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PREPARATION, CHARACTERISATION AND COOKOFF BEHAVIOUR OF BOOSTER COMPOSITIONS PREPARED FROM RDX AND ACRYLIC OR ETHYLENE-VINYL ACETATE DISPERSIONS

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ABSTRACT

A range of potential booster explosives has been prepared using RDX and commercially available acrylic and ethylene-vinyl acetate dispersions. The dispersions were coagulated in hot aqueous slurries of RDX by electrolyte addition and the use of thermal coagulation aids, a process compatible with existing Australian production plant and methods. Generally the polymer coatings on the RDX crystals were porous and the precipitated particles had not coalesced to form polymer films. In these cases the compositions were no less sensitive to impact than RDX. Several softer polymers gave effective coatings and desensitised the RDX. Film formation was enhanced using plasticizers and flow properties were improved by applying an additional coating of zinc stearate. Vacuum thermal stability in most cases was excellent. The six compositions rated as most promising were prepared on a larger scale and their impact sensitiveness, shock sensitivity and cookoff behaviour have been assessed. Compositions with sensitiveness less than that of tetryl have been produced; however further desensitization, particularly to slow cookoff, must be achieved to produce an acceptable insensitive booster composition. The use of plasticizers to enhance coating efficiencies and the incorporation of other explosives to modify cookoff response should be examined.

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PREPARATION, CHARACTERISATION AND COOKOFF BEHAVIOUR OF BOOSTER COMPOSITIONS PREPARED FROM RDX AND ACRYLIC OR ETHYLENE-VINYL ACETATE DISPERSIONS

1. INTRODUCTION

Two insensitive PBX main charge fillings (PBXN-107 and PBXW-115) are being evaluated at MRL for use in new Australian ordnance. These compositions are less vulnerable to unplanned hazardous stimuli (i.e. bullet/fragment impact, sympathetic detonation and fuel-fire cookoff) than currently used TNT-based fillings. To fully realise the benefits of this reduced vulnerability, it is essential that the fuze booster compositions used in conjunction with these fillings have similar low vulnerability. As yet no compositions meeting this requirement have been qualified overseas. Since the problems of formulating satisfactory insensitive booster compositions have not been solved a project has been commenced at MRL to develop a suitable composition for use in Australian production and filling.

The first stage in this project has involved identifying the polymeric binder and coating method for preparing RDX/polymer (95:5) compositions which give the best (mildest) responses when assessed using a small scale cookoff test. The results obtained to date have shown that certain ethylene-vinyl acetate (EVA) copolymers can give compositions exhibiting mild cookoff responses if the RDX crystals are well coated [1]. These RDX/EVA compositions and the slurry coating method used for their preparation are the subject of continuing investigations. However, the method for producing these compositions and other promising overseas compositions, e.g. PBXW-7 (RDX/TATB/Viton A 35:60:5) would require major plant and operational changes if they were to be introduced into Australian defence production. The slurry coating technique involves the removal of flammable organic solvent from a vigorously agitated aqueous slurry of RDX containing an immiscible solution of the polymer in organic solvent [2]. It requires special, expensive processing equipment not currently used for explosives in Australia and introduces the additional hazard of toxic, flammable solvent into explosives processing plants.

These problems can be avoided and existing equipment can be utilized if the compositions are prepared using aqueous polymer dispersions. These are coagulated, often by electrolyte addition [3, 4] or by using thermal coagulation aids [5], in an aqueous RDX slurry to give moulding granules. This technique has been previously studied using several polymer dispersions including polyacrylates [3] and polyurethanes [4, 5], however EVA dispersions have not been examined and compositions prepared by this method have not been evaluated for use as insensitive boosters.

This report describes the evaluation of both EVA and polyacrylate dispersions for the preparation of insensitive booster compositions. Acrylic polymers were included in this study because a binder of this type is used in the insensitive main charge filling PBXN-107. The study was undertaken both to assess the viability of preparing compositions by the aqueous dispersion technique and to characterise promising compositions, in particular their cookoff behaviour using small scale tests.

2. EXPERIMENTAL APPROACH

Electrolyte solutions and thermal coagulation aids were used to induce coagulation of acrylic and EVA dispersions in aqueous RDX slurries for the preparation of RDX/polymer (95:5) compositions. The impact sensitiveness of these compositions was determined and the relative efficiencies of the polymer coatings were qualitatively assessed using scanning electron microscopy.

Polymers which desensitized RDX were identified for further formulation studies. Because some polymers conferred poor flow properties on the compositions and/or failed to desensitize RDX, two formulating approaches were investigated. Firstly, two flow modifiers (Tullanox 500 and zinc stearate) were incorporated and their effects were compared. Secondly, the effect of various plasticizers and coalescing aids on the coating efficiencies of acrylic polymers and the impact sensitiveness of the resulting compositions was determined. Improved compositions resulting from these studies were prepared on a larger scale and their shock sensitivities and cookoff behaviour were determined. The compatibility of all polymers, coagulation aids and additives with RDX was assessed using a vacuum thermal stability test.

3. EXPERIMENTAL

3.1 Materials

RDX Grade A Class 1 (recrystallized) was used in all experiments. It was received wet from Albion Explosives Factory, Melbourne and dried at the pump prior to use.

All the aqueous polymer dispersions examined were commercially available. The acrylic dispersions were selected from the Acronal (BASF), Diofan (BASF) and Rhoplex (Rohm and Haas) ranges of products. Selected Mowilith (Hoechst) dispersions and a Vinnapas (Wacker) EVA dispersion were also examined. Table 1 lists the dispersions studied, the properties of these commercial products and the properties of the resulting dried polymer films.

Polymer additives used in these experiments included plasticizers, coalescing aids and flow modifiers. These additives and their manufacturers are listed in Table 2. Cartafix U Liquid (Sandoz) and Basensol DS 2171 (BASF) were used in some experiments to lower the temperature required for coagulation of the dispersions. Manoxol OT 60, obtained from Harcros Industrial Chemicals, is a 60% w/w solution of an anionic surfactant (dioctyl sodium sulphosuccinate).

The sodium stearate used was laboratory reagent grade. All inorganic salts used were laboratory reagent or analytical reagent grades. In all the preparations distilled water was used.

3.2 Preparation of RDX/Polymer (95:5) Compositions

These compositions were prepared by the following general method. The specific experimental condition employed for the different dispersions are given in Table 3.

A slurry of RDX (31.67 g) and water (30 g) was mechanically stirred for 15 min at room temperature then the dispersion (containing 1.67 g of polymer by weight) was added. The thermal coagulation aid (if required) was added then the temperature of the slurry was raised and a solution of the electrolyte in water was added dropwise at a constant rate over the addition period. In most experiments the slurry was then maintained at a higher temperature (typically 80-100°C) until coagulation was complete and the aqueous phase was clear. The stirred slurry was chilled in ice-water then the product was collected by filtration and washed thoroughly with water. The product was dried initially at the pump and finally at 60°C in a vacuum over silica gel.

3.3 Preparation of RDX/Acrylic Polymer/Additive (95:5:0.5) Compositions (Small Batches)

These compositions were usually prepared by the general method described in Section 3.2. In all cases the plasticizers or coalescing aids were stirred with the dispersions for at least 24 h at room temperature prior to the coating experiments. Details of the specific experimental conditions for the preparation of each composition are given in Table 4.

3.4 Preparation of RDX/Vinnapas EV 2/Additive Compositions (Small Batches)

A small batch (33.3 g) of RDX/Vinnapas EV 2/zinc stearate (95:4:1) was prepared by following the method described in Section 3.5 and altering the quantities.

The RDX/Vinnapas EV 2/Tullanox 500 (97:2:1) composition was prepared by the following method.

A slurry of RDX (31.67 g) and water (25 g) was stirred for 15 min at room temperature then Vinnapas EV 2 (1.33 g) diluted with water (10 mL) was added. The mixture was heated to 70°C and an aqueous sodium sulphate solution (20% w/w, 50 mL) was added at a constant rate over 40 min. The mixture was then heated to 90°C and Manoxol OT 60 (0.053 g) in water (5 g) was added. Tullanox 500 (0.317 g) was added after 15 min and the stirred mixture was heated for a further 30 min then cooled. The product was collected and dried at the pump, then drying was completed in a vacuum oven at 60°C over silica gel.

3.5 Preparation of RDX/Polymer/Additive Compositions (Large Batches)

The methods for preparing these compositions are variations on the general approach described below. If plasticizers were used, these were stirred with the dispersions for a minimum of 24 h prior to preparation of the compositions. The various compositions prepared and the specific experimental conditions are listed in Table 5.

RDX (300 g) and water (300 g) were stirred mechanically for 15 min and the dispersion (containing plasticizer if required) was added. The thermal coagulation aid (if required) was added then the slurry was stirred for a further 15 min, the temperature was raised and the electrolyte solution was added at a constant rate over the stated time. This was usually followed by further heating at a high temperature to complete thermal coagulation. The mixture was then cooled to 20°C and the collected product was washed with water (minimum of 2.5 L). If an additional coating of zinc stearate was required the material was maintained in a damp state after washing and was then stirred with distilled water (180 mL) in the mixing vessel and heated to 80-90°C. A hot aqueous solution of sodium stearate (6% w/w) was added (51 mL was used when the final composition was required to contain 1% zinc stearate). After 15 min an aqueous solution of zinc sulphate (2% w/w) was added at a constant rate over the stated addition time. The mixture was then cooled to 20°C and the composition was collected, washed thoroughly with water (minimum of 4 L) and dried at the pump. Drying was then completed by maintaining the composition at 60°C under vacuum over silica gel for several hours.

3.6 Characterization

3.6.1 Scanning Electron Microscopy

A Cambridge Instruments Model S250 Mk II scanning electron microscope with a tungsten electron gun was used to obtain the micrographs. The instrument was operated at 15-21 kV in the secondary electron mode and a tilt angle of 30 degrees was generally used. The samples were mounted using PVA adhesive and were sputter coated with a conducting film of gold. Coating efficiencies were determined qualitatively after a thorough examination of the sample and several representative micrographs.

3.6.2 Rotter Impact Sensitiveness : Figure of Insensitiveness (F of I)

Impact sensitiveness was determined using a Rotter Apparatus [6]. The F of I values were determined using the Bruceton procedure to analyse results. Unless indicated otherwise these were obtained using 50 caps. The values quoted are relative to RDX Grade F = 80 and are rounded to the nearest five units. They are derived from the height for a 50% probability of initiation. Average gas volumes for positive results are also quoted.

3.6.3 Shock Sensitivity : Small Scale Gap Test

Shock sensitivity data was obtained using the MRL small scale gap test (SSGT) [7]. The donor used was a UK Mk 3 exploding bridge wire detonator attenuated by brass shim. The acceptor was two 12.7 mm diameter x 12.7 mm height cold pressed cylinders of the explosive under study. A detonation was confirmed using a mild steel witness block. The results were obtained from 20-30 firings using the Bruceton staircase method and are quoted in mm of brass shim for a 50% detonation probability, 95% confidence limits and standard deviation.

3.6.4 Vacuum Thermal Stability Test

Standard Australian procedures [8] were followed in performing this test. The results quoted are the average volume (mL) of gas evolved at 120°C over 40 h for duplicate 5 g samples. Tests were performed on RDX/polymer (95:5) compositions and RDX/additive (95:5) physical mixtures, together with control tests using 0.25 g of the additives alone.

3.6.5 Cookoff Test

The cookoff behaviour of the compositions was assessed using the Super Small-scale Cookoff Bomb (SSCB) [9, 10]. The SSCB uses an explosive sample 16 mm diameter and 64 mm long, with a total mass of approximately 20 g, consisting of four pellets. All compositions were pressed to 90 %TMD for testing. Duplicate tests were conducted on all samples at a fast heating rate (approximately 1°C/s), and compositions which showed mild responses were subjected to further tests at both fast and slow (approximately 0.1°C/s) heating rates. The results presented include the type of cookoff response obtained, the explosive surface temperature at reaction, and the time to reaction.

4. RESULTS AND DISCUSSION

4.1 RDX/Polymer (95:5) Compositions

Experimental conditions were devised for preparing RDX/polymer (95:5) compositions using nine acrylic and four EVA copolymers (Section 3.2 and Table 3). The dispersions were coagulated using polyvalent electrolyte solutions. In difficult cases, thermal coagulation aids were also added; these aids are polymers and the principles of the action of this class of compounds have been reviewed [11]. In preliminary experiments some compositions were prepared using only thermal coagulation aids (no electrolyte) but these were found to have inferior coating efficiencies. Coagulation of all dispersions was enhanced when higher temperatures were used but for some dispersions it was necessary to operate at reduced temperatures to prevent agglomeration during processing.

Several of the compositions where the polymer was "soft" had a tendency to form clumps during the drying process, which made them difficult to handle. This problem was most severe with Vinnapas EV 2 and the RDX/Vinnapas EV 2 (95:5) composition was not characterized because of its poor handling properties. However a free flowing composition, RDX/Vinnapas EV 2/zinc stearate (95:4:1), was prepared by washing the polymer-coated RDX to remove electrolyte and applying a zinc stearate coating in a second coating operation (Section 3.4). An alternative, less successful approach to solving this problem was also briefly investigated. Immediately after coagulation of the Vinnapas EV 2 dispersion, an anionic surfactant and then a hydrophobic fumed silica, Tullanox 500, were added to the hot slurry. The surfactant aided deposition of the Tullanox 500, a flow modifier, on the exterior of the polymer coated RDX particles and the composition containing RDX/Vinnapas EV 2/Tullanox 500 (97:2:1) prepared in this one step coating process (Section 3.4) had adequate flow properties. However the Tullanox 500 made this composition extremely sensitive to impact (F of I = 30) so this approach was not further pursued.

Rotter impact sensitiveness data for all these compositions are compared in Table 6. Nine of the compositions are as sensitive or slightly more sensitive to impact than the original uncoated RDX Grade A (F of I = 80), and one composition was extremely sensitive. The three least sensitive compositions contained either Mowilith DM 120, Rhoplex HA-24 or Vinnapas EV 2 zinc stearate, with Fs of I ranging from 90-115. The impact sensitiveness of the RDX polymer compositions will be influenced by the fracture, physical and thermal properties of the polymers [12] and differences in polymer coating efficiencies [4]. Because the polymers vary greatly in chemical structure and since their properties are not well characterized (see Table 1) it is not possible to evaluate the effects of polymer properties on impact sensitiveness, however in general those polymers which desensitized RDX were comparatively soft.

Differences in coating efficiencies appeared to have the most pronounced effect on impact sensitiveness. The coating efficiencies of compositions prepared from all dispersions, except Mowilith DM 105 and Mowilith DM 130, were qualitatively assessed using scanning electron microscopy (SEM). Some representative micrographs of some of these compositions and RDX Grade A crystals (Micrograph A) are compared in Figure 1. The texture of the polymer coatings and the bare RDX crystal surfaces differ markedly and differences in coating efficiency were easily assessed.

The RDX/Vinnapas EV 2/zinc stearate (95:4:1) composition was least sensitive to impact and SEM examination of a Vinnapas EV 2 coating, prior to addition of the zinc stearate, showed that the polymer was deposited in a uniform film over the surfaces of the crystals to give an excellent coating (Micrograph B). The compositions prepared using Rhoplex HA-24 and Mowilith DM 120 (Fs of I ~ 95 and 90) were also significantly less sensitive than RDX but in these cases the polymer coatings were not uniform (Micrographs C and D). The polymers were deposited in clumps across the crystal surfaces. Although there was evidence of coalescence of the precipitate, some areas of exposed RDX crystal were clearly visible.

The remaining polymers gave compositions that had similar or greater impact sensitiveness than RDX. Coating efficiency was poor, with [illegible] and coarse precipitated polymer particles typically being deposited across the crystal surfaces to form a porous, open mat coating. This is illustrated in micrographs for the RDX/Acronal 230 D (95:5) composition (Micrographs E and F) and the RDX/Diofan A 880 S (95:5) composition (Micrograph G). The coating has been disrupted in one case and the exposed bare crystal surface at the centre of micrograph F is clearly distinguished from the more textured, porous polymer coating. The worst coating was obtained using Acronal 250 D where the composition more closely approximated a physical mixture.

In an effort to reduce the impact sensitiveness of compositions prepared from these dispersions, both coalescing aids and plasticizers were incorporated to soften the precipitated dispersions, induce their coalescence and promote polymer film formation on the surfaces of the crystals.

4.2 RDX/Acrylic/Additive (95:5:0.5) Compositions

Many compositions were prepared from RDX by using five acrylic dispersions modified with various plasticizers and coalescing aids. The dispersions and additives were routinely premixed prior to the coagulation experiment so that the polymer particles were completely softened. Since these additives affected the stability of the dispersions it was necessary to modify the coagulation conditions when different additives were used with the same dispersion (Table 4).

Rotter impact sensitiveness data for RDX/acrylic/additive (95:5.0.5) compositions are shown in Table 7. The results show that, without plasticizers, Acronal 230 D sensitizes RDX to give an F of I value of 70, but the incorporation of different plasticizers has a range of desensitizing effects. The most pronounced effects were obtained using Reofos 65 (F of I = 100) and Fyrol FR-2 (F of I = 90). Limited experiments using plasticizers with Acronal 250 D, Acronal 627, Acronal S 704 and Diofan A 880 S produced only small changes in impact sensitiveness. In some cases the plasticized compositions were more sensitive. The experiments confirmed that for each dispersion it was necessary to empirically determine the best plasticizer for reducing impact sensitiveness.

The relative coating efficiencies of many RDX/Acronal 230 D/additive (95:5:0.5) compositions were compared using scanning electron microscopy. As the impact sensitiveness of the compositions decreased (F of I increased) the degree of coalescence of precipitated polymer increased and more uniform coating films were obtained. This effect was most pronounced for the composition containing Reofos 65 (Micrograph H) where the crystals were well covered with a quite uniform polymer film. This good coating is in marked contrast to the poor coatings obtained using Acronal 230 D without plasticizer (Micrographs E and F). Similarly, the precipitated polymer particles in the RDX/Acronal 627 (95:5) composition (Micrograph I) partly coalesced when Reofos 65 was added as a plasticizer (Micrograph J). In this case film formation was not complete and the coating efficiency was assessed as only fair.

The plasticizer experiments identified three dispersion/additive combinations — Acronal 230 D/Reofos 65, Acronal 250 D/DOP and Acronal 627/Reofos 65 — which partly coated the RDX and gave desensitized moulding powders. These mixtures, together with Rhoplex HA-24, Mowilith DM 120 and Vinnapas EV 2, which also markedly reduced impact sensitiveness, were used to prepare larger batches of compositions for evaluation as insensitive booster compositions. The RDX/Acronal 250 D/DOP (95:5:0.5) composition could not be considered for fuzing applications because it was too impact sensitive (F of I = 85, minimum requirement 90 [13]); it was further studied here primarily to determine the effect of this polymer/plasticizer combination on cookoff behaviour. The zinc stearate coating technique which improved the RDX/Vinnapas EV 2 composition was also used with the compositions containing RDX with Acronal 230 D/Reofos 65, Rhoplex HA-24 and Mowilith DM 120 to improve the flow properties of the moulding powders.

4.3 Insensitive Booster Compositions (Large Batches)

Large batches of the six compositions identified in the preliminary evaluation were prepared using the experimental conditions given in Table 5. The composition containing Acronal 230 D and Reofos 65 was difficult to consistently produce and the large batch was more sensitive to impact (Table 8, F of I = 90) than a small batch prepared without an additional zinc stearate coating (Table 7, F of I = 100). The other large batches containing plasticized acrylic polymers had F of I values (Table 8) consistent with those obtained for earlier small scale batches (Table 7), while the large batches of the formulations containing unplasticized coatings but with zinc stearate coatings were all less sensitive to impact than the small-scale batches of RDX/polymer alone (Table 6).

The shock sensitivities of all the compositions were determined using compacts pressed to 90 %TMD and the results are shown in Table 8. All compositions are less shock sensitive than granular tetryl and more shock sensitive than the insensitive booster composition PBXW-7. The compositions containing plasticized acrylic polymers have comparatively high $M_{50\%}$ values ranging from 2.65 to 2.30 mm while the other compositions, containing Rhoplex HA-24, Mowilith DM 120 and Vinnapas EV 2, have similar (2.18 - 2.17 mm) low $M_{50\%}$ values. The latter compositions are also less sensitive to impact.

The compositions prepared from the harder polymers (i.e. Acronal 230D, Acronal 250D, and Acronal 627) softened with plasticizers gave less cohesive pellets on compaction than did the other compositions. The SSCB test pellets prepared from Acronal 230D were quite crumbly, and those prepared from Acronal 250D grew considerably on storage (diameter increased from 15.875 mm to approximately 16.3 mm within 6 months under ambient conditions) and showed pronounced distortion and severe roughening of all surfaces. All the other compositions gave satisfactory pellets.

4.3.1 Cookoff Behaviour

The results of SSCB tests on the six booster compositions are shown in Table 9, together with results for tetryl and PBXW-7 Type II [10] for comparison. Fast heating rate tests were performed on all compositions, and slow heating rate tests were carried out on those compositions which gave mild reactions at the fast heating rate.

Two of the compositions containing plasticized acrylic polymers (Acronal 230D and Acronal 627) gave violent cookoff reactions (detonations) at the fast heating rate, and were not examined further. The other plasticized acrylic polymer, Acronal 250D with DOP, gave mild explosions at the fast heating rate, and detonation at the slow heating rate. This result was somewhat surprising, since the Acronal 250D composition had a poorer coating efficiency than the other plasticized acrylic polymers (reflected in its higher shock and impact sensitivities), and gave poor quality pressed pellets which could be expected to break up more readily on heating, resulting in a larger exposed surface area and leading to more rapid and violent reaction.

The other composition containing an acrylic polymer, Rhoplex HA-24, gave deflagration or mild explosion reactions at the fast heating rate; at the slow heating rate explosions varying from mild to extremely violent (close to detonation - the SSCB baseplate was cracked through but not holed) were obtained.

Both the compositions using EVA copolymer coatings (Mowilith DM 120 and Vinnapas EV 2) gave widely varying responses at the fast heating rate, ranging from burning (with residues of unconsumed explosive being recovered) through to detonation; and both compositions gave detonations at the slow heating rate. Both of these compositions had good quality coatings, and were expected to give mild cookoff reactions. The reason for the variable responses at the fast heating rate is unknown; generally good agreement is obtained between replicate tests at a given heating rate [1, 9, 10].

Four of the six compositions produced have improved cookoff behaviour over that of the current booster explosive, tetryl, at the fast heating rate; however, two of these compositions give variable responses to this test. Only one composition (containing Rhoplex HA-24) gave any reduction in cookoff response at the slow heating rate. None of the compositions give cookoff responses as mild as those of the US insensitive booster composition PBXW-7 Type II; however, it should be noted that other results for materials with the same nominal composition as PBXW-7 (RDX/TATB/Viton A 35:60.5) have shown violent responses at both fast and slow heating rates [9, 10].

4.4 Vacuum Thermal Stability

Vacuum thermal stability tests at 120°C were conducted on 5 g quantities of the RDX/Polymer (95:5) compositions, and on physical mixtures (95:5) of coagulants or additives with RDX and control samples (0.25 g) of the coagulation aids and additives alone. The results of these tests are shown in Tables 10 and 11. The pass fail criterion for compatibility with RDX is that gas evolution should be less than 5 mL over 40 h [14].

All the acrylic and EVA polymers examined in this study show good compatibility with RDX and all gas volumes, except that for the RDX/Diofan A 880 S composition, are below 1 mL (Table 10). The Diofan copolymer contains vinylidene chloride which has comparatively poor thermal stability and this may contribute to the comparatively high result of 1.6 mL.

The results for the physical mixtures of RDX with the coagulation aids and additives (Table 11) show evolved gas volumes in excess of 5 mL for Cartafix U Liquid, the thermal coagulation aid, and two plasticizers, Re-plas 1360 and Reoplex 400, indicating poor compatibility with RDX. In the case of Cartafix U Liquid the poor thermal stability of this material is contributing to the high result; under the test conditions the decomposition of this additive alone gave an evolved gas volume of 3.56 mL. Cartafix U Liquid contains amine functions [15] and these characteristically show poor compatibility with RDX. The poor compatibility is not a problem during normal processing to prepare the moulding powders; the low evolved gas volumes for RDX/polymer (95:5) compositions (Table 10) prepared using this coagulation aid indicate that this material does not seriously contaminate the final washed product. All other coagulation aids and additives had quite low evolved gas volumes indicating satisfactory compatibility with RDX.

5. CONCLUSIONS

Explosive formulations suitable for use in fuze booster applications can be prepared by adding certain commercially available acrylic and EVA dispersions to hot aqueous RDX slurries and coagulating the dispersions. The coagulation is induced by addition of electrolyte solutions and can be enhanced using thermal coagulating agents. Such a process could be carried out on existing Australian production equipment.

The impact sensitiveness of the resulting compositions is strongly influenced by the efficiency of the polymer coating of the RDX crystals. Generally, under optimized coagulation conditions, the fine polymer precipitate is deposited across the surfaces of the crystals but these particles fail to coalesce to form a uniform coating; instead, a porous, open mat coating is formed. These compositions are no less sensitive to impact than RDX. Three dispersions - Vinnapas EV 2, Rhoplex HA-24 and Mowilith DM 120 - gave soft precipitates that underwent partial coalescence to give greater coating efficiency. These RDX/polymer (95:5) compositions had F of I values of at least 90 (RDX = 80). The relatively poor flow properties of these compositions were enhanced by applying a zinc stearate coating in a second operation, and these compositions were evaluated as insensitive booster compositions.

Plasticizers and coalescing aids were used to modify unsatisfactory dispersions and promote film formation, and in several cases gave compositions which were less impact sensitive than those where the dispersion was used alone. Three acrylic/plasticizer (10:1) combinations - Acronal 230D/Reofos 65, Acronal 250D/DOP, and Acronal 627/Reofos 65 - which gave improved coatings and desensitized the RDX were formulated to give booster compositions with acceptable flow properties.

All of the acrylic and EVA polymers examined were found to be compatible with RDX, and gave RDX compositions with satisfactory vacuum thermal stability. Most additives and plasticizers examined were also found to be compatible with RDX, although two plasticizers (Re-plas 1360 and Reoplex 400) were incompatible. The thermal coagulation aid Cartafix U Liquid was also found to have poor compatibility with RDX; however, this material is used in low concentrations and does not appear to contaminate the final product, and compositions prepared using this material all showed satisfactory vacuum thermal stability.

In addition to impact sensitiveness, shock sensitivity and cookoff behaviour were assessed on pressed charges. The compositions prepared from the unplasticized soft polymers (i.e. Rhoplex HA-24, Mowilith DM 120 and Vinnapas EV 2) all gave higher quality pressed pellets than did the compositions containing plasticizers. These formulations also showed reduced impact sensitiveness, shock sensitivity, and cookoff response relative to the compositions prepared with plasticized acrylic polymer coatings. The six formulations selected as most promising from the initial small-scale experiments were more extensively examined.

All the formulations were less impact sensitive than RDX, and all except the RDX/Acronal 250D/DOP composition had F of I values greater than 90, and would therefore be acceptable for fuze booster applications. All the formulations had shock sensitivities intermediate between those of tetryl, the most widely used booster explosive, and PBXW-7 Type II, an insensitive US booster explosive currently being qualified for general use. Four of the six formulations gave less violent cookoff reactions than tetryl under fast heating conditions; only one (containing Rhoplex HA-24) showed any reduction in cookoff response at a slower heating rate. None of the formulations gave cookoff responses as mild as those of PBXW-7 Type II; however, this was not unexpected, as this composition contains 60% of the insensitive explosive TATB to give the desired moderation of cookoff response. Incorporation of TATB into the compositions examined here should further reduce the violence of their cookoff reactions.

The most promising formulation produced was RDX/Rhoplex HA-24/zinc stearate 95:4:1. This material has an F of I of 110, a shock sensitivity of 2.17 mm (SSGT) at 90 %TMD, and exhibits mild cookoff behaviour at a fast heating rate, although producing more violent reactions than desired at a slow heating rate. Such a composition, although less sensitive than tetryl, is not an acceptable insensitive booster explosive.

The use of plasticizers to enhance the coating efficiency of polymers which produce some desensitisation, and the incorporation of other explosives to modify the cookoff response, should be further examined as methods of producing an acceptable insensitive booster explosive.

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TABLE 1 SOME PROPERTIES OF THE ACRYLIC AND EVA DISPERSIONS AND THE DRIED FILMS OF THESE POLYMERS

	DISPERSION			DRIED FILM			
Material	Non Volatile (%)	Dispersion Type	Composition	Density (Mg/m^3)	Tensile Strength (MPa)	Elongation at Break (%)	Tg (°C)
Acronal 230 D	50	Anionic	Acrylic/ styrene	1.115[a]	15	200	30
Acronal 250 D	50	Anionic	Ethyl acrylate/ acrylonitrile	1.188[a]	15	200	24
Acronal 627	50	Anionic	Methyl methacrylate/ butyl acrylate	1.105[a]	12	90	-20
Acronal S 704	50	Anionic	Acrylic/ styrene	1.08	8	520	22
Diofan A 880 S	45	Anionic	Acrylic/ vinylidene chloride	1.50	17	312	28
Rhoplex HA-12	45	Nonionic	Acrylic	NA	NA	NA	9
Rhoplex HA-24	44.5	Anionic	Acrylic	1.160[a]	NA	NA	-15
Rhoplex P-376	50	Anionic	Acrylic/ styrene	NA	NA	NA	6
Rhoplex B-1139	38	Anionic	Acrylic	NA	NA	NA	25
Mowilith DM 105	55	NA	Ethylene/ vinyl acetate	NA	5	600	5
Mowilith DM 120	50	NA	Ethylene/ vinyl acetate/ vinyl chloride	1.208[a]	5	500	12
Mowilith DM 130	50	NA	Ethylene/ vinyl acetate	NA	2.5	500	-7
Vinnapas EV 2	50	NA	Ethylene/ vinyl acetate	1.190[a]	8	500	-10

a Densities determined in this study using a gas pycnometer at 20 ± 2°C.
NA Not available

TABLE 2 FUNCTIONS, CHEMICAL DESCRIPTIONS AND MANUFACTURERS OF ADDITIVES USED IN COATING EXPERIMENTS

Material	Chemical Description	Function	Manufacturer
DOA[a]	Dioctyl adipate	Plasticizer	CSR Chemicals
DOP	Dioctyl phthalate	Plasticizer	CSR Chemicals
Edenol DCHP	Dicyclohexyl phthalate	Solid plasticizer	Henkel
Fyrol FR-2	Tris(β,β'-dichloro-isopropyl) phosphate	Flame-retardant plasticizer	Stauffer Chemical Company
Methyl di-icinol	Diethylene glycol monomethyl ether	Coalescing aid	ICI
Plastilit 3060	Polypropylene glycol alkylphenyl ether	Plasticizer	BASF
PEG 12000	Polyethylene glycol (molecular weight 12 000)	Plasticizer	ICI
Reofos 65[b]	Triaryl phosphate ester	Flame-retardant plasticizer	Ciba-Geigy
Re-plas 1360	High molecular weight long chain polyester	Plasticizer	Townsend Chemicals
Reoplex 400	Polymeric	Plasticizer	Ciba-Geigy
Texanol	2,2,4-trimethyl-1,3-pentanediol monoisobutyrate	Coalescing aid	Eastman
Tullanox 500	Trimethylsilated fumed silica	Flow modifier	Tulco Inc.

[a] Density at 24°C is 0.981 Mg/m^3

[b] Density at 25°C is 1.164 Mg/m^3

TABLE 3 EXPERIMENTAL CONDITIONS FOR THE PREPARATION OF 33.3 GRAM BATCHES OF RDX/ACRYLIC (95:5) AND RDX/EVA (95:5) COMPOSITIONS

Polymer Dispersion Used	Thermal Coagulation Aid (wt g)	Addition of Aqueous Electrolyte Solution				Additional Heating for Thermal Coagulation	
		Type (conc w/w)	Weight Added (g)	Time Taken (min)	Temperature (°C)	Time (min)	Temperature (°C)
ACRYLIC							
Acronal 290 D	Cartafix U (0.5)	$CaCl_2$ (25%)	40[a]	-	25	30	80-85
Acronal 250 D	Cartafix U (0.34)	$CaCl_2$ (5%)	30[a]	-	25	40	90-95
Acronal 627	-	$CaCl_2$ (10%)	23	30	80	-	-
Acronal S 704	Cartafix U (0.34)	$CaCl_2$ (25%)	10	30	60	5	95-100
Diofan A 880 S	-	$CaCl_2$ (25%)	7	30	60	15	75
Rhoplex HA-12	-	$Al_2(SO_4)_3$ (25%)	10	25	45	15	95
Rhoplex HA-24	-	$Al_2(SO_4)_3$ (25%)	7	25	45	10	75
Rhoplex P-376	-	$Al_2(SO_4)_3$ (25%)	4	15	35	10	85
Rhoplex B-1339	Basensol DS 2171 (0.34)	$Al_2(SO_4)_3$ (25%)	6	20	45	10	55
EVA							
Mowilith DM 105	Basensol DS 2171 (0.34)	$Al_2(SO_4)_3$ (25%)	33	30	70	20	95-100
Mowilith DM 120	Basensol DS 2171 (0.34)	$Al_2(SO_4)_3$ (25%)	33	30	70	20	95-100
Mowilith DM 130	Basensol DS 2171 (0.34)	$Al_2(SO_4)_3$ (25%)	32	30	70	20	95-100
Vinnapas EV 2	-	Na_2SO_4 (20%)	50	30	75	-	-

[a] The electrolyte solution was used in place of water to prepare the aqueous slurry.

TABLE 4 EXPERIMENTAL CONDITIONS FOR THE PREPARATION OF 33.3 GRAM BATCHES OF RDX/ACRYLIC/ADDITIVE (95:5:0.5) COMPOSITIONS

Polymer Dispersion	Plasticizer	Thermal Coagulation Aid (wt, g)	Addition of Aqueous Electrolyte Solution				Heating for Thermal Coagulation	
			Type (25% w/w)	Weight Added (g)	Time Taken (min)	Temperature (°C)	Time (min)	Temperature (°C)
Acronal 230 D	Dioctyl adipate	Cartafix U (0.34)	$CaCl_2$	10	15	70	30	95-100
	Edenol DCHP (a)	Cartafix U (0.34)	$Al_2(SO_4)_3$	25	30	70	30	95-100
	Pyrol FR-2	Cartafix U (0.34)	$CaCl_2$	10	10	70	20	90-95
	Methyl di-icinol(b)	Cartafix U (0.34)	$CaCl_2$	10	12	90-95	40	90-95
	Plastilit 3060 (c,d)	Cartafix U (0.34)	$CaCl_2$	37	-	25	30	85-90
	PEG 12000	Cartafix U (0.34)	$CaCl_2$	10	12	95-100	15	95-100
	Reofos 65	Cartafix U (0.34)	$CaCl_2$	12	20	70	45	95-100
	Re-plas 1360	Cartafix U (0.34)	$Al_2(SO_4)_3$	30	30	70	20	95-100
	Reoplex 400 (b,d)	Cartafix U (0.34)	$CaCl_2$	6	7	90-95	20	90-95
	Texanol	Cartafix U (0.34)	$CaCl_2$	10	15	70	30	95-100
Acronal 250 D	Dioctyl adipate	Cartafix U (0.34)	$Al_2(SO_4)_3$	35	30	70	30	95-100
	Dioctyl phthalate	Cartafix U (0.34)	$Al_2(SO_4)_3$	35	30	70	30	95-100
Acronal 627	Reofos 65	Cartafix U (0.34)	$Al_2(SO_4)_3$	10	12	70	15	90
Acronal S 704	Dioctyl adipate	Cartafix U (0.34)	$CaCl_2$	30	25	75	30	95-100
	Dioctyl phthalate	Cartafix U (0.34)	$CaCl_2$	33	30	70	20	95-100
	Reofos 65	Cartafix U (0.34)	$Al_2(SO_4)_3$	4	8	70	20	95
Diofan A 690 S	Dioctyl adipate	-	$CaCl_2$	10	12	75	20	85-90
	Dioctyl phthalate	-	$CaCl_2$	7	12	70	25	90
	Reofos 65	Cartafix U (0.34)	$Al_2(SO_4)_3$	5	10	70	15	85-90

a Acronal 230 D was treated with a solution of Edenol DCHP in methylene chloride (40% w/w) for 24 h prior to this experiment.

b In this experiment heating for coagulation was carried out prior to electrolyte addition.

c The electrolyte solution was used in place of water to prepare the aqueous slurry.

d Composition ratios were 95:5:1.5

TABLE 5 EXPERIMENTAL CONDITIONS FOR THE PREPARATION OF RDX/POLYMER/ADDITIVE (LARGE BATCHES)

Composition	Thermal Coagulation Aid (g)	Addition of Aqueous Electrolyte Solution				Heating for Thermal Coagulation		Zinc Stearate Coating Conditions		
		Type (% w/w)	Quantity Added	Time Taken (min)	Temperature (°C)	Time (min)	Temperature (°C)	Vol of $ZnSO_4$ Soln (28 w/w) (mL)	Addition Time (min)	Temperature (°C)
RDX/Acronal 230 D/Reofos 65/zinc stearate (95:4:0.4:1)	Cartafix U (3.22)	$CaCl_2$ (25)	300 g	80	70	50	95	200	70	80
RDX/Acronal 250 D/DOP (95:5:0.5)	Cartafix U (3.22)	$Al_2(SO_4)_3$ (25)	300 mL	95	70	50	95	-	-	-
RDX/Acronal 627/Reofos 65 (95:5:0.5)	Cartafix U (3.22)	$Al_2(SO_4)_3$ (25)	20 g	25	75	40	90	-	-	-
RDX/Rhoplex HA-24/zinc stearate (95:4:1)	-	$Al_2(SO_4)_3$ (25)	66 g	25	90	-		200	80	90
RDX/Mowilith DM 120/zinc stearate[a] (95:4:1)	Basensol DS 2171 (3.22)	$Al_2(SO_4)_3$ (25)	300 g	100	70	30	95	300	100	80
RDX/Vinnapas EV 2/zinc stearate[b] (95:3.5:1.5)	-	Na_2SO_4 (20)	475 mL	60	75	-	-	200	20	90

[a] The mixture was maintained at 95°C for 20 min after the addition of the zinc sulphate solution

[b] The Vinnapas EV 2 dispersion was diluted with water (90 mL) prior to addition

TABLE 6 IMPACT SENSITIVENESS OF RDX/ACRYLIC (95:5) AND RDX/EVA (95:5) COMPOSITIONS

Composition and Class of Binder	F of I[a]	Gas Evolution (mL)
ACRYLIC		
RDX/Acronal 230 D	70	15
RDX/Acronal 250 D	80	17
RDX/Acronal 627	80	16
RDX/Acronal S 704	75	15
RDX/Diofan A 880 S	70	15
RDX/Rhoplex HA-12	85	15
RDX/Rhoplex HA-24	95	13
RDX/Rhoplex P-376	75	16
RDX/Rhoplex B-1339	75	16
EVA		
RDX/Mowilith DM 105	75	14
RDX/Mowilith DM 120	90	14
RDX/Mowilith DM 130	75	14
RDX/Vinnapas EV 2/zinc stearate[b]	115	15
RDX/Vinnapas EV 2/Tullanox 500[c]	30	10

[a] Relative to RDX Grade F = 80

[b] Composition ratio was 95:4:1

[c] Composition ratio was 97:2:1

TABLE 7 IMPACT SENSITIVENESS OF VARIOUS RDX/ACRYLIC/ADDITIVE (95:5:0.5) COMPOSITIONS

Polymer and Plasticizer	F of I [a]	Gas Evolution (mL)
ACRONAL 230 D		
-	70	15
Methyl di-icinol	70	16
Reoplex 400 [b]	70	15
Polyethylene glycol 12000	75	16
DOA	80	15
Edenol DCHP	80[c]	14
Re-plas 1360	80	11
Texanol	80	16
Plastilit 3060 [b]	85	14
Fyrol FR-2	90	15
Reofos 65	100	14
ACRONAL 250 D		
-	80	17
DOA	85[c]	15
DOP	85[c]	13
ACRONAL 627		
-	80	16
Reofos 65	90	10
ACRONAL S 704		
-	75	15
Reofos 65	65[c]	13
DOP	70[c]	14
DOA	75[c]	15
DIOFAN A 880 S		
-	70	15
Reofos 65	60[c]	13
DOP	70[c]	14
DOA	80[c]	13

[a] Relative to RDX Grade F = 80

[b] Composition ratios were 95:5:1.5

[c] Results obtained using 25 caps only

TABLE 8 IMPACT SENSITIVENESS AND SHOCK SENSITIVITY (SSGT) OF RDX/POLYMER/ADDITIVE COMPOSITIONS

Composition and Class of Binder Used		Impact Sensitiveness			Shock Sensitivity (mm)		
		F of I[a]	Gas Evolved (ml)	Relative Density (%TMD)	$M_{50\%}$	Range $L_{95\%}$	Standard Deviation
ACRYLIC							
RDX/Acronal 230 D/Reofos 35/zinc stearate	(95:4:0.4:1)	90	12	90.03	2.63	2.71 2.54	0.039
RDX/Acronal 250 D/DOP	(95:5:0.5)	8[illegible]	15	90.06	2.65	2.77 2.54	0.052
RDX/Acronal 627/Reofos 65	(95:5:0.5)	90	10	90.01	2.30	2.35 2.26	0.022
RDX/Rhoplex HA 24/zinc stearate	(95:4:1)	110	13	90.00	2.17	2.22 2.13	0.021
EVA							
RDX/Mowilith DM 120/zinc stearate	(95:4:1)	105	11	90.02	2.18	2.25 2.11	0.032
RDX/Vinnapas EV 2/zinc stearate	(95:3.5:1.5)	125	13	90.00	2.17	2.24 2.11	0.028
OTHER BOOSTER COMPOSITIONS							
Tetryl, granular[b]		110	16	90.0	1.259	1.315 1.203	0.026
PBXW-7 Type II[b] (RDX/TATB/Viton A)	(35:60:5)	90	4	90.0	1.415	1.446 1.382	0.015

a Relative to RDX Grade F - 80 and determined using 25 caps only

b Data from reference 13

TABLE 9 RESULTS OF COOKOFF TESTS (SSCB) OF RDX/POLYMER/ADDITIVE COMPOSITIONS
ALL TESTS CONDUCTED AT FAST HEATING RATE, EXCEPT WHERE INDICATED

Composition	Explosive Surface Temperature (°C)	Time (s)	Cookoff Reaction
ACRYLIC POLYMER			
RDX/Acronal 230D/	227	248	Detonation
Reofos 65/ZnSt	242	251	Detonation
95:4:0.4:1			
RDX/Acronal 250D/DOP	242	266	Explosion (mild)
95:5:0.5	238	238	Explosion (mild)
Slow Heating	220	1642	Detonation
RDX/Acronal 627/Reofos 65	243	-	Detonation
95:5:0.5	236	256	Detonation
RDX/Rhoplex HA-24/ZnSt	234	255	Explosion (mild)
95:4:1	228	239	Deflagration (b)
Slow Heating	223	1573	Explosion (mild) (b)
Slow Heating	223	1570	Explosion (violent)
EVA POLYMER			
RDX/Mowilith DM120/ZnSt	224	235	Burning (a, b)
95:4:1	237	240	Detonation
	242	263	Deflagration (b)
Slow Heating	222	1484	Detonation
RDX/Vinnapas EV2/ZnSt	241	267	Burning (a, b)
95:3.5:1.5	242	252	Detonation
	237	276	Deflagration (b)
Slow Heating	217	1662	Detonation
OTHER BOOSTER COMPOSITIONS			
Tetryl [10]	257	239	Detonation
	238	240	Detonation
PBXW-7 Type II [10]	265	266	Burning (a, b)
Slow Heating	213	1657	Deflagration (b)

(a) Appreciable amounts (> 2 g) of unconsumed explosive recovered after test.
(b) Traces of explosive on parts after test.

TABLE 10 VACUUM THERMAL STABILITY RESULTS OF RDX/ACRYLIC (95:5) AND RDX/EVA (95:5) COMPOSITIONS

Composition and Class of Binder Used	Average Gas Evolved (mL) after 40 h at 120°C
ACRYLIC	
RDX/Acronal 230D	0.41
RDX/Acronal 250D	0.98
RDX/Acronal 627	0.76
RDX/Acronal S704	0.86
RDX/Diofan A 880 S	1.60
RDX/Rhoplex HA-12	0.14
RDX/Rhoplex HA-24	0.20
RDX/Rhoplex P-376	0.11
RDX/Rhoplex B-1339	0.40
EVA	
RDX/Mowilith DM 105	0.10
RDX/Mowilith DM 120	0.70
RDX/Mowilith DM 130	0.05
RDX/Vinnapas EV 2/zinc stearate[a]	0.10
RDX/Vinnapas EV 2/Tullanox 500[b]	0.00

a. Composition ratio was 95:4:1

b. Composition ratio was 97:2:1

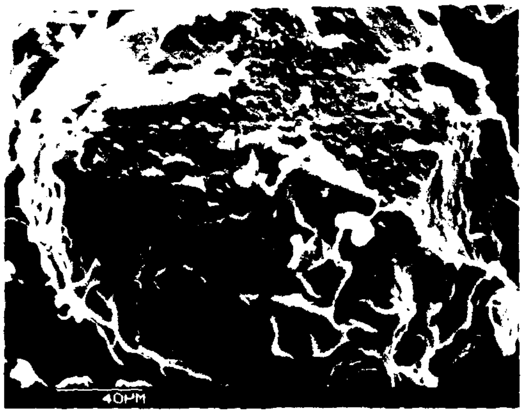
TABLE 11 VACUUM THERMAL STABILITY RESULTS FOR RDX/ADDITIVE (95:5) PHYSICAL MIXTURES AND THE ADDITIVES ALONE

Additive	Average Gas Evolved (mL) after 40 h at 120°C	
	Physical Mixtures (5 g)	Additive Only (0.25 g)
Basensol DS 2171	1.28	0.18
Cartafix U Liquid	8.95	3.56
Dioctyl adipate	0.10	0.0
Dioctyl phthalate	0.10	0.0
Edenol DCHP	0.20	0.0
Fyrol FR-2	0.40	-0.02
Methyl di-icinol	2.55	-0.30
Plastilit 3060	0.63	-0.14
PEG 12000	0.00	0.0
Reofos 65	0.13	0.05
Re-plas 1360	5.00	-0.16
Reoplex 400	8.05	-0.03
Texanol	-0.10	-0.20

Micrograph A *RDX Grade A Class 1 (recrystallized).*

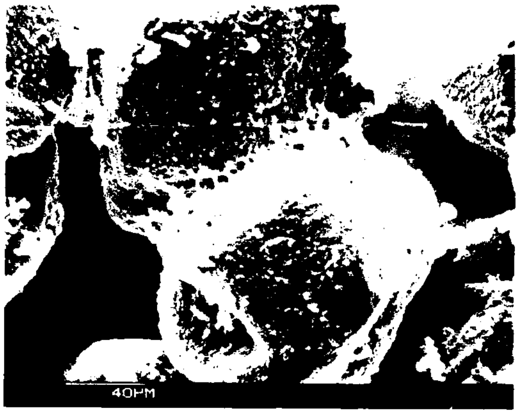
Micrograph B *RDX/Vinnapas EV 2 (98:2). Crystals are well coated with polymer.*

Figure 1 *Scanning electron photomicrographs of various RDX-based compositions*



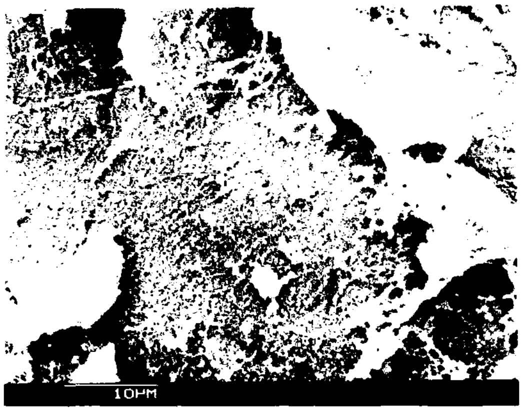
Micrograph C *RDX/Rhoplex HA-24 (95:5). Precipitated polymer deposited in clumps with some evidence of coalescence.*

Micrograph D *RDX/Mowilith DM 120 (95:5). Precipitated polymer particles have partially coalesced to give a fair coating.*



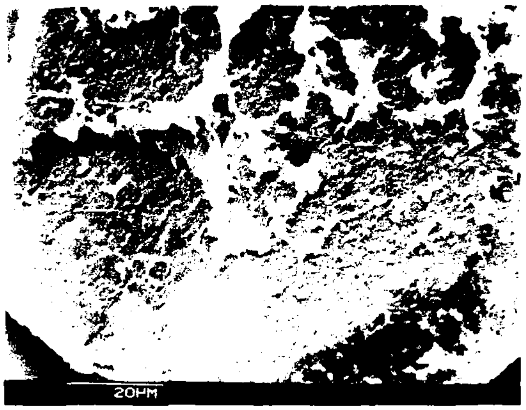
Micrograph E RDX/Acronal 230 D (95:5). The deposited polymer particles form a porous, open mat coating.

Micrograph F RDX/Acronal 230 D (95:5). The coating is disrupted in the centre of the micrograph exposing bare crystal.



Micrograph G *RDX/Diofan A 880 S (95:5). Fine and coarse precipitated polymer particles cover the crystal surface. Film formation has not occurred.*

Micrograph H *RDX/Acronal 230 D/Reofos 65 (95:5:0.5). The precipitated polymer has formed a uniform film giving a good coating.*



Micrograph I RDX/Acronal 627 (95:5). Poor coating with fine polymer particles distributed across the crystal surface.

Micrograph J RDX/Acronal 627/Reofos 65 (95:5:0.5). The precipitated particles have partially coalesced as a result of plasticizer addition.

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ABSTRACT

A range of potential booster explosives has been prepared using RDX and commercially available acrylic and ethylene-vinyl acetate dispersions. The dispersions were coagulated in hot aqueous slurries of RDX by electrolyte addition and the use of thermal coagulation aids, a process compatible with existing Australian production plant and methods. Generally the polymer coatings on the RDX crystals were porous and the precipitated particles had not coalesced to form polymer films. In these cases the compositions were no less sensitive to impact than RDX. Several softer polymers gave effective coatings and desensitised the RDX. Film formation was enhanced using plasticizers and flow properties were improved by applying an additional coating of zinc stearate. Vacuum thermal stability in most cases was excellent. The six compositions rated as most promising were prepared on a larger scale and their impact sensitiveness, shock sensitivity and cookoff behaviour have been assessed. Compositions with sensitiveness less than that of tetryl have been produced; however further desensitization, particularly to slow cookoff, must be achieved to produce an acceptable insensitive booster composition. The use of plasticizers to enhance coating efficiencies and the incorporation of other explosives to modify cookoff response should be examined.