PROCESS OPTIMIZATION TO IMPROVE PRODUCT QUALITY AND CONSISTENCY OF ENERGETIC MATERIALS AT HSAAP

Insensitive Munitions & Energetic Materials Technology Symposium 2013

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Briefing Outline

• Introduction and Program Overview
• Recrystallization of Crude HMX
  • Effect of process parameters on particle size distribution
  • Effect of process parameters on particle shape and crystal morphology
• Results and Path Forward
• Concluding Remarks
• Acknowledgements
Introduction / Program Overview

- BAE Systems manufactured a wide range of energetic ingredients at HSAAP
  - Legacy: RDX/HMX (various classes); Comp B/C-4
  - Next Generation: NTO/TATB/DNAN/IMX family
- Robust manufacturing processes established
- Continuous Improvement effort between R&D, Operations and QA to further improve product quality & consistency via process parameters change
  - DOE / lab scale experiments
  - Extensive characterization
  - Production scale confirmation experiment
- HMX recrystallization targeted first
  - Other products will be studied
HMX Manufacturing Process at HSAAP

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HMX Class 1

HMX Class 3

Nitration

\[
\begin{align*}
\text{Hexamine} & \quad \xrightarrow{\text{nitric acid}} \\
& \quad \xRightarrow{\text{ammonium nitrate, acetic anhydride, acetic acid}} \\
\text{Crude HMX} & \quad \xrightarrow{\text{Acetone/Cyclohexanone}} \\
\end{align*}
\]

Recrystallization

Dewatering

Grinding
HMX Recrystallization with Acetone

- Exclusively used as the solvent to produce HMX Class 1 and HMX 80s
- Project Goals
  - 1. Particle Size Distribution Improvements
    - Determine which processing parameter(s) contributed to changes in the particle size distribution of HMX Class 1 and 80s
    - Once identified, focus on optimizing specific processing parameter(s) in the HMX Recrystallization to further improve consistency in particle size distribution
  - 2. Crystal morphology (Quality) Improvements
    - Adjust processing parameters to produce rounded HMX crystals for commercial applications (HMX mains).
Experiment Set Up

- All experiments were conducted initially in lab scale, with equipment simulating production scale
  - Two 13 Liter Glass Vessel with steam/water jacket; agitator; solvent recovery
  - HMX crude (dry) dissolved in acetone/water slurry in the dissolver (top)
  - Saturated HMX slurry gravity fed into recrystallization vessel (bottom)
  - Recrystallized HMX product filtered and oven dried
  - Recrystallized HMX product characterized by Malvern (PSD) and Digital Microscopy (Shape/Morphology)
Project 1: HMX Class 1/80S PSD Improvement

- A Design of Experiment focusing on the major processing parameters affecting the HMX PSD was conducted:
  - Solids Concentration (amount of HMX crude in the slurry)
  - Solvent to Water Ratio
  - Agitation Rate in Recrystallization Vessel
  - Reaction Time at various stages during dissolution/recrystallization
  - Water presence in Recrystallization Vessel during the charge of HMX crude feed
- A thirty-two run experiment was designed and conducted
- Particle size distribution of HMX crystals from each run was analyzed
### Design of Experiment (HMX Dissolution / Recrystallization)

#### Run Order & Process Parameters

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#### HMX Particle Size Distribution by Malvern Particle Size Analyzer

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Design of Experiment Statistical Results

- The DOE was deemed a success
- After analyzing all the PSD results, statistically significant models were generated which determined that two processing parameters impacted the HMX PSD more than others
- Due to the complexity of the DOE, large amount of noise was detected in the measurement
  - Gave directional trends rather than accurately predicted values
- Additional lab scale experiments were conducted to validate the models further (additional runs focusing in the two parameters)
- In 5 out of 6 experiments, the PSD of the final product met the predicted ranges
- Valuable findings in the effort to improve PSD consistency of HMX crystals
Project 1 Path Forward

- A second DOE is recommended to further optimize the two variables
- Incorporate minor changes in other parameters without affecting PSD results
- Upon satisfactory result, scale up to full size production with optimized processing parameters
- Conduct similar study for RDX Recrystallization with Cyclohexanone
Project 2: HMX Crystal Morphology (Quality) Improvements

- HMX Class 3 recrystallized in Cyclohexanone
- HMX Class 3 used in various commercial products
- Crystal Morphology not ideal (twinning / rough edges)
- Customer requested a different (improved) morphology without changes in particle size characteristics
  - Improvement in product sensitivity and flow properties
  - Higher Bulk Density with rounded crystals
- Evaluate recrystallization with an alternate solvent
  - Larger HMX crystals to mimic the Class 3 PSD profile
Project 2: HMX Crystal Morphology (Quality) Improvements

- Conduct lab scale experiments to adjust processing parameters from Project 1
  - Different solubility levels
  - Distillation rate
- Significant improvement in crystal shape (more rounded, less twinning)

Typical HMX Class 3 Crystals

Lab Scale Improved HMX Crystals
Project 2: HMX Crystal Morphology (Quality) Improvements

- Process improvements evaluated in large scale production
  - 8 batches manufactured
  - Significant improvement in crystal morphology (more rounded)
  - Reduction in Friction Sensitivity (BAM) observed
    - HMX Class 3 (50% ~ 167 N); Rounded HMX Class 3 (50% ~ 204 N)
Project 2: HMX Crystal Morphology (Quality) Improvements

Typical HMX Class 3 Crystal

Rounded HMX Class 3 Crystal
Project 2: Path Forward

- Further optimize the improved production scale HMX Class 3 process
- Gather end-user feedback upon evaluation of the new products
  - Processability of PBXN-110 from CXM-10
    - Lower end-of-mix viscosity due to improved crystal morphology
    - Impact/Friction Sensitivity improvement in PBXN-110
  - Coated HMX products from the Oil and Gas industries (HMX Main)
    - Flow properties improvement (benefit high volume loading in feed hopper)
    - Lower friction sensitivity
Concluding Remarks

- Currently lots of emphasis focused on the next generation insensitive ingredients
- However, BAE Systems are committed to improving the legacy ingredient products
- Good collaboration between R&D and Operations to evaluate product improvement through well-thought DOE
- Significant improvement in crystal quality in HMX Class 3 achieved with recrystallization using an alternate solvent
- Potential improvement in HMX product consistencies after DOE runs
- Further process improvement projects planned for other products (e.g. RDX & NTO)
Acknowledgements

- Process Operators and Engineers for technical advise and support
- Operation Analysts for analytical support
- OSI senior management for financial support
- Customers (Oil and Gas Industries) for end-users’ evaluation feedback