

MOLECULAR AND MICROSCOPIC STRUCTURAL EFFECTS IN THE DETONATION OF FLUID EXPLOSIVES

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The detonation behaviour of a model emulsion explosive was investigated to yield the heat release rate as a function of ionic structure of the oxidiser phase and degree of mixedness of the emulsion. Neutron diffraction studies were undertaken to characterise the oxidiser phase structure between molar ratios of solute to solvent of 1 and 3. Degree of mixedness was characterised using optical microscopy to measure the number average droplet size of the oxidiser phase which ranged over a factor of 2.5. At low solvent concentration marked ion pairing was evident between the ammonium and nitrate ions in solution and a strong correlation was found between chemical reactivity and the microscopic ion-pair structure. The concentration at which ion pairing no longer was evident corresponded closely to that where the critical diameter of the explosive increased markedly. The nature of the reaction varied from hot spot and grain burning-like at low water contents to thermal explosion-like at high water contents. Changes to the degree of fuel-oxidiser mixedness revealed a strong quantitative relationship between reaction rate and mixedness. Experiments repeated at varying levels of sensitising voidage demonstrated that the level of voidage had a pronounced effect on systems of low mixedness thus providing clear evidence of mixing occurring during the shock compression process.

INTRODUCTION

The effect of gross structural properties on the detonation behaviour of heterogeneous fluid explosives has been well researched. For systems comprising a reactive liquid sensitised with high or low density particles, the relationship between detonation performance and number density and size of inclusions has been elucidated by several workers¹⁻³. Surprisingly however, little or no published data exists on the effect of molecular structure of the fluid on detonation performance or on the effect of mixedness in fluids comprising immiscible fuel and oxidiser phases.

The first discussion of such effects in aqueous explosives was by Sujansky⁴, who found changes in

the detonation behaviour of hydrazine mono-nitrate solution below 80% w/w solution. Also one of the authors has discussed possible kinetic changes in crystallised emulsions where the water from the crystallised droplets diffused under osmotic pressure to the non-crystallised droplets thus reducing the ionic strength therein⁵. This work concentrates on investigating the effect of changes in oxidiser strength and liquid microstructure on chemical reactivity under detonation conditions.

Emulsion explosives typify fluid explosives. They have become widely used as commercial blasting agents, largely displacing nitro-glycerine and watergel products for factory manufacture and supplementing ANFO in wet ground applications and where extra strength is required. The detonation kinetics of these

products as a function of void size have been described by the authors in a previous paper⁶. This paper discusses the detonation kinetics of emulsions with water contents of between about 16% by weight and a level where failure to propagate in small diameter charges would be expected.

Additionally the effect of mixedness, that is to say the proximity of fuel and oxidiser components, was investigated by altering the number average droplet size of the discontinuous oxidiser phase in the oil. The effect of voidage on mixedness effects was also characterised to investigate the level of shock induced mixing within the reaction zone of the explosive.

EXPERIMENTAL METHODS

The model explosive comprised an aqueous solution of ammonium nitrate emulsified into mineral oil and sensitised with glass micro-balloons. The chemical composition was kept approximately constant and the fluid structure was manipulated at the molecular level by altering the ionic concentration of the oxidiser solution and on the microscopic level by changing the degree of mixedness of the emulsion.

The oxidiser was an aqueous solution of pure ammonium nitrate and the fuel phase was a mixture of refined mineral oil and surfactant the ratio of which was adjusted so as to maintain approximate oxygen balance over all the formulations. Typical compositions used are given in Table 1. It should be noted that because of the difference in molecular weights between NH_4NO_3 and water there is a considerable change in molar ratio over the relatively small range of mass ratios used in the experiments, Table 2.

	A	B	C	D	E	F
NH_4NO_3	76.4	74.7	69.5	65.1	60.6	56.1
H_2O	15.8	17.7	23.2	27.9	32.6	37.4
Oil	3.8	3.6	3.3	3.0	2.8	2.5
Surfactant	1.5	1.5	1.5	1.5	1.5	1.5
Micro-balloons	2.5	2.5	2.5	2.5	2.5	2.5

TABLE 1: FORMULATION OF EXPLOSIVE FOR OXIDISER STRUCTURE WORK

% NH_4NO_3 by mass	Mole Ratio AN : Water
80	1.11
75	1.48
70	1.905
65	2.39
60	2.96

TABLE 2: MASS FRACTION MOLE RATIO

Glass micro-balloons of type C15/250 manufactured by the 3M Corporation were used for density control. These had been previously floated on industrial methylated spirits to remove solid and high density particles. The number average micro-balloon size was about 50 microns.

All emulsions were mixed on a Hobart N90 industrial kitchen mixer using the whisk as the mixing tool in batch sizes of 1 or 2 kg. During preparation, the oxidiser solution was heated to 20 K above its crystallisation point and added slowly to a mixture of oil and surfactant in the steam heated mixer bowl with the mixer running at speed 2. This effected formation of a crude emulsion. When all the oxidiser solution had been added the mixer was then run at speed 2 or 3 to refine the emulsion further. Running for 5 minutes at speed 3 gave the highest refinement, running at speed 2 the lowest. Control could therefore be exerted over droplet size and hence mixedness between fuel and oxidiser phases. Droplet sizes were determined by optical microscopy using a reflectance technique. A low degree of mixing tended to produce a bimodal distribution of droplet size, tending towards mono-disperse with increased mixing. Typical size distributions for various degrees of mixing are given below, Figures 1 through 5.

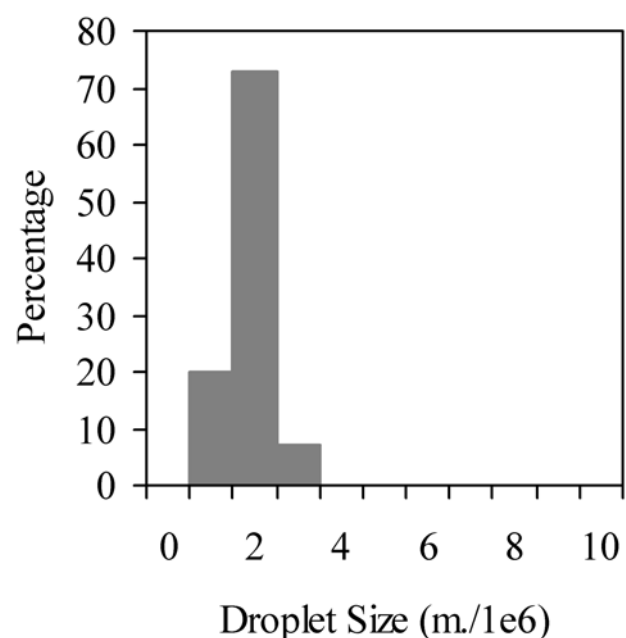


FIGURE 1: DROPLET SIZE DISTRIBUTION FOR LONGEST MIXING

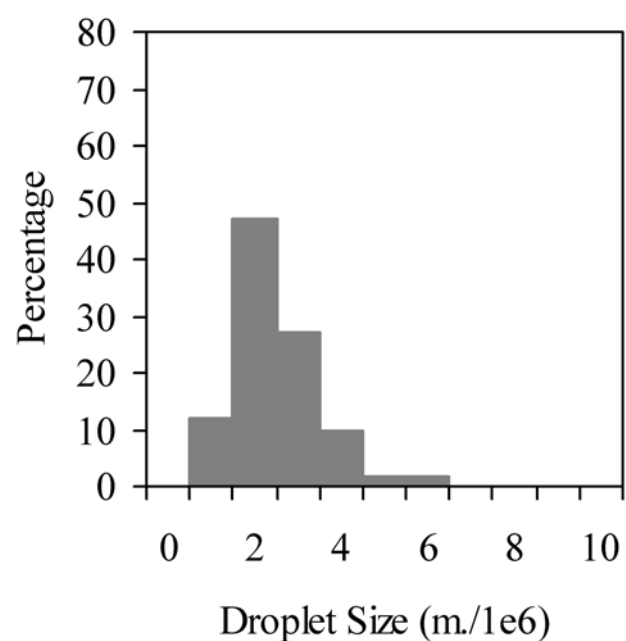


FIGURE 2: DROPLET SIZE DISTRIBUTION FOR LONGER MIXING

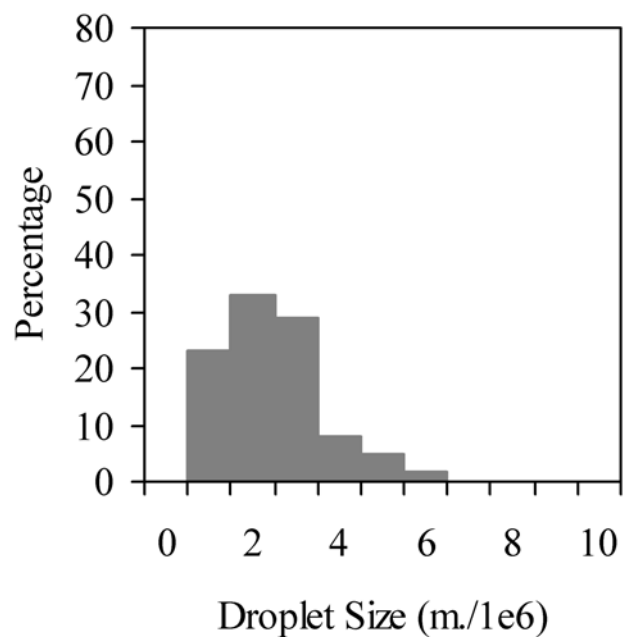


FIGURE 3: DROPLET SIZE DISTRIBUTION FOR INTERMEDIATE MIXING

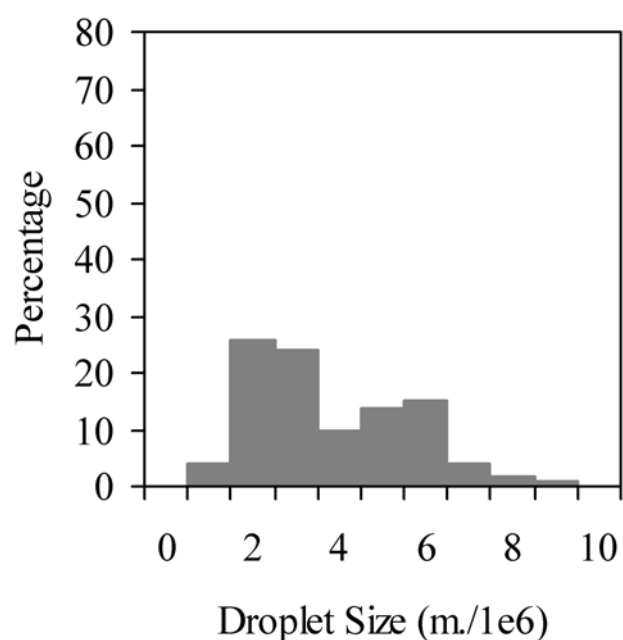


FIGURE 4: DROPLET SIZE DISTRIBUTION FOR SHORTER MIXING

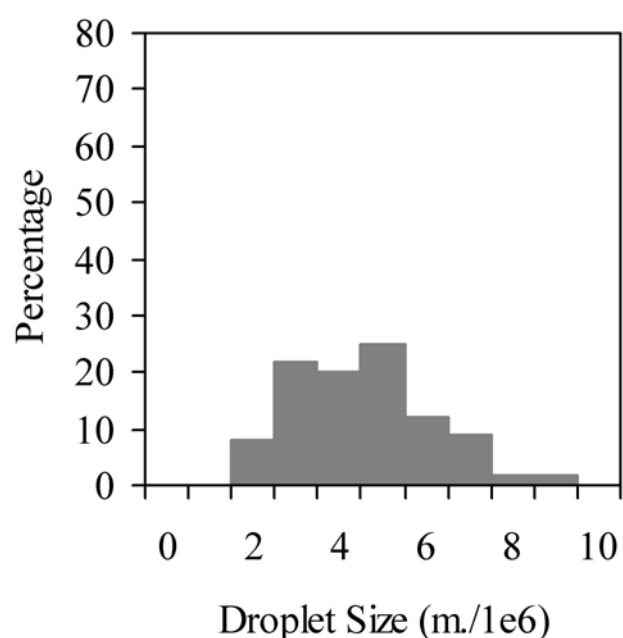


FIGURE 5: DROPLET SIZE DISTRIBUTION FOR SHORTEST MIXING

For the ionic strength studies, the emulsion was refined to the highest possible level giving a number average droplet size, N_A , in the region of 1.47 microns. For the mixedness studies which were based on Formulation A in Table 1, the emulsification and density control was as in Table 3.

	Minutes @ 125 rpm	Minutes @ 285 rpm	% C15/250	N_A 1e-6/m
X1	.5	0	1.25	3.64
X2	3	0	1.25	1.90
X3	3	0	1.25	1.47
Y1	.5	0	1.80	3.64
Y2	1	0	1.80	3.22
Y3	3	0	1.80	1.90
Y4	3	5	1.80	1.47
Z1	.5	0	2.50	3.64
Z2	3	0	2.50	1.90
Z3	3	5	2.50	1.47

TABLE 3: FORMULATION OF EMULSION EXPLOSIVE FOR MIXEDNESS STUDIES

Formulations were immediately packaged in paper shells. After cooling naturally to 25C the charges were initiated with a detonator having a base charge of 0.8g PETN, in combination with a 4g Pentolite primer. This eliminated possible effects of low emulsion sensitivity at higher water contents. It was found that Formulation F did not detonate in 30 or 38mm diameter charges even when primed with 28g Pentolite and no further work was carried out on this formulation.

The Charge Diameter Effect, that is the variation of detonation velocity with charge diameter, was measured for the various compositions used. All data were obtained from time of flight measurements over a distance of 0.2 metres. Measurements were made only once the detonation had become stable, i.e. after at least six charge diameters from the initiator. In separate tests the critical diameter was measured using a pass/fail criterion on un-instrumented cartridges. This eliminated the possibility of premature failure in charges close to the critical diameter due to perturbation of the hydrodynamic flow by the time of flight probes.

Critical initiation pressures were measured using the DRG gap test⁷, a variant of the NOL Gap test procedure. Acceptor charges were 24mm in diameter, 120mm long and aerated with 2.5% C15/250 micro-balloons.

CHARGE DIAMETER EFFECT ANALYSIS

The Charge Diameter Effect data were analysed using the CPeX small divergent flow model to yield the heat release rate of the chemical reaction occurring in the hydrodynamic flow. The CPeX detonation model⁸ has previously been used in this fashion⁹⁻¹⁶ and therefore the analysis will only be briefly described herein.

Firstly, thermo-hydrodynamic calculations for the ideal Chapman - Jouguet states were carried out using the JCZ3 equation of state¹⁷, Table 4

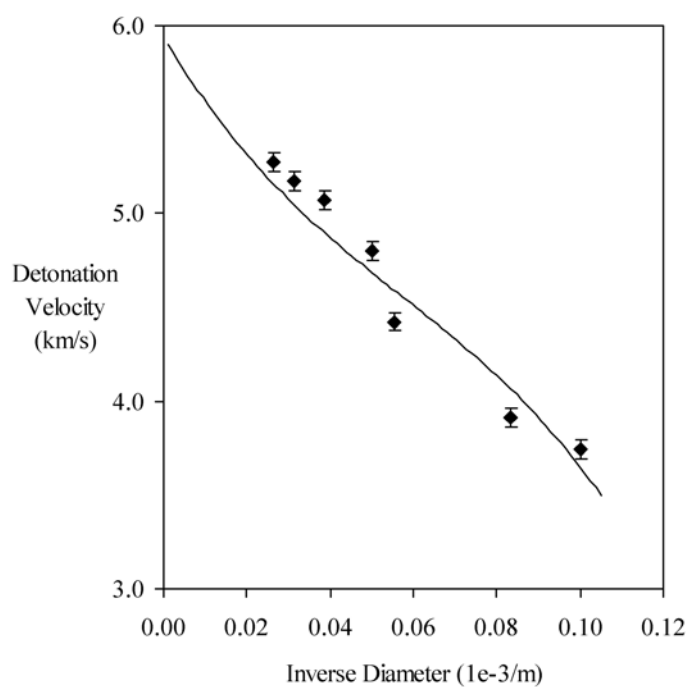


FIGURE 6: CHARGE DIAMETER EFFECT FOR COMPOSITION A

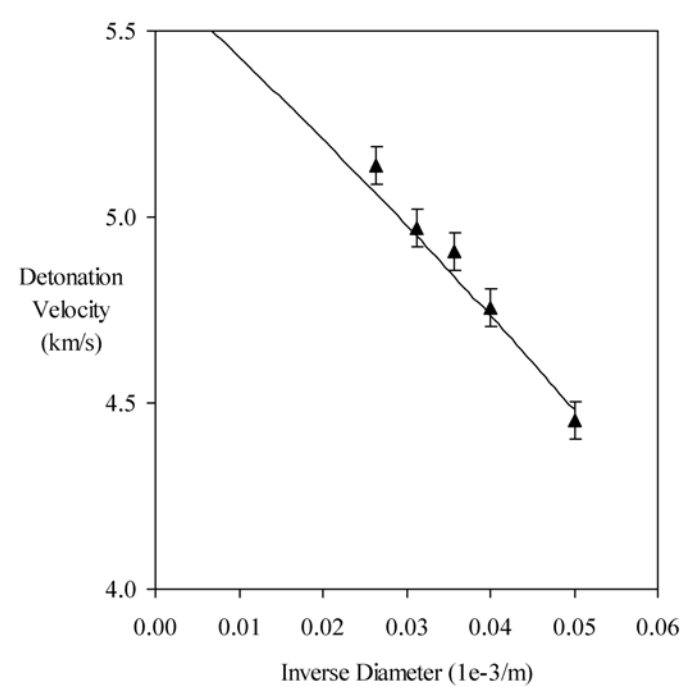


FIGURE 8: CHARGE DIAMETER EFFECT FOR COMPOSITION D

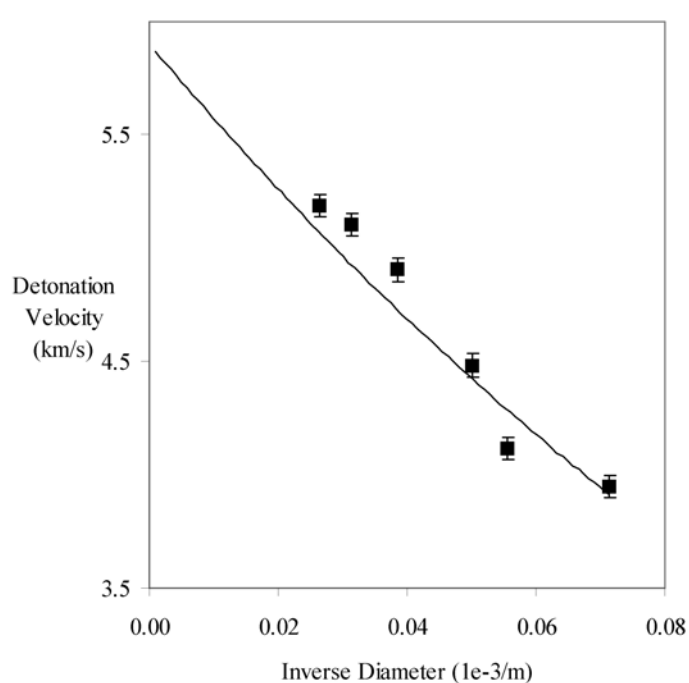


FIGURE 7: CHARGE DIAMETER EFFECT FOR COMPOSITION B

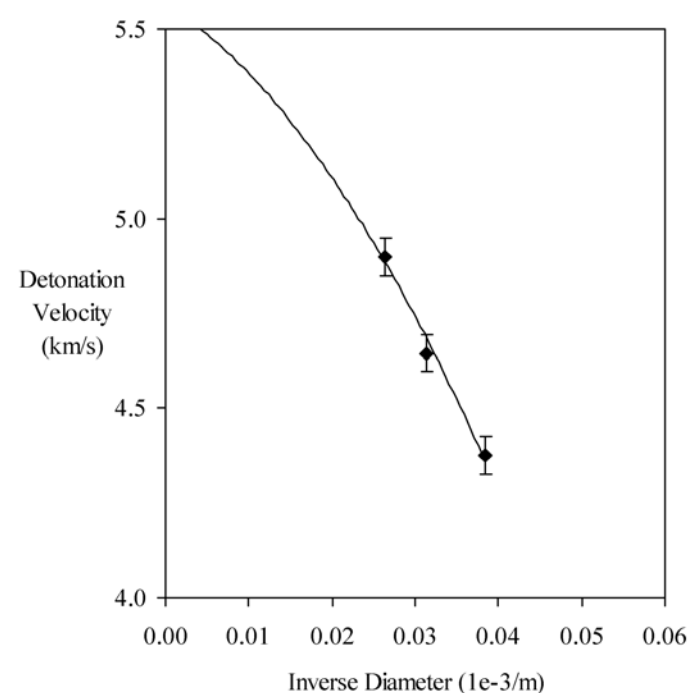


FIGURE 9: CHARGE DIAMETER EFFECT FOR COMPOSITION E

	A	B	C	D	E	F
Density kg/m ³	1011	1090	1070	1050	1050	1020
Ideal VoD km/s	6.08	5.94	5.81	5.69	5.6	5.42

TABLE 4: IDEAL DETONATION VELOCITIES

The small divergent flow based detonation model, CPeX, was used to reproduce the measured diameter effect curves by manipulation of three fitting constants in the CPeX chemical kinetic (heat release rate) function:

$$\dot{\lambda} = (1 - \lambda) \left(a_h \frac{(p - p_c)^{b_h}}{\tau_h} + a_l \frac{p^{b_l}}{\tau_l} + a_s \frac{p^{b_s}}{\tau_s} \right)$$

where λ denotes the mass fraction reacted; τ time

differentiation; p pressure; a_i shape functions dependent only on the initial formulation; b_i , pressure exponents; τ_i time constants; p_c critical pressure; and the subscripts $i = h, l, s$ refer to various phases in the explosive, nominally hot spot, liquid and solid. The three fitting parameters were $\tau_{h,l}$ and p_c , a_s being identically zero as no solid was present and b_i being left at the normal value of unity.

It should be noted that there is substantial evidence for a functional form of this type. Initiation behaviour shows a threshold shock pressure in granular explosives consistent with a critical hot spot concept and particle size effects have already been associated with a grain burning mechanism¹⁸⁻²¹. The form is established in the literature for modelling the performance of multi-component, multi-phase explosives⁹.

An automatic fitting algorithm, based on a line search followed by a constrained minimisation, was

used to generate a least squares fit to the experimental data with a critical point lying marginally below that measured experimentally.

The experimental data and CPeX fits were in good agreement over the range of voidages used. Examples are given in Figures 6 through 9.

ANALYSIS OF GAP TEST DATA

The initiation behaviour was characterised using a variant of the small scale gap test as referenced above. The initiation data was analysed by hugoniot shock impedance matching techniques to give the critical initiation pressure in the emulsion as given in Table 5.

Mole Ratio AN aq: H ₂ O	Critical Initiation Pressure GPa.
1.00	1.23
1.20	1.62
1.65	1.81
2.00	2.65

TABLE 5: CRITICAL INITIATION PRESSURE AS A FUNCTION OF OXIDISER MOLALITY

SIGNIFICANCE OF THE HEAT RELEASE FUNCTION

Every explosive shows a detonation velocity decrement in finite diameters. The decrement is both chemical kinetic and hydrodynamic in origin. From consideration of the Chapman-Jouguet criteria^{3,8,22,23}, it can be shown that there must be sufficient energy released in the reaction zone to support the shock and the rate of release of energy at the sonic point must equal that of energy loss. The Charge Diameter Effect curve is thus a measure of both the degree of reaction and the instantaneous reaction rate at the sonic point at each charge diameter. By analysing the empirical charge diameter effect in terms of the heat release rate, a large section of the reaction rate, degree of reaction, pressure hyper-space can be probed.

From previous studies¹⁰ it has been shown that the heat release function can vary from thermal explosion-like to hot spot and grain burning-like in nature. Thermal explosion is characterised by a low initial rate followed by a very rapid reaction during which almost all of the explosive is consumed. The greatest variation in the reaction rate occurs at very high or very low degrees of reaction. In the case of hot spot and grain burning, the reaction rate varies smoothly and slowly with the degree of reaction and there is little variation between initial and peak rates.

For the compositions being tested, CPeX derived reaction profiles were calculated at a reference pressure of 5 GPa. Figure 10 shows the CPeX degree of reaction data for formulations A to E. It can be seen that there is a progression from the smooth curve for A, which is typical of a hot spot and grain burning type mechanism to E, which shows little reaction until about 0.7 microseconds, when there is an abrupt rapid progression to complete reaction, typical of a thermal explosion type mechanism.

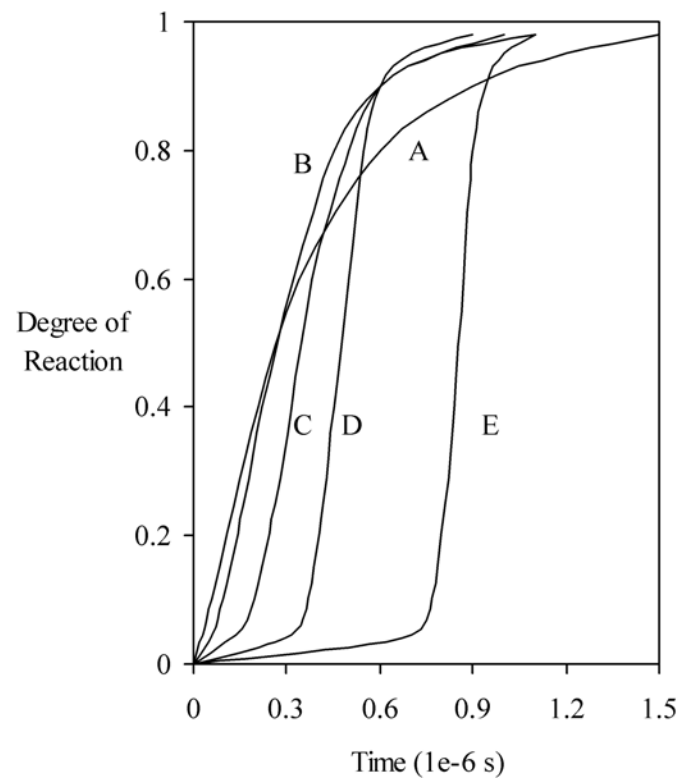


FIGURE 10: HEAT RELEASE CALCULATED FROM DIVERGENT FLOW MODEL

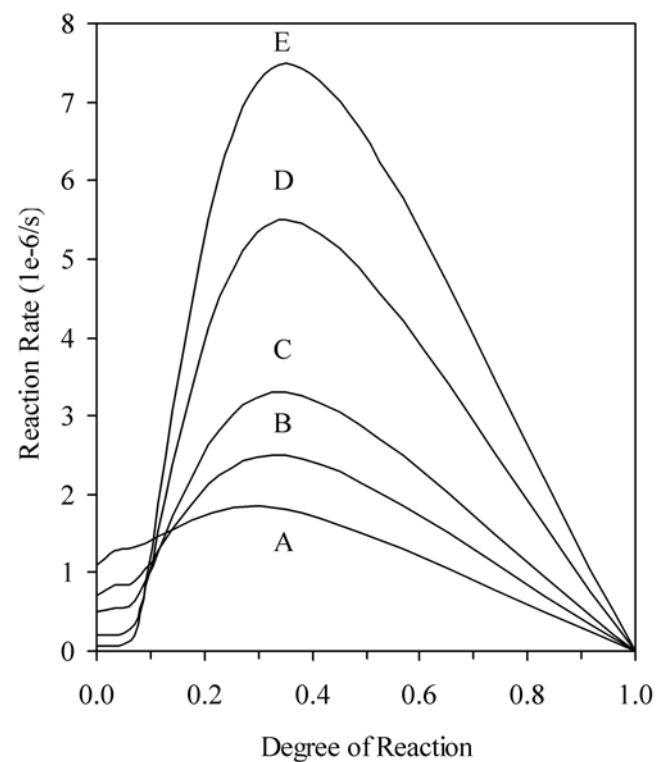


FIGURE 11: HEAT RELEASE RATES FROM DIVERGENT FLOW MODEL

Figure 11 shows the corresponding reaction rate versus degree of reaction curves. For A the initial rate is almost as high as the peak rate. For B through E the initial rate progressively falls to a very low value whilst the peak rates progressively rise to a very high value. These progressions signify a change to a thermal explosion type mechanism.

STRUCTURE OF AMMONIUM NITRATE SOLUTIONS

In order to study the structure of the oxidiser phase, a first order isotopic difference method of neutron diffraction was applied to both ¹⁴N and ¹⁵N in both the ammonium and nitrate ions. The solutions used were 12 molal and 18 molal in deuterium oxide.

In both solutions the ND_4^+ structure exhibited a strong intra-molecular peak at 1.04 Angstroms. At the higher concentration a small peak at 2.15 Angstroms was observed in the radial distribution function, $G(r)$, Figures 12 and 13. If this peak is assumed to contain oxygen atoms, a co-ordination number of 1 is obtained. This could be the oxygen atom of a near neighbour water molecule or the oxygen atom of a NO_3^- anion. As the peak is absent in the 12 molal solution it is logically assigned to the anionic interaction. The results suggest therefore that there is ion pairing in the 18 molal solution.

The longer range structure of the two solutions is similar with broad peaks centred at 3.0 and 3.4 Angstroms. In the more concentrated solution the peak at 3.0 Angstroms is calculated to contain 1 nitrogen and about 6 oxygen atoms that belong to the nearest neighbour water molecules. When integrated over the range 3.2 to 4.1 Angstroms, the peak centred at 3.4 Angstroms can readily accommodate the two remaining oxygen atoms of the nitrate ion, 12 deuterium atoms of the nearest neighbour hydration shell and about 5 or 4.5 oxygen or deuterium atoms respectively.

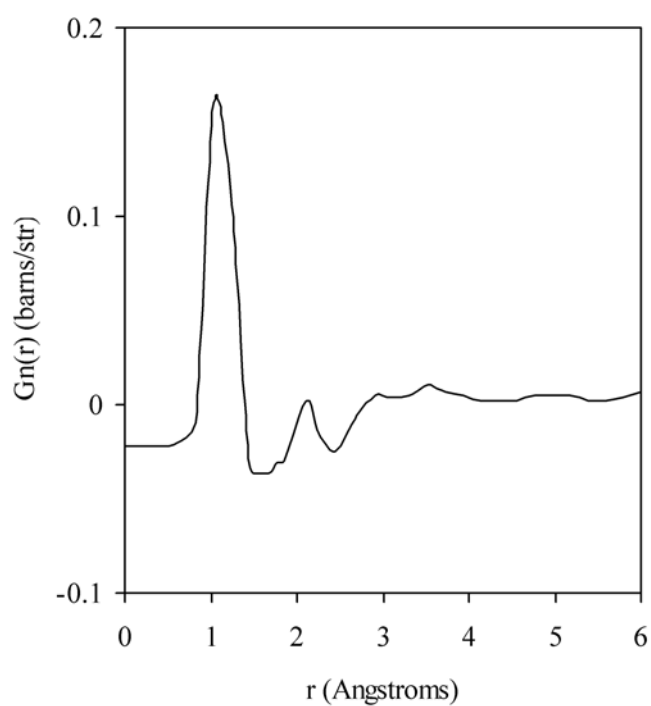


FIGURE 12: AUTO-CORRELATION FUNCTION

There is no peak at 2.15 Angstroms in the 12 molal solution. However, there is a relative increase in the size of the peak at 3.2 Angstroms, ascribed to an oxygen belonging to the nitrate anion. The rest of the peak can accommodate 6 or 7 other oxygen atoms forming a near neighbour weakly defined hydration shell. The peak centred at 3.4 Angstroms is similar to that in the 18 molal case and there is sufficient area under it to contain 14 deuterium atoms, 1 nitrogen atom, 2 oxygen atoms and a remaining 4 other atoms.

The above data refers to solutions in deuterium oxide. To change to light water as used in the detonation experiments the molality must be corrected: 18 molal ND_4NO_3 in D_2O is 60.19% w/w ND_4NO_3 or 1 mole ND_4NO_3 per 2.776 moles D_2O . For NH_4NO_3 in H_2O at the same molecular ratio the solution would be 61.5% w/w NH_3NO_3 . The corresponding calculations

for 12 molal solutions are 1 mole ND_4NO_3 to 4.16 moles D_2O and 51.60% w/w NH_4NO_3 .

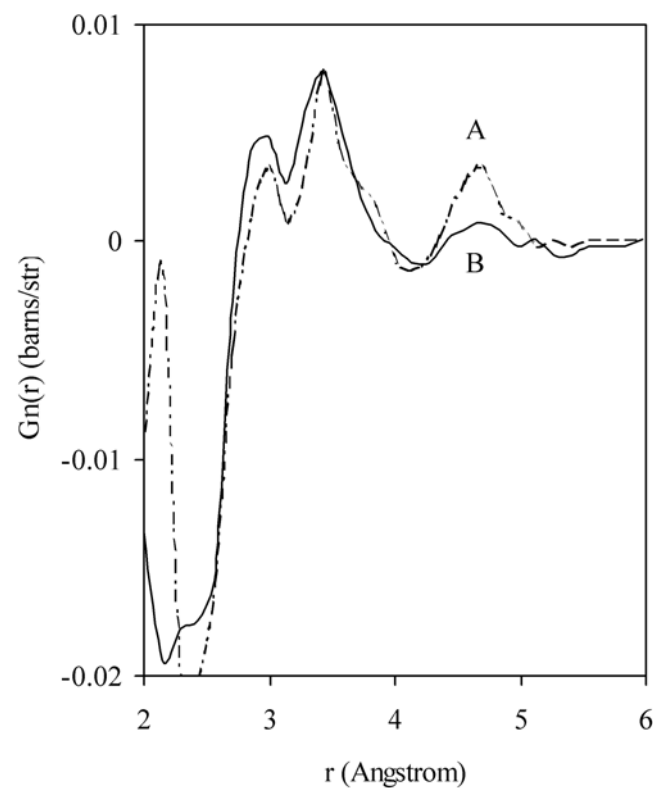


FIGURE 13: EXPANSION OF AUTO-CORRELATION FUNCTION SHOWING ION PAIRING

A suggested structure for the ammonium nitrate ion pair is shown in Figure 14.

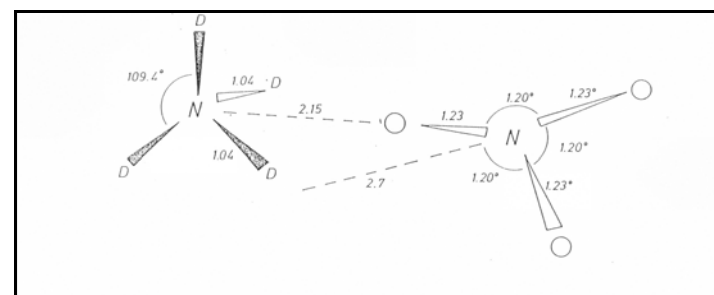


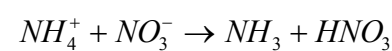
FIGURE 14: STRUCTURE OF AMMONIUM NITRATE ION PAIR IN SOLUTION

DISCUSSION ON IONIC STRENGTH EFFECTS

From the experimental work and data reduction it has been shown that higher water content leads to a change from hot-spot and grain burning to thermal explosion, and to higher critical initiation pressures. Ion pairing has been found to occur in solutions of greater than ca. 61% w/w ammonium nitrate. These factors may be rationalised by considering the possible affects of increasing the water content in emulsions.

The emulsion specific heat is increased. This may be expected to increase the detonation velocity at failure as a higher shock pressures will be required to achieve any given shock temperature. This can be seen in the progression of failure velocities in Figures 6 through 9.

The solution structure is changed, in particular less ion pairing is expected. This may have consequences for important ion-ion reactions such as:



From the small divergent flow analysis, the reaction time has been found to be significantly less than 800 ns. It is interesting to speculate whether ion pairing is essential to enable reaction in such a short period. It is noted that at 65% by mass ammonium nitrate there is strong evidence of ion pairing in the neutron diffraction data and that the ion pairing ceases below 61.5% ammonium nitrate. That the critical diameter of the explosive increases markedly as the oxidiser strength falls from 65% and 60% is evidence of the importance of ion pairing to the reaction mechanism under detonation conditions.

Finally, excess water may inhibit free radical reactions that are important in detonation and there also will be changes in the Hugoniot, though that is probably a relatively small effect.

The progression from hotspot and grain burning towards thermal explosion type behaviour at increased water content is worthy of further discussion. It is proposed that the change over from a typical hot spot liquid burning mechanism to thermal explosion can be explained by considering the progress of a slowly reacting hot spot resulting from the lower hot spot temperature in the high water content emulsions. If the hot spot fails to react immediately its energy will be dissipated into the bulk of the explosive through both thermal diffusivity and turbulent flow induced by void collapse. In the limit this will lead to failure caused by hydrodynamic expansion. However, there is an intermediate case where much of the hot spot energy is dissipated into the bulk but the rapidly cooling hot spots finally ignite and induce thermal explosion in the bulk that is now at much above its initial shock temperature due to influx of hot spot energy. This high bulk temperature requires considerable dissipation of hot spot energy so the thermal explosion mechanism is only prevalent at relatively high void contents and at high water contents where the hot spot reaction is retarded.

Shock temperatures have been calculated for the case of discreet hot spots in bulk material and for the case where the hot spot energy is distributed uniformly throughout the shock heated zone, i.e. an "average" temperature.

The condensed phase hugoniot was estimated from the chemical composition of the explosive²⁴ and the shock temperature calculated by the method of Walsh and Christian²⁵. The effect of porosity was included by the method of Afanasenkov et al²⁶ as modified in reference 10 ibid. The hot spot temperature was calculated from the bulk shock temperature, the hugoniot excess energy due to void collapse and the hot spot volume, assumed to equal the initial void volume. Typical input parameters are given below along with the results of the calculations.

It can be seen that the hot spot and average temperatures rise more rapidly than the bulk temperature as shock pressure is increased. A pressure of 5.07 GPa approximates to the reference pressure chosen for the CPeX heat release calculations. It is noted that at this pressure, the average shock temperature is sufficient to drive the entire reaction zone into a thermal explosion type reaction²⁷,

validating the proposed mechanism.

Quantity	Value	Units
Specific Heat	2.2E-3	MJ/kg/K
dP/dT	1.75E6	Pa/K
Density	1.3E3	Kg/m3
Initial Temperature	298	K
Hugoniot A	2.27E3	m/s
Hugoniot B	1.63	

TABLE 6: DATA FOR SHOCK TEMPERATURE CALCULATIONS

Shock Pressure	Bulk Temp.	Hot Spot Temp.	Average Temp.
GPa.	K	K	K
2.005	340	963	460
3.104	366	1223	577
4.039	391	1646	705
5.07	422	1998	816
7.417	504	2810	1081
10.15	616	3770	1405

TABLE 7: CALCULATED SHOCK TEMPERATURES

MIXEDNESS EFFECTS

The emulsion formulation designated "A" in the ionic strength studies was mixed for various times and sensitised with micro-balloons as before. Mixing times, voidage levels and number average droplet sizes are given in Table 3 above.

Critical diameters, Table 8, were found to be a strong function of voidage and droplet size. The effect of voidage was found to become progressively weaker as mixing times were increased, i.e. the droplet size of the emulsion was refined and mixedness increased. This provided strong evidence of shock induced mixing of the explosive on the microscopic scale during detonation.

Formulation	Critical Diameter mm.
X1	22
X2	16
X3	13
Y1	17
Y2	14
Y3	12
Y4	10
Z1	10
Z2	8
Z3	8

TABLE 8: CRITICAL DIAMETER DATA

Charge Diameter Effect data were measured at all levels of mixedness and voidage given in Table 3 and the resulting data analysed using the CPeX small divergent flow model to yield the heat release rate function as before, Graph 15.

Previously, the authors have shown that the heat release functions found in such tests depend on the

interaction of fuel and oxidiser components and that such an interaction is influenced both by diffusion and by the hydrodynamic field created by the shock induced collapse of the porous explosive itself. Based on this work, the authors postulated that the effect of mixedness on detonation performance would be most evident at low voidage where shock induced mixing would be minimised and least evident at high voidages where shock induced mixing would be greatest. This is as has been found in practice.

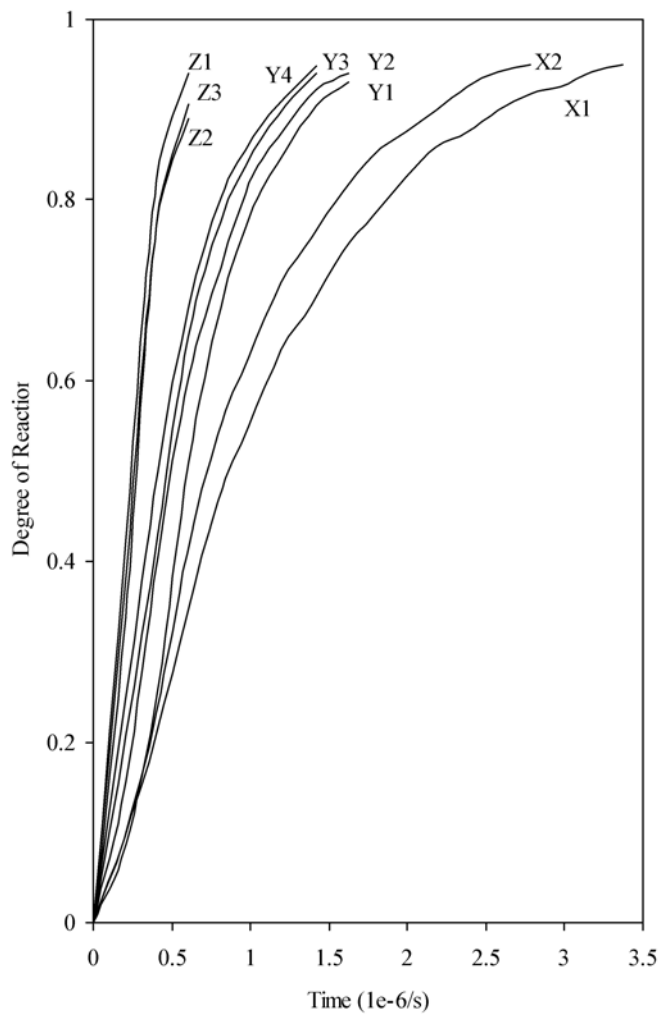


FIGURE 15: RESULTANT HEAT RELEASE CALCULATED FROM DIVERGENT FLOW MODEL

For the X series of formulations (low voidage), the degree of reaction plots show a significant dependence on level of mixedness, indicating little occurrence of secondary mixing during shock compression. For the Z formulations (high voidage), the degree of reaction plots are almost identical, indicating that intense mixing has occurred as a consequence of shock induced void collapse. The mixing has refined the emulsion and greatly reduced the effect of diffusion on reaction rates as seen in formulation X1 or X2.

Based on the data a crude estimate of the diffusion rate has been made that is the droplet diameter divided by the reaction time, i.e. of the order of microns per microsecond. This is in approximate agreement with results published by other workers^{24,27}.

CONCLUSION

In this study a simple emulsion type explosive was used to investigate both molecular and micro-structural effects on detonation performance.

At low water contents marked ion pairing was

evident between the ammonium and nitrate ions in solution. This pairing disappeared at higher water levels. A strong correlation between chemical reactivity and the microscopic ion-pair structure was found. The concentration at which ion pairing was no longer evident corresponded closely to that where the explosive became non-detonable in small diameter charges. Additionally the nature of the reaction varied from hot spot and grain burning at low water contents to thermal explosion like at high water contents. This unexpected result was interpreted as the consequence of a retarded hot spot reaction due to the higher water content increasing the diffusion of hot spot energy into the bulk phase.

Changes to the degree of fuel-oxidiser mixedness were effected by altering the energy input to the emulsion during preparation. A strong quantitative relationship was found between reaction rate and degree of mixedness. Surprisingly the level of voidage was shown to have a pronounced effect on systems of low mixedness but not on systems of high mixedness, providing clear evidence of mixing occurring during the shock compression process.

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