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SPEEDMIXER PROCESSING OF ENERGETIC MATERIALS

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U.S. ARMY COMBAT CAPABILITIES DEVELOPMENT
COMMAND ARMAMENTS CENTER

Munitions Engineering and Technology Center

Picatinny Arsenal, New Jersey

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14. ABSTRACT Energetic materials present numerous processing challenges. Physical, thermal, and electrical insults can result in material degradation or catastrophic events. SpeedMixers offer unique mixing capabilities that enable safe and rapid combination of formulation constituents. This report will outline SpeedMixer functions, safe operation protocols, and numerous energetic formulations successfully processed with the SpeedMixer.						
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INTRODUCTION

The SpeedMixer, a dual asymmetric centrifuge (DAC) mixer, is a bladeless mixer capable of creating uniform and homogenous blended materials with simultaneous air removal. Two complementary forces are applied to the mixing cup in the SpeedMixer: (1) the centrifugal force from the mixing cup orbiting the SpeedMixer's central axis, and (2) the centrifugal force from the mixing cup rotating around its own central axis in an angular position (fig. 1). These forces create unique flow patterns resulting in superior mixing and disruption of air-cell formation when compared to traditional invasive mixing methods. The SpeedMixer can mix materials with a wide range of viscosities, ranging from water to thick pastes and semi-solids like putties (>1 M cP). Broadly speaking, liquids of all viscosities, pastes, semi-solids, powders, and any combination of these types of materials may be processed.

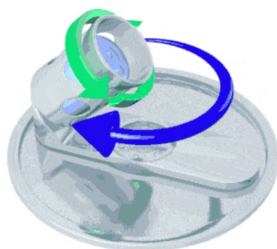


Figure 1
SpeedMixer rotation diagram

The U.S. Army Combat Capabilities Development Command (DEVCOM) Armaments Center (AC), Picatinny Arsenal, NJ, uses vacuum SpeedMixers, which perform this same DAC process but with the addition of an in-situ vacuum inside a chamber within the SpeedMixer to quickly remove all desired volatiles from the mixture. SpeedMixers are capable of mixing at very small scale and can mix batches as small as 100 mg or less depending on the material, cup, and model selection. Mixing at small scale is an attractive method to quickly and safely process experimental materials. Once a desired experimental formulation has been identified, it is easy to scale up to large batch sizes of up to 20 kg. SpeedMixers may be operated with or without vacuum. In the vacuum programmable models, mixing can occur inside a sealed chamber either under a controlled vacuum or under controlled inert gas.

Energetic materials found in explosive, propellant, and pyrotechnic formulations present unique processing challenges. Physical, thermal, and electrostatic insults to the material can create a range of detrimental effects. Minor physical stimuli, such as crystal fractures that alter the particle size¹, may result in benign effects that are manifested by poor or out-of-specification mix products, but they typically do not result in catastrophic failures. If the physical insult is sufficient to initiate the energetic material, catastrophic responses can occur. Effects of thermal insults also depend upon the magnitude of the insult. Minor thermal stimuli typically result in benign responses, potentially causing materials to decompose or evaporate, and thus altering the chemical composition of the final product. Significant thermal events can cause materials to ignite and even experience deflagration-to-detonation transitions (DDT) (ref. 1). Electrostatic effects are less intuitive, more difficult to characterize and observe, and typically result in singular go/no-go events, which also potentially result in catastrophic responses. Safe processing with energetic materials requires careful consideration of all potential sources of stimuli.

¹ Brousseau, P., Personal communication, DRDC Valcartier Research Centre, Montreal, Canada, 2020.

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Formulating raw energetic materials is necessary to stabilize the individual ingredients and enable their use in various military applications. Secondary high explosives are typically processed into three types of formulations: melt-cast, cast-cure, and pressed. Melt-cast formulations involve a solid-phase binder that melts at moderate to high temperatures and the melt phase is typically combined with a nonmelttable crystalline explosive to increase the overall performance of the formulation. These materials are processed at elevated temperature (60 to 110 °C), loaded into fixtures, and cooled to produce a dense, solid mass. Extended processing at high temperatures prevents melt-cast explosive processing with the current configurations of the SpeedMixer. However, some melt-cast materials may be first preheated and quickly loaded and mixed for homogenization. The quick mixing time of the SpeedMixer allows the melt-cast little time to cool down and appropriate processing may be attainable. Cast-cure formulations incorporate solid, crystalline, explosive ingredients into a liquid-phase binder containing reactive moieties that cure to a solid, polymeric matrix upon chemical or thermal initiation. Traditionally, cast-cure formulations are a flowable paste-like consistency prior to cure to allow for gravity-fed or vacuum-assisted loading of the end-items. Pressed explosives are produced in two phases: (1) coating of the solid or crystalline ingredients with a polymeric binder, and (2) high-temperature and/or pressure compaction to produce high-density, uniform billets. Cast-cure and pressed explosives have the possibility to be processed with the SpeedMixer.

Research involving new and novel energetic materials comes with additional processing risks because the materials are sometimes not fully characterized. While iterative, scale-up protocols exist for operations incorporating new materials, assumption of additional risk is necessary while performing initial mixes to produce homogeneous material for evaluation. The SpeedMixer has enabled a level of safe operation within the energetics research field in several ways, most notably by supporting consistent, remote-operated processing of small mixes (5 to 10 g) that historically required hand mixing (ref. 2). This report will discuss the safe operation of a SpeedMixer when processing energetic materials and some successfully employed mix procedures.

SAFE OPERATION

Ingredient Characterization

Characterization of raw ingredients is the primary safety protocol in place to protect energetic materials, processing equipment, and operators. Differential scanning calorimetry (DSC) and thermal gravimetric analysis (TGA) provide indication of thermal stability and threshold temperatures to avoid during processing (ref. 3). Handling hazards are evaluated with friction, impact, and electrostatic discharge (ESD) testing (ref. 3). These preliminary characterizations require only milligrams of material that can be safely handled with proper shielding and personal protective equipment (PPE). Following individual characterizations, combinations of materials are additionally screened for chemical compatibility via DSC experiments. Occasionally, vacuum thermal stability (VTS) is necessary to confirm compatibility and ingredient combination stability. Only materials deemed chemically compatible are mixed above the minimum amounts necessary to execute these tests. Beyond the chemical compatibility of the formulation ingredients, additional compatibilities must be executed for any material that the energetics may contact. Of specific relevance to this report, mix constituents must be tested against polypropylene to ensure compatibility with the standard SpeedMixing cups. If materials are deemed incompatible with polypropylene, mixing cups made of other plastics, glass, metal, or other custom materials may be used (e.g., custom holders to adapt ESD containers).

Electrostatic Discharge Generation

The SpeedMixer is grounded through the housing and mechanical components. However, it is not possible to attach a physical grounding wire to the mix cup due to the rotational motion of the mixing mechanism. Electrostatic dissipative or metal cups may reduce the electrical build-up during mix operations. Additionally, mixes performed under inert, nonoxidative atmosphere (N₂, Ar, etc.) with capable models can help reduce potential ignition sources.

Thermal Considerations

All materials processed in the SpeedMixer will experience a shear-mixing environment due to the counter-centrifugal motion of the mixer. The amount of shear generated is correlated with the viscosity of the in-process mix and the revolutions per minute (RPM) of the mixing vessel, in which higher viscosity materials at higher RPMs will experience significantly higher shear. If the shear environment is not properly controlled, shear mixing can increase the bulk temperature of the mix. To control the temperature of the mix, several best-practice protocols are required. Initially, using minimal amounts of material provides insight into new mixes and provides an opportunity to qualitatively evaluate the resulting viscosity.

In order to properly mix a thick material, one must find the minimum speed that will generate enough force for the material to flow and therefore mix within the cup. Mixing at high speeds for extended periods can potentially generate high temperatures. To avoid high temperatures, start with a process ramp to help determine the minimum speeds and times required to homogenize the desired materials. First, begin with low mixing speeds (e.g., ~1,000 RPM in a DAC-150 mixer) and short mix times (~20 sec). After each mix cycle, check the temperature of the material with an infrared (IR) temperature sensor or thermocouple to ensure that it stays within safe limits. Increase the speed of each cycle by a few hundred RPMs and repeat until the optimal speed is found. A good indication of adequate speed is when the material begins climbing the sidewall of the container (also known as the mix line). Once the mix line begins to form, increased speeds are generally unnecessary and only serve to excessively increase material temperature. Typically, once a mix line forms, only increased time is necessary to achieve full homogenization. It can also be useful to experiment with decreasing speeds once a mix line has formed. In some materials, once the material begins to move and is nearly homogenized, it is possible to decrease speeds further while maintaining the same motion/mixing in the cup, which further decreases the potential for excessive heat generation. All mixes should be processed 50 °C below the decomposition onset temperature for any energetic ingredient, as determined via DSC analysis. If temperature increases are suspected, a cool-down period should be observed post-mix. Cragun et al. presented an example of mix duration, mix speed, and resulting temperatures on a composite formulation (ref. 2). However, it should be noted that all composite mixes behave differently and must be characterized and processed accordingly. When necessary, programming mixing and resting profiles may aid temperature control.

In the case of extreme temperature increases, the mixing cup may act as a failure mechanism. Polypropylene melts at approximately 160 °C and transitions from a hard solid to a rubbery material (glass transition temperature) at approximately 120 °C. At the glass transition temperature, changes to the integrity of the mixing cup will result in weight shifting. This weight shifting will generate unbalanced rotations triggering the automatic shutdown. This fail-safe is only applicable for ingredients with decomposition onsets above 210 °C and should be considered a worst-case-scenario option. Previously mentioned control of the mix cycle speed and duration should be considered the primary control mechanism for shear heating and thermal considerations. A secondary containment system can also be used to help reduce the impact of mixing cup softening inside the machine (i.e., a polypropylene cup can be mixed inside of another mixing cup with FlackTek's cup-in-a-cup holder system).

FORMULATION PROCESSING

Cast-cure Formulations

Legacy cast-cure explosive processing utilizes high-shear bladed mixers, which can suffer from several limitations. These limitations include, but are not limited to, mix homogeneity due to dead spots in the mix vessel and end-of-mix (EOM) viscosity constraints (formulations need to be of a sufficiently low viscosity such that the blades are able to move through material). Generation of the shear regimes necessary to achieve homogeneity requires extremely small blade and bowl separation tolerances and any tolerance alteration can have detrimental effects on the mix. Contact between mixer components can lead to impact or frictional insults sufficient to initiate energetic materials. Inversely, over-spaced mixers will not sufficiently disperse formulation constituents. Additionally, the portion of the mix experiencing the high-shear mixing regime is limited to the space between the blades. Material outside this domain is not aggressively mixed for extended periods. Reduced mixing efficiency extends the processing time for a typical cast-cure formulation to several hours. As such, a wide range of applications has been pursued with SpeedMixing of cast-cure energetic materials. SpeedMixing retains the ability to generate high-shear mixing while eliminating blade tolerance concerns. Moreover, the SpeedMixer mixing mechanism generates a more uniform shear environment capable of achieving uniformity on the timescale of minutes rather than hours required with a bladed mixer.

Bladed mixers also face significant mix quantity limitations. Small amounts of material (<10 g) are of insufficient volume to properly interact with mixing blades. Exploratory mixes, especially with new materials, must be performed by hand prior to scaling to sufficient volumes to process in a traditional bladed mixer. Hand mixes introduce the potential for insufficient homogeneity, potentially affecting analyses associated with the formulation composition. Insufficient homogeneity is especially concerning with complex formulations containing small fractions of ingredients. Furthermore, material stuck to the blades and bowl also generates significant waste losses for the traditional process. Quantities as small as 5 g net explosive weight (NEW) have been successfully mixed in the SpeedMixer with all of the resulting material immediately available for experimental evaluation because the polypropylene cup is disposable. Use of disposable cups also allows for rapid transition between formulations without the risk of mixing vessel contamination.

Binder materials typically include a range of liquid viscosities and, occasionally, some solid ingredients. Investigation of the thermal and mechanical properties of these binder systems can be difficult without homogeneous samples to support consistent data acquisition. The SpeedMixer has been used to process cast-cure binders in two ways: (1) to generate samples for binder studies, and (2) to create homogeneous stock binder blends for incorporation into formulations. Binder studies benefit from the rapid throughput of mixing numerous samples of varying composition (refs. 4 and 5). Use of homogeneous binder stocks ensures that replicate formulation mixes retain a consistent composition rather than suffer batch-to-batch error associated with individual-ingredient measurements.

Processing compositions of liquid and solid ingredients may require trial-and-error alterations to SpeedMixer cycles (speed and duration). While mixes within the weight limitations of the mixer are physically possible, ideal mixing conditions exist with 1/2 to 1/3 of the mixing cup volume filled with material. Typical cast-cure formulations are best processed by loading liquid ingredients into the bottom of the mixing cup followed by large-particle-size solids with smaller particle sizes on the top. Separation of the small-particle-size material from the liquid constituents in the mix cup allows for more rapid processing of the material. Large surface areas of small particles absorb liquids quickly, potentially generating extremely high-viscosity regions early in the mixing cycle that are difficult to disperse. By allowing the liquids to absorb to the larger particle (lower surface area) material first, slower viscosity increases occur during mixing, so homogeneity is achieved faster. Additionally, if the

liquid is loaded on top of the powders, the mixture is more likely to end up with agglomerates due to the liquids being forced downwards at the beginning of the mix cycle, compacting the powders together and making them more difficult to shear apart later in the process. Sealed mixing containers should be employed until all dry ingredients are wetted down through preliminary mix cycles. The addition of vacuum following initial powder wet-out phases can help remove entrained air from dry particle/powder surfaces, thus allowing for quicker and more intimate contact of the liquid with the solid phase. Wetted composition viscosity then dictates the intensity and duration of subsequent mixing necessary to achieve uniformity while avoiding thermal limitations. Vacuum capable SpeedMixers can produce air-free formulations if vacuum is applied to the mix after the wet-down is complete. Energetic composites including nitramines, organic, and inorganic additives, and various liquid binders have been successfully mixed with the DAC-150 and DAC-600 mixers. While numerous mixers exist to produce similar materials, the small-scale (<10 g) mixes have reduced processing time, increased homogeneity and repeatability compared to hand mixes, and enabled remote execution for increased safety.

The SpeedMixer and supporting accessories provide unique processing opportunities for cast-cure explosives. Homogeneous, de-gassed mix products achieved with the mixer may be processed directly inside right circular cylinder containers to support explosive characterization testing. This eliminates the need for transfer of pristine material from a mix cup to a test fixture where void generation or air entrapment can occur. Custom designed holders may be necessary to properly position fixtures within the SpeedMixer basket. Similarly, custom fixtures may be held within the standard mix cups for use of the traditional basket holders referred to as a cup-in-a-cup configuration (fig. 2).

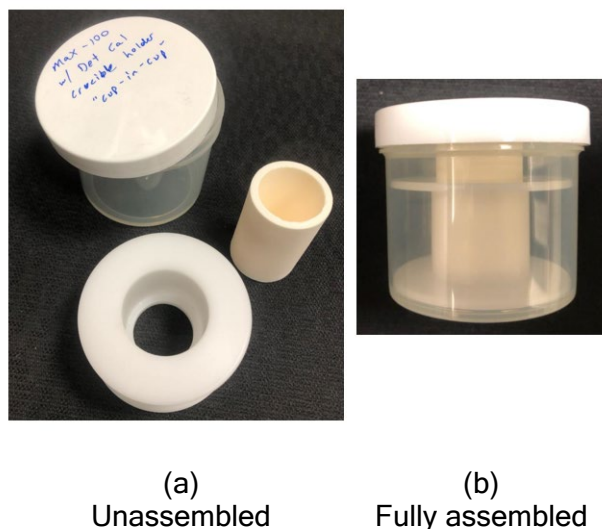


Figure 2
Custom SpeedMixer cup-in-a-cup fixture, (a) unassembled and (b) fully assembled in mixing configuration

Total assembly of the fixtures, holders, and mix constituents must fall within the operational weight for the mixing arm and must not extend above the center of rotation. For test fixtures that exceed the limits of the mixer, post-processing is still necessary. FlackTek SpeedDisks provide a simple method for transferring from SpeedMixer cups to other vessels. SpeedDisks can be customized for a wide range of cartridges, syringes, metal tubes, and other testing fixtures used in the explosives industry. SpeedDisks allow users to transfer fully mixed formulations from as-processed SpeedMixer cups into a syringe/cartridge/canister of smaller diameter while reducing the potential of adding air/bubbles to the system during transfer. This approach, coupled with design of

custom parts, has successfully produced extremely high-quality loadings of numerous cast-cure explosives. Both approaches eliminate the need for vacuum casting material post-mixing.

Pressed Formulations

Pressing powders are produced in large quantities through a slurry coating technique (fig. 3). Solvent-based lacquers of polymeric binder systems are added to solid ingredients suspended in a stirring nonsolvent. Distillation of the lacquer solvent causes binder deposition onto the solid particle surfaces creating agglomerates, which can be filtered and dried to remove all processing liquids. Scaling down this operation to investigate small quantities of new materials suitable for these formulations is difficult because mixing regimes in large vessels are difficult to replicate in small volume reactors.



Figure 3
Slurry still used to produce pressing powders

Reliable screening for pressed formulation ingredients and compositions can be achieved with the SpeedMixer at small scales (5 to 10 g). To do so, only the lacquer solvent is required and no anti-solvent is necessary. Lacquers, solids, and additives are placed in the mixing cup, with the mixing cup having a hole drilled in the top to facilitate escaping solvent vapors. Mixing profiles and observations vary based on the composition being investigated. A standard mix proceeds iteratively with frequent observations of the material consistency directing the intensity and duration of the subsequent mix cycle. Initially, the high-solvent concentration will result in a free-flowing suspension of solid particles. As mixing progresses, and the solvent begins to evaporate, the material will become a soft but solid mass. At this point, the mass behaves similarly to the aforementioned cast-cure formulations where high-shear mixing of a viscous material results in rapid and effective separation of particle agglomerates. Continued mixing and subsequent heating from shear mixing of an increasingly viscous material drives off additional lacquer solvent. When only trace solvent remains, the material will begin to crumble and break into smaller pieces. At this point, mixing is complete, having achieved uniform distribution of all ingredients and evaporation of most processing solvent. The material may be further crumbled manually, panned out, and dried in an oven to ensure complete solvent evaporation.

The final product does not resemble the agglomerated, usually rounded, particles produced by slurry coating. Bulk density and particle-size distributions will differ significantly from those produced in a slurry process. However, the composition of the resulting material will be identical and can be used to support small-scale analyses. Smaller particles, or crumbles, of material are sufficient

for DSC and other thermal analyses. Larger particles may be milled and sieved to achieve desired particle sizes, like those required for hazard testing (ref. 3).

Unique Formulations

Primary Explosive Formulations

Developmental processing of a primary explosive-based slurry is traditionally performed in 3 to 4-g batches requiring a 4 to 5-hr period for each batch. The SpeedMixer has been used to safely prepare slightly larger batches (15 g per batch) of slurry with reduced processing time (under an hour). SpeedMixer processing reduced expensive hazardous material waste generation and eliminated potential batch-to-batch mixing variations. This increase in batch size produces sufficient material to be considered a commercial production lot.

Light-curing Formulations

As energetic material requirements change and evolve, processing technologies need to evolve along with the requirements. One of these evolutions is the need for light-curable binder systems for cast-cure formulations. These binder systems offer unique handling challenges, as the material will cure during loading and mixing if exposed to natural or ambient light. To prevent ambient light from entering the mixing cavity of the SpeedMixer, a light-absorbing filter was adhered to the SpeedMixer's polycarbonate top door. The filter absorbs ultraviolet (UV) light through the blue region, as most light initiating materials react with exposure to this area of the light spectrum. Nevertheless, the filter does not block all the visible light, so visualization of the mixing cavity is still possible with a remote-operated camera. If visualization of the mixing cavity is not required, options exist to use UV-safe black containers or otherwise UV-impenetrable containers. Once the sample is removed from the mixing cavity, polypropylene cups are immediately wrapped in aluminum foil or placed into an ammunition can to avoid any stray light exposures during transportation. With the light filter in place, all standard mixing protocols can be performed on these unique formulations.

Pre-made Formulations

Recently, a need has emerged for the incorporation of inert solid powders into already formulated paste-like explosive materials to perform developmental proof-of-concept testing. For this application, the existing formulation needs to be warmed during the mixing process to allow for ease of powder incorporation. The warming, achieved by DAC shear mixing, reduces the viscosity of the paste-like formulation leading to a more flowable material. To perform the mixing, the paste-like material is added to the mixing cup first due to the fact that it contains the liquid binder constituents, and the solid powder is added on top of the existing material. Multiple mixing iterations, with reconsolidation of the material to the bottom of the cup between iterations, may be necessary for the full incorporation. Final temperature of the fully mixed and incorporated material typically is between 50 and 70 °C.

CONCLUSIONS

Mixing energetic materials with the SpeedMixer is safer, it is more efficient, and it allows for greater creative control than legacy processing. This report outlined key considerations necessary for safe operation of the SpeedMixer with energetics. Cast-cure formulations, including the binder constituents, are most directly applicable to the SpeedMixer, and numerous compositions fit the capability. A modified procedure for producing pressing powders is also sufficient for early-screening experiments and safety analyses. Small-scale mixes, especially those containing new ingredients and novel formulation combinations, benefit the most from this mixing technology.

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FUTURE WORK

Proposed studies for comparing quantitative metrics for mixes performed in the SpeedMixer and a traditional blade mixer are being considered. If the studies receive funding, future results will be published accordingly.

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