A**) Typical visual observations for a "Go" (left) and "No-Go" (right) events. (**B**) Typical pressure records for "Go" & "No-Go" events. Please click here to view a larger version of this figure.

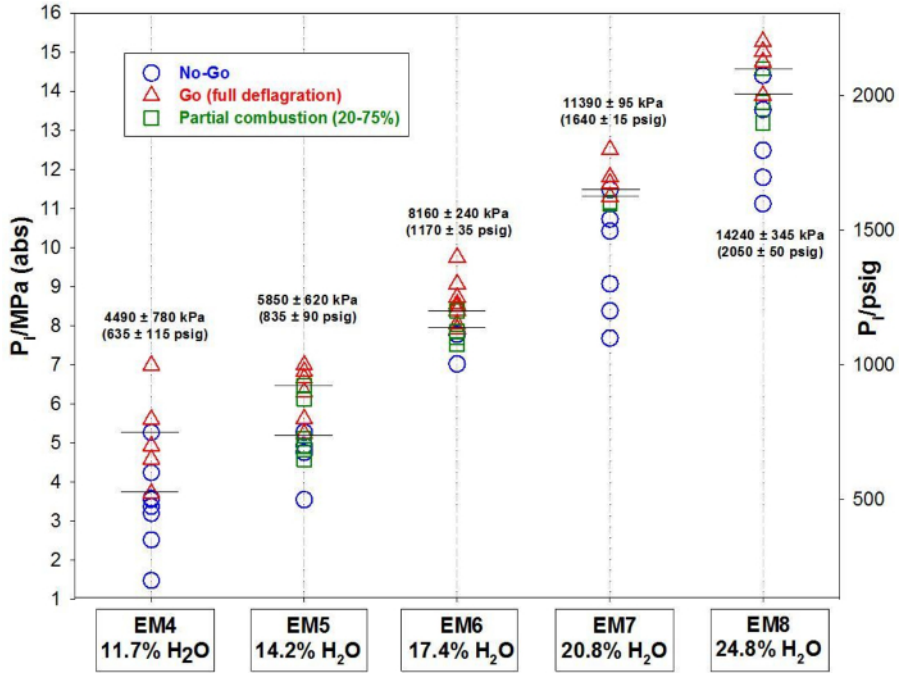

Figure 5: Summary of results for the MBP measurements of ammonium nitrate/water AWE formulae. Please click here to view a larger version of this figure.

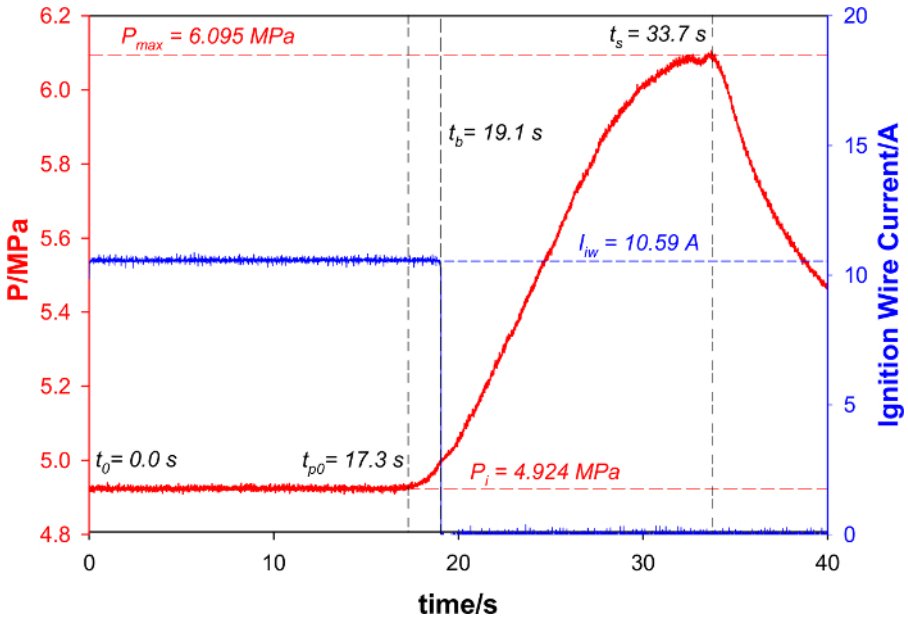

Figure 6: Example of an analyzed MBP experiment. Please click here to view a larger version of this figure.

Discussion

Our work demonstrated that the linear hot-wire geometry with 0.5 mm diameter NiCr straight wire and 10 to 16 A ignition current was adequate to ignite AWEs with water contents up to 25 mass %. For high viscosity formulations (such as packaged emulsion products), horizontal and vertical configurations provide almost identical results [17]. However, for low viscosity formulae (such as bulk emulsion products) gravity effects in vertical configuration induce emulsion flow which disturbs the ignition process. In these cases, the horizontal configuration was found to provide valid and reproducible results [17]. It should be noticed that the MBP values obtained in the present work for high water content emulsions are much lower than those reported by Wang [7] for similar products. This difference is probably due to the fact that, in his case, the ignition source had a coil geometry, which is less efficient to transfer energy to the emulsion, compared with the straight cylindrical geometry used in the present work. Also, if the coil is made out of too small diameter wire or if the loops are too close to each other, the ignition coil may burn prematurely, before the emulsion can be ignited. In such a case, it is very likely that failure to ignite may have been confused with failure to propagate.

As an example, for a typical surface bulk AWE such as EM6 (17.4 % water, **Figure 5**), the MBP measured with the present cylindrical geometry is 8.2 MPa. The MBP quoted by Wang for a similar product with less water (16.0 % water), using the coil geometry, was 15.2 MPa [7], which is

almost twice as high. Moreover, using the coil geometry with the same nichrome wire used in the present work, it has been found that a similar emulsion with 16.8 % water could not be ignited to sustained combustion even at initial pressure up to 15.8 MPa [9]. In comparison, the emulsions investigated in the present work, which had water contents as high as 24.8 %, could all be ignited to sustained combustion at pressures below 15 MPa.

As expected, the data obtained in the present work clearly demonstrates that water content is the major ingredient controlling the MBP of AWEs. The role of several other ingredients has also been investigated in some detail. However, many unexpected effects of some ingredients (sodium nitrate and glass microspheres, for examples) have been evidenced [14] and more research would be required to fully understand how their presence affects the ignition and propagation of combustion in these AWE systems.

The test, as described in the above protocol, has been added to the requirements for the authorisation of high explosives in Canada by the Explosives Regulatory Division of Natural Resources Canada [18]. It became an authorisation test for the acceptance of explosives handled using pumps or augers. This test has also been proposed as an alternative to the UN TDG Series 8c Test (Koenen test) [19] for AWEs. The acceptance of the test is currently pending further discussion within the informal correspondence group being led by Canada [20]. This group consists of seven international Competent Authorities and four non-Government Organizations. More detailed information on the above protocol can be obtained by contacting the authors.

Disclosures

The authors declare that they have no competing financial interests.

Acknowledgements

The development of the testing protocol reported in this publication results from a joint research project between Natural Resources Canada (CanmetCERL, Explosives R&D Section) and Orica Mining Services. Permission of Orica Mining Services to publish non-proprietary information on this subject is fully acknowledged. The participation of CanmetCERL's Analytical Section to the physical characterization of the various AWEs prepared throughout the present work is also gratefully acknowledged.

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