Manufacturing of PAX-3 High Explosive

Authors: Sean Swaszek, Phil Samuels, Dr. Paul Anderson, Katherine Guraini

Abstract

The manufacturing methods of processing controls of explosives play an important role in maintaining the quality of explosive formulations. PAX-3 is an explosive formulation that is of interest to the Army for use in gun launch munitions and grenades. PAX-3 shows improved shock and Insensitive Munition (IM) response as compared with traditional explosive fills. ARDEC developed the explosive formulation PAX-3 utilizing a twin screw extrusion mixing method. This is a continuous process in which a two part mixture is fed into the extruder, which uses high shear mixing to produce PAX-3. Currently, ARDEC is also pursuing an alternate formulation process of PAX-3 production in a single batch 500 gallon slurry coating process. Efforts for qualification of explosives often requires a significant amount of resources to complete all required testing. ARDEC is evaluating the material produced from these two methods to assess PAX-3 safety, long-term aging, and maintaining similar sensitivity and performance characteristics according to AOP-7. Cost and acquisition time are also responses of interest.

Background

The explosive composition PAX-3 is a high blast explosive that maintains metal pushing capability for applications. The formulation is composed of HMX, aluminum, binder, and plasticizer. In processing the material, the HMX and aluminum is coated in the polymer binder. The PAX-3 molding powder is pressed into items achieving high densities for specific applications. Efforts to produce and qualify PAX-3 molding powder using a slurry coating is being pursued by ARDEC. The ingredients such as final granule particle size and composition for the PAX-3 remains the same between both processes. Standards such as AOP-7 dictate that undergoing any process change may cause for requiring a requalification of a process.

The method of twin screw extrusion was pursued as a larger scale manufacturing method to produce PAX-3 high explosive. Early laboratory studies had shown the capability to produce PAX-3 using twin screw extrusion process using a non-aluminized analogous formulation and Aluminum. This is a 2 step process in which the non-aluminized analogous formulation is first produced using a slurry coating process and then used as part of a feed mix with aluminum in the twin screw. The twin screw extrusion process is a continuous method in which the ingredients are fed at a constant rate to produce the final product. This allows for flexibility to produce variant formulations containing different ratios of constituent materials. Efforts to transition the twin screw technology to larger scale manufacturing was funded under the ManTech program in 2009. Using a 19mm die twin screw extruder at Milan AAP, PAX-3 was produced in large scale quantities and used for characterization testing at ARDEC. This batch of material was used for qualification efforts and is now an accepted process to produce PAX-3.

Recently, work was pursued to produce PAX-3 using the slurry coating in a one-step single batch process. BAE Holston developed a process to produce 500gallon batches of PAX-3 using this method. Slurry coating is a process in which a binder-lacquer is dissolved in a solvent.
Energetic materials such as RDX or HMX along with Aluminum are mixed in an aqueous solution with the dissolved mixture in solvent. Upon heating the solution, the solvent is distilled off and the binder-lacquer mix coats individual particles. The benefit of this reduces the process from a 2-step using twin screw extrusion to a 1-step procedure for cost savings. The final product of the slurry process appears to be similar to material made in the twin screw extruder shown in figure 1.

![Figure 1: Granules of PAX-3 produced in a twin screw extruder (left) and slurry process (right)](image)

When undergoing changes to a production process for explosive such as PAX-3 there is concern materials will have different properties or characteristics. Examples of parameters that could affect the behavior include varying amounts of polymer coating of energetic particles, residual solvents or water remaining in the composition, foreign contaminants, or a physical change such as particle size or morphology. To ensure that the PAX-3 produced in the 1-step slurry process is comparable to the 2-step twin screw extrusion, it is being evaluated through a series of characterization tests. Running a complete series of qualification testing requires a significant amount of time and costs associated with the effort. ARDEC has therefore chosen a reduced set of tests to show the slurry produced PAX-3 maintains similar characteristics to the qualified twin screw extrusion process.

**Scanning Electron Microscopy (SEM)**

SEM images were obtained using a JEOL JCM 5700 tungsten filament scanning electron microscope using palladium/gold-coated samples in high vacuum mode. SEM images from PAX-3 water slurry method are shown in figure 2.

![SEM images of PAX-3 granules produced using the slurry process](image)

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SAFETY TESTS

The basic sensitivity tests to evaluate a materials sensitivity include impact, friction, ESD, and shock sensitivity were conducted on PAX-3. Tests were performed according to AOP-7 and STANAG 4489 ED1. Samples were prepared by grinding raw material in a Wiley Mill until it passed through a 25 mesh screen and dried at 120°F to a constant weight. The results of the sensitivity tests

Explosives Research Laboratory (ERL) Impact Test

The ARDEC ERL type 12 impact tester using a 2 ½-kg drop weight was used to determine the impact sensitivity of the sample. The drop height corresponding to the 50% probability of initiation measures impact sensitivity. The test method is described in STANAG 4489 Ed.1 "Explosives, Impact Sensitivity Tests." The PAX-3 did not react at a drop height of 21.6 cm for the water slurry material and 40 cm for the twin screw extrusion material. This range of results is on part with secondary explosive materials.

BAM Friction

The large BAM friction test method is described in AOP-7, 201.02.006, “BAM Friction Test.” The porcelain pin is lowered onto the sample, and a weight placed on the arm to produce the desired load. The tester was activated, and the porcelain plate was reciprocated once to and fro. The results are observed as either a reaction (i.e., flash, smoke, and/or audible report) or no reaction. Testing begins at the maximum load of the apparatus (360 N) or lower if experience warrants it. If a reaction occurs in ten trials, the load is reduced until there are no reactions observed in the ten trials. The slurry material had 0/10 no-go reactions at 288N and a go reaction at 320N. The BAM impact was conducted on the twin screw extrusion material and results were 0/10 no-go reactions at 288N and a go reaction at 320N.

Shock Sensitivity (LSGT)

Large Scale Gap Testing (LSGT) was performed in accordance with AOP-7, 201.04.001. The PAX-3 test samples were pressed into free standing pellets and then stacked up into a 1.5” diameter by 5.0” long steel tubing which was supported a 0.375” thick witness plate. A detonator sat on top of booster pellets that were separated from test sample by a series of card gaps as shown in Figure 3. The clear cut hole on the witness plate determines whether the test is a “go” or “no go”. The 50% point between “go” and “no go” for PAX-3 water slurry method was 155 cards (36.8 kbar). The result of the PAX-3 from the twin screw extrusion method was 143 cards (43.4 kbar).

<table>
<thead>
<tr>
<th>Safety Test Results</th>
<th>PAX-3 Slurry</th>
<th>PAX-3 Twin Screw</th>
</tr>
</thead>
<tbody>
<tr>
<td>Test</td>
<td>ERL Impact</td>
<td>BAM Friction</td>
</tr>
<tr>
<td></td>
<td>21.6cm (0/10) no-go</td>
<td>288 (0/10) no-go</td>
</tr>
<tr>
<td></td>
<td>40cm (0/10) no-go</td>
<td>288 (0/10) no-go</td>
</tr>
<tr>
<td>LSMG</td>
<td>320 reaction</td>
<td>320 reaction</td>
</tr>
<tr>
<td></td>
<td>155 cards</td>
<td>143 cards</td>
</tr>
</tbody>
</table>

Figure 3: Results from sensitivity testing on PAX-3
Thermal Characterization

Additional thermal characterization tests were performed to determine the materials response undergoing heating. Specific tests chosen were to look at off-gassing, residual solvents, reaction due to heating, and compatibility. The vacuum thermal stability, thermal stability, small scale burn, and differential scanning calorimetry were performed on the PAX-3 produced by the slurry process.

The vacuum thermal stability test was performed in accordance to AOP-7 202.01.01. A 5g sample of PAX-3 to 80°C for 48hrs. Post test results showed a 0.1% change in mass due to off-gas which is considered a passing score, the twin screw extrusion material had a similar score of 0.1% change in mass. These results are below the 2mL/g of gas evolved passing criteria.

The thermal stability test was conducted in accordance with TB700-2 where a 50g sample of explosive molding powder. The sample was heated to 75°C for a duration of 48 hours. Visual inspection of the PAX-3 explosive showed no signs of reaction or burning due to heating similar to the twin screw extrusion material.

Differential Scanning Calorimetry (DSC) testing was conducted on PAX-3 powder in accordance with STANAG 4515 where a small sample had undergone heating of 5°C per minute. The average onset and peak temperatures were 277.4°C and 279.2°C for slurry PAX-3. The reference results from PAX-3 produced using the twin screw extrusion method were 275.4°C for onset and 277.1°C for reaction.

![DSC results of PAX-3 subject to heating rate of 5°C/min.](image)

Physical Characteristics

Using the twin screw extrusion process the PAX-3 was extruded continuously from a 19mm diameter die, cut and grouped to achieve smaller agglomeration of particles, and then used in molding powder for pressing. In the slurry coating process, the individual particles crash out of solution and range over a size distribution. The bulk density of molding powder produced using the slurry process batches 0.76-0.78g/cc compared to 0.85g/cc from the produced the twin screw extrusion. The composition analysis of the slurry material has the HMX, aluminum, binder, and plasticizer contents within the spec. Compression testing was performed to evaluate the mechanical behavior of the materials. The material was evaluated in accordance to the uniaxial
compression test STANAG 4443. The PAX-3 molding powder was pressed into billets and machined into samples with a 1.5” length and 0.75” diameter. The material was conditioned at 23°C and placed on an Instron 5969 material testing device. The samples were compressed at a rate of 0.015in/min recording the displacement and load. The data was used to calculate stress and strain profiles characterizing the behavior of PAX-3 under compression. The peak compressive strength for slurry PAX-3 was 2848PSI and the twin screw extrusion PAX-3 was 3002PSI. The PAX-3 molding powder was shown to achieve similar densities under similar pressing conditions.

The formulation PAX-3 is currently of interest and being evaluated in grenade and tank ammunition applications. Gun launch munitions undergo significant loading during the acceleration of the projectile. Additional studies were performed to evaluate the response of potential defects under loading due to setback. The PAX-3 produced using the twin screw extrusion process was tested in the ARDEC Setback Test Equipment. The new PAX-3 produced in the slurry process has been evaluated in using the Indian Head setback test. The devices can apply loading rates on explosives samples. Cavities are machined into explosive billets to resemble defects such as gaps, cavities, and cracks. The loading rate and defect sizes are increased in order to obtain a reaction from the explosive.

Summary

While undergoing different processing methods to produce the PAX-3 formulation, initial testing results show slurry produced material maintains similar characteristics to the twin screw extrusion produced material. Standard tests used to look at the thermal behavior, sensitivity, and
physical characteristics were evaluated and showed no notable differences in results changing the processing method between twin screw extrusion and slurry coating. Additional studies are being conducted on aged samples of PAX-3 slurry material. Using this data, ARDEC will qualify the slurry process by the “delta qualification” process with a reduced amount of testing. This will provide additional cost savings reducing additional testing while maintaining the rigor associated with the energetics qualification process. Results thus far show that the PAX-3 molding powder from the slurry process maintains its sensitivity and key characteristics after undergoing the processing method change.
References

2) Twin Screw Extrusion of Thermobaric Explosives. Fair, M. 2006 IMEM
Acknowledgements

RDECOM – ARDEC
BAE Holston
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PEO AMMO
ATK Plymouth