

Improved Particle Size Reduction & Evaluation Technology for Energetics at HSAAP

IMEMTS 2021

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Overview

- The final particle size of traditional crystalline explosives (RDX/HMX) at Holston is controlled through a number of technologies:
 - Solvent Recrystallization
 - Wet-grinding
 - Fluid Energy Milling
- Evaluation of newer technologies could lead to reduced processing time and better control of process
- Technology requirements:
 - Ability to safely process explosives
 - Improvement in processing time/control vs. legacy technology
 - Ability to meet particle size/morphology requirements to serve as drop-in replacement
 - Cost to develop / implement technology

RDX/HMX Particle Size Technology

RDX/HMX Particle Size

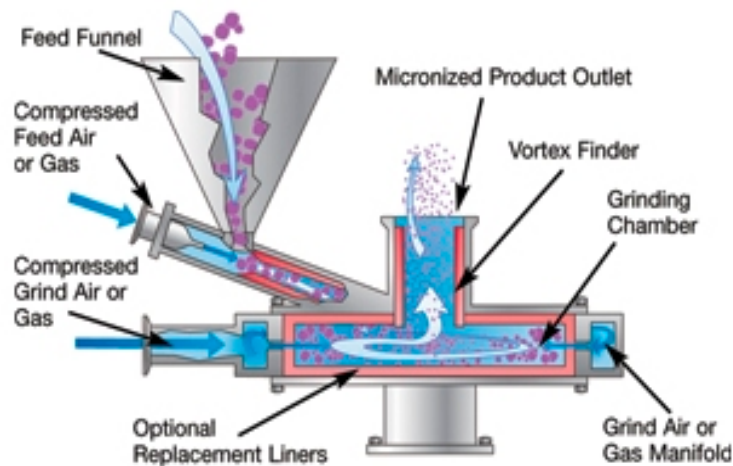


Solvent based Recrystallization

Traditional:

Wet Grinding

Fluid Energy Milling



Fluid Energy Mill

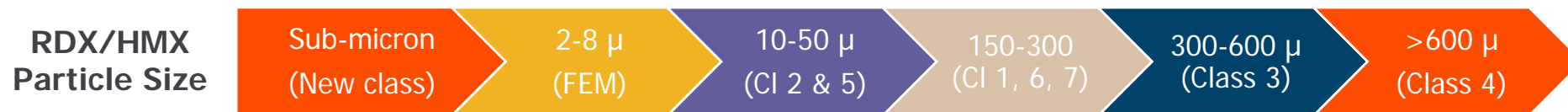


Cowles Grinder

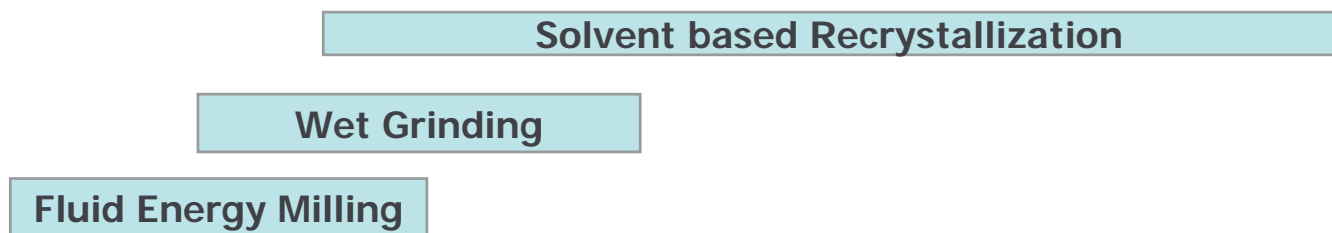


Recrystallization Vessel

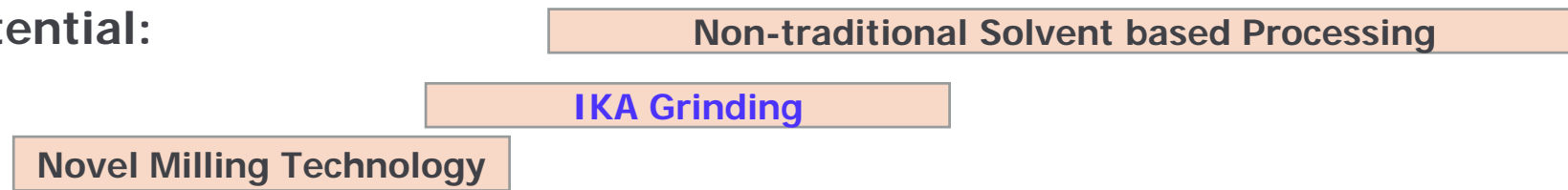
RDX/HMX Particle Size Technology



Traditional:



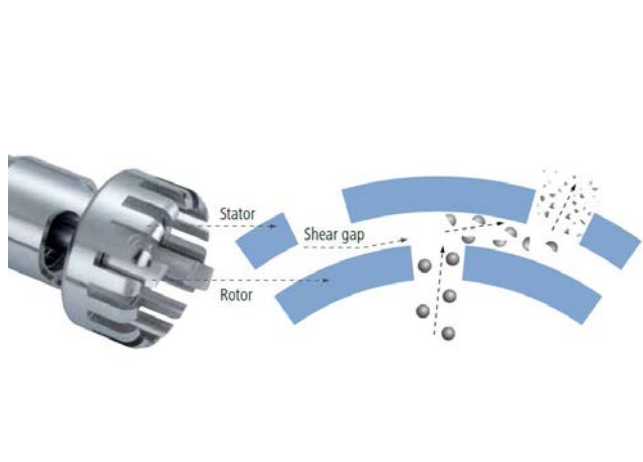
Potential:



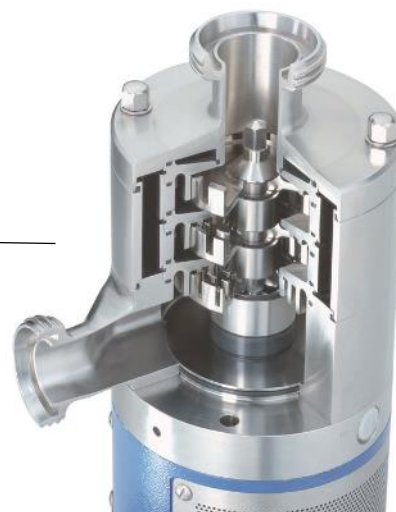
IKA Grinder

IKA Grinder: Evaluation for Replacement of Traditional Wet-Grinding Technology

RDX/HMX IKA Particle Size Reduction Technology



High-Shear Dispersion



3-Stage Rotor-Stator Head

- Slurry-based particle size reduction technology
- Three-stage rotor-stator heads in series
 - Multiple size heads allow targeted PSD reduction
- Ability to vary power setting, recirculate slurry, and multiple units in series allows for further targeted PSD reduction
- Flexible low-cost technology
- Throughput: 500 L/hr (pilot unit)

IKA Grinder: High shear wet-grinding technique for safe particle size reduction of energetic crystals

Case Study: HMX Class 5 (Pump Grinder)

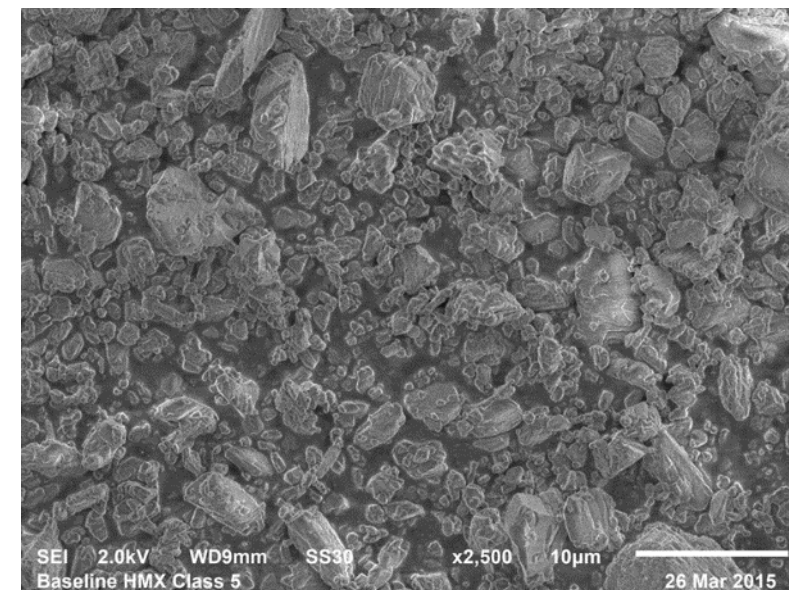
- HMX Class 5 (Pump Grinder) used in number of legacy HSAAP Products
 - Time / Energy **inefficient** process
 - Difficulty in reliably meeting targeted particle size



Pump Grinder

Particle Size Attributes:

Analytical Test	Value
Malvern, 10th Percentile	1.57 μm
Malvern, 50th Percentile	4.11 μm
Malvern, 90th Percentile	16.64 μm
BET Surface Area	2.57 m^2/g
Density (He Gas Pycnometer)	1.91 g/mL



HMX Class 5

Case Study: HMX Class 5 (Pump Ground)

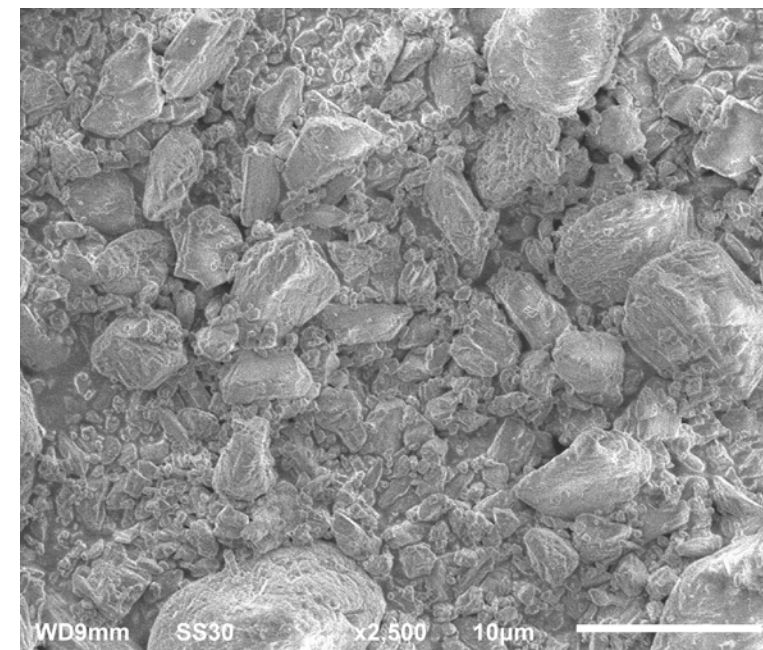
- Evaluation of **IKA Grinder** as replacement technology for pump grinder:
 - Reduction in processing time by >90%
 - Particle size/shape similar to pump ground material
 - Process to be fine-tuned in terms of concentration, power settings, recirculation time

Particle Size Attributes:

<u>Analytical Test</u>	<u>IKA Ground</u>	<u>Pump Ground</u>
Malvern, 10th Percentile	3.60 μm	1.57 μm
Malvern, 50th Percentile	7.10 μm	4.11 μm
Malvern, 90th Percentile	22.60 μm	16.64 μm
BET Surface Area	1.30 m^2/g	2.57 m^2/g
Density (He Gas Pycnometer)	1.91 g/mL	1.91 g/mL



IKA Grinder



HMX Class 5 with IKA Grinder

Case Study: HMX Class 5 (Pump Ground)

Evaluation of alternate particle size reduction technology to replace pump grinder:

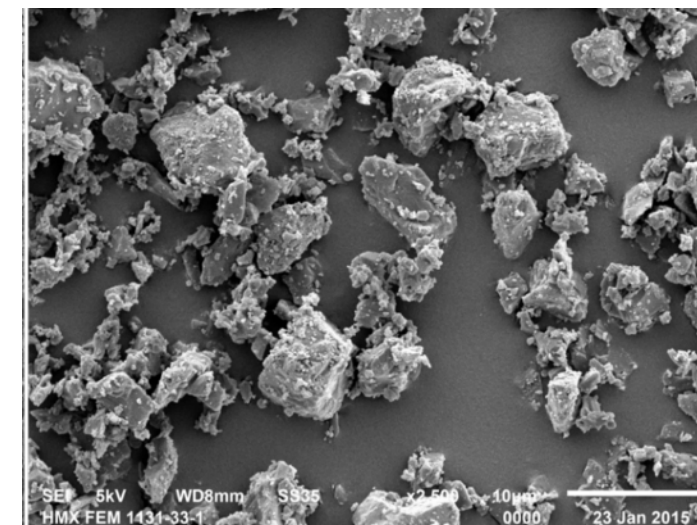
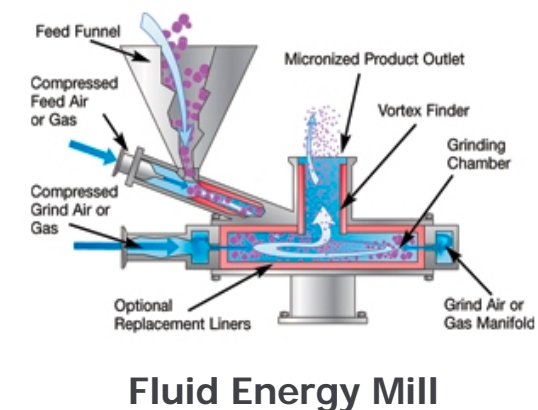
- Fluid energy mill
- Crash precipitation from solvent
- Spray atomization
- Lab RAM
- Ultrasonic Treatment
- Roll Mill
- Cowles Grinder
- Fluidized bed mill
- Planetary ball mill
- Super critical CO₂

Case Study: HMX Class 5 (Pump Ground)

- Evaluation of FEM as replacement technology for pump grinder:
 - Mature particle size reduction technology at HSAAP
 - Particle size/shape similar to pump ground material with material slightly finer
 - Viable processing alternative with some downsides:
 - Dry milling vs. wet milling
 - No improvement in processing time

Particle Size Attributes:

<u>Analytical Test</u>	<u>FEM</u>	<u>Pump Ground</u>
Malvern, 10th Percentile	1.64 μm	1.57 μm
Malvern, 50th Percentile	3.49 μm	4.11 μm
Malvern, 90th Percentile	7.35 μm	16.64 μm
BET Surface Area	3.24 m^2/g	2.57 m^2/g
Density (He Gas Pycnometer)	1.91 g/mL	1.91 g/mL



HMX Class 5 with FEM

Case Study: HMX Class 5 (Pump Ground)

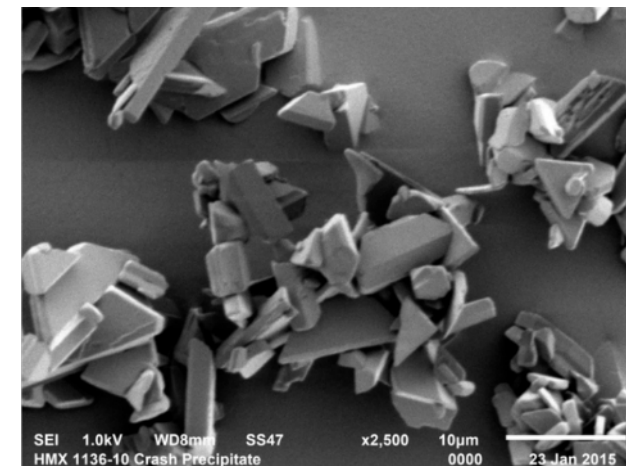
- Evaluation of **Solvent Crash Precipitation** as replacement technology for pump grinder:
 - Evaluation of batch/continuous processing
 - Particle size slightly larger than pump ground material w/ different morphology
 - Viable processing alternative with potential downsides:
 - Potential presence of impact-sensitive HMX polymorphs
 - Ability to control particle size at production scale



Recrystallization Vessel

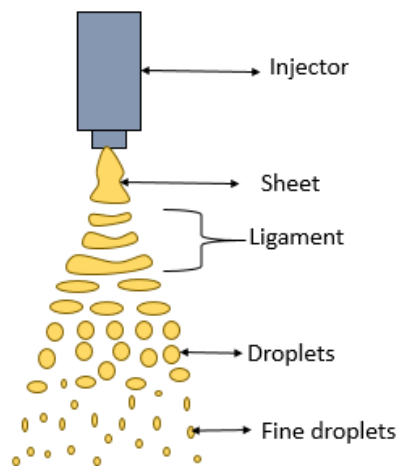
Particle Size Attributes:

<u>Analytical Test</u>	<u>FEM</u>	<u>Pump Ground</u>
Malvern, 10th Percentile	3.47 μm	1.57 μm
Malvern, 50th Percentile	7.08 μm	4.11 μm
Malvern, 90th Percentile	16.02 μm	16.64 μm
BET Surface Area	1.40 m^2/g	2.57 m^2/g
Density (He Gas Pycnometer)	1.82 g/mL	1.91 g/mL



HMX Class 5 through Crash Precipitation

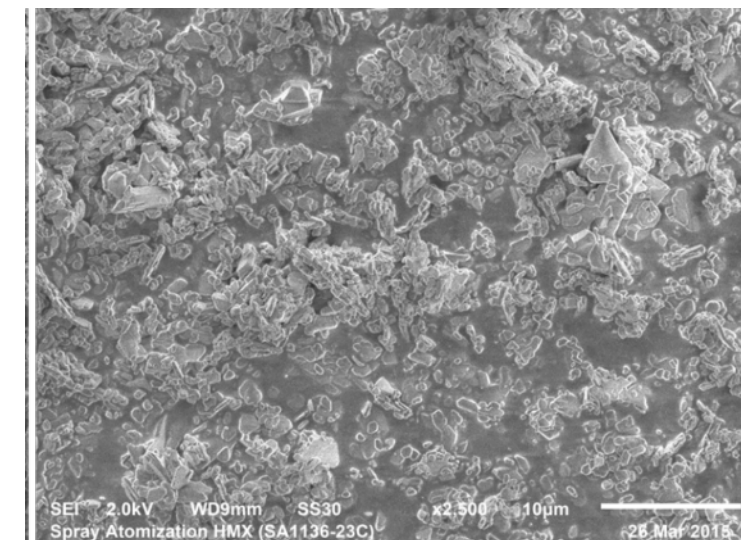
Case Study: HMX Class 5 (Pump Ground)



- Evaluation of **Spray Atomization** as replacement technology for pump grinder:
 - Use of solvent/anti-solvent spray atomization process
 - Particle size/shape similar to pump ground material
 - Viable processing alternative with potential downsides:
 - Unreliable control on particle size at laboratory scale
 - Large amount of solvent recovery required at production scale

Particle Size Attributes:

<u>Analytical Test</u>	<u>FEM</u>	<u>Pump Ground</u>
Malvern, 10th Percentile	1.80 μm	1.57 μm
Malvern, 50th Percentile	4.18 μm	4.11 μm
Malvern, 90th Percentile	11.91 μm	16.64 μm
BET Surface Area	3.82 m^2/g	2.57 m^2/g
Density (He Gas Pycnometer)	1.91 g/mL	1.91 g/mL



HMX Class 5 through Spray Atomization



Spray Atomization

Case Study: HMX Class 5 (Pump Ground)

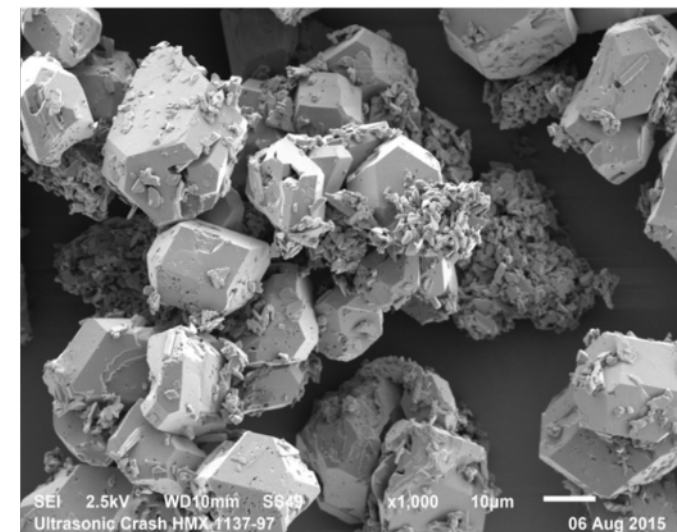
- Evaluation of **Ultrasonic Probe** as replacement technology for pump grinder:
 - Use of ultrasonic probe in combination with solvent/anti-solvent process
 - Particle size/shape larger than pump ground material
 - **Significant engineering required** to overcome processing hurdles:
 - Significant particle size growth if material filtered slowly
 - Fine-tuning of processing parameters and probe required



Ultrasonic Probe

Particle Size Attributes:

<u>Analytical Test</u>	<u>FEM</u>	<u>Pump Ground</u>
Malvern, 10th Percentile	9.59 μm	1.57 μm
Malvern, 50th Percentile	21.26 μm	4.11 μm
Malvern, 90th Percentile	38.05 μm	16.64 μm
BET Surface Area	0.52 m^2/g	2.57 m^2/g
Density (He Gas Pycnometer)	1.88 g/mL	1.91 g/mL



HMX Class 5 through Ultrasonic Probe

Case Study: HMX Class 5 (Pump Ground)

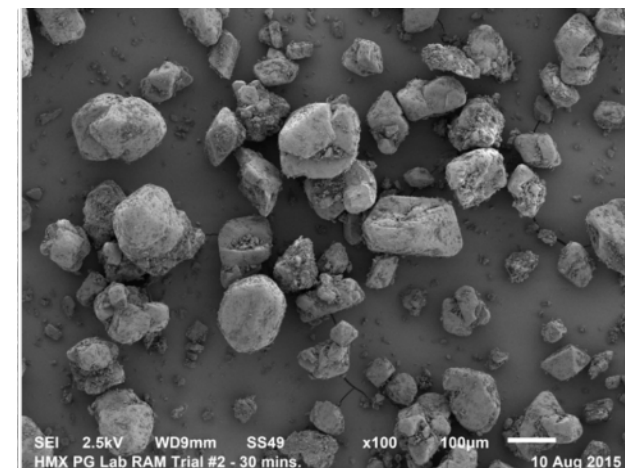
- Evaluation of LabRAM as replacement technology for pump grinder:
 - Use of resonant acoustic energy to cause particle size attrition
 - Not a viable option for Pump grinder replacement
 - Further evaluation of LabRAM with solvent/antisolvent system a possibility

Particle Size Attributes:

<u>Analytical Test</u>	<u>FEM</u>	<u>Pump Ground</u>
Malvern, 10th Percentile	5.67 μm	1.57 μm
Malvern, 50th Percentile	67.32 μm	4.11 μm
Malvern, 90th Percentile	199.01 μm	16.64 μm



LabRAM



HMX Class 5 through LabRAM

Case Study: LLM-105

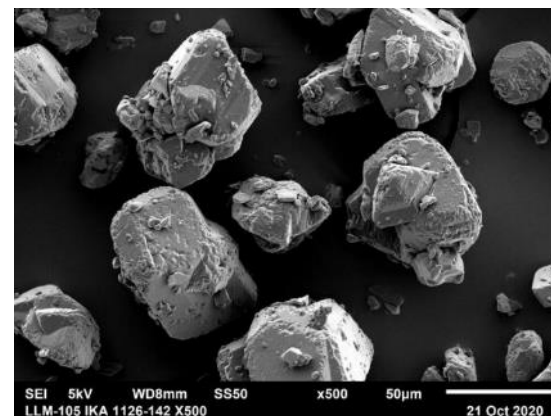
- Goal: Reduction in LLM-105 particle size through wet-milling technology
 - Cowles Grinder
 - IKA Grinder
- Targeting d_{50} of ~60 microns
- While both technologies successful, **IKA grinder was >90% faster** to meet particle size target



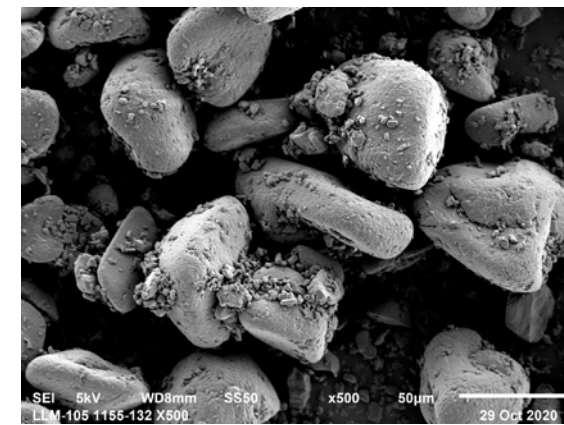
Cowles Grinder

Particle Size Attributes:

<u>Analytical Test</u>	<u>IKA Grinder</u>	<u>Cowles Grinder</u>
Malvern, 10th Percentile	29.01 μm	7.62 μm
Malvern, 50th Percentile	57.16 μm	48.01 μm
Malvern, 90th Percentile	98.74 μm	92.01 μm



LLM-105 (IKA)



LLM-105 (Cowles)

Summary and Path Forward

- Use alternate technologies to replace HMX Class 5 (Pump Ground)
 - IKA Grinder shows good promise as newer wet-milling technology
 - FEM HMX also shows promise and has production scale maturity
- Path Forward (IKA Grinder)
 - Further evaluation of alternate materials / particle size classes
 - Evaluation of IKA-ground material is various formulations
 - Further development of technology from pilot to production scale

Acknowledgements

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■ Thank You



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