FINAL REPORT

Environmentally Conscious Process Development for the Production of Composite Propellants and Explosives

SERDP Project WP-2605

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14. ABSTRACT

A prototype Continuous Acoustic Mixing (CAM) device was redesigned to improve Clean-In-Place (CIP) capabilities based on RAM technology. The CAM-CIP was also designed to allow for temperature control of the mixed material and rated for mixing energetic material. A surrogate energetic formulation using water insoluble materials was developed by NAWCWD China Lake to simulate the viscosity and density of a standard PBX used by the US Navy. The continuous mixing and CIP processes were characterized on the prototype CAM-CIP system using measurements of the produced material's density, solids composition, maximum stress, maximum strain, and hardness. The models were developed to model the mixing and CIP behavior, and the models were used to design and manufacture an energetics rated CAM-CIP with temperature measurement and control capability. The new energetics rated CAM-CIP was characterized and optimized to produce surrogate energetic material at a maximum rate of 3 kg/min while providing good homogeneity and consistency. The CAM-CIP produces a significantly reduced waste stream (both volume and toxicity) during production and cleaning. The CAM-CIP also results in improved operator safety. The next phase of the project is to transition the CAM-CIP equipment and knowledge to NAWCWD China Lake to produce high energy formulations used by the DoD.

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ACRONYMS

CAM	Continuous Acoustic Mixer
CAM-CIP	Continuous Acoustic Mixer configured for clean-in-place
CIP	clean-in-place
CN/OH	cyano-to-hydroxyl ratio
DNSA	dinitrosalicylic acid
DOA	dioctyl adipate
DoD	Department of Defense
g	gravity, acceleration due to
g/cc	grams per cubic centimeter
gm	Grams
gm/min	grams per minute
HGS	hollow glass spheres
HMX	cyclotetramethylene tetranitramine
HSD	honest statistical difference
HTPB	hydroxyl-terminated polybutadiene
Hz	Hertz
IPA	isopropyl alcohol
IPDI	isophorone diisocyanate
kP	Kilopoise
L	Liters
lbs	Pounds
mg	Milligrams
min	Minutes
NAWCWD	Naval Air Warfare Center Weapons Division
PBX	plastic-bonded explosive
PPE	personal protective equipment
psi	pounds per square inch
RAM	ResonantAcoustic® Mixer or ResonantAcoustic® Mixing
RDX	cyclotrimethylene trinitramine
RSD	relative standard deviation
RTD	resistance temperature device
SBIR	Small Business Innovation Research

SERDP	Strategic Environmental Research and Development Program
SGB	solid glass beads
TGA	thermal gravimetric analysis
TPB	triphenyl bismuth
v/v	volume-to-volume
VOC	volatile organic compound

KEY WORDS

Acoustic Mixing
Clean-In-Place
Continuous Acoustic Mixing
Continuous Mixing
High Solids Loading Paste Mixing
High Viscosity Mixing
High Viscosity Paste
Mixing
Paste
PBX Mixing
Resodyn
Resonant Acoustic®
Resonant Acoustic® Mixing

ABSTRACT

The technology and equipment developed addresses the substantial waste stream associated with the annual manufacture of millions of pounds of energetic materials for Department of Defense (DoD) use. [1] The objective of this project is to develop a manufacturing process enabled by a Continuous Acoustic Mixer (CAM) using technology that reduces the waste stream and improves worker safety when processing rocket propellants, explosives and pyrotechnic formulations for DoD munitions.

A prototype CAM device was redesigned to improve Clean-In-Place (CIP) capabilities (referred to as the CAM-CIP) based on RAM technology that has been repeatedly demonstrated to be the superior mixing technology for processing energetic materials.[2-7] The CAM-CIP realizes these same advantages while enabling continuous manufacturing of energetic material using a RAM environment. The CAM-CIP was also designed to allow for temperature control of the mixed material and rated for mixing energetic material.

A surrogate energetic formulation was developed by NAWCWD China Lake to simulate the viscosity and density of a standard PBX used by the US Navy. The surrogate formulation was also developed with water insoluble materials to accurately reflect the PBX material behavior during a CIP process.

The continuous mixing and CIP processes were characterized on the prototype CAM-CIP system using measurements of the produced material's density, solids composition, maximum stress, maximum strain, and hardness. The knowledge gained from the characterization was used to model the mixing and CIP behavior, and the models were used to design and manufacture an energetics rated CAM-CIP with temperature measurement and control capability.

The new energetics rated CAM-CIP was characterized and optimized to produce surrogate energetic material at a maximum rate of 3 kg/min while providing good homogeneity and consistency. The CAM-CIP produces a significantly reduced waste stream (both volume and toxicity) during production and cleaning. The CAM-CIP also results in improved operator safety. The key is the reduced CAM volume (surface area requiring cleaning) relative to batch mixing vessels, and the ability of the RAM environment to enable limited amounts of soap and hot water (not volatile or hazardous solvents) to effectively clean the mixing components.

The next phase of the project is to transition the CAM-CIP equipment and knowledge to NAWCWD China Lake to produce high energy formulations used by the DoD. Ultimately, this effort will quantifiably answer the questions as to the extent to which the implementation of the CAM-CIP system will reduce environmental, safety and occupational health impacts associated with the large scale manufacturing of energetic formulations. It is anticipated that implementation of the CAM-CIP technology will significantly reduce downstream waste produced in current batch production operations.

The CAM-CIP technology will benefit the DoD by providing a functional, cost effective, scalable mixing process and methodology for manufacturing energetic formulations that will reduce energetic material waste, cleaning waste, solvent use, water use, and energy use in manufacturing.

EXECUTIVE SUMMARY

Introduction

The production of composite rocket propellants and plastic bonded explosives (PBXs) for use in DoD weapon systems often requires the use of hazardous materials during the manufacturing and cleanup steps. Depending on the size of the batch processor, this waste stream can result in hundreds or thousands of pounds of explosive hazardous waste produced per batch. To reduce these waste streams and improve personnel safety, new processing technology needs to be developed.

As an alternative to the legacy planetary mixers, ResonantAcoustic® mixing (RAM) technology is making fundamental changes to the way energetic materials are formulated and manufactured. RAM technology has the unique capability to rapidly mix high solids filled, high viscosity energetic materials (such as composite propellants and PBXs) without the need for solvents to thin the mixture. No mechanical elements enter the mix vessel eliminating the need for blades and scrapers used in conventional mixers, thus eliminating clean-up steps and minimizing waste generation.

Batch style RAM mixers have been developed and demonstrated with energetic compositions at both the laboratory scale (LabRAM) and the production scale (RAM 5) at a variety of DoD, DoE and industry facilities including NAWCWD China Lake, McAlester Army Ammunition Plant (MCAAP), and the Naval Sea Warfare Center Indian Head Explosive Ordnance Disposal Technology Division NSWC-IHEODTD.

The available batch capacities of the RAM machines are small when compared with some industrial scale mixers and large DoD munitions systems. The Continuous Acoustic Mixer (CAM) was conceived to address high volume production needs that currently exist in the energetics manufacturing industry nationwide.

The CAM concept consists of a series of stacked cascading plates. The plates are configured at an angle, which allow material to flow from the top of the stack to the bottom. The entire stack for this project is designed to be mounted on a RAM 5 acoustic mixer which provides the RAM environment that drives the mixing. Figure 1 illustrates the basic CAM components.

The key features of the proposed CAM development that contribute to waste reduction and improved operations safety are summarized below:

- The reduced volume of the CAM relative to a conventional batch vessel reduces explosion hazard and cleaning.
- The CAM is able to mix only the volume of material needed for a given production run.

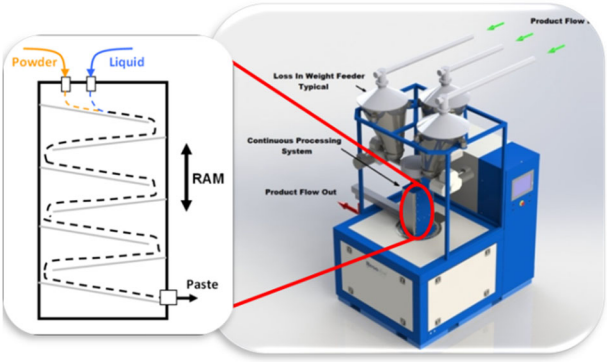

Figure 1. RAM 5 mounted Continuous Acoustic Mixer (CAM) system concept

- The CAM allows the utilization of benign cleaning solutions, reducing or even eliminating VOC issues.
- The CAM system reduces or eliminates a secondary waste stream of contaminated rags, solvent, gloves and masks.
- The CAM provides for the reduction or even elimination of worker exposure to waste materials and hazardous solvents.
- The RAM based CAM technology eliminates moving parts in the mixing vessel that are potential ignition sources.

This project continued the development of the CAM technology so that it can be used for the production of current and future DoD formulations and how the technology can reduce the downstream production waste on a scale applicable to the largest of the DoD tactical weapons platforms.

Objectives

The objective of WP-2605 is to develop a manufacturing process enabled by the new CAM integrated with existing RAM technology that reduces the waste stream and improves worker safety when processing rocket propellants, explosives, and pyrotechnic formulations for DoD munitions. This objective was addressed by a three-phase project, as outlined in the following paragraphs.

In Phase 1, the first goal of the project focused on creating inert composite formulations that represent the behavior of energetic composite formulations during a CIP process. The second goal of Phase 1 of the project was to use a prototype CAM to develop an energetics rated CAM configured for a CIP process that promotes the environmentally benign and efficient production of high viscosity formulations.

After the CAM-CIP is manufactured at the start of Phase 2, the next goal of the project was to optimize the mixing and CIP processes of the continuous mixing module using the surrogate formulation then transition that knowledge with the CAM-CIP mixing module to NAWCWD China Lake.

In Phase 3, the final goal of the project is to continuously produce energetic material using the CAM-CIP followed by a CIP process at NAWCWD China Lake. The mixed, degassed, cast, and cured material will be tested for stress, strain, hardness, and output to demonstrate that the continuously mixed energetic material is equivalent to, or better than, material produced using a legacy batch mixing process. The CAM-CIP will also be evaluated for cleaning efficiency and the amount of waste produced compared to cleaning a legacy batch mixing process.

Ultimately, this effort will quantify the extent to which the implementation of the CAM-CIP system will reduce environmental, safety, and occupational health impacts associated with the manufacturing of energetic formulations. It is anticipated that implementation of the CAM-CIP technology will significantly reduce downstream waste on a production scale commensurate with current production operations.

Technical Approach

The Phase 1 technical work completed at Resodyn focused on the fundamental science of the CAM technology with regards to mixing and cleaning viscous composite pastes. The objective was to use the unique characteristics of RAM for both efficient mixing and more efficient cleaning operations. Additional hardware development work undertaken allowed for temperature monitoring inside the CAM modular stack and temperature control of the continuously mixed material.

Preliminary to CAM and CIP process development work undertaken at Resodyn, inert formulations were developed by the researchers at NAWCWD China Lake to match the material properties of current U.S. Navy qualified plastic-bonded explosives (PBXs). The down-selected inert formulation for this project was prepared to match the density and rheological properties of one of the following explosives: PBXN-110, PBXN-112, or PBXN-109. Of the explosives chosen for evaluation in this project, PBXN-110 has the highest end-of-mix viscosity when prepared by batch RAM and legacy bladed mixers and was thought to be the most difficult to mix with the new CAM technology.

With a working batch RAM mixing process and surrogate formulation specified, work began on developing a continuous mixing module and process with an associated CIP process. Fundamental aspects of the CAM process had already been established via other research projects, in particular the U.S. Air Force Small Business Innovation Research (SBIR) Phase II and Phase II Enhancement contract number FA9300-11-C-3011. This work was extended in the first phase of this Strategic Environmental Research and Development Program (SERDP) project by using the knowledge to design a prototype CAM-CIP.

The prototype CAM-CIP is a series of cascading plates into which materials enter at the top and exit at the bottom (Figure 2). The CAM-CIP stack of plates is mounted on a RAM 5 device so that each plate in the cascade acts as a RAM acoustic transducer that produces the desired mixing.

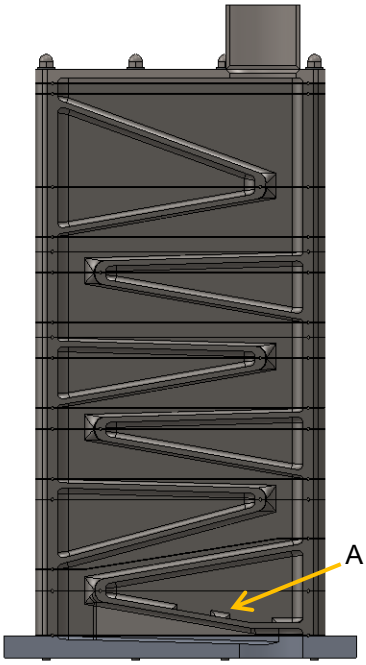

Figure 2. Cross sectional view of the CAM-CIP prototype.

The CAM itself is part of a continuous mixing process shown in Figure 3 comprising three operational stages: material addition, material mixing, and product extraction. The material addition step was accomplished using pumps for liquids and loss-in-weight feeders for powdered solids. Progressive cavity pumps were used to add liquids at constant rates with low amounts of variation in the flow rate.

The mixing process was then investigated in several designed experiments to determine the effect of several factors on two different responses. Tests were also conducted to determine the feasibility of controlling the temperature of the material in the mixing process. At the end of each mixing run, the CIP process was adjusted to achieve 100% cleaning efficiency while creating a minimum of waste.

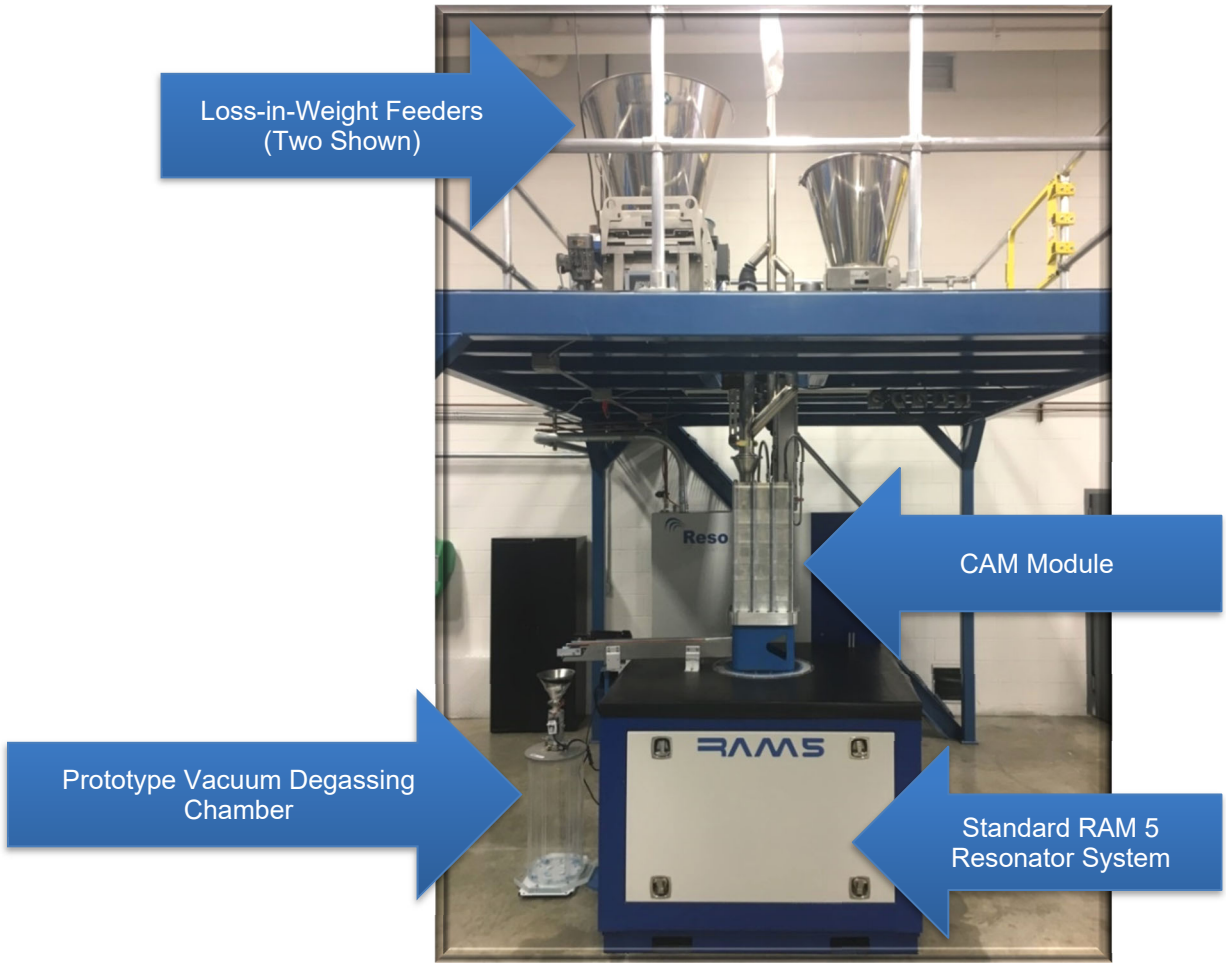

Figure 3. CAM process setup.

The knowledge and experience gained with the prototype CAM-CIP were used to design an energetics capable CAM-CIP with temperature measurement and control capability.

The third generation continuous mixing module was then characterized for consistency and homogeneity using a custom designed experiment methodology using surrogate energetic material. Concurrent with the mixing process development of the CAM-CIP, the CIP process developed on the prototype CAM-CIP was further refined on the energetics capable CAM-CIP.

Results and Discussion

Task 1.1 CAM-CIP Prototype Design for Effective Cleaning

A prototype CAM-CIP was designed and manufactured with rounded features to eliminate 90-degree corners where material would be prone to accumulate termed “dead zones.” A flat gasket type seal using a soft, compliant silicone material was designed to keep material from migrating from the processing regions of a CAM-CIP module and implemented. Testing done at Resodyn on the prototype CAM-CIP and the CAM-CIP has shown zero failures and leaks using the designed seals.

Task 1.2 CAM-CIP Prototype Design for Temperature Control

Two plates of the prototype CAM were modified with thermowells, and a plate with heat transfer channels was manufactured to test the concept of a temperature controlled CAM-CIP. The experiments showed that the concept was feasible, and the design was implemented in the CAM-CIP.

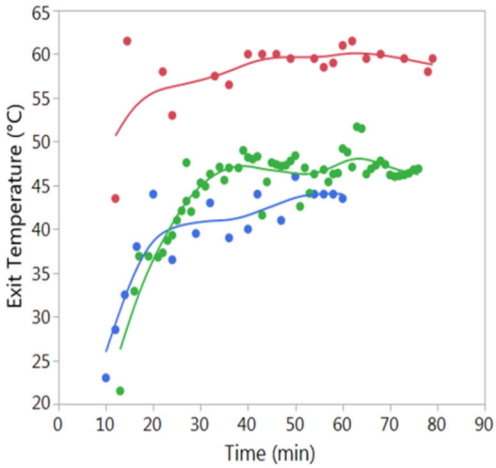

Figure 5. PBXN-110 surrogate material exit temperature for cooling, heating, and no control.

Based on the prototype results, the CAM-CIP was designed with five plates capable of heating or cooling material (shown in Figure 4) being mixed in the CAM-CIP. Several continuous mixing runs have been completed with the CAM-CIP. The temperature of the mixed material exiting the CAM-CIP over time for three different runs is shown in Figure 5. The data in Figure 5 show that it is possible to control the temperature of the material exiting the CAM-CIP. With appropriate equipment, it is possible to independently control the temperature of the material on each plate inside the CAM-CIP.

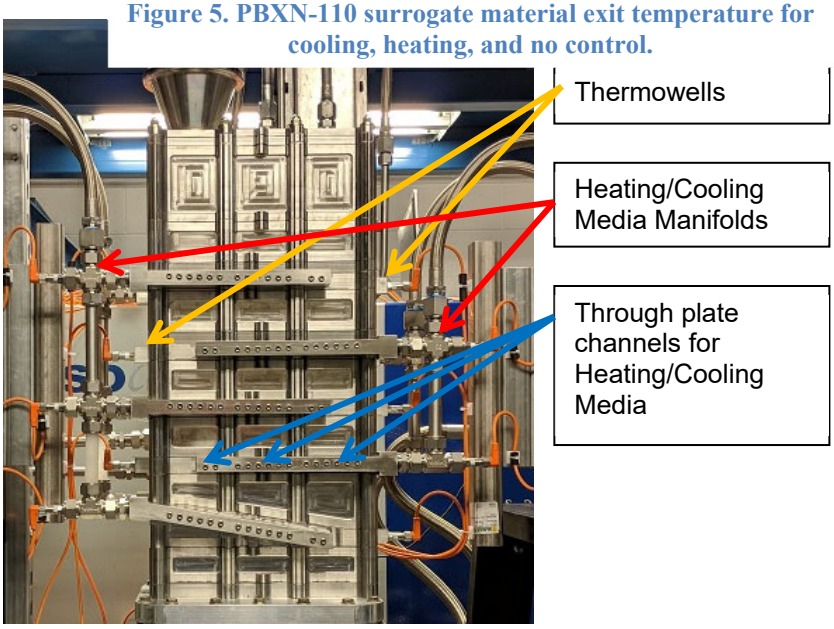

Figure 4. CAM-CIP showing thermowells and heating cooling plates.

Task 1.3 CAM-CIP Prototype Design for Pressure Control

As material is continuously processed in the CAM-CIP, air will be incorporated into the mixed material. The incorporated air must be removed from the mixed material before the material is cast and cured to prevent the cured material from containing voids or air bubbles.

A prototype degassing chamber was designed and is shown in Figure 6. Dog bones cut from cured material degassed using the prototype chamber were X-rayed to visualize the presence of any voids or cavities. An image of a typical dog bone section along with its X-ray is shown in Figure 7 showing no voids.

Task 1.4 CAM-CIP Prototype Testing and Material Evaluation

By measuring the solids loading of material exiting the CAM-CIP, the consistency of the produced material can be measured over time. An example of TGA measurements of mixed material over time is shown in Figure 8. The data shown in Figure 8 represent a solids fraction RSD of 0.5% indicating a process that is in control. Furthermore, a process with an RSD of 0.5% can also be described as being significantly better than a 3 sigma process when the solids percentage requirement is ±1.5%.

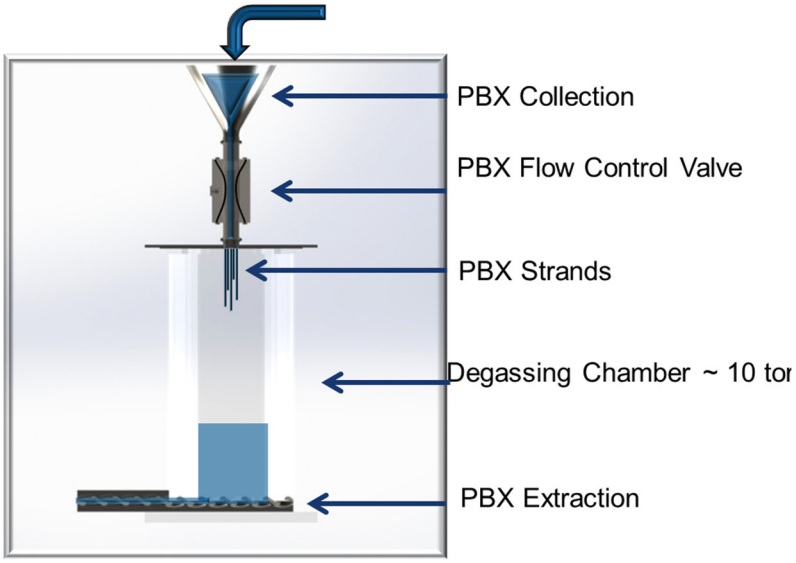

Figure 6. Prototype degassing chamber.

Figure 7. Typical cured PBXN-110 surrogate dog bone (left) and X-ray image of the same (right).

Task 1.5 CAM-CIP Prototype Evaluation for Efficient Cleaning

After NAWCWD China Lake personnel developed an inert surrogate formulation for PBXN-110, batch testing was conducted to identify an aqueous detergent that would clean the surrogate material off a polypropylene surface in a RAM environment. Undiluted Liquinox provided the highest cleaning efficiency (higher even than mineral spirits) and was used as the detergent for the remainder of the project.

Different concentrations of Liquinox in water were then tested in the manner previously described to determine the concentration that provided the highest cleaning efficiency. A concentration of 35 v/v% Liquinox in water was found to provide the highest cleaning efficiency. For experimental efficiency in solution preparation, a Liquinox 33 v/v% in water solution was used for CIP process development.

In order to show that the glass bead based surrogate material accurately represented the behavior of the energetic material with respect to CIP processes, an experiment was conducted at NAWCWD China Lake.

The images in Figure 9 show that the glass bead surrogate material represents the CIP behavior of the energetic material. In Figure 9, the residual energetic material left after the CIP process is shown on the left. The residual surrogate material left after the CIP process is shown on the right.

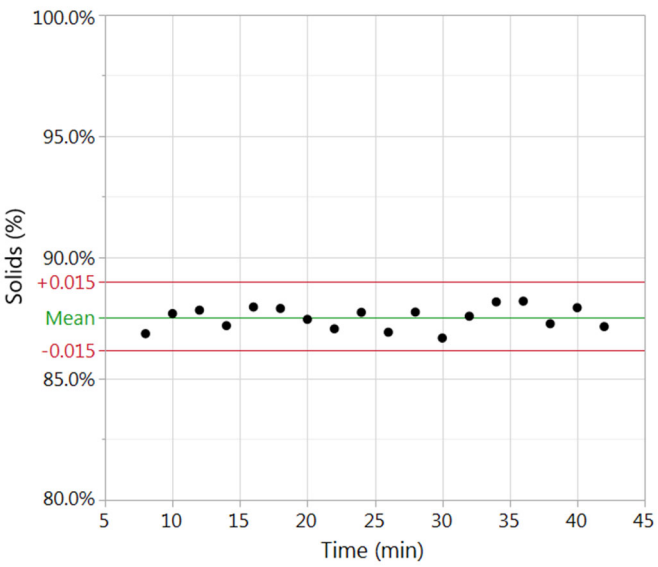

Figure 8 Solids percentage as measured by TGA over time of PBXN-110 surrogate mixed material.

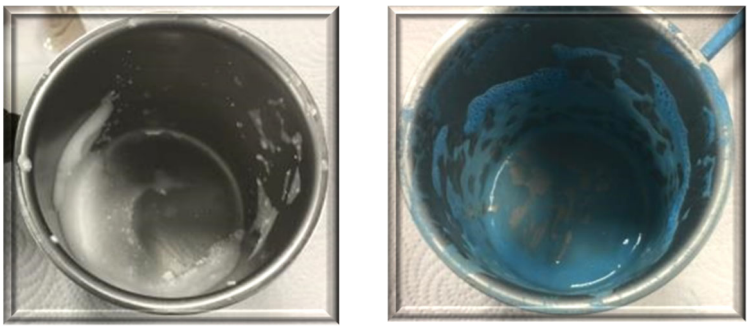

Figure 9. Post CIP images of energetic material (left) and surrogate material (right).

The inert PBX mixing in Task 1.4 using the prototype CAM-CIP set the stage to evaluate the effect of changes in the CIP process after each continuous mixing run. After each mixing run using the prototype CAM-CIP, a CIP was performed under different time, temperature, acceleration, and flow rate conditions.

The final CIP process has been used in over 25 subsequent continuous CAM-CIP mixing runs, and the cleaning efficiency of each run has been 100%. The amount of detergent solution used in the scrubbing cycle is 3.6 liters (L), and the amount of rinse water used is 4.7 L. It is important to note that the amount of detergent and rinse water used is decoupled from the amount of mixed material produced due to the nature of a continuous mixing process. As more material is mixed, the amount of CIP waste created remains constant; therefore, the ratio of CIP waste to mixed material produced decreases at a rate proportional to the inverse of the production rate.

Task 2 Inert Formulation Development

Preliminary to CAM and CIP process development, inert formulations were developed by the researchers at NAWCWD China Lake to match the material properties of current U.S. Navy qualified plastic-bonded explosives (PBXs). At the beginning of the project, surrogate formulations were prepared to match the density and rheological properties of the following explosives: PBXN-110, PBXN-112, or PBXN-109.

Finally, a combination of hollow glass spheres (HGS) and solid glass beads (SGB) gave an inert material that best matched the density and end-of-mix viscosity of PBXN-110. The final inert formulation is given in Table 1.

Table 1. Glass bead inert surrogate formulation for PBXN-110.

Material	Weight %
R45 HTPB	12 – 14
DiOctyl Adipate (DOA)	
Lecithin	
DiNitroSalicylic Acid (DNSA)	
TriPhenyl Bismuth (TPB)	
IsoPhorone DiIsocyanate (IPDI)	
Ethanox 702	
Cyan Blue	
HGS	86 – 88
SGB	
Cyano-to-Hydroxyl Ratio (CN/OH)	1.0
End-of-Mix Viscosity (80-140°F)	10 – 30 kP

The PBXN-110 inert formulation was prepared with conventional batch mixers, as well as batch style RAM mixers and the material properties validated against the Military Specifications for the analogous qualified PBXs. Once it had been determined that the material processed using the RAM technology met the material property specifications for standard PBXN-110, the formulations were given to the researchers at Resodyn Corporation for evaluation in the CAM configuration.

Task 3 Optimize CAM-CIP Operation

The CAM-CIP module rated for live energetic mixing and configured for temperature control was received, assembled, and ready for testing at Resodyn on 16 February 2019. The CAM-CIP module was characterized according to a designed experiment methodology. The performance of the CAM-CIP was characterized with respect to three main factors. The factors were total mass flow rate, RAM acceleration, and percent solids. The responses that were modeled were material homogeneity, material compositional consistency, and material flow rate.

The modeling shows that at the ideal operating conditions investigated in the designed experiment of 1,250 gm/min, 80 g of acceleration, and 86% solids, the relative standard deviation of the solids percentage in the collected sample was 0.1% RSD. The worst recorded RSD occurred at 93% solids and was 3% RSD.

The characterization of the CAM-CIP shows that at the optimized operating conditions the CAM-CIP produces material with a high level of homogeneity.

Next, the material consistency of the mixed paste exiting the CAM-CIP was sampled and measured. A chart of the solids fraction from a typical run is shown in Figure 10. The green line represents the average amount of solids for the run. The red lines represent 3 standard deviations from the mean. The RSD of the solids percentage of the mixed material for the run in Figure 10 was 0.8% indicating that the CAM-CIP module is reducing the variation of the solids percentage in the mixed material imparted by the loss-in-weight feeders.

The CAM-CIP was designed with cross-flow channels in five plates to allow hot or cold water to be passed in a cross-flow manner through the plates indirectly heating or cooling the mixing material akin to a shell and tube heat exchanger. The CAM-CIP was also designed to accommodate RTDs to measure the inlet and outlet temperature of the fluid used for heating a cooling the material as well as the temperature of the mixed paste as it enters and exits the sections of the CAM-CIP that contain the plates with cross-flow channels.

A series of experiments were conducted to measure the temperature of the material as it progressed through the CAM-CIP as well as the temperature of the water as it entered and exited each cross-flow plate in the CAM-CIP. The temperature traces from a typical run using 75 °C water in the cross-flow channels are shown in Figure 11 and Figure 12.

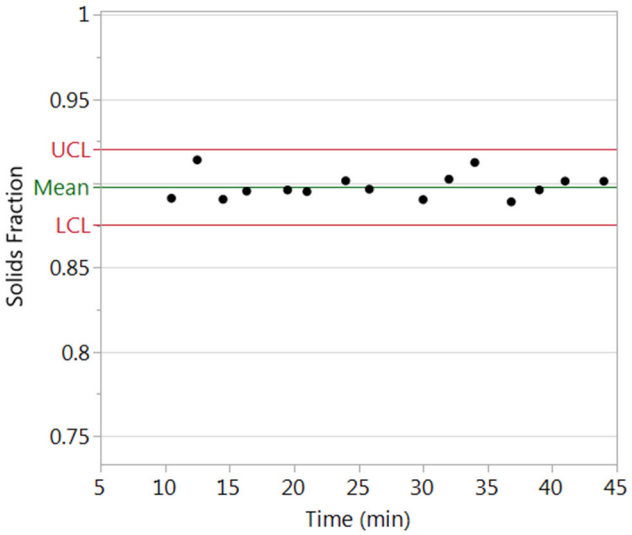

Figure 10. TGA measurements over time for a typical CAM-CIP run

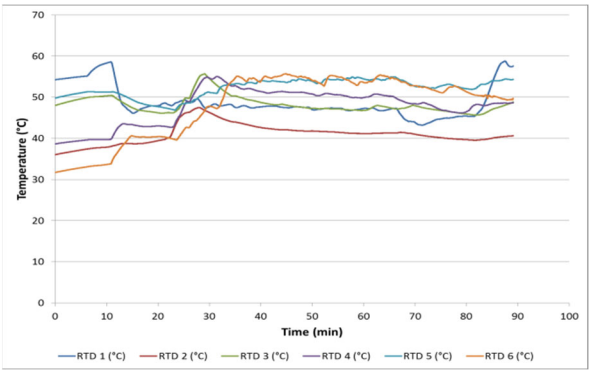

Figure 11. Temperature traces showing the RTDs measurements of the paste temperature

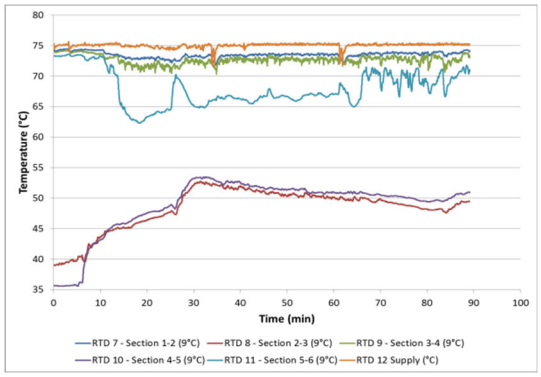

Figure 12. Temperature traces of the heating wat outlet temperatures for each section, RTD 12 is th inlet temperature for each section

The average amount of power transferred from the hot water varies from section to section due to the different paste temperatures, different levels of mixedness, and different viscosities of the pastes in each section. The average power transferred from the water ranged from 100 W at the top of the CAM-CIP to 800 W at the bottom of the CAM-CIP.

A series of 4 continuous runs were conducted with IPDI added to the DOA liquid reservoir. The mixed paste produced during these runs was degassed, cast, and cured so that the physical properties of the surrogate PBX material could be measured and analyzed for consistency and homogeneity.

The cured material was sectioned into disks. Four dog bones from each disk were cut and labeled A through D where dog bones A and D were on the outside of the disk and B and C were in the inside of the disk. The maximum stress and maximum strain of each dog bone were tested according to the JANNAF dog bone standards and STANAG 4506 method. Examples of the average maximum stress and average maximum strain over the dog bones A, B, C, and D for each disk are shown in Figure 13 and 14 respectively. The physical data shows that the physical properties of the material produced by the CAM were not consistent.

Examples of the average maximum stress and strain by dog bone A, B, C and D averages over all disks for a continuous mixing run are shown in Figure 15. A Tukey HSD test was conducted on the data shown in Figure 15 to compare the average maximum stress and strain measurements. The analysis showed that the material was homogeneously mixed, just not consistently mixed. In other words, the material in each disk was the same. The material from disk to disk varied however.

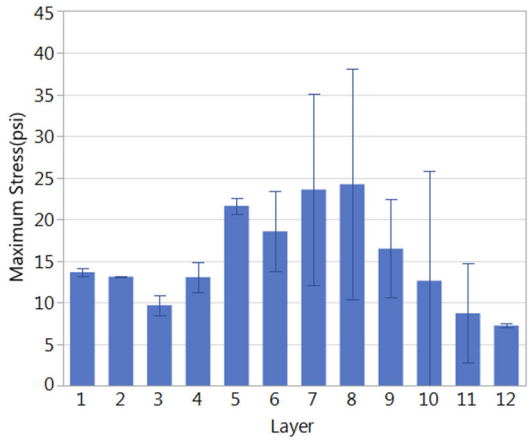

Figure 13. Maximum stress versus layer (error bars represent one standard deviation)

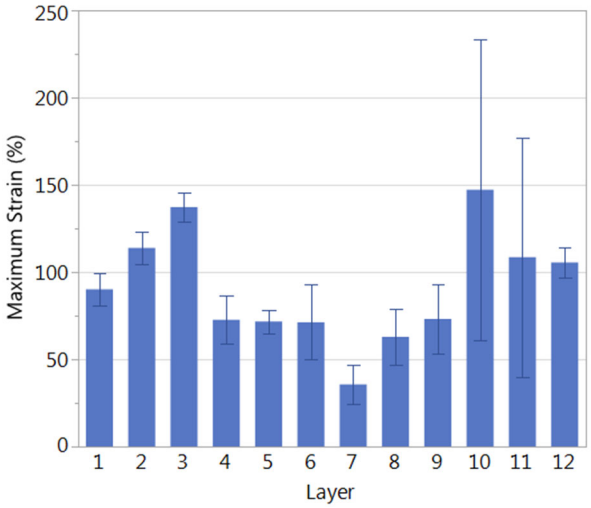

Figure 14. Maximum strain versus layer (error bars represent one standard deviation)

After discussions with NAWCWD China Lake, the probable cause of the inconsistency in the maximum stress and maximum strain of the continuously mixed and cured material is that the IPDI is reacting with itself or water in the air over the course of a mixing run (3 hours minimum) and changing the effective CN/OH ratio of the cured surrogate PBX material over time.

Before transitioning the CAM-CIP to China Lake, the method of adding IPDI to the mixture will be modified to correct the issue. IPDI will be separately metered and kept under an inert gas blanket during the mixing run to prevent any competing reaction or change of the CN/OH ratio over time.

Task 4 Optimize CAM-CIP Cleaning Procedure

During the 14 runs conducted with the CAM-CIP, the cleaning efficiency and the robustness of the CIP process was confirmed. The CAM-CIP was 100% clean after every run as long as the CAM-CIP was kept air tight during the entire CIP process. If the CAM-CIP was not airtight, the cleaning efficiency would be 99% instead of 100% due to residual material left on the lid similar to early efforts in Task 1.5. To ensure the CAM-CIP was airtight for each run, the gasket seals between each plate were redesigned as part of this task.

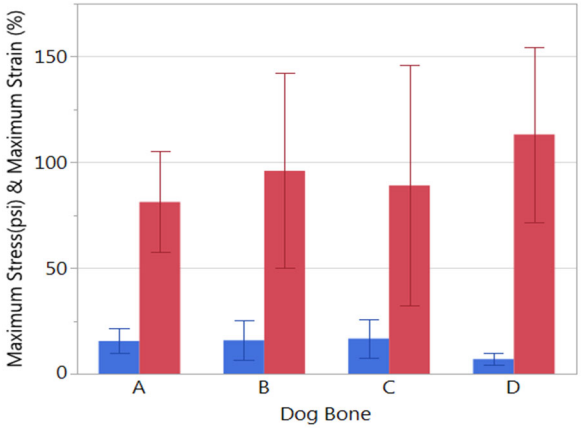

Figure 15. Maximum stress and maximum strain versus dog bone (error bars represent one standard deviation)

Task 5 Establish Design Requirements for Interface Hardware for Downstream Processing

After material exits the CAM-CIP, it needs to be moved to vacuum degassing equipment. The

requirements for hardware to transition from the CAM-CIP to the vacuum degassing equipment are Class I Div I compliant, low profile or able to fit between the CAM-CIP and RAM 5 resonator plate, and sealed to prevent material leaks or CIP splashing. Extra funds have been granted to China Lake to design, fabricate, and test a solution involving a Class I Div I progressive cavity (PC) pump.

Implications for Future Research and Benefits

The RAM continuous mixing process has been successfully developed to mix surrogate PBXN-110 material. Phase 3 of the project involves transferring the CAM-CIP to NAWCWD China Lake to demonstrate the continuous mixing process and CIP using energetic material. The CAM-CIP is ready to transfer at this time, however, transitioning the CAM-CIP equipment and process to NAWCWD China Lake facility to mix energetic material will require some development of the current ancillary operations.

First, an improved method of conveying material from the exit of the CAM-CIP to the vacuum degassing operation is required. The method will need to be rated for energetic service and able to handle highly loaded and viscous paste.

Second, a method of diverting off specification material that can be operated remotely is needed. During the startup of the continuous mixing process, a small amount of DOA rich material is produced. The DOA rich material needs to be segregated from the in specification material produced later. The diversion method needs to be energetic material rated, and remotely operated.

Third, a continuous vacuum degassing unit that can handle the nominal maximum flow rate of the CAM-CIP is needed. The prototype unit developed as part of Tasks 1.4 and 3 has a processing rate of approximately 380 gm/min. The development of the prototype degassing unit provided the operation and process requirements for a continuous degassing system. The degassing unit needs to be energetics material rated.

When NAWCWD China Lake is ready to receive the CAM-CIP at the end of December 2019, Task 7 can begin in earnest. The other preparatory tasks of Task 7 have recently been completed including training of NAWCWD China Lake personnel.

OBJECTIVE

The proposed technology development project addresses the substantial waste stream and improves worker safety associated with the annual manufacture of millions of pounds of energetic materials for Department of Defense (DoD) use. The objective of WP-2605 is to develop a manufacturing process enabled by the new Continuous Acoustic Mixer (CAM) integrated with existing Resonant Acoustic® Mixing (RAM) technology that reduces the waste stream and improves worker safety when processing rocket propellants, explosives, and pyrotechnic formulations for DoD munitions. This objective was addressed by a three-phase project, as outlined in the following paragraphs.

In Phase 1, the first goal of the project focused on creating inert composite formulations that represent the behavior of energetic composite formulations during a clean-in-place (CIP) process. The second goal of Phase 1 of the project was to develop an energetics rated CAM configured for a CIP process that promotes the environmentally benign and efficient production of high viscosity formulations.

Phase 2 began with designing and manufacturing an energetics rated CAM-CIP. After the CAM-CIP was manufactured, the next goal of the project was to optimize the mixing and CIP processes of the continuous mixing module using the surrogate formulation then transition that knowledge with the CAM-CIP mixing module to NAWCWD China Lake.

In Phase 3, the final goal of the project is to continuously produce energetic material using the CAM-CIP followed by a CIP process at NAWCWD China Lake. The mixed, degassed, cast, and cured material will be tested for stress, strain, hardness, and output to demonstrate that the continuously mixed energetic material is equivalent to, or better than, material produced using a legacy batch mixing process. The CAM-CIP will also be evaluated for cleaning efficiency and the amount of waste produced compared to cleaning a legacy batch mixing process.

Ultimately, this effort will quantify the extent to which the implementation of the CAM-CIP system will reduce environmental, safety, and occupational health impacts associated with the manufacturing of energetic formulations. It is anticipated that implementation of the CAM-CIP technology will significantly reduce downstream waste on a production scale commensurate with current production operations.

BACKGROUND

The production of composite rocket propellants and plastic bonded explosives (PBXs) for use in DoD weapon systems often requires the use of hazardous materials, during the manufacturing and cleanup steps. The current manufacturing process requires workers to handle material, or to be in close contact with the formulations at different stages of the mixing process throughout the final formulation loading operation and batch mixer cleanup operations. Many solvents used in both the mixing and cleaning operations exhibit a high vapor pressure, which increases the inhalation exposure of the workers in those operations.[1] In addition, the legacy

batch style processing technology results in a loss of the material that remains adhered to the bowl and mixing blades after the processing and loading operations are complete. This "hold on" material represents a significant portion of the waste stream associated with energetic materials production and has been measured to be between 5-20% of the total batch.[2] Depending on the size of the batch processor, this waste stream can result in hundreds or thousands of pounds of explosive hazardous waste produced per batch. To reduce these waste streams and improve personnel safety, new processing technology needs to be developed.

As an alternative to the legacy planetary mixers, ResonantAcoustic® mixing (RAM) technology is making fundamental changes to the way energetic materials are formulated and manufactured.[3-8] RAM technology has the unique capability of rapidly mixing high solids filled, high viscosity energetic materials (such as composite propellants and PBXs) without the need for solvents to thin the mixture. No mechanical elements enter the mix vessel eliminating the need for blades and scrappers used in conventional mixers, thus eliminating clean-up steps and thereby minimizing waste generation. Mixing can occur in the mold or even in the end use container, thus eliminating casting or machining steps that generate waste material and personnel exposure.[9]

The mixing principal for RAM is illustrated in Figure 16, which shows a vessel being subjected to a low-frequency acoustic field in the vertical direction. The mixing vessel acoustic field is generated by the moving boundary condition of the vessel moving up and down at nominally 60 Hz and up to 100 g of acceleration.

The acoustic field is generated throughout the mixing vessel by pressure waves from the bottom of the mixing vessel into the media. Because the acoustic field forms throughout the mixing vessel, the shear forces that facilitate mixing are also distributed throughout. It is important to note that low-frequency RAM mixing is not "ultrasonic" mixing, and no cavitation is generated by the RAM mixing process.

Batch style RAM mixers have been developed and demonstrated with energetic compositions at both the laboratory scale (LabRAM) and the production scale (RAM 5) at a variety of DoD, DoE and industry facilities including NAWCWD China Lake, McAlester Army Ammunition Plant (MCAAP), and the Naval Sea Warfare Center Indian Head Explosive Ordnance Disposal Technology Division NSWC-IHEODTD. Because the RAM technology mixes efficiently without high shear paddles, it reduces the operator's exposure to the material; there is no need to scrape down the paddles or the bowl in order to ensure complete incorporation of the ingredients in the composite. Furthermore, the elimination of the mixing blades has opened up the ability to mix explosives *in situ*, or within the case of the desired munition item. This eliminates the casting operation and the "hold on" waste associated with cleanup of the mixing bowl.

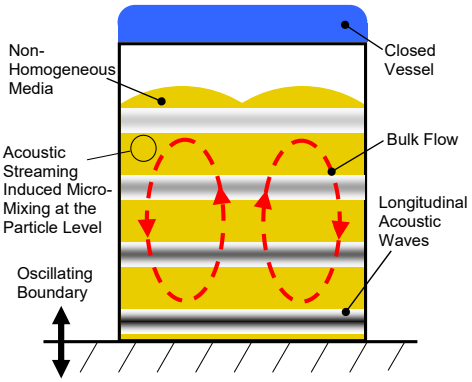

Figure 16. The ResonantAcoustic Mixer uses acoustic energy to provide both bulk and micro-mixing

RAM batch processing equipment is currently produced in three sizes, the LabRAM (2 pound capacity batch mixer) for bench top experimental work, the RAM 5 (80 pound capacity batch mixer) and the RAM 55 (960 pound capacity batch mixer). The available batch capacities of the RAM machines are small when

compared with some industrial scale mixers and large DoD munitions systems. For example, the McAlester Army Ammunition Plant routinely processes PBXs in an industry standard 600 gallon Baker Perkins dual blade mixer. This is used to produce 7,000 to 13,000 pounds of high explosives in a single batch. At these scales, the advantages of the batch style RAM technology may be easily trumped by the required scale of production. The CAM was conceived to address high volume production needs that currently exist in the energetics manufacturing industry nationwide.

An important feature of the CAM technology is that it uses the same platform as the batch mixer vessel. Hence, the end user can use the RAM 5, or RAM 55 technology, interchangeably, for either batch, or continuous processing. The CAM consists of a series of stacked cascading plates. The plates are configured at an angle, which allow material to flow from the top of the stack to the bottom. The entire stack is designed to be mounted on a RAM 5 acoustic mixer which provides the RAM environment that drives the mixing. Figure 17 illustrates the basic CAM components.

The prototype CAM has been demonstrated with generic dry powder blends and composite pastes. The results of this early development testing suggest that once optimized, the CAM technology can produce composite propellant material at rates comparable to industry standard 600 gallon mixers. Based on experiments utilizing a typical inert PBX like formulation, a RAM 5 based CAM can theoretically produce at a rate of 560 lbs/hr while a RAM 55 based CAM could produce material at a rate of 6,500 lbs/hr.

The key features of the proposed CAM development that contribute to waste reduction and improved operations safety are summarized below:

The reduced volume of the CAM relative to a conventional batch vessel reduces explosion hazard and cleaning burden. The CAM cascading plate arrangement is designed to hold only 20-60 lbs of in-process material at any time. This small volume of material actively being mixed is made possible by the reduced time required for mixing in a RAM system. Regardless of how many 1,000s of pounds of energetic material are being produced, the amount of in-process material does not increase. This will significantly improve the safety of the operation and reduce the amount of energetic material that is lost to cleaning of the mixer and must be disposed of as hazardous waste.

The CAM is able to mix only the volume of material needed for a given production run. Currently, the conventional planetary mixers in use have specific capacity ranges. Capital investment decisions dictate which capacity ranges are available, as opposed to operational need. For instance, a gap may exist between the capacities of a conventional 5 gallon mixer and a 30 gallon mixer. If an operational need arose that required an amount between those two ranges, either more than one mix would need

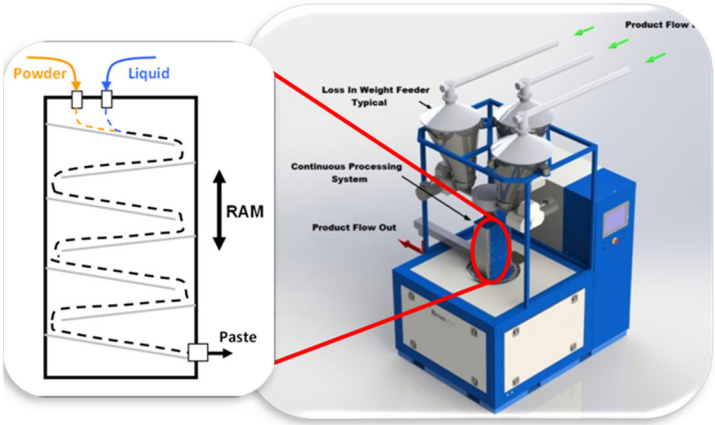

Figure 17. RAM 5 mounted Continuous Acoustic Mixer (CAM) system concept.

to be performed, or significantly more material would be produced than necessary. CAM technology obviates this waste of labor and material by providing a single-machine solution with continuous and functionally unlimited capacity.

The CAM allows the utilization of benign cleaning solutions, reducing or even eliminating VOC issues. Removal of the "hold-on" energetic material from the CAM will use far less cleaning material per pound of energetic material produced. It has been demonstrated that simple hot water and detergent are sufficient to remove an inert hydroxyl terminated polybutadiene (HTPB) based composite formulation from a sealed container in the RAM environment.

The CAM system reduces or eliminates a secondary waste stream. The enclosed system and internal cleaning action of the CAM reduces or eliminates the need for the operator to take apart the mixer and clean by hand. Thus a secondary waste stream of contaminated rags, solvent, gloves and masks is avoided or limited.

The CAM provides for the reduction or even elimination of worker exposure to waste materials and hazardous solvents. Cleaning the CAM is envisioned as a fully contained procedure that does not require operator contact. This allows for CIP capabilities which afford a significant improvement in operator safety. Experimentation with early prototypes of the CAM (see the photo in Figure 18) clearly shows the vigorous scrubbing action of water and air passing through the prototype CAM. The plan is to utilize hot water and detergent in this manner as the primary cleaning process for the proposed CAM-CIP system.

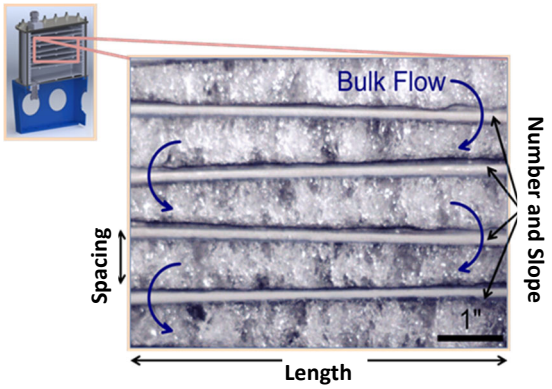

Figure 18. Prototype CAM processing water and air, significant geometric characteristics are notated

The RAM based CAM technology eliminates moving parts in the mixing vessel that are potential ignition sources. The CAM approach for continuous mixing, like the RAM batch mixing, offers inherent processing safety. RAM is a low shear approach to mixing that does not use impellers, scrapers or other internal moving parts. This removes the ignition hazard associated with concentrated high shear zones. The low frequency of RAM also eliminates the possibility of cavitation.

This project proposes to continue the development of the CAM technology so that it can be used for the production of current and future DoD formulations and how the technology can reduce the downstream production waste on a scale applicable to the largest of the DoD tactical weapons platforms.

MATERIALS AND METHODS

The Phase 1 technical work focused on the fundamental science of the CAM technology with regards to mixing and cleaning viscous composite pastes. The objective was to use the unique characteristics of RAM for both efficient mixing and more efficient cleaning operations.

Additional hardware development work undertaken allowed for temperature monitoring inside the CAM modular stack and temperature control of the continuously mixed material.

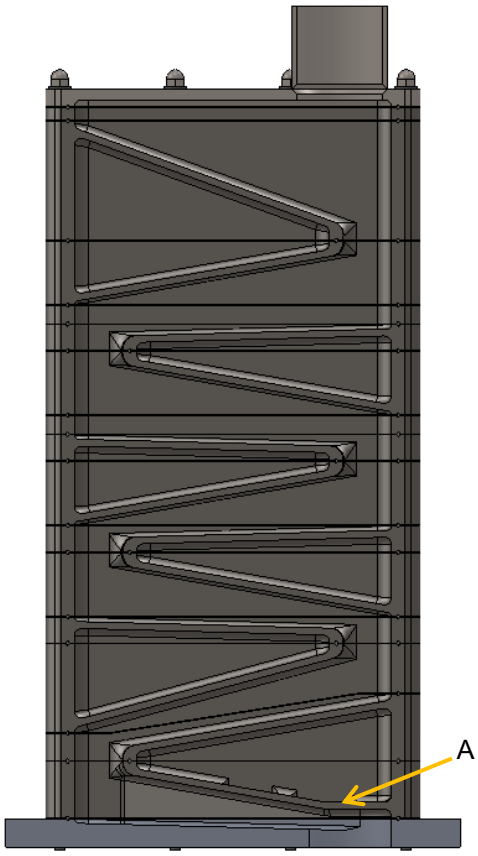

Figure 19. Cross sectional view of the CAM-CIP prototype.

Preliminary to CAM and CIP process development, inert formulations were developed by the researchers at NAWCWD China Lake to match the material properties of current U.S. Navy qualified plastic-bonded explosives (PBXs). The down-selected inert formulation for this project was prepared to match the density and rheological properties of one of the following explosives: PBXN-110, PBXN-112, or PBXN-109. Of the explosives chosen for evaluation in this project, PBXN-110 has the highest end-of-mix viscosity when prepared by batch RAM and legacy bladed mixers and was thought to be the most difficult to mix with the new CAM technology.

Several different detergents were tested using the PBXN-110 surrogate formulation to determine the most effective detergent for this project by mixing the inert formulation in a batch vessel. The mixed material was poured out of the vessel and a detergent solution was added and "scrubbed" under RAM motion. The cleaning solution was poured out of the vessel, and the mass of material remaining was measured to determine the cleaning efficiency according to Equation 1. Liquinox provided the highest cleaning efficiency and was used as the detergent for the remainder of the project.

Different concentrations of Liquinox in water were then tested in the manner previously described to determine the concentration that provided the highest cleaning efficiency. A concentration of 33% Liquinox in water was found to provide the highest cleaning efficiency and was used for CIP process development in subsequent tasks.

$$\left\{1 - \frac{(Final\ Mass - Empty\ Vessel\ Mass)}{Empty\ Vessel\ Mass}\right\} * 100 = Cleaning\ Efficiency$$

Equation 1. Cleaning efficiency

With a working batch RAM mixing process and cleaning detergent specified, work began on developing a continuous mixing process and associated CIP process. Fundamental aspects of the CAM process had already been established via other research projects, in particular the U.S. Air Force Small Business Innovation Research (SBIR) Phase II and Phase II Enhancement contract number FA9300-11-C-3011. This work was extended in the first phase of this Strategic Environmental Research and Development Program (SERDP) project by using the knowledge to design a prototype CAM-CIP.

The prototype CAM-CIP is a series of cascading plates into which materials enter at the top and exit at the bottom (Figure 19). The CAM-CIP stack of plates is mounted on a RAM device so that each plate in the cascade acts as a RAM acoustic transducer that creates a nominal 60 Hz sinusoidal motion with peak-to-peak displacements up to 0.55 inch when operating at 100 *g* of acceleration. Material interaction with these acoustic transducers produces the desired mixing. In the CAM configuration, the materials being mixed pass over a number of these active transducers as the materials flow through the system.

The prototype CAM-CIP was designed with rounded features as shown in Figure 20 to eliminate dead spots that can produce poorly mixed material and are hard to clean. The prototype CAM-CIP was also designed with sanitary type bulb seals between each plate to prevent metal-to-metal contact and to prevent material from migrating outside the wetted process area.

The CAM itself is part of a continuous mixing process shown in Figure 21 comprising three operational stages:

1. Material addition
2. Material mixing
3. Product extraction

The material addition step was accomplished using pumps for liquids and loss-in-weight feeders for powdered solids. Progressive cavity pumps were used to add liquids at constant rates with low amounts of variation in the flow rate.

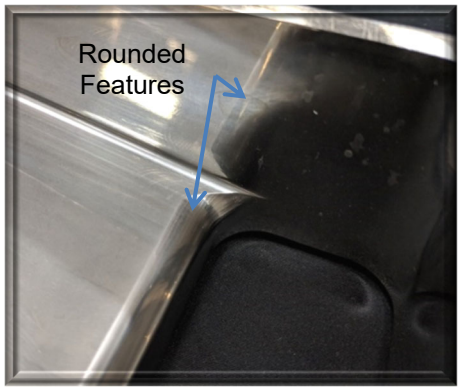

Figure 20. Example of a CAM plate with rounded features.

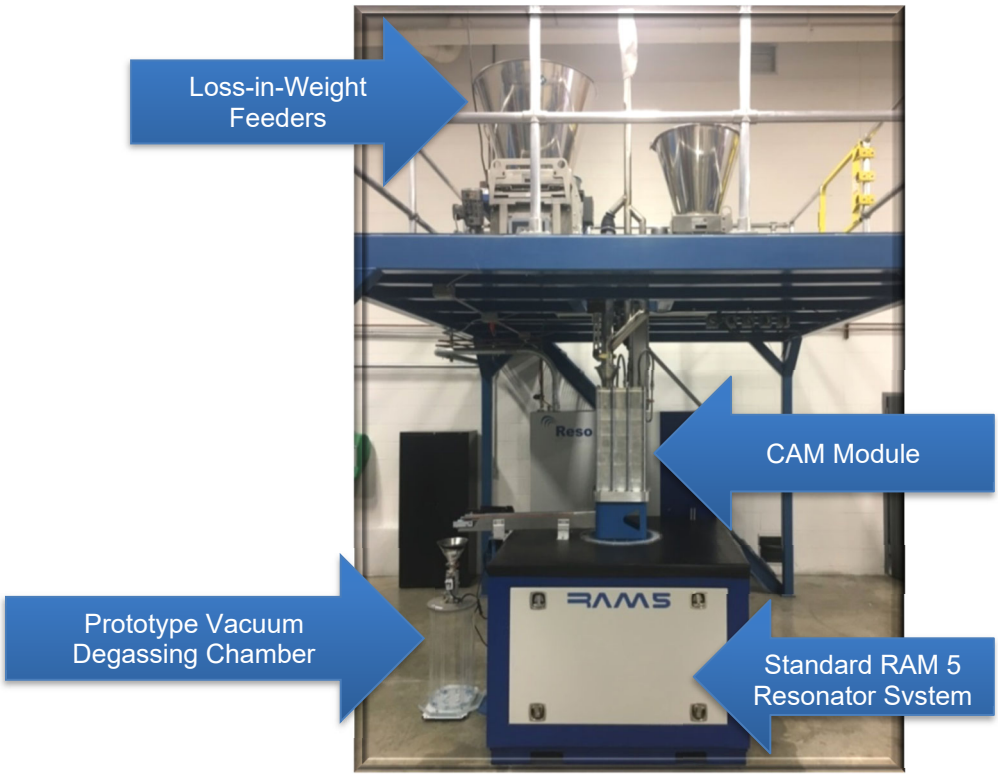

Figure 21. CAM process setup.

The loss-in-weight feeders were modified to reduce the amount of variation in the feed rate around the set point. At the beginning of the mixing process development, the feeders were supplying powder to the prototype CAM-CIP with a relative standard deviation (RSD) of 3.0% about the set point. This amount of variation in the solids content produced material that was unacceptable.

After modifying the feeders, the feeders were able to supply powder to the prototype CAM-CIP with a RSD of 1.0% about the set point. The feeders were modified by replacing the open flight feeder screws with solid screws to prevent the SGB from flowing too quickly into the prototype CAM-CIP. The feeders were also modified by adding mesh to the outlet nozzles to prevent the HGS from being fed in large compacted clumps. Furthermore, the feeders were placed on metal plates resting on vibration dampening feet to eliminate each feeder agitator from disturbing the other feeder. Finally, the automatic feedback control loop parameters (gain and reset time) of each feeder was tuned for minimum variation in feed rate for the feeder material and feed rate. A cartoon showing these modifications is shown in Figure 22.

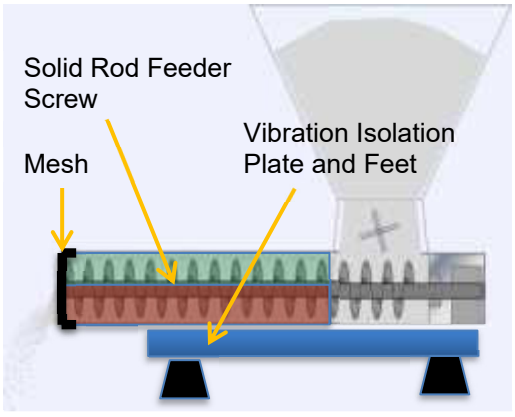

Figure 22. Loss-in-weight feeder showing modifications made to reduce feed variations.

The mixing process was then investigated in several designed experiments to determine the effect of several

factors on two different responses. The factors that were varied were the prototype CAM-CIP configuration, the RAM 5 acceleration, the mass flow rate, and the solids loading of the mixed material. The responses that were measured and modeled were the consistency of the material produced in the continuous mixing process and the homogeneity of the material produced.

In the context of this discussion, the homogeneity of the material is referring to how well the material exiting the prototype CAM-CIP is mixed at any given instant of time and is an indication of how well the prototype CAM-CIP is mixing material. The homogeneity measurements were made using several thermal gravimetric analysis (TGA) measurements to measure the amount of solids in small (~100 milligram (mg)) samples taken from different locations of a 100 gram (gm) sample collected at a certain time during a mixing run. The variation in the amount of solids over these small samples (range, standard deviation, or the like) was used to determine the homogeneity of the material produced by the prototype CAM-CIP at the time the 100 gm sample was taken.

The consistency of the mixed material is referring to the amount of variation in a property of the exit material over time and an indication of how well controlled the continuous mixing process is. TGA was used to measure the solids loading over time to measure the consistency of the CAM process.

Two different prototype CAM-CIP configurations were tested. At the prototype CAM-CIP exit, there is a pinch point (labeled point A in Figure 19). Tests were conducted with and without a ½ inch spacer to determine what affect, if any, the presence of the spacer had on the CAM process.

The RAM 5 acceleration was varied from 60 to 80 *g* to measure the effect acceleration had on the mixedness of the material and the consistency of the CAM process.

The processing factors that were varied were total mass flow rate and solids loading. The flow rate was varied from 1,000 to 6,000 grams per minute (gm/min) in the prototype CAM-CIP. The solids loading was varied from 83% to 92% solids with most testing concentrating on 86% to 89%.

Tests were also conducted to determine the feasibility of controlling the temperature of the material in the mixing process. One plate of the prototype CAM stack was designed and manufactured with 14 channels through the plate (shown in Figure 23). Hot or cold water can be fed through the channels to heat or cool the plate (and the material being mixed in the plate's section of the CAM stack) without the material and water coming into contact.

Two plates of a CAM stack were modified with thermowells as shown in Figure 24 so that the material temperature could be measured with a resistance temperature device (RTD) without the material and the RTD coming into contact. These modified plates were sufficient to show temperature control feasibility, but characterization testing had to wait for the energetic rated CAM-CIP (reported in the Task 1.2 section).

An investigation was conducted into minimizing the off specification material produced at the beginning of the continuous mixing process. To establish steady state mixing in the prototype CAM, liquid must be introduced to the CAM before the solids. The liquid creates a region in the CAM that captures the powder to create a paste that establishes good mixing. In the absence of

the initial liquid region, powder will proceed through the CAM-CIP at a high rate causing excessive dusting at the CAM-CIP exit and requiring a long period of time to establish good mixing. Unfortunately, the liquid priming creates a certain amount of liquid rich off specification material at the beginning of the mixing run. At the beginning of each mixing run, the amount of liquid used to prime the prototype CAM-CIP was varied to measure the effect of the amount of liquid prime on the amount of off specification material produced.

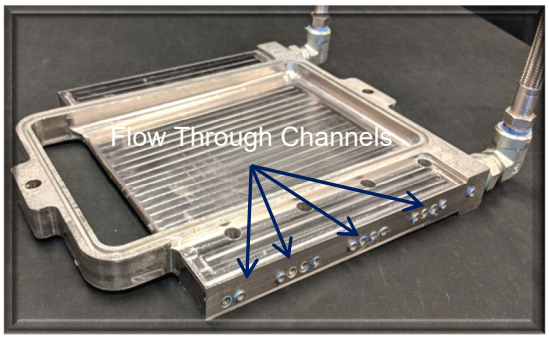

Figure 23. Prototype temperature control plate for feasibility testing.

At the end of each mixing run, the CIP process was adjusted to achieve 100% cleaning efficiency while creating a minimum of waste. The temperature of the detergent, the RAM 5 acceleration, the cleaning duration, and the flow rate of detergent were changed during the scrubbing cycle. The temperature of the rinse water, the rinse duration, and the flow rate of rinse water were also varied for each rinse cycle.

The knowledge and experience gained with the prototype CAM-CIP were used to design an energetics capable CAM-CIP with temperature measurement and control capability. The finished module was then characterized for consistency and homogeneity using a custom designed experiment methodology.

The heating and cooling capability of 5 cross-flow channel plates were also investigated using the information from 13 RTDs to measure the temperature of the paste and heating/cooling water inlet and outlet temperatures.

Four runs were conducted with curable material in the CAM-CIP. The material was degassed in a prototype degassing chamber and cured to measure the physical properties of the resulting surrogate PBX material. The measurements were then used to analyze the homogeneity and consistency of the cured material produced by the CAM-CIP.

Concurrent with the mixing process development of the CAM-CIP, the CIP process developed on the prototype CAM-CIP was further refined on the energetics capable CAM-CIP.

RESULTS AND DISCUSSION

Task 1.1 CAM-CIP Prototype Design for Effective Cleaning

The existing CAM cascade plate design needed to be modified to eliminate dead zones, improve transition sections, and seal joints

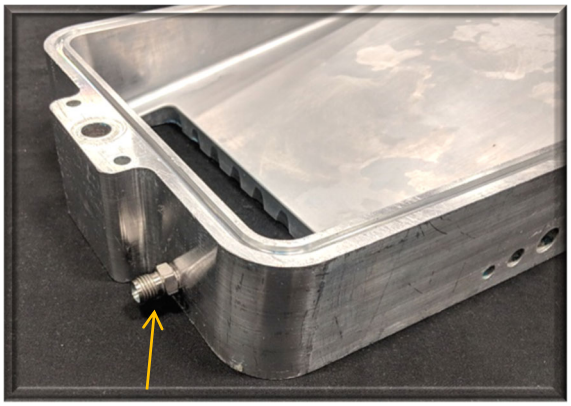

Figure 24. Thermowell for RTD measurement.

between plates to prevent material accumulating during mixing. Additionally, it was necessary to modify the existing CAM to enable the addition of the cleaning solution.

A prototype CAM-CIP was designed and manufactured with rounded features to eliminate 90-degree corners where material would be prone to accumulate termed "dead zones." Material that accumulates in dead zones can periodically be released into the process resulting in off specification material. Dead zones are also areas difficult to clean in a CIP process. Close-ups of these rounded features are shown in Figure 20 above.

A flat gasket type seal using a soft, compliant silicone material was designed to keep material from migrating from the processing regions of a CAM-CIP module and implemented. A silicone gasket design was chosen as shown in Figure 25 for implementation in the CAM-CIP. The gasket shown in Figure 25 was imaged after a continuous mixing run followed by a CIP. Testing on the prototype CAM-CIP and the CAM-CIP has shown zero failures and leaks using the designed seals.

Task 1.2 CAM-CIP Prototype Design for Temperature Control

Temperature control is necessary during a continuous mixing process. The temperature of the material being mixed must be kept below a maximum temperature determined by the curing agent and binder system being used for the composite PBX material. If the material exceeds the maximum temperature, the material would cure prematurely, possibly inside the mixer.

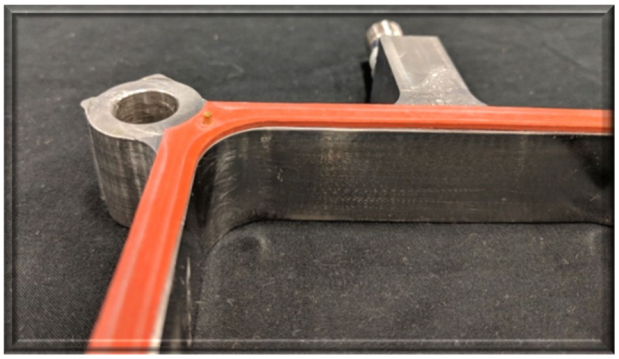

Figure 25. Silicone gasket post CIP process.

The temperature can also be controlled during mixing to stabilize and enhance the mixing process. The material temperature along with other factors such as composition and processing conditions determine the material rheology including the viscosity. Controlling the temperature of the material as it mixes allows the material properties to be adjusted to enable the best properties for mixing such as a lower viscosity or shear thinning rheology.

Temperature control during the CIP process may also be critical to prevent material from curing in the mixer during the CIP process. The CIP process is also benefitted by increasing the temperature of the residual material thus lowering the residual material's viscosity making it easier to clean out of a CAM-CIP stack.

Temperature control is achieved by passing hot or cold water through channels embedded in the plates of the CAM module. The temperature of the material is measured using RTDs in thermowells at each end of the plates.

The prototype CAM was modified with thermowells, and a plate with heat transfer channels was manufactured to test the concept of a temperature controlled CAM-CIP. Images of the modifications are shown in Figures 23 and 24. The temperature of the mixed material before and after the heat transfer plate as well as the temperature of the cooling water, in this case, before and

after the heat transfer tubes during a continuous mixing run is shown in Figure 26. The experiment showed that the concept was feasible, and the design was implemented in the CAM-CIP.

Based on the prototype results, the CAM-CIP was designed with five plates capable of heating or cooling material (shown in Figure 27) being mixed in the CAM-CIP. Several continuous mixing runs have been completed with the CAM-CIP. The temperature of the mixed material exiting the CAM-CIP over time for three different runs is shown in Figure 28. The data in Figure 28 show that it is possible to control the temperature of the material exiting the CAM-CIP from 40 to 75°C. With appropriate equipment, it is possible to independently control the temperature of the material on each plate inside the CAM-CIP.

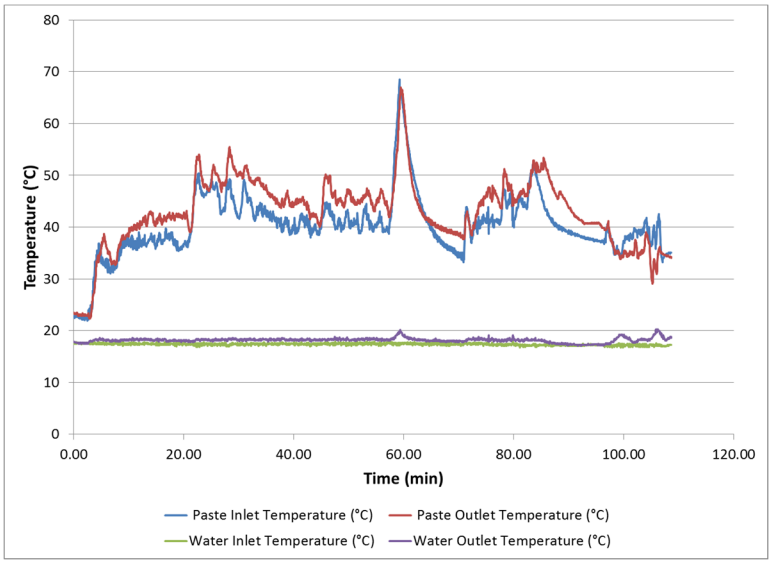

Figure 26. PBXN-110 surrogate and cooling water temperatures of prototype CAM-CIP with single heat transfer plate.

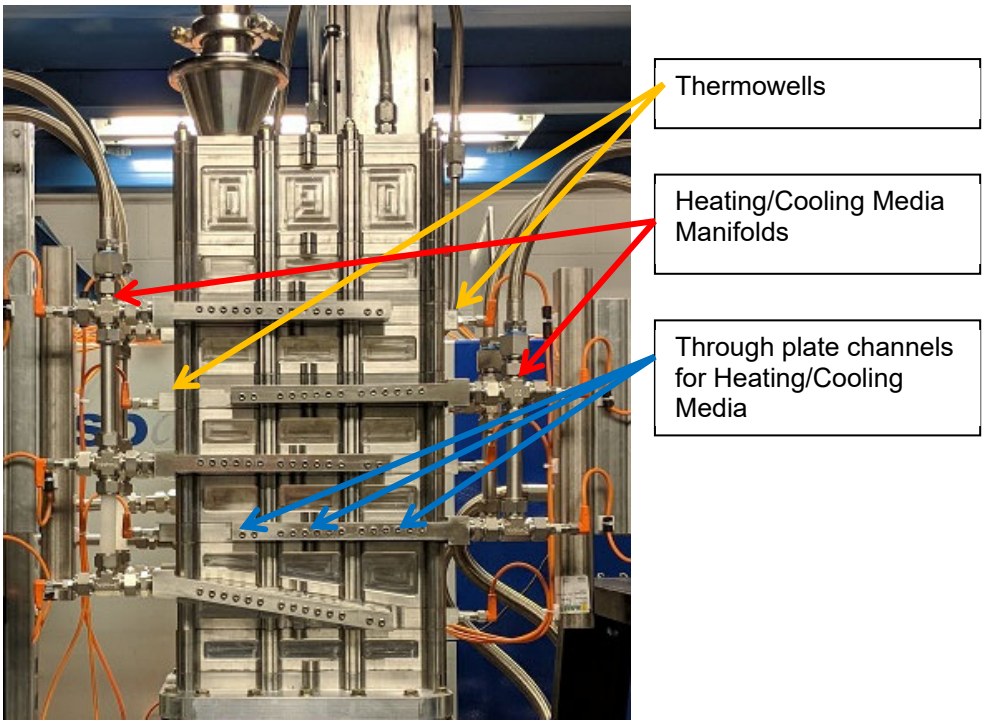

Figure 27. CAM-CIP showing thermowells and heating cooling plates.

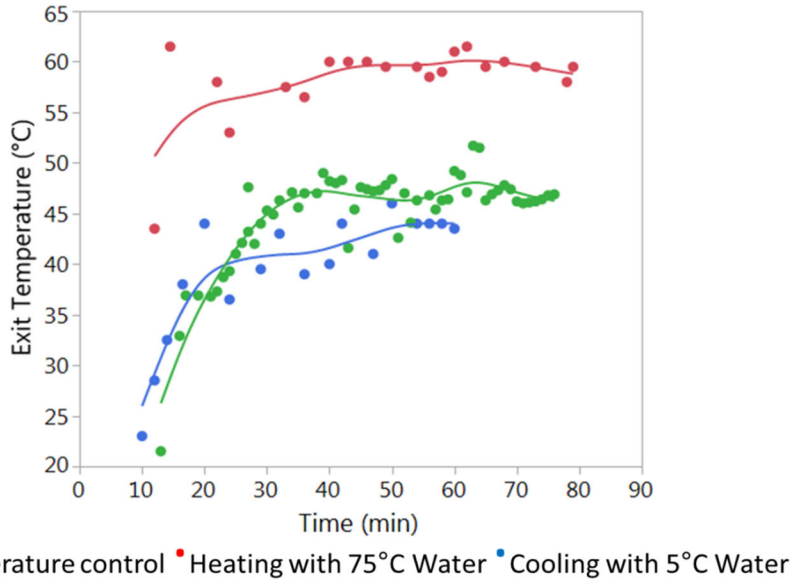

Figure 28. PBXN-110 surrogate material exit temperature for cooling, heating, and no control.

Task 1.3 CAM-CIP Prototype Design for Pressure Control

As material is continuously processed in the CAM-CIP, air will be incorporated into the mixed material. The incorporated air must be removed from the mixed material before the material is cast and cured to prevent the cured material from containing voids or air bubbles. Voids and air bubbles are unacceptable in the cured energetic material.

Degassing the material as it moves through the CAM-CIP was found to be impractical because the absolute pressure required for degassing was too low (~ 10 Torr) to allow material to continue to exit the CAM-CIP. It was decided to develop a continuous degassing process to allow material to be simultaneously degassed and cast into a vessel for later curing and testing. The operational knowledge gained from developing a prototype semi-continuous process will be used to develop a continuous degassing process for commercial production.

A prototype degassing chamber was designed and is shown in Figure 29. Material is added to a hopper and is then sucked through a variable control valve so that the material flow rate can be controlled. After the control valve, the material passes through a strainer to create strands of material and increase the surface area to volume ratio of the material. The higher surface area to volume ratio allows a higher degassing rate. The strands of material shown in Figure 30 then fall into the mold being used to cast the degassed material. After the material has been degassed, the vacuum in the chamber is broken collapsing any remaining voids in the cast material. The mold is then cured in an oven at a minimum of 60°C for 5 days.

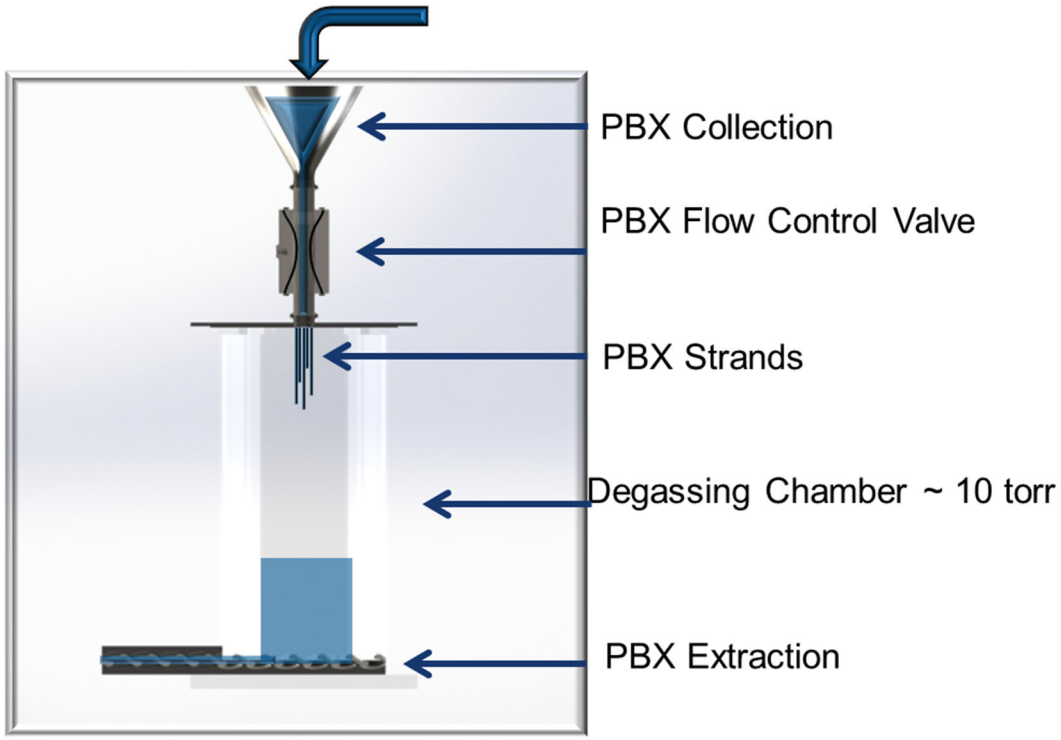

Figure 29. Prototype degassing chamber.

A 5 gallon batch of PBXN-110 surrogate material was mixed on a RAM 5 to test the degassing process. The material was degassed and cast into a mold 4" x 4" x 7". Once the material in the mold was degassed, cast, and cured at 60°C for 7 days, it was sectioned into plates. Three dog bones were cut from each of the plates and tested for strain and hardness.

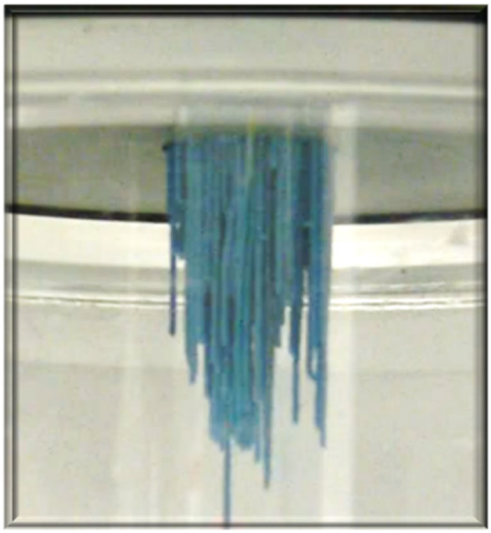

Figure 30. Strands of PBXN-110 surrogate entering the degassing chamber.

The dog bones were also X-rayed to visualize the presence of any voids or cavities. An image of a typical dog bone section along with its X-ray is shown in Figure 31. According to the X-rays, no voids or cavities were found indicating the prototype semi-continuous vacuum degassing process was adequate to degas the PBXN-110 surrogate material.

The average hardness from each plate is shown in Figure 32 with error bars showing one standard deviation. The material appeared homogeneous, and a Tukey HSD test comparing the means confirmed that the material hardness of the samples from each plate was statistically identical.

The average strain at break from each plate is shown in Figure 33 with error bars showing one standard deviation. The strain measurements show wider variation than the hardness measurements due to the more sensitive nature of the strain testing; however, a Tukey HSD test comparing the means confirmed that the material from each plate strain at break was statistically identical.

The hardness and strain results shown in Figures 32 and 33 confirmed that the semi-continuous degassing chamber and associated process were capable of degassing the homogeneously mixed PBXN-110 surrogate material. One of the goals of further testing with the prototype CAM-CIP and the CAM-CIP will be to determine the necessary operating conditions of a continuous degassing process.

Figure 31. Typical cured PBXN-110 surrogate dog bone (left) and X-ray image of the same (right).

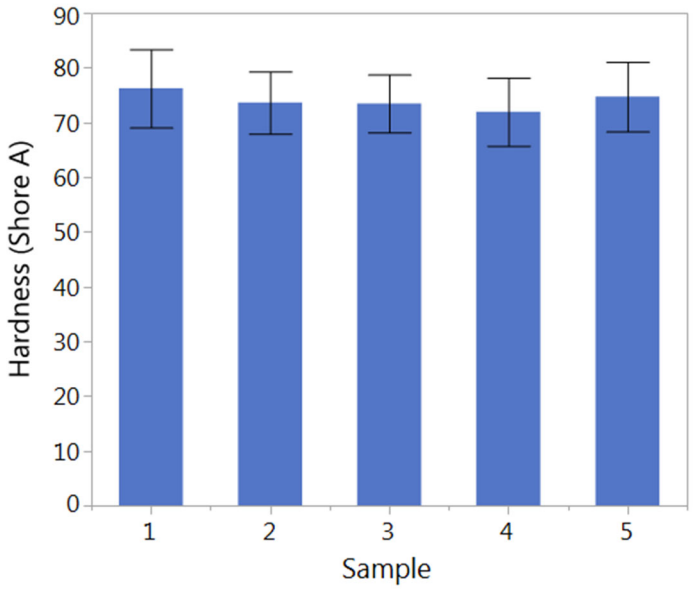

Figure 32. Average hardness of cured PBXN-110 surrogate samples from a single RAM 5 batch mix (error bars represent one standard deviation).

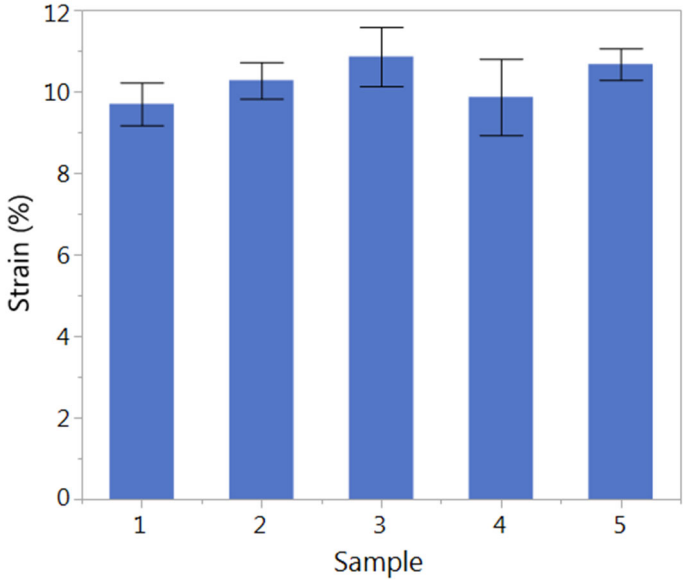

Figure 33. Average strain at break of cured PBXN-110 surrogate samples from a single RAM 5 batch mix (error bars represent one standard deviation).

Task 1.4 CAM-CIP Prototype Testing and Material Evaluation

The prototype CAM-CIP is one part of the continuous acoustic mixing process. As such, to characterize and understand the performance of the prototype CAM, the individual processes upstream of the prototype CAM-CIP need to be characterized and understood to deconvolute the upstream operations from the mixer.

To characterize the entire mixing process, each operation involved in the process, both upstream and downstream of the prototype CAM-CIP, needs to be characterized. Once each operation involved in the CAM process is characterized, the entire process can be improved to the maximum capability of the individual components. If the process needs further improvement, sufficient understanding then exists to confidently design improvements to the entire process to increase the CAM process capability.

The first individual operation in the continuous mixing process is material addition to the CAM-CIP. Material addition is accomplished with progressive cavity pumps for the liquids (R45 HTPB and DOA slurry) and loss-in-weight feeders for the powders (SGB and HGS).

The progressive cavity pumps were calibrated gravimetrically at various flow rates. The process revealed that the pumps were stable and fed the liquids at the specified flow rate with a relative standard deviation (RSD) of 0.5% each.

The loss-in-weight feeders are more sophisticated and required more effort to characterize and optimize. At the beginning of testing the CAM process with SGB and HGS, the mass flow rate of the un-optimized feeders varied about the mass flow rate set point with an RSD of 3%. The amount of variation in the feeder's flow rate produced PBXN-110 surrogate with solid fractions outside the specified limits of 86% to 89% and was unacceptable. To reduce the mass flow rate variation of the feeders, a combination of modifications was used to reduce the variation in the powder flow rate.

Solid flight feeder screws were installed on both feeders to eliminate the possibility of material free-flowing through the center of an open flight feeder screw. This effect is a possibility with free-flowing powders and was seen with the SGB. An example of one of the solid rod feeder screws is shown in Figure 34.

Mesh screens were added to the nozzle outlets as shown in Figure 35 in order to simultaneously add back pressure on the material in the feeder nozzle and break up any lumps caused by the increased back pressure. This effect is a possibility with low bulk density powders such as the HGS.

Figure 34. Solid rod feeder screw.

Vibration isolation plates were placed under the feeders to prevent the agitators in each feeder from affecting the other feeder. During operation, the loss-in-weight feeders can be mechanically agitated to prevent rat-holing and bridging. The SGB feeder does not need this agitation due to the free-flowing nature of the SGB, but the HGS feeder does require the agitation to keep the HGS from readily rat-holing. The vibration isolation plates prevented the HGS feeder from interfering with the SGB feeder operation.

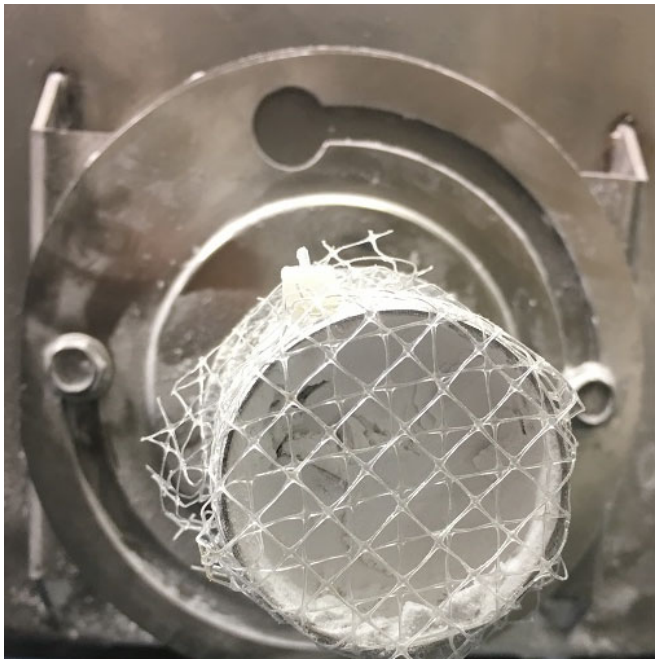

Figure 35. Mesh screen installed on the HGS feeder.

The gain and reset time constants of the feeder feedback control loops were also tuned for the materials and flow rates used in the experiments.

After the feeders were optimized, the variability of the feeder flow rates was measured at 1% (RSD). Charts of the feeder flow rates over time before and after the optimization modifications are shown in Figure 36. With a powder flow rate RSD of 1%, the CAM process produced mixed material with solid fractions between the specified limit of 86% to 89%.

TGA was chosen to measure the solids loading of the material exiting the CAM-CIP. Once the feeder flow rate was optimized, a method of measuring the performance of the continuous mixing process was needed to optimize the operation of the CAM-CIP. TGA measures the solids loading by pyrolyzing all the ingredients of the mixed material except the SGB and the HGS. By comparing the mass of a sample before and after the pyrolysis process, the combined amount of HGS and SGB can be calculated as the solids loading (as a fraction or percentage) of the material.

By measuring the solids loading of material exiting the CAM-CIP, the consistency of the produced material can be measured over time. An example of TGA measurements of mixed material over time is shown in Figure 37. The data shown in Figure 37 represent a solids fraction RSD of 0.5% indicating a process that is in control. Furthermore, a process with an RSD of 0.5% can also be described as being significantly better than a 3 sigma process in this case.

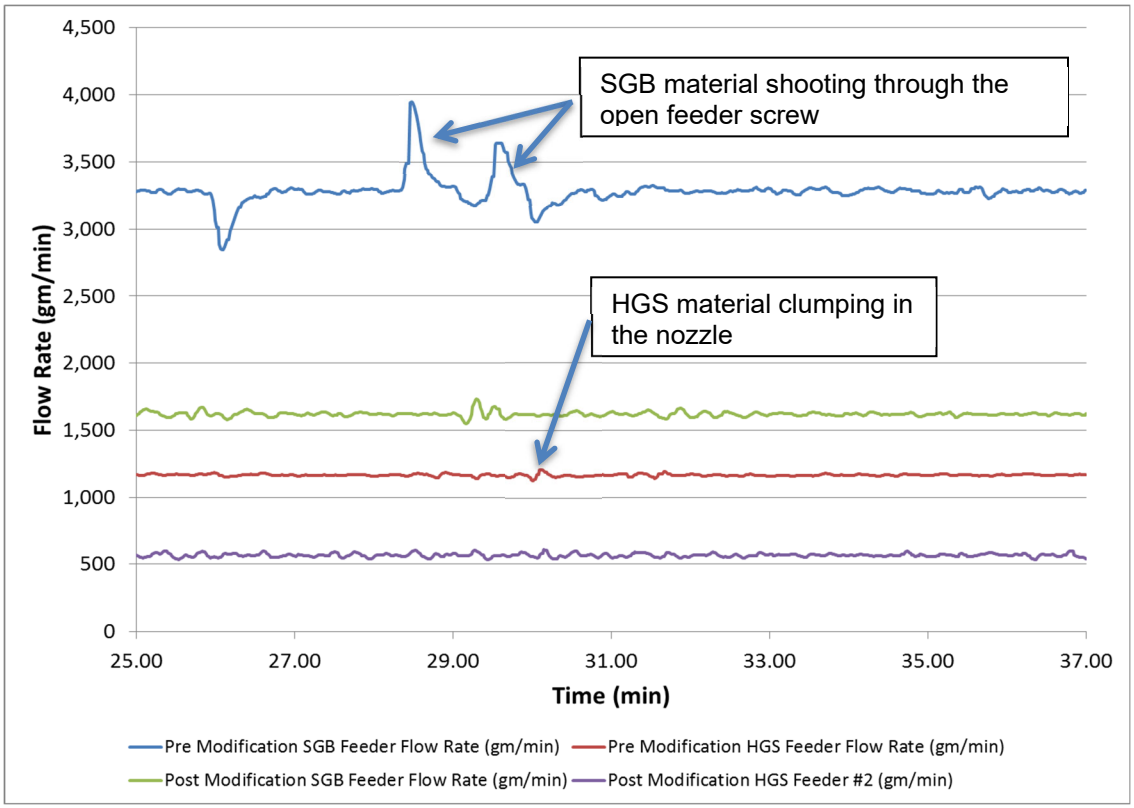

Figure 36 Feeder flow rates during continuous mixing runs before and after feeder modifications.

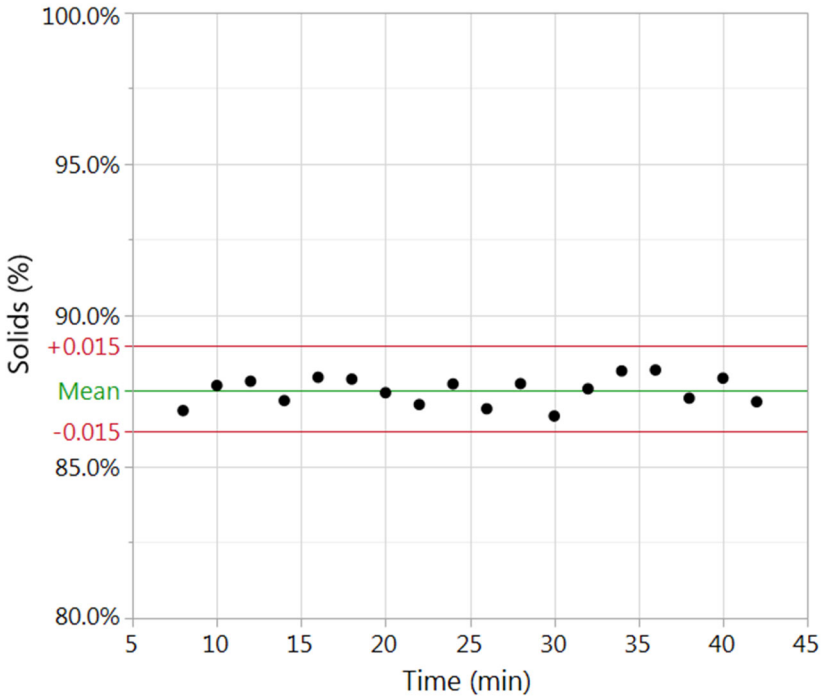

Figure 37. Solids percentage as measured by TGA over time of PBXN-110 surrogate mixed material.

Once it was shown that TGA provided a reliable and repeatable measure of the CAM-CIP performance, the CAM-CIP's response to changing process variables with respect to consistency

was measured and modeled. The model was then used to optimize the performance of the CAM-CIP. Designed experiments were conducted where the acceleration, mass flow rate, and solids loading of the mixing process were varied.

Table 2 shows the different accelerations, mass flow rates, and solids loadings used in the designed experiment. Also shown in Table 2, in the right most column, is the response measured during each test in the experiment.

Table 2. Test conditions and measured responses for a designed experiment to characterize the CAM-CIP.

Test	Solids Loading, %	Acceleration, *g*	Flow Rate, gm/min	Solids Loading Range, %
1	92.6	60	932	0.4097
2	92.6	80	932	3.175
3	88.6	70	535	1.044
4	89.0	60	970	0.5808
5	89.0	80	970	0.6595
6	86.0	70	1,500	0.00989
7	87.5	60	1,760	0.1824
8	89.0	70	2,500	0.1230
9	87.5	80	2,500	0.5012
10	86.0	60	2,500	0.4531
11	87.5	70	1,250	0.1617

During each condition in the designed experiment, the mixer was allowed to reach steady state (approximately 10 minutes of run time) then three samples of material were collected 2 minutes apart. The solids loading of each sample was measured using TGA, and the range (maximum – minimum) of the three samples was calculated and recorded in the right most column of Table 2. As such, the effect of acceleration, mass flow rate, and solids loading on the consistency of the CAM-CIP can be and was modeled.

Shown in Figures 38, 39, and 40 are three graphical representations of a model of the effect that nominal solids loading (as a percentage), acceleration, and flow rate have on the consistency (as a range in solids loading over time) of the mixed material exiting the CAM-CIP. The three images show how the homogeneity of the mixed material responds to changes in the solids loading and flow rate at three different accelerations (60, 70, and 80 *g*).

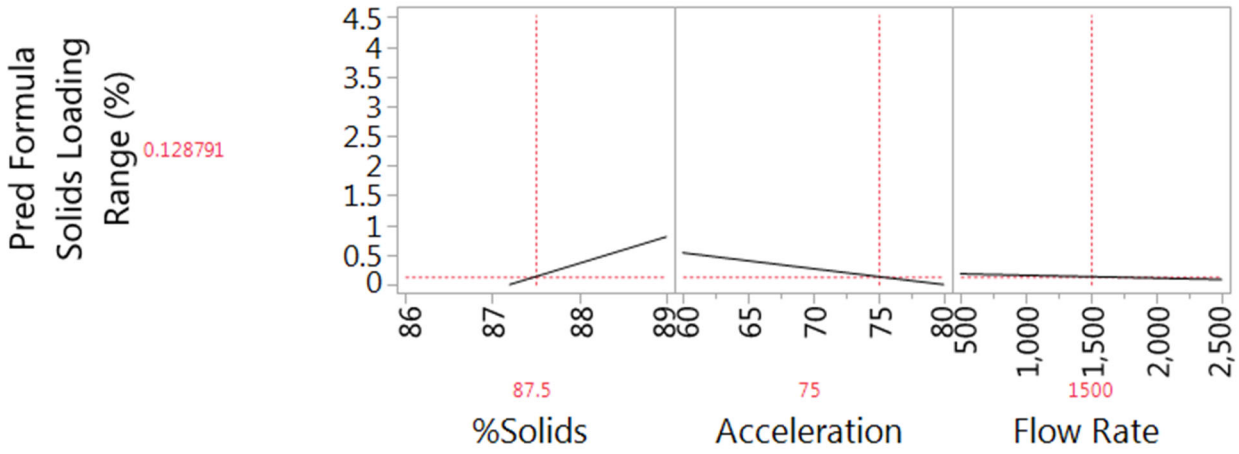

Figure 38. Model of solids loading response to % solids, acceleration, and flow rate for the CAM-CIP mixing PBXN-110 at 75 *g*.

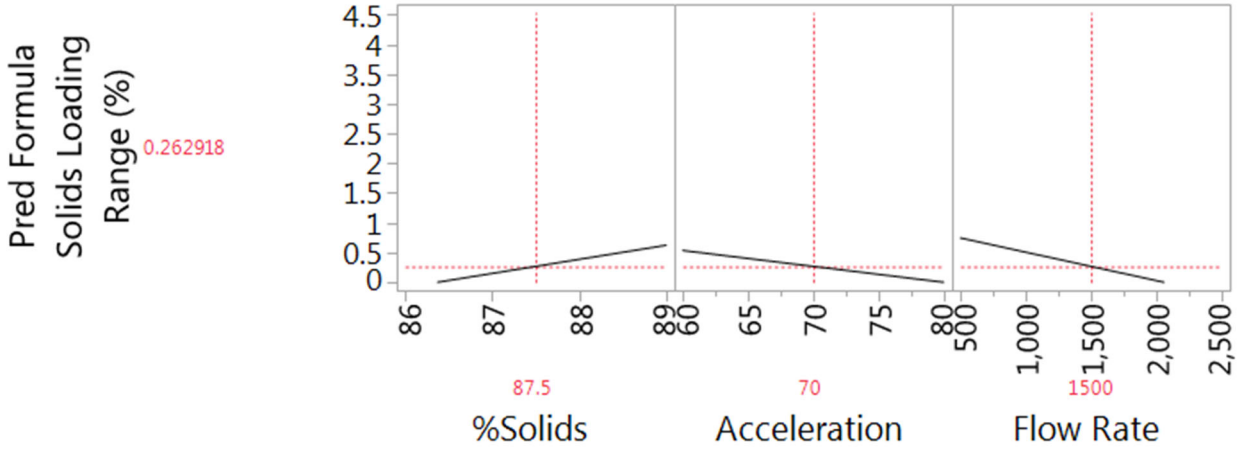

Figure 39. Model of solids loading response to % solids, acceleration, and flow rate for the CAM-CIP mixing PBXN-110 at 70 *g*.

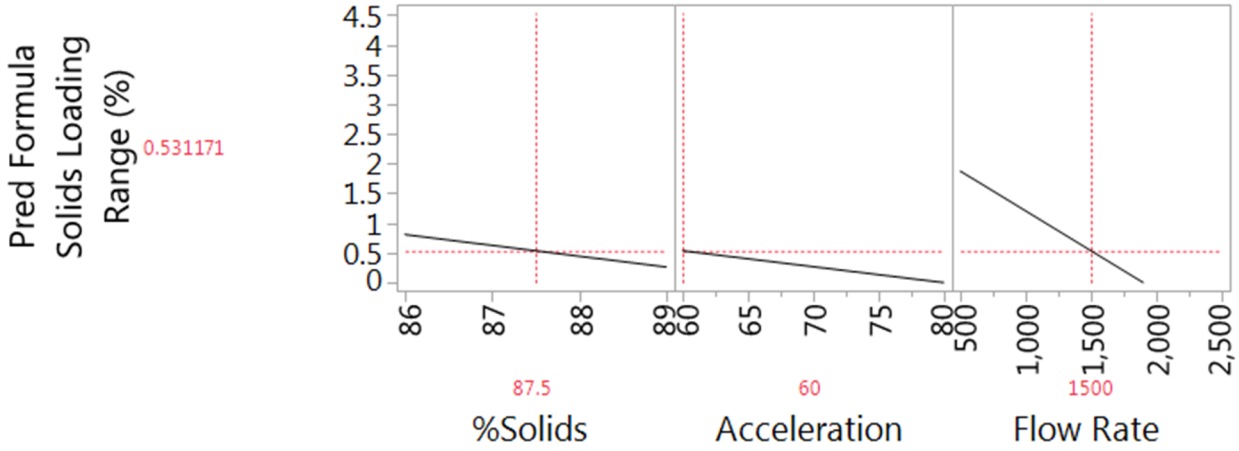

Figure 40. Model of solids loading response to % solids, acceleration, and flow rate for the CAM-CIP mixing PBXN-110 at 60 *g*.

The nature of the effect of solids loading and flow rate on the solids loading range depends on the acceleration indicating that the solids percentage and flow rate interact with the acceleration to affect the homogeneity of the mixed material exiting the CAM-CIP. At 60 *g* of acceleration

(see Figure 40, above), there is a negative relationship between the solid percentage and the solids loading range. At 75 *g* of acceleration (see Figure 39, above), the effect of solids percentage on solids loading range becomes positive. Similarly, the negative relationship of flow rate on the solids loading range at 60 *g* becomes minimal at 75 *g* of acceleration (see Figure 38 above).

The interaction between acceleration and solids loading and flow rate has important consequences for commercial production of energetic material. The solids percentage and flow rate of material being mixed cannot be arbitrarily set or changed for a given acceleration. Conversely, there is an ideal acceleration to mix energetic material at a given material formulation.

For a given mixing process, there will be operating conditions that provide insensitivity to certain types of variation upstream of the mixture. For example, if the process flow rate of a mixing process was variable during the process then for a given solids percentage there may exist an acceleration where the homogeneity of the material is insensitive to flow rate.

TGA can also be used to measure the solids loading of a single sample at several points. The homogeneity of the samples can then be quantified as a range, standard deviation, or RSD of the solids loading in the sample. The effect of acceleration, mass flow rate, and solids loading on the homogeneity of the material exiting the CAM-CIP at any moment can then be characterized and modeled. The model can then be used to optimize the mixedness of material exiting the CAM-CIP.

Figure 41 shows the RSD of the range of solids loading over the first nine different operating conditions as described in Table 2, above. The variation of the RSD over time is due to the changing conditions. While the homogeneity of the mixed material does vary, the different RSDs are all low in an absolute sense, and the RSDs from the best operating conditions are exceptionally low.

Figure 42 shows the average solids loading of two samples collected during a continuous mixing run using optimized process conditions. The solids loading was measured using TGA at five separate locations in each collected sample indicating the material is well mixed as it exits the CAM-CIP. The samples were taken from a run at 80 *g* of acceleration, and 1,250 gm/min with a solids loading of 88%. The RSD of the solids loading for both samples is 0.15% and 0.11% showing that the material that exits the CAM-CIP at any given moment of time is extremely well mixed.

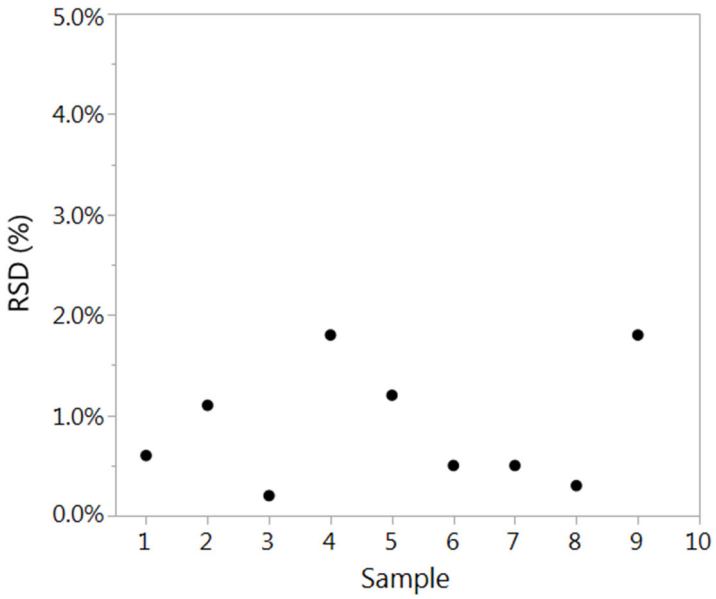

Figure 41. RSD of mixed material at nine different operating conditions.

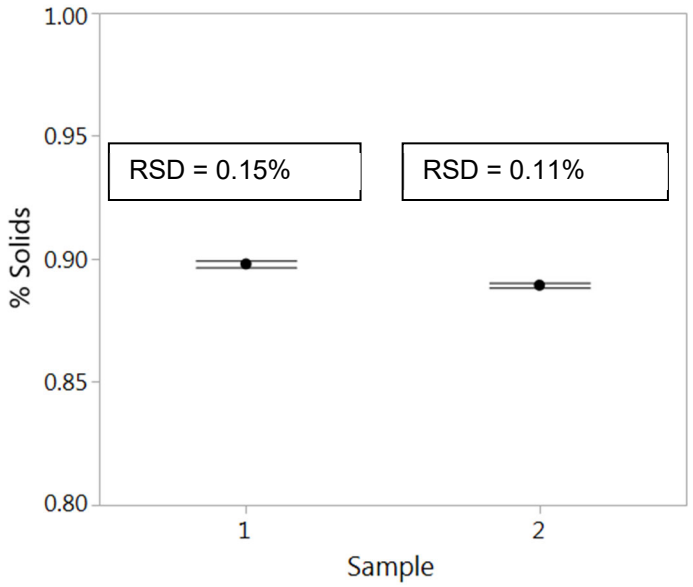

Figure 42. Average solids loading of two samples collected during a continuous mixing run (error bars represent one standard deviation of the material collected).

Task 1.5 CAM- CIP Prototype Evaluation for Efficient Cleaning

After NAWCWD China Lake personnel developed an inert surrogate formulation for PBXN-110, batch testing was conducted to identify an aqueous detergent that would clean the surrogate material off a polypropylene surface in a RAM environment.

The tests were conducted by mixing surrogate PBXN-110 material in a pre-weighed vessel then pouring it out. After the dirty vessel was again weighed, a detergent solution was added and "scrubbed" under RAM motion. The cleaning solution was poured out of the vessel, and the mass of material remaining was measured to determine the final mass. The cleaning efficiency was then calculated using Equation 1, above. A test was also conducted using mineral spirits as a control.

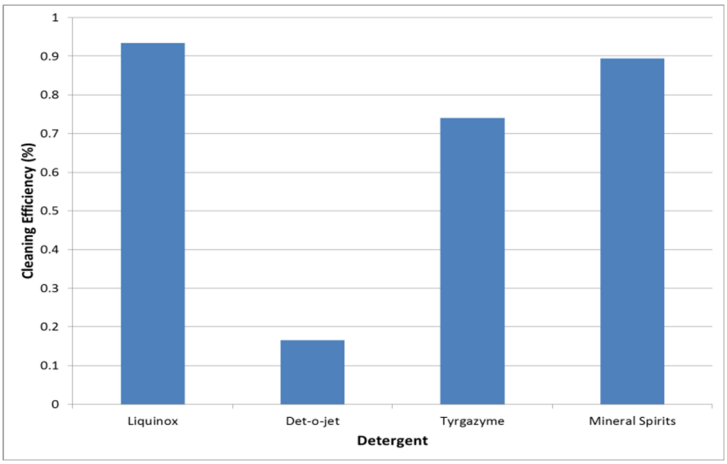

Figure 43. Cleaning efficiencies of different detergents in batch CIP testing.

The results of the experiment are shown in Table 3 and Figure 43. Undiluted Liquinox provided the highest cleaning efficiency (higher even than mineral spirits) and was used as the detergent for the remainder of the project. The decision to down-select the detergent to Liquinox was made while keeping in mind that the decision was made using data that might possibly be confounded by the concentration of detergent, the temperature of the detergent, the surface to be cleaned (polypropylene in this experiment versus stainless steel in the CAM-CIP), and most importantly the material being cleaned (a sugar/gypsum surrogate for PBXN-110 in this case).

Table 3. Cleaning efficiency results of batch RAM CIP testing.

Detergent	Mass of Material to be Cleaned, gm	Mass of Material After CIP, gm	Cleaning Efficiency, %
Liquinox	35.18	7.43	93
Det-o-jet	68.97	97.12	17
Tyrgazyme	64.54	29.02	74
Mineral Spirits	63.73	12.29	89

Different concentrations of Liquinox in water were then tested in the manner previously described to determine the concentration that provided the highest cleaning efficiency. Table 4 and Figure 44 show the results from the experiment.

Table 4. Cleaning efficiencies tabulated for different Liquinox in water concentrations.

Liquinox Concentration, v/v %	Mass of Material After CIP, gm	Cleaning Efficiency, %
15	10.3	91.0
25	9.8	91.4
35	8.1	92.9
50	15.3	86.6
75	13.6	88.1
100	24.3	78.6

The cleaning efficiency results versus Liquinox concentration is shown in Figure 44 for Liquinox concentrations from 15% to 100% on a volume-to-volume basis (v/v%). A concentration of 35 v/v% Liquinox in water was found to provide the highest cleaning efficiency. For experimental efficiency in solution preparation, a Liquinox 33 v/v% in water solution was used for CIP process development.

In order to show that the glass bead based surrogate material accurately represented the behavior of the energetic material with respect to CIP processes, an experiment was conducted at NAWCWD China Lake. Energetic material was mixed in a batch vessel. The material was poured, but not scraped out leaving residual material in the container. A detergent solution of 33 v/v% Liquinox in water was added to the vessel, and the vessel was agitated in the RAM mixer at 95 *g* of acceleration. The detergent solution was poured out and the residual material imaged. The test was repeated using surrogate material, and the images are shown in Figure 45. Efforts to quantify the mass of material remaining after each step and calculate the cleaning efficiency were confounded by a leak in the vessel lid seal.

The images show that the surrogate material represents the CIP behavior of the energetic material. In Figure 45, the residual energetic material left after the CIP process is shown on the left. The residual surrogate material left after the CIP process is shown on the right.

The inert PBX mixing in Task 1.4 using the prototype CAM-CIP set the stage to evaluate the effect of changes in the CIP process after each continuous mixing run. After each mixing run using the prototype CAM-CIP, a CIP was performed under different time, temperature, acceleration, and flow rate conditions.

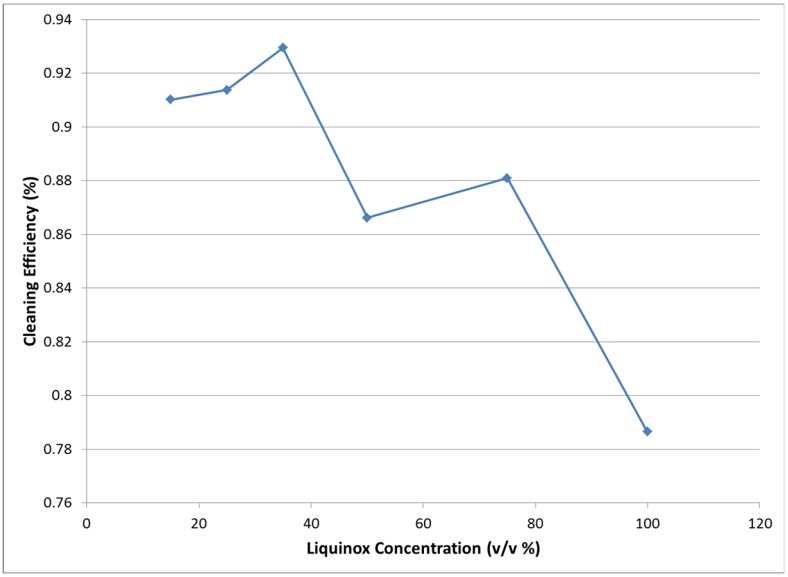

Figure 44. Cleaning efficiencies for different concentrations of Liquinox in water in batch CIP testing.

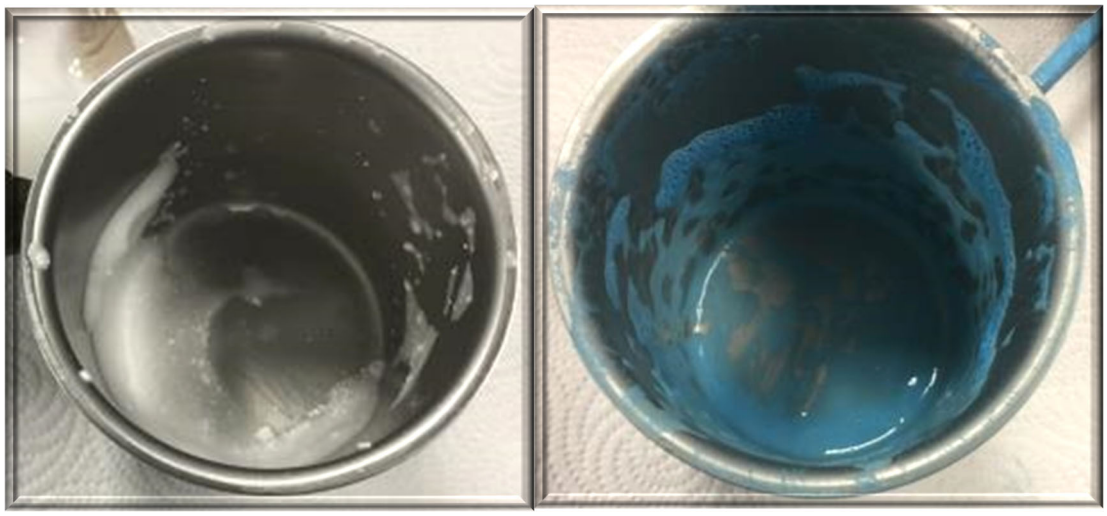

Figure 45. Post CIP images of energetic material (left) and surrogate material (right).

To evaluate the effect of changes in the various CIP factors, the cleaning efficiency was measured as well as the volume of cleaning solution and rinse solution used. The results of these evaluations were used in iterative design modifications to the CAM-CIP to enhance the cleaning effectiveness.

Over 11 separate tests, the time spent feeding 33 v/v% Liquinox into the prototype CAM-CIP was varied from 5 to 15 minutes to measure the effect of scrubbing time on the cleaning efficiency. A weak positive effect was discovered at low scrubbing times (5 to 8 minutes), and the cleaning efficiency did not increase for scrubbing times greater than 8 minutes.

The temperature of the 33 v/v% Liquinox solution was varied from 18 to 60°C, and a positive effect of the temperature on the cleaning efficiency was discovered. Higher temperatures were not tested because the surrogate material might cure at temperature higher than 60°C. For subsequent testing, the Liquinox solution was fed at 60°C into the CAM modules.

The RAM 5 acceleration was tested at set points of 80 and 95 *g* of acceleration to measure any effect of acceleration on cleaning efficiency. A strong positive relationship was discovered as expected, and the scrubbing process was conducted at 95 *g* of acceleration for the remaining CIP development.

The time, temperature, and acceleration of the rinse process were tested in separate experiments to measure the effects on the cleaning efficiency and eliminate any confounding effects on the scrubbing testing. The same general trends were discovered with the rinse process as with the scrubbing process with one difference. The maximum effective time of the rinse process was determined to be 5 minutes.

After the testing previously described, the best CIP process is listed in Table 5. Typical cleaning efficiencies achieved with the process in Table 5 were greater than 99.9%, but a 100% cleaning efficiency was unachievable. Paradigm shifting attempts were made to achieve a 100% cleaning efficiency with the most promising attempt coming from power washing the prototype CAM-CIP in place.

Table 5. First optimal CIP process.

Cycle	Feed	Time, min	Temperature, °C	Acceleration, *g*
Scrub	33 v/v% Liquinox	8	60	95
Rinse	Water	5	60	95

An elegant solution was discovered during a process change where the liquid feed lines were disconnected and the powder feed tube was sealed before the CIP process was started on the prototype CAM-CIP. The isolated CAM-CIP module is essentially air and water tight except for the module exit. During the scrubbing cycle, the detergent solution forms foam under the RAM motion. The foam that forms creates an airtight seal at the CAM module exit. As more foam is formed from detergent being fed to the CAM-CIP module, the foam fills the CAM-CIP module. When the CAM-CIP module is full of foam, the liquid detergent feed can be turned off while the CAM-CIP module continues to scrub itself minimizing the amount of detergent solution used. At the end of the scrubbing process, compressed air at about 0.1 pounds per square inch (psi) is used to blow the foam out of the CAM-CIP module.

During the rinse cycle, the same effect occurs with foam filling the entire CAM-CIP during the process. The foam is a lower concentration in detergent than the foam created during the scrubbing cycle, but it too must be blown out of the CAM-CIP module with compressed air. A final rinse at lower acceleration (35 *g*) is used to remove the last of the foam from the CAM-CIP module.

The final CIP process is described in Table 6. It has been used in over 25 subsequent continuous CAM-CIP mixing runs, and the cleaning efficiency of each run has been 100%.

The amount of detergent solution used in the scrubbing cycle is 3.6 liters (L), and the amount of rinse water used is 4.7 L. It is important to note that the amount of detergent and rinse water used is decoupled from the amount of mixed material produced due to the nature of a continuous mixing process. As more material is mixed, the amount of CIP waste created remains constant; therefore, the ratio of CIP waste to mixed material produced decreases at a rate proportional to the inverse of the production rate.

Images of the plates of the prototype CAM-CIP are shown in Figure 46. The images show the 100% cleaning efficiency of the CIP process in Table 6.

Table 6. Final optimized CIP process conditions.

Cycle	Feed	Time, min	Temperature, °C	Acceleration, *g*	Volume Used, L
Scrub	33 v/v% Liquinox	8	60	95	3.6
Rinse	Water	5	60	95	4.7

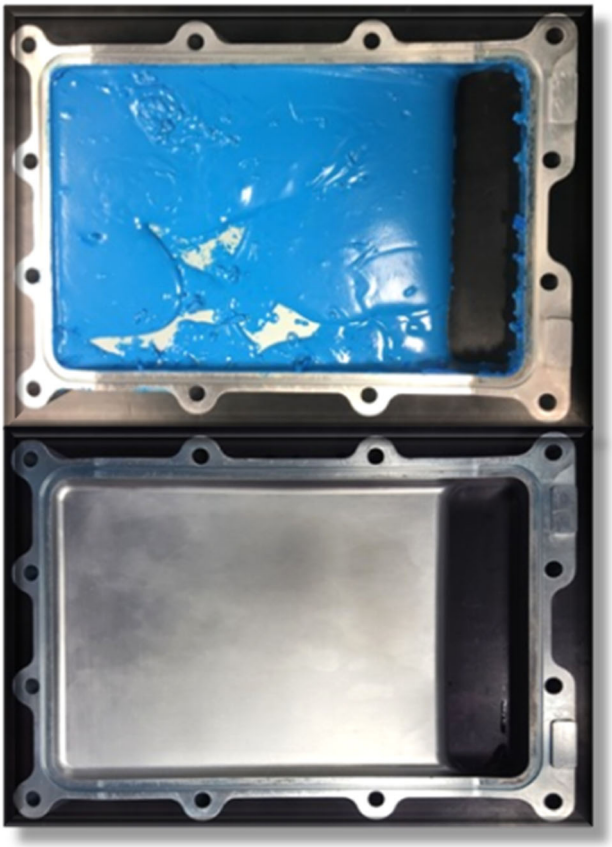

Figure 46. Images of a CAM-CIP plate before (top) and after (bottom) the CIP process.

Task 2 Inert Formulation Development

Preliminary to CAM and CIP process development, inert formulations were developed by the researchers at NAWCWD China Lake to match the material properties of current U.S. Navy qualified plastic-bonded explosives (PBXs). At the beginning of the project, surrogate formulations were prepared to match the density and rheological properties of the following explosives: PBXN-110, PBXN-112, or PBXN-109.

A number of nonreactive solids were evaluated as surrogates of cyclotetramethylene tetranitramine (HMX) including granulated sugar, powdered sugar, gypsum, potassium chloride, and aluminum powder. The combination of the laurel methacrylate binder with sugar and gypsum used to mimic PBXN-112 produced a non-Newtonian shear thickening material that did not process well during preliminary tests in the batch RAM environment. The combination of aluminum powder and sugar in R45 to mimic PBXN-109 initially proved very difficult to match the density of PBXN-109 with an acceptable end-of-mix viscosity.

The first formulation to give a close balance of material density and end-of-mix viscosity used a combination of granulated sugar and gypsum with hydroxyl-terminated polybutadiene (HTPB) to mimic the formulation of PBXN-110. This material gave a density that was a little bit lower

than PBXN-110 but matched very well the end-of-mix viscosity. The surrogate formulation produced a very granular looking final product that visually matches the look of PBXN-110.

In addition to the low density (1.62 grams per cubic centimeter (g/cc) for the down-selected PBXN-110 formulation), the incorporation of the gypsum caused the final mixed product material to flow chaotically through the casting funnel when placed under reduced pressure and vibration. It was also noted at this point in the project that because the granulated sugar was water soluble, it may artificially enhance the ability of the CIP process to effectively remove the residual product from the CAM stack. The decision was made to abandon the initial inert surrogate in favor of one where both particle size solid components are insoluble in water.

For the second, water-insoluble version of the inert material, melamine was evaluated that can be procured from OCI Nitrogen Inc. in a variety of particle sizes. Glass spheres were also evaluated that are also available in a variety of particle sizes as well as a variety of densities since they come in both hollow and solid particle form.

The advantage of melamine is that it is an organic molecule with a structure that resembles the nitramines in HMX and cyclotrimethylene trinitramine (RDX) and, as such, should offer similar particle-binder wetting interactions that should mimic the mixing of the material using the RAM environment. The difficultly with using melamine is that the density is quite low (1.57 g/cc) when compared to HMX. While the material end-of-mix viscosity of PBXN-110 could be matched very well, the composition density could not.

Finally, a combination of hollow glass spheres (HGS) and solid glass beads (SGB) gave an inert material that best matched the density and end-of-mix viscosity of PBXN-110. The final inert formulation is given in Table 7.

One drawback to using the glass beads is that because of their pure spherical nature, the final product produced does not match the granular appearance of PBXN-110. The mixed PBXN-110 surrogate material appears to be very smooth. In all other ways, it has proven to be a very good material surrogate of PBXN-110, and all of the solid components are insoluble in water which should provide the proper environment for the evaluation of the CIP process.

The PBXN-110 inert formulation was prepared with conventional batch mixers, as well as batch style RAM mixers and the material properties validated against the Military Specifications for the analogous qualified PBXs. Once it had been determined that the material processed using the RAM technology met the material property specifications for standard PBXN-110, the formulations were given to the researchers at Resodyn Corporation for evaluation in the CAM configuration.

Table 7. Glass bead inert surrogate formulation for PBXN-110.

Material	Weight %
R45 HTPB	12 – 14
DiOctyl Adipate (DOA)	
Lecithin	
DiNitroSalicylic Acid (DNSA)	
TriPhenyl Bismuth (TPB)	

IsoPhorone DiIsocyanate (IPDI)	
Ethanox 702	
Cyan Blue	
HGS	86 – 88
SGB	
Cyano-to-Hydroxyl Ratio (CN/OH)	1.0
End-of-Mix Viscosity (80-140°F)	10 – 30 kP

Task 3 Optimize CAM-CIP Operation

The CAM-CIP module rated for live energetic mixing and configured for temperature control was received, assembled, and ready for testing at Resodyn on 16 February 2019.

The CAM-CIP module was characterized according to the designed experiment detailed in Section 1.4. The performance of the CAM-CIP was characterized with respect to three main factors. The factors were total mass flow rate, RAM acceleration, and percent solids.

The responses that were modeled were material homogeneity, material compositional consistency, and material flow rate.

Typical continuous mixing runs took a total of six hours to complete. The longest runs involving inspecting the CAM-CIP module after a hard stop mid-mixing took up to 10 hours including the additional time to clean the CAM-CIP module by hand. The shortest runs of 40 minutes involved running four mixing/CIP runs in one day.

The 6 hour time included one half hour of filling the loss-in-weight feeder hoppers and liquid pump reservoirs and one half hour of assembling the CAM-CIP module on the RAM 5 for a total of one hour of setup. Next, one half hour of additional heating was required for the HTPB to finish preheating.

The actual average run time for the mixing process was 90 to 110 minutes with the shortest time of about 40 minutes and the longest time of about 150 minutes. The startup process typically took 10 minutes after which well mixed paste starts exiting the CAM-CIP. In about half of the continuous mixing runs (those conducted for Task 3 with the CAM-CIP), the run would proceed for one hour until the loss-in-weight feeders and the DOA reservoir were empty. In the other half of the mixing runs (those conducted in Task 1.4 with the prototype CAM-CIP), the runs would proceed for 10 to 20 minutes longer because the loss-in-weight feeders would be refilled about 40 minutes into the process.

The average CIP process took 24 minutes. The scrubbing phase using detergent lasted 14 minutes and the rinse phase using water lasted 10 minutes. Before the CIP process was optimized, the longest CIP time was 45 minutes. Disassembling the CAM-CIP module for inspection typically took 15 minutes, and cleaning the downstream equipment (conveyor belt and vacuum degassing valve) took a final two to two and a half hours.

As described above, the material homogeneity is the instantaneous homogeneity of the material exiting the CAM-CIP. Stated another way, material homogeneity is how well mixed the

material that exits that CAM-CIP is at any one moment. Material homogeneity is measured using TGA. A sample of material is collected (typically 200 mL representing 15 seconds worth of produced material). The amount of solids in the sample is measured by conducting TGA measurements on material from 5 different locations in the sample a shown in Figure 47.

The modeling shows that at the ideal operating conditions investigated in the designed experiment of 1,250 gm/min, 80 g of acceleration, and 86% solids, the relative standard deviation of the solids percentage in the collected sample was 0.1% RSD. The worst recorded RSD occurred at 93% solids and was 3% RSD.

The characterization of the CAM-CIP shows that at the optimized operating conditions the CAM-CIP produces material with a high level of homogeneity.

The material consistency is how the composition of the material exiting the CAM-CIP changes over time. Material consistency is also measured using TGA. A sample of material is collected every 3 to 5 minutes. The amount of solids in the sample is measured by conducting TGA measurements on the samples taken over time.

The material consistency is not a measure of just the CAM-CIP performance. The material consistency also depends on the upstream operations that affect the composition of the produced paste. In this case, the material consistency depends on the pumps that deliver the R45 HTPB and the DOA/Lecithin/Blue Dye as well as the loss-in-weight feeders that deliver the HGBs and the SGSs.

The first step in measuring the material consistency of the CAM-CIP was to tune or optimize the loss-in-weight feeders for the lower feed rates used for the CAM-CIP as opposed to the prototype CAM-CIP. The feed rate consistency of the loss-in-weight feeders were then measured while the feeders were operating during a typical continuous mixing run. The RSD of the loss-in-weight feeder feed rates were 1.0%.

Next, the material consistency of the mixed paste exiting the CAM-CIP was sampled and measured as described above. A chart of the solids fraction from a typical run is shown in Figure 48. The green line represents the average amount of solids for the run. The red lines represent 3 standard deviations from the mean. The RSD of the solids percentage of the mixed material for the run in Figure 23 was 0.8% indicating that the CAM-CIP module is reducing the variation of the solids percentage in the mixed material imparted by the loss-in-weight feeders.

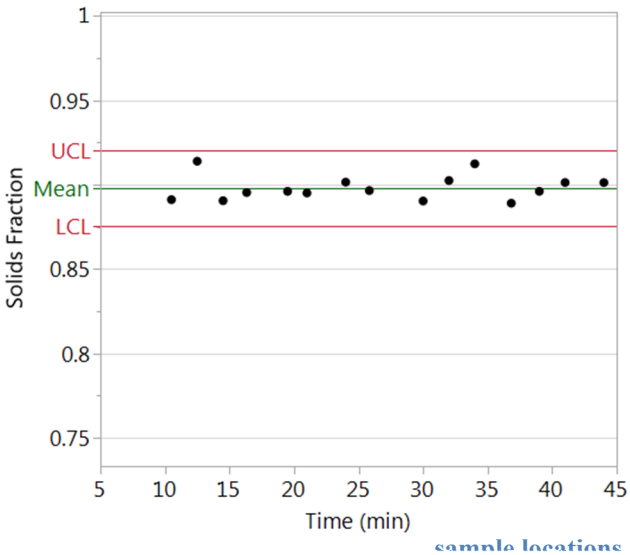

Figure 48. TGA measurements over time for a typical CAM-CIP run

The CAM-CIP was designed with cross-flow channels in five plates to allow hot or cold water to be passed in a cross-flow manner

through the plates indirectly heating or cooling the mixing material akin to a shell and tube heat exchanger. The CAM-CIP was also designed to accommodate RTDs to measure the inlet and outlet temperature of the fluid used for heating or cooling the material as well as the temperature of the mixed paste as it enters and exits the sections of the CAM-CIP that contain the plates with cross-flow channels.

A series of experiments were conducted to measure the temperature of the material as it progressed through the CAM-CIP as well as the temperature of the water as it entered and exited each cross-flow plate in the CAM-CIP. The temperature traces from a typical run using 75 °C water in the cross-flow channels are shown in Figure 49 and Figure 50.

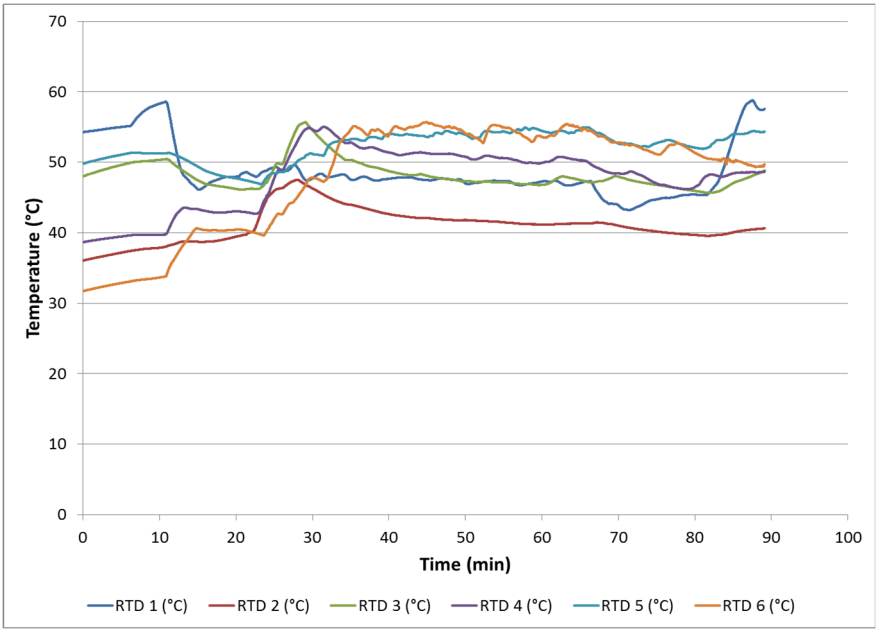

Figure 49. Temperature traces showing the RTDs measurements of the paste temperature

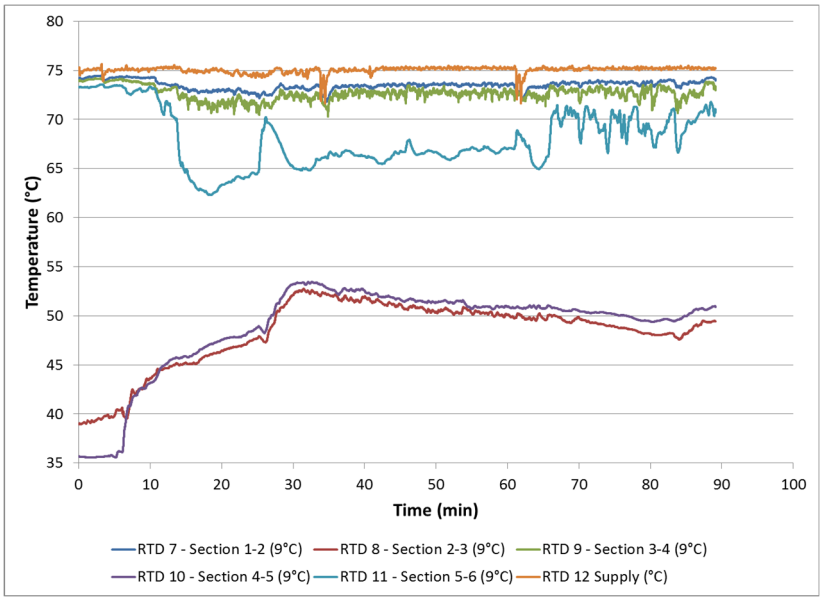

Figure 50. Temperature traces of the heating water outlet temperatures for each section, RTD 12 is the inlet temperature for each section

The locations of the numbered RTDs in the CAM-CIP are shown in Figure 51.

Since it is not possible at this time to measure the amount of surface area of the cross-flow plate that is in contact with the mixed material, it is not possible to calculate the heat transfer coefficient of the paste material being mixed in the CAM-CIP. Due to the variable nature of the flow rate of material moving through the CAM-CIP, the instantaneous measurement the heat resistance from the cross-flow plates was not practical or useful. The power transferred into the material from the water can be calculated however.

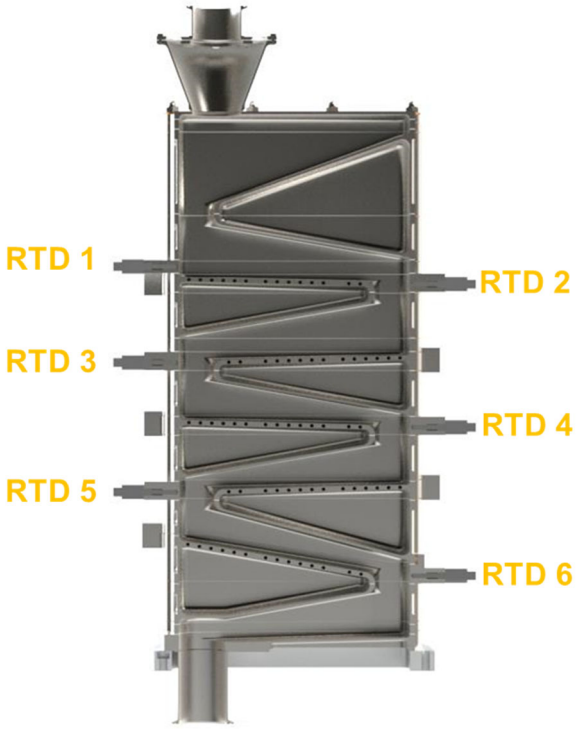

Figure 51. CAM-CIP rendering showing the location of the RTDs used to measure the paste temperature

The average amount of power transferred from the hot water varies from section to section due to the different paste temperatures, different levels of mixedness, and different viscosities of the pastes in each section. The average power transferred from the water ranged from 100 W at the top of the CAM-CIP to 800 W at the bottom of the CAM-CIP.

A series of 4 continuous runs were conducted with IPDI, TPB, and DNSA added to the liquid reservoir containing the DOA, lecithin, and blue dye. The mixed paste produced during these runs was degassed, cast, and cured so that the physical properties of the surrogate PBX material could be measured and analyzed for consistency and homogeneity.

The first run was conducted with the prototype CAM module. Mixed material was produced at 3,000 gm/min. Approximately 800 mL of material was collected every two minutes and degassed in the prototype degassing chamber shown before and repeated in Figure 52. The degassed material was collected inside the degassing chamber in a cylinder 8" in diameter and 18" tall. Once the material was degassed, the cylinder was cured at 65°C for 7 days.

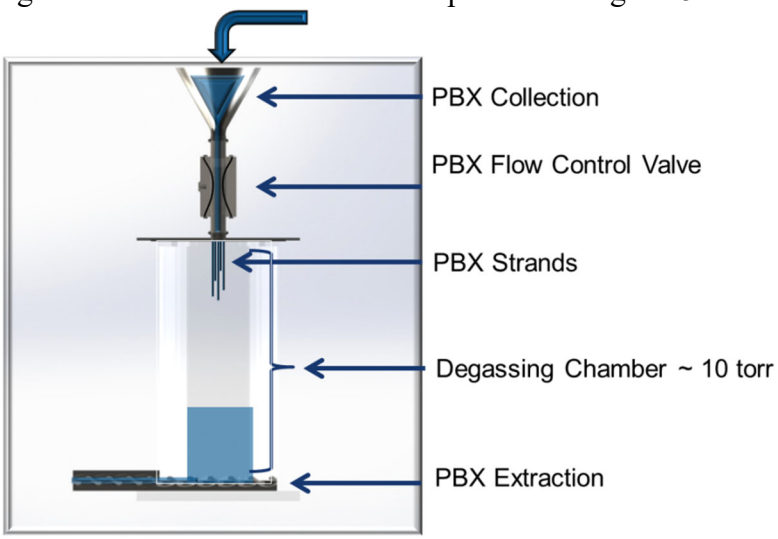

Figure 52. Cartoon showing the prototype vacuum degassing chamber

Between samples collected for degassing, 100 mL samples of material were collected for TGA testing. The TGA results are shown in Figure 53 and indicate that the material composition consistency was good with an RSD (around the mean of 87.5% solids) of 0.5%.

The cylinder containing the cured material was sectioned into disks about 0.75 in thick. The disks were then trimmed to a thickness of 0.5 in on a lathe. Four dog bones from each disk were cut and labeled A through D where dog bones A and D were on the outside of the disk and B and C were in the inside of the disk.

The maximum stress and maximum strain of each dog bone were tested using an MTS Alliance RT/100 according to JANNAF dog bone standards and STANAG 5406. The maximum stress and maximum strain are shown in Figure 54 and 55 respectively. The physical data shows that the physical properties of the material produced by the CAM were not consistent.

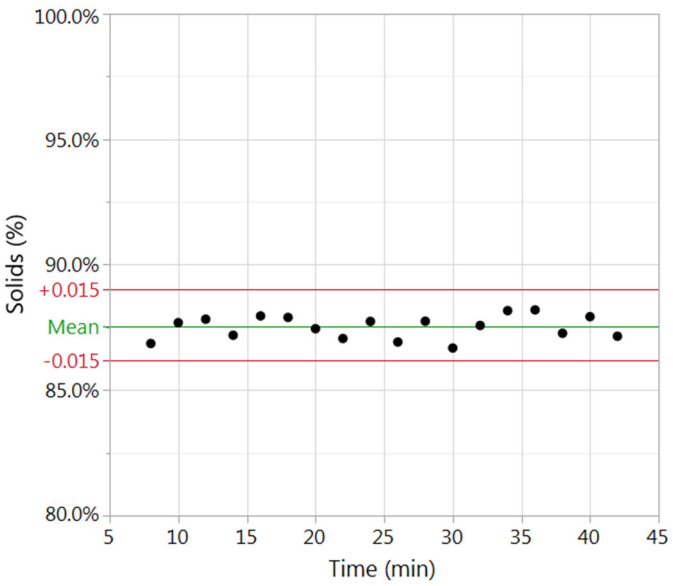

Figure 53. Solids percentage of samples collected over time

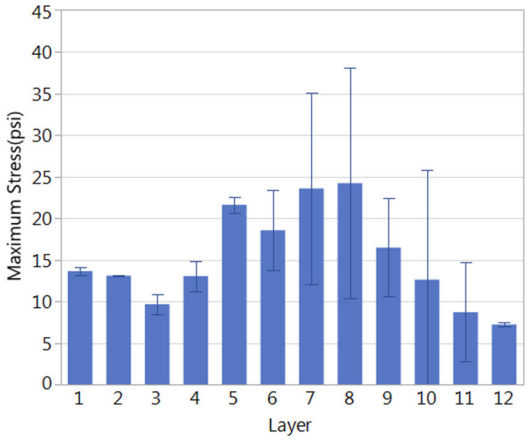

Figure 54. Maximum stress versus layer (error bars represent one standard deviation)

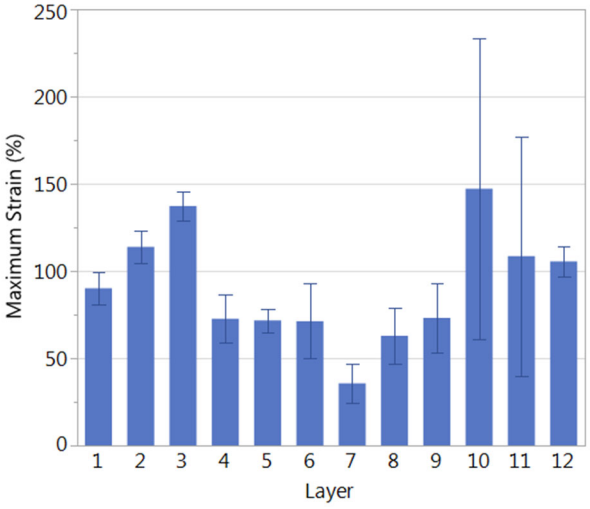

Figure 55. Maximum strain versus layer (error bars represent one standard deviation)

Subsequent checks of the curative chemicals, the solids and liquids feeding equipment, and batch mixing tests revealed no apparent cause of the poor cured material consistency.

The average maximum stress and strain by dog bone are shown in Figure 56. A Tukey HSD test was conducted on the data shown in Figure 56 to compare the average maximum stress and strain measurements. The analysis showed that the material was homogeneously mixed, just not

Figure 57. Second curative run materia

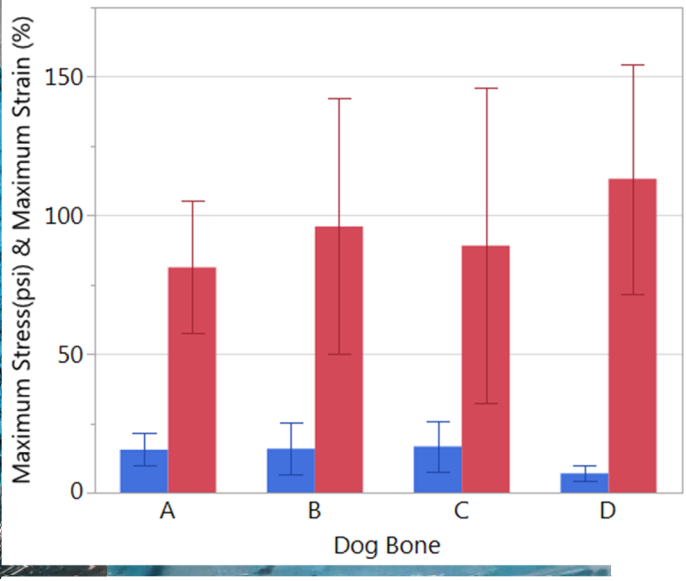

Figure 56. Maximum stress and maximum strain versus dog bone (error bars represent one standard deviation)

consistently mixed. In other words, the material in each disk was the same. The material from disk to disk varied however.

The second run was conducted with the CAM-CIP and hot water (75°C) heating plates 2 and 4. Technicians from NAWCWD China Lake were operating the continuous mixing equipment during the run as the final run of a 3 run series intended to train the China Lake technicians before transitioning the CAM-CIP equipment to China Lake for energetic material mixing.

Before the curative run, the formulation was changed (the amount of lecithin was increased). The DOA mixture was significantly more viscous than expected, and therefore, the flow rate of the DOA mixture was less than called for in the test formulation. The resulting mixed paste produced by the CAM-CIP was more viscous than was expected. The end result was that the prototype degassing unit was unable to adequately degas the material.

Regardless, the material was collected in the degassing cylinder and cured, but there were too many cavities and voids as shown in Figure 57. The material was not sectioned or tested for physical properties, but the composition of the mixed material over time was measured using TGA. The results are shown in Figure 58. The average solids percentage of 90% was higher than expected because the viscosity of the liquid containing lecithin was higher than usual reducing the liquid flow rate enough to affect the total solids percentage. The RSD of the material about the mean was 1%. The red lines in Figure 58 are the same ±0.15% solids from the mean as shown in Figure 53 showing the consistency of the continuous process during the second run with more viscous material was somewhat lower.

The third curative run was conducted with the CAM-CIP with no heating or cooling. The CAM-CIP produced 1,250 gm/minute material. Unfortunately, the lecithin concentration was again high enough to affect the final solids percentage. The average solids percentage was 89.8% instead of 87.5% solids.

The TGA results are shown in Figure 59. Once again, the red lines in Figure 59 are the same ±0.15% solids from the mean as shown in Figure 53, above, showing the consistency of the continuous process during the third run with more viscous material was lower. However, the continuous mixing process was still in control.

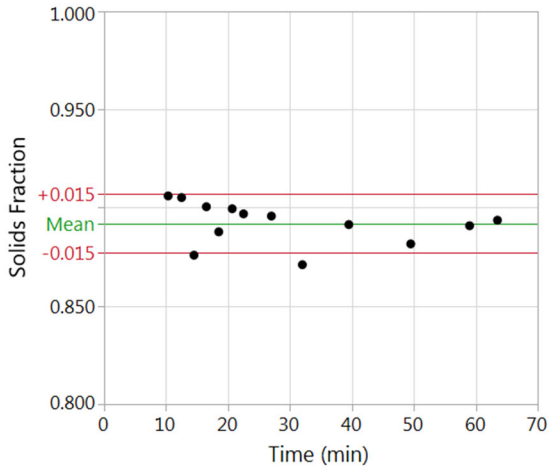

Figure 58. Solids percentage over time for the second curative run

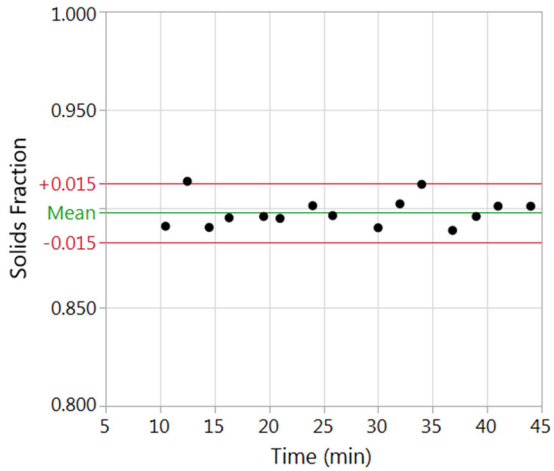

Figure 59. Solids percentage over time for the third curative run

The average maximum stress versus slice (slice is equivalent to time for the collected mixed material) and dog bone are shown in Figures 60 and 61 for the third curable run. The variableness of the maximum stress over time showed that the cured material physical properties were not consistent over time nor did they match the nominal properties expected from a CN/OH ratio of 1.0. The maximum stress versus dog bone data shown in Figure 61 however show that the material was homogeneous.

The average maximum strain versus slice (slice is equivalent to time for the collected mixed material) and dog bone are shown in Figures 62 and 63. The variableness of the maximum strain over time showed that the cured material physical properties were not consistent over time nor did they match the nominal properties expected from a CN/OH ratio of 1.0. The maximum strain versus dog bone data shown in Figure 63 however show that the material was homogeneous.

The fourth curative run was conducted with the CAM-CIP with no heating or cooling. The CAM-CIP produced 1,250 gm/min of paste material with an average solids percentage of the mixed material of 88.7%.

During the fourth curative run, samples for TGA analysis were not taken of separate material during the run. Instead, the solids percentage and density of the cured material was measured on each disk at the positions shown in Figure 64.

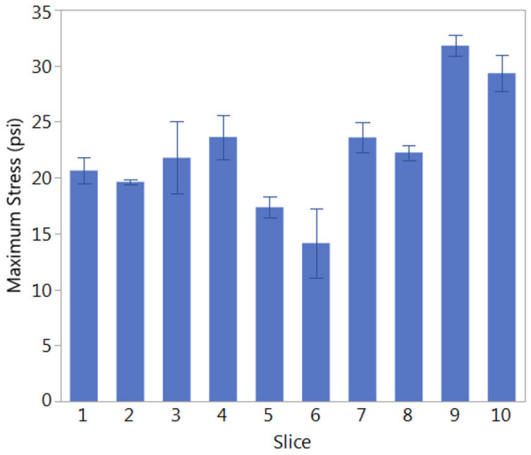

Figure 60. Maximum Stress versus slice (time) for third curative run (error bars show one standard deviation)

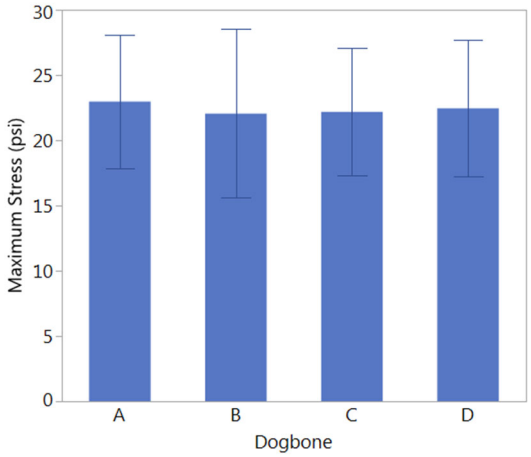

Figure 61. Maximum Stress versus dog bone for third curative run (error bars show one standard deviation)

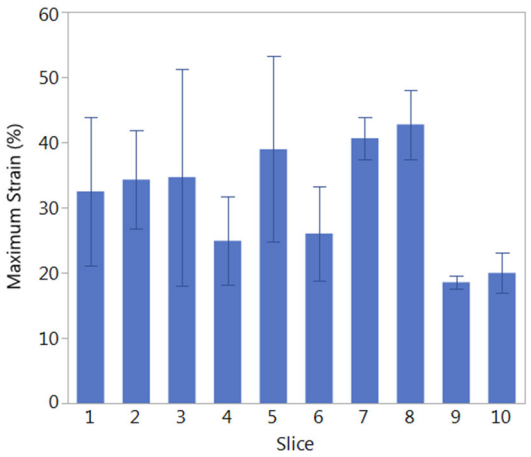

Figure 62. Maximum Strain versus slice (time) for third curative run (error bars show one standard deviation)

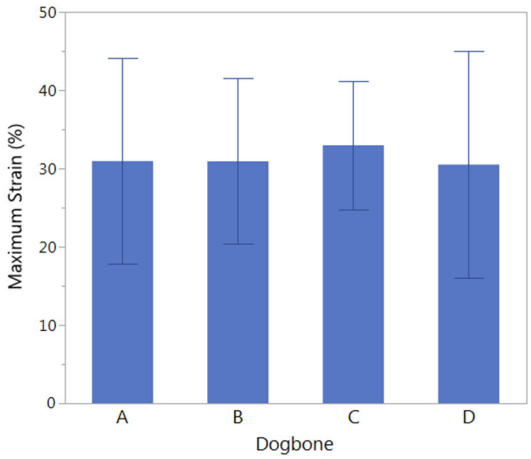

Figure 63. Maximum Strain versus dog bone for third curative run (error bars show one standard deviation)

The average solids percentage versus slice measured by TGA is shown in Figure 65. The TGA results show that the material produced by the CAM-CIP in terms of solids percentage has a high level of consistency.

The average solids percentage versus location is shown in Figure 66. The location results show that the material produced by the CAM-CIP in terms of solids percentage has a high level of homogeneity.

The average density versus slice measured by the ASTM method D792 is shown in Figure 67. The density results also show that the material produced by the CAM-CIP in terms of density has a high level of consistency.

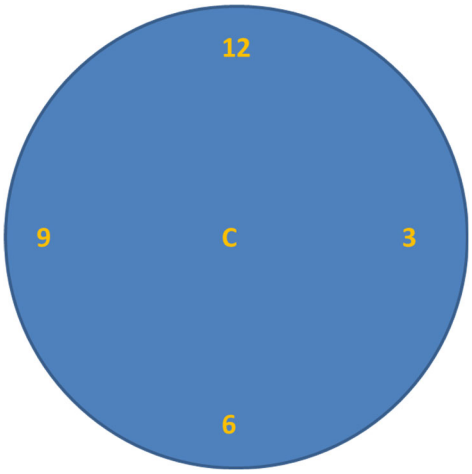

Figure 64. Sample map showing locations of TGA and density measurements

The average density versus location is shown in Figure 68. The location results show that the material produced by the CAM-CIP in terms of density has a high level of homogeneity.

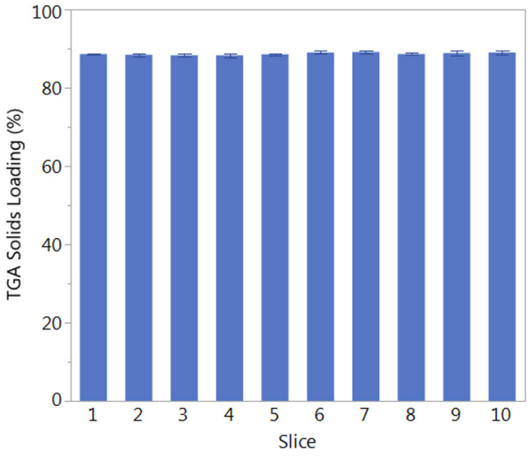

Figure 65. Solids percentage versus slice (time) for fourth curative run (error bars show one standard deviation)

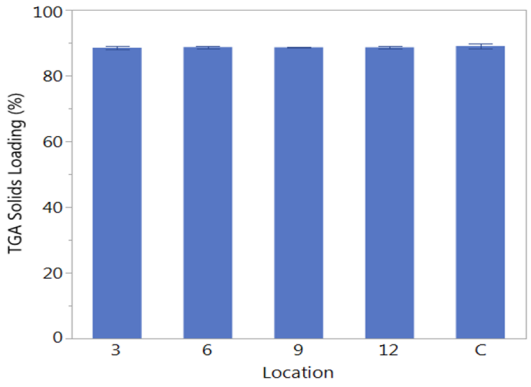

Figure 66. Solids percentage versus location for fourth curative run (error bars show one standard deviation)

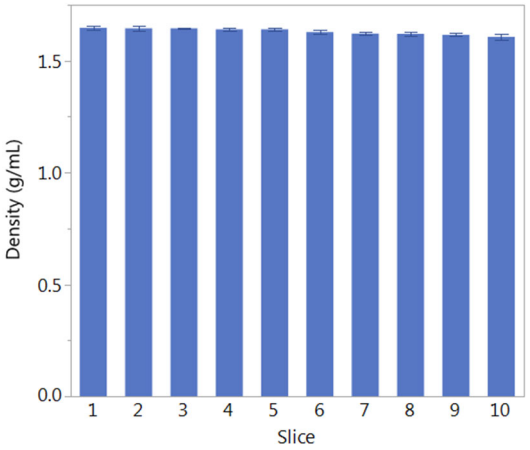

Figure 67. Density versus slice (time) for fourth curative run (error bars show one standard deviation)

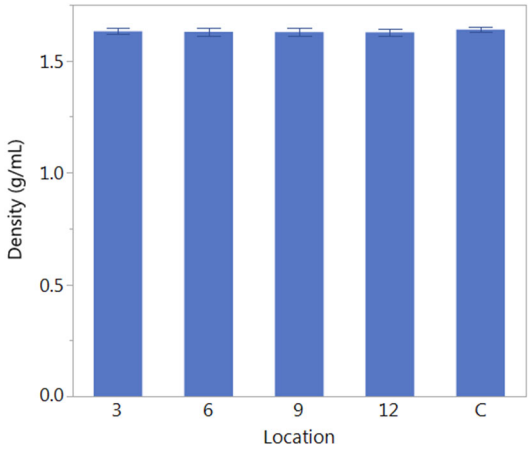

Figure 68. Density versus location for fourth curative run (error bars show one standard deviation)

The average maximum stress versus slice for the first 5 slices (slice is equivalent to time for the collected mixed material) and dog bone are shown in Figures 69 and 70. Only the first 5 slices were tested because the material in the column above slice 5 was not solid enough to make stress and strain measurements possible. The variableness of the maximum stress over time showed that the cured material physical properties were not consistent over time nor did they match the nominal properties expected from a CN/OH ratio of 1.0. The maximum stress versus dog bone data shown in Figure 70 however show that the material was homogeneous.

The average maximum strain versus slice for the first 5 slices (slice is equivalent to time for the collected mixed material) and dog bone are shown in Figures 71 and 72. The variableness of the maximum strain over time showed that the cured material physical properties were not consistent over time nor did they match the nominal properties expected from a CN/OH ratio of 1.0. A Tukey HSD test comparing the maximum strain versus dog bone data shown in Figure 72 however shows that the material was homogeneous.

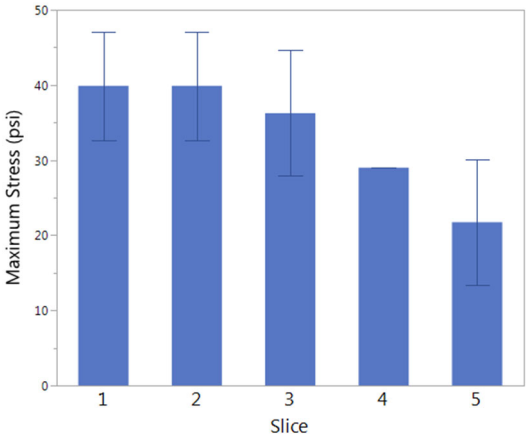

Figure 69. Maximum Stress versus slice (time) for fourth curative run (error bars show one standard deviation)

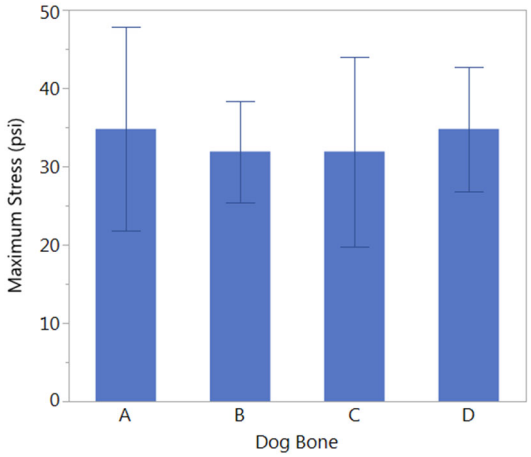

Figure 70. Maximum Stress versus dog bone for fourth curative run (error bars show one standard deviation)

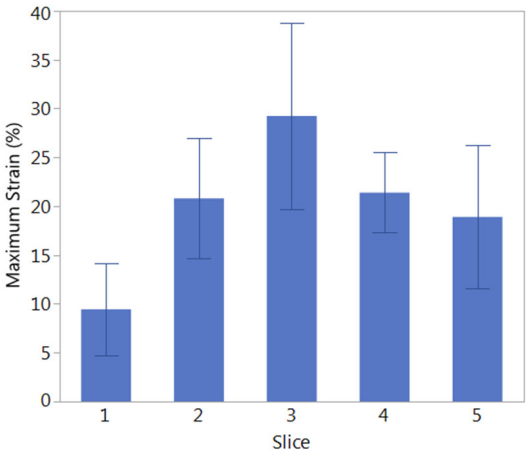

Figure 71. Maximum Strain versus slice (time) for fourth curative run (error bars show one standard deviation)

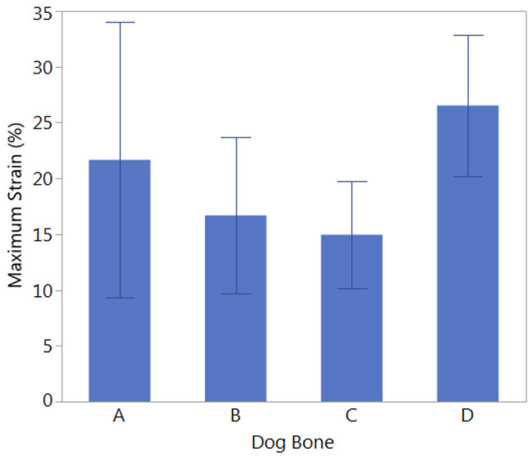

Figure 72. Maximum Strain versus dog bone for fourth curative run (error bars show one standard deviation)

Other large scale batch mixes of surrogate energetic material were produced for other projects at the same time as the continuous runs detailed above. The material was tested in the same manner as the CAM-CIP cured samples. The material created in a batch manner had a high level of consistency according to density, composition, maximum stress, maximum strain, and hardness.

The poor consistency of the continuously produced cured material physical properties were shown to depend on some factor other than the curing oven used, the degassing procedure used, the curing chemicals used, and the batch size. The cause was unique to the continuous process that somehow affected the CN/OH ratio (the ratio of cyano functional groups to hydroxyl functional groups) of the material over time. Because the cyano functional groups are on the molecules of IPDI and the hydroxyl functional groups are on the molecules of HTPB, the effect had to be related to a system involving one or both of those two chemicals.

After discussions with NAWCWD China Lake, the probable cause of the inconsistency in the maximum stress and maximum strain of the continuously mixed and cured material is that the IPDI is reacting with itself or water in the air over the course of a mixing run (3 hours minimum) and lowering the effective CN/OH ratio of the cured surrogate PBX material over time.

Before transitioning the CAM-CIP to China Lake, the method of adding IPDI to the mixture will be modified to correct the issue. IPDI will be separately metered and kept under an inert gas blanket during the mixing run to prevent any competing reaction or change of the CN/OH ratio over time.

Task 4 Optimize CAM-CIP Cleaning Procedure

A CIP procedure, described in section 1.5, was developed for the prototype CAM that produced a 100% clean mixing module. When the CAM-CIP was received 16 February 2019, the same cleaning procedure was to confirm that the procedure still resulted in a 100% cleaning efficiency. The CIP procedure was confirmed to work equally well.

During the 14 runs conducted with the CAM-CIP, the cleaning efficiency and the robustness of the CIP process was confirmed. The CAM-CIP was 100% clean after every run as long as the CAM-CIP was kept air tight during the entire CIP process. There were no failures or anomalies in the testing. If the CAM-CIP was not airtight, the cleaning efficiency would be 99% instead of 100% due to residual material left on the lid similar to early efforts in Task 1.5.

During 3 of the first four runs with the CAM-CIP, a problem was discovered with the gasket seals on the CAM-CIP during the mixing process. When the mixing was started, a small amount of liquid (the liquid was DOA with blue dye and lecithin) would leak from between the CAM-CIP plates where a gasket had slipped. Once the powder flow was started, the leak would stop because the viscosity of the paste was too thick to leak through the small gap between the plates.

When the CIP process was started, the combination of the detergent solution with the motion of the RAM at 95 g of acceleration created a foam as has been described above. When the foam formed, the pressure inside the CAM-CIP increased and forced the foam out of the gap between the plates where the gasket had slipped. This prevented the foam from filling the CAM-CIP above the location of the gap. Therefore, the CAM-CIP was not cleaned above the gap, and the resulting cleaning efficiency would drop to 99% from 100% similar to early CIP efforts during Task 1.5.

The cause of the leaks where the gasket had slipped was determined to occur during the CAM-CIP assembly process. The CAM-CIP is assembled from the bottom plate up with a gasket between each plate or spacer. If, during assembly, the stack moves, the gap between plates could grow too large and the gasket would no longer be fully engaged between two plates allowing the gasket to slip.

To solve the problem, the gaskets were redesigned to be thicker and made with a more compliant material. The gaskets made with the new material stayed fully engaged with the top and bottom of the plates during the CAM-CIP assembly process. Leaks were eliminated, and all subsequent runs proceeded with 100% cleaning efficiency. It should be noted that the gaskets are wear items, and it is required to use new gaskets for each run. It is possible that used gaskets may

not be thick enough or compliant enough to keep from slipping during the CAM-CIP assembly process. For the purposes of gasket replacement, a run consists of a mixing cycle followed by a CIP cycle.

Before transitioning the CAM-CIP module to China Lake, two changes have been proposed. Currently, the line used to deliver the DOA solution is the same line used to deliver the Liquinox solution during the CIP process. The first consequence of using the same line is that an amount of DOA is pushed into the CAM-CIP at the beginning of the CIP process.

The second consequence is that when air is used to blow out the CAM-CIP of foam, an amount of detergent or rinse water that is still in the line is pushed into the CAM-CIP first and is wasted artificially increasing the amount of detergent or rinse water needed. By installing a separate, short as possible, line for the CIP process, DOA, detergent, and water waste will be minimized. The CIP process will also take less time.

The second proposed change is to install an automated blade valve in the powder drop tube and automated valves in the DOA, R45, and CIP lines. The automated valves will allow the CIP process to be performed without any human interaction in an energetic environment

Task 5 Establish Design Requirements for Interface Hardware for Downstream Processing

After material exits the CAM-CIP, it needs to be moved to vacuum degassing equipment. Currently, a flat bellows is used to connect the CAM-CIP exit which is moving under RAM motion to a plate adapter above a conveyor belt.

The current method involves several shortcomings. The system is not sealed and a small amount of splashing can occur during the CIP process. The conveyor belt involves exposed gears that are not safe in an energetic environment. The conveyor belt is being run using a motor that is not Class I Div I compliant, and therefore, cannot be used at China Lake.

The requirements for hardware to transition from the CAM-CIP to the vacuum degassing equipment are Class I Div I compliant, low profile or able to fit between the CAM-CIP and resonator plate, and sealed to prevent material leaks or CIP splashing.

Extra funds have been granted to China Lake to design, fabricate, and test a solution involving a Class I Div I progressive cavity (PC) pump or a vibrating, inclined trough.

Task 6 Documentation of CAM-CIP Operation

The operations manual was developed during the course of the prototype CAM-CIP and CAM-CIP module testing. Input on the mixing and CIP procedure was obtained from China Lake engineers and technicians during their training visit in May 2018.

The operations manual is available concurrently with this report.

Task 7 Installation and Verification of CAM-CIP Hardware and Processes at NAWCWD

At the time of this report, design and development of equipment that will be needed at NAWCWD China Lake to continuously mix energetic material are underway. The processes that are to be automated for energetics mixing are material conveyance from the exit of the CAM-CIP to continuous vacuum degassing, off-specification material diversion, and continuous vacuum degassing.

Task 7 is scheduled to begin in December 2019. Equipment including the CAM-CIP module, the material conveyance system, the continuous vacuum degassing system, and the off-specification material system will delivered to NAWCWD China Lake and installed by Resodyn and NAWCWD personnel.

Task 8 CAM-CIP Production of Live PBX

Production of energetic material is scheduled to begin in March of 2020. Once the CAM-CIP hardware and processes are installed, energetic material will be continuously mixed and produced. The current equipment at NAWCWD China Lake is sized for a continuous run of 76 minutes at flow rate of 1.2 kg/min and will produce 90 kg of material. At the end of the continuous mixing run, the CAM-CIP module will be cleaned in place.

Task 9 Characterize CAM-CIP Produced Live PBX

A main goal and requirement of the project is to continuously mix energetic material equivalent to or better than that produced using legacy methods. Once energetic material is produced in Task 8, it will be tested for homogeneity and consistency.

The continuously mixed material will be tested by measuring the end of mix viscosity, density, composition, maximum stress, maximum strain, and hardness. These same tests were conducted at Resodyn, but the material produced at Resodyn was a surrogate material. Resodyn's testing was to measure material consistency and homogeneity as a response to characterize and optimize the CAM-CIP. The testing on the energetic material will not only confirm the CAM-CIP produces consistent and homogenous energetic material, but that the material produced is equivalent to that produced using legacy mixing processes.

To further confirm the CAM-CIP produced material is equivalent to or better than material produced using legacy systems, the material produced will be tested for thermal stability, accelerated aging, detonation velocity, and detonation pressure tests along with large scale gap testing to assess the performance output with respect to shock sensitivity.

Task 10 Evaluation of CAM-CIP Waste Streams for Live PBX Production

The other goal of the project is to measure the waste stream produced during CAM-CIP continuous production of energetic material including wasted energetic material, waste cleaning solutions, and waste cleaning material. Once the waste streams are measured, the results will be

compared to the same waste streams created during legacy mixing processes to evaluate the environmental and economic benefits of the CAM-CIP.

CONCLUSIONS AND IMPLICATIONS FOR FUTURE IMPLEMENTATION

The RAM continuous mixing process has been successfully developed to mix surrogate PBXN-110 material. As part of the development effort, a CAM-CIP has been designed, manufactured, and qualified at Resodyn. The CAM-CIP was designed to mix energetic material continuously using a RAM 5 resonator platform.

The CAM-CIP was also designed with the ability to control the temperature of the mixed material. The CAM-CIP includes five plates with the ability to independently heat or cool the mixed material on each plate.

The CAM-CIP mixing process has been modeled using a designed experiment methodology for PBXN-110 surrogate material. The experiment investigated the effects of acceleration, solids loading, mass flow rate, and CAM-CIP configuration on the consistency and the homogeneity of the continuously mixed material.

The investigation determined that for a given material rheology (composition, temperature, and shear history) the homogeneity and consistency of the continuously mixed material varied in a predictable but complicated way. At ideal mixing conditions, the homogeneity of the material expressed as the RSD of the solids loading of a sample tested at five different locations is on the order of 0.2%. The consistency of the continuously mixed material expressed as the RSD of solids loading of samples collected every 2 minutes about the average solids loading is less than 1.0%.

The designed experiment also revealed that the processing conditions of solids loading and mass flow rate interact with the acceleration when it comes to the produced material homogeneity and consistency.

A prototype vacuum degassing system was also designed, fabricated, assembled, and tested. The heavily manual operation of semi-continuously degassing mixed material for casting and curing assisted in the development of design requirements for an automated continuous degassing unit.

The degassed and cured material was analyzed for consistency and homogeneity by measuring the physical characteristics of the material. The analysis showed that the method of premixing DOA and lecithin with the curative chemical, IPDI, causes inconsistent maximum stress and maximum strain measurements. However, analysis of the density and TGA measurements showed that the material is well mixed with high levels of homogeneity and consistency. The method of feeding liquids into the CAM-CIP will be redesigned before transitioning the CAM-CIP to NAWCWD China Lake.

The CAM-CIP module was also designed as the central component of a CIP process. The CIP process has been optimized to clean surrogate material from the CAM-CIP with 100% cleaning

efficiency using an aqueous detergent solution. The CIP capability of the CAM-CIP will allow energetics manufacturers to reduce their environmental footprint by eliminating the use of organic solvents from their legacy cleaning processes.

The continuous CAM-CIP process also reduces the amount of waste that is produced during energetics manufacturing. The absolute amount of wasted energetic material is small (nominally half the volume of the CAM-CIP) providing an economic benefit. The continuous RAM mixing process also allows the amount of material wasted during cleaning to be decoupled from the amount of material produced providing additional economic benefit to making larger amounts of material at a time. The amount of PBX and cleaning waste for batch mixing compared to waste from a continuous CAM-CIP process is shown in Figure 73.

Before transitioning the CAM-CIP to China Lake, another liquid delivery line and port in the CAM-CIP will be added to further minimize the CIP waste created and minimize the detergent and rinse water used.

A continuous process also gives manufacturers the ability to make only the amount of material required preventing wasted material required by batch overruns.

The CIP process also prevents workers from exposure to organic solvents and associated vapors that are generally toxic as well as hazardous. The improvement in personnel safety is a large incentive to developing CIP processes, but the reduced amount of personal protective equipment (PPE) and cleaning articles (wash clothes etc.) provide environmental and cost benefits as well.

Phase 3 of the project involves transferring the CAM-CIP to NAWCWD China Lake to demonstrate the continuous mixing process and CIP using energetic material. The CAM-CIP is ready to transfer at this time, however, transitioning the CAM-CIP equipment and process to NAWCWD China Lake facility to mix energetic material will require some development of the current ancillary operations.

First, an improved method of conveying material from the exit of the CAM-CIP to the vacuum degassing operation is required. The method will need to be rated for energetic service and able to handle highly loaded and viscous paste.

Second, a method of diverting off specification material that can be operated remotely is needed. During the startup of the continuous mixing process, a small amount of DOA rich material is produced. The DOA rich material needs to be segregated from the in specification material produced later. The diversion method needs to be energetic material rated, and remotely operated.

Third, a continuous vacuum degassing unit that can handle the nominal maximum flow rate of the CAM-CIP is needed. The prototype unit developed as part of Tasks 1.4 and 3 has a processing rate of approximately 380 gm/min. The development of the prototype degassing unit provided the operation and process requirements for a continuous degassing system. The degassing unit needs to be energetics material rated.

When NAWCWD China Lake is ready to receive the CAM-CIP, Task 7 can begin in earnest. The preparatory tasks of Task 7 have recently been completed including training of NAWCWD China Lake personnel.

The completion of the testing at NAWCWD China Lake and subsequent analysis of the data collected will bring the continous RAM mixing process several steps steps closer to a process qualified for commercial production of energetic materails for the DoD or hard to mix materials in other industries. Other industries that currently use RAM technology for mixing and have expressed interest in a continuous process to meet production goals are pharmaceutical production, reachargeable battery manufacturing, and petrochemical additive formulation.

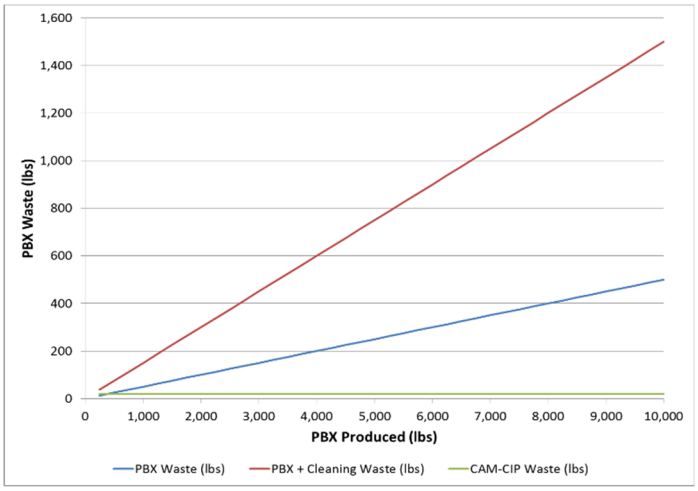

Figure 73. Batch PBX waste and cleaning waste versus waste from a continuous CAM-CIP based process.

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