Propellants, Explosives, Pyrotechnics

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Resonant Acoustic® Mixing: Processing and Safety

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Abstract: New processing technologies are allowing researchers, industry and academia to probe new materials space not previously achievable. These technologies include additive manufacturing and Resonant Acoustic® Mixing (RAM) which are being demonstrated to reduce processing times, environmental impact and of course cost. With the introduction of any new technology it is imperative that users, managers and national bodies provide the resources and time to determine, understand and provide guidance associated with the safe operating envelope. Combining the RAM with energetic materials requires numerous steps and iterations to generate the required knowledge.

This paper is divided into three parts; the first provides a comparison of existing process technologies for energetic materials and how the community has approached using RAM for each of the energetic material sectors. The second part of the paper provides a summary of a review on how RAM users had provided safety assurance in using the technology with energetic materials. The third part covers how the community is using fundamental and applied research to continue in understanding the technology and where the benefits may lie. It is noted that this field is young therefore information contained herein will change in the future.

Keywords: Resonant Acoustic® Mixing · RAM · safety assessment · mixing classification

1 Introduction

New processing technologies are allowing researchers, industry and academia to probe new materials space not previously achievable. These technologies include additive manufacturing [1] and Resonant Acoustic® Mixing (RAM) [2] both of which have expanded in their use and application over the last 15–20 years. For additive manufacturing alone there are over 171 companies involved in manufacturing, equipment, and software with an increase of 80% in the last year [1]. A subset of additive manufacturing techniques applied to energetic materials manufacture have shown great potential for bespoke properties and performance of energetic materials [3]. For RAM there is only one company involved in the manufacture of the apparatus but the number of applications and publications associated with the technology is constantly increasing; Figure 1.

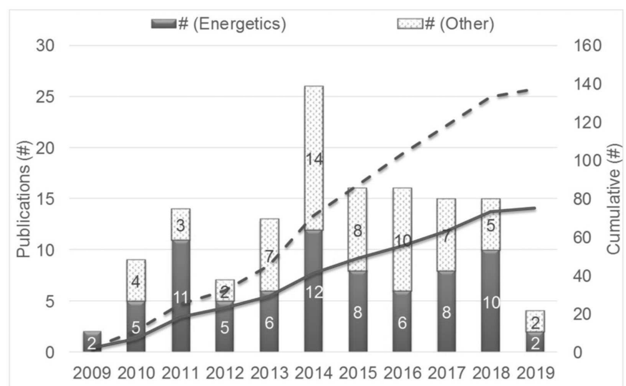

Figure 1. Number of publications associated with RAM.

In the last 10 years the number of energetics-related publications has also slowly increased (Figure 1) as the technology is being accepted by the community. Looking to the energetics related publications, the topics within the field that have been tackled are diverse, from Al/Bi_2O_3 nanothermites [4] to co-crystallisation [5,6], castable propellants [7] to pyrotechnics [8] and Al-ice suspensions [9]. The drive to use a new technology originates from the want to reduce processing times, environmental impact and costs or to access new material formulations not previously achievable. This can be achieved for the RAM so long as it is not being done at the expense of safety.

Each organisation that wishes to use the RAM with energetic materials (EM) has to generate enough information/evidence to satisfy their safety authorities that the process can be carried out safely and protect the lives of the operators. With each organisation starting from the same point,

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albeit with different applications in mind, this process of evidence gathering is being repeated. Several organisations have joined together to better pool their resources and share their experiences of using the RAM with energetic materials. MSIAC has also provided a forum for its member nations to discuss aspects relating to safety and qualification of RAM produced energetic materials.

Combining the RAM with EM requires a comprehensive understanding of the hazards associated with the equipment on the energetic materials and vice versa. This process takes time and resources, but understanding how to effectively process materials on the RAM requires knowledge gained from experience, applied to the generation of theory and tested with simulation. These aspects of safety and design, Figure 2, are part of the wider risk management process that is needed to safely produce energetic materials.

This paper will outline the development of the safe use of RAM technology and the same necessary steps to be applied to any other innovative processing technology to manufacture novel and safe energetic formulations

2 Existing Process Technology

The processing of energetic materials is linked to their family i.e. high explosive, gun propellant, rocket propellant, and their applications. A review of these processes discusses typical operation, advantages and disadvantages for a given family of materials [10]. The example provided in Figure 3 shows the three main types of processing techniques for secondary explosives.

For some materials there are multiple stages that must be carried out to generate the final product. This is best highlighted by the eighteen process steps for the manufacture of cast double base (CDB) rocket motor propellants [11]. The processes illustrated in Figure 3 tend to be the final stage in filling a munition or generating the final shape/form prior to assembly. The differentiating factor for the use of each process is the physical state of the formulation i.e. solid (powder, flake), liquid (molten, viscous polymer).

A definition for mixing is a unit operation that involves manipulation of a heterogeneous physical system with the intent to make it more homogeneous [12]. The initial process of mixing the raw ingredients into a formulation requires the use of a mixer. This stage is generally the most hazardous as raw, energetic materials are being handled and subsequently coated, gelled, melted and/or frozen. These processes are designed to reduce the hazard of the starting materials, improve mechanical properties and provide a material that meets design and performance specifications.

The process of mixing can be classified by the physical materials that are being processed; *Rotational – Blade* (R-B); *Rotational – Vessel* (R-V); *Translational – Vessel* (T-V) [13]. The choice of mixer is intimately linked to the material being processed. Assessments were carried out by several organisations to show that planetary mixers (R-B) [14,15], extrusion mixers (R-B) [16,17], milling (R-V) [18] and bi-axial shakers (R-V) [19] were suitable for use with energetic materials. The hazards included clearance of the blades/screws (where present) to the inside vessel surface, vessel & blade material, temperature control (flow of heat in/out of vessel), electrostatic discharge (ESD), impact, friction, over pressure, and seals for electrical components.

Continuous Mixer & Extruder Users Group (CMEUG) is an example of where, over a period of 20 years, a multinational group of users came together to share their experiences of using twin screw extrusion (TSE) with energetic materials.

Resonant Acoustic® Mixing (RAM), a T-V process, uses distributive mixing and relies on modest frequency and

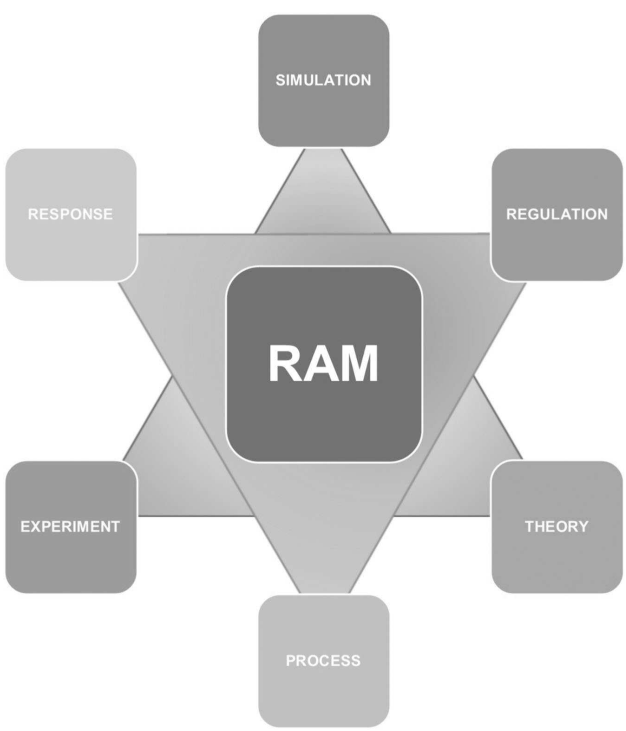

Figure 2. Safety and design considerations for RAM.

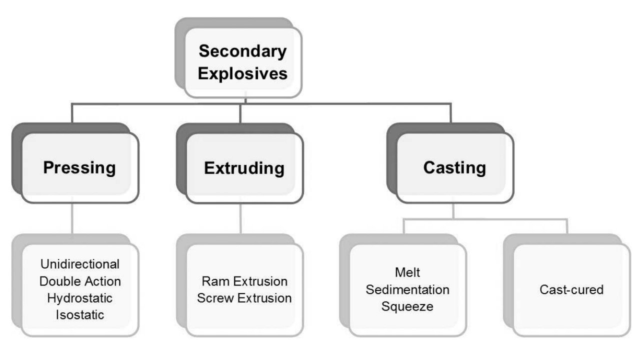

Figure 3. Classification of processing secondary explosives [10].

large displacements of platform to achieve high acceleration (< 100 G). The principle of RAM is to mix by high amplitude oscillation, acoustic streaming and particle collisions. But the most important part of RAM is the resonator. Its aim is to create high amplitude oscillation efficiently. Reaching mechanical resonance is the key because it means reaching a virtually lossless transfer of the mix system's mechanical energy into the material [20].

Several user groups have formed to help the community understand and share information on the use of RAM. These include the Technical Interchange hosted by Resodyn, a UK-based user group, and a US-based user group.

Comparing each family of mixers the translational – vessel does not have any internal moving parts or addition of media to transfer energy to the mix. This is a big advantage with regards to safety. The work carried out by the users have to show that friction, electrostatic and thermal induced initiation are not hazards for a given set of energetic materials.

2.1 Hazard Analysis

So, for a new processing technique or technology, what mechanisms are in place to obtain safety-related evidence?

At the top level there is national **regulation** that provides guidance on how the safety provisions should be met for those who are manufacturing and storing energetic materials. Examples of which are provided in Table 1.

However, the regulations in Table 1 do not articulate how to meet the regulations, just that they must be met. Codes of practice help in providing some practical aspects of meeting legislation.

Processing of **energetic materials** safely requires an understanding of how the energy from the mixer is transferred to the mixed media, what are the modes of initiation and their level of response. Work by Guymon [27] broke the process safety problem down by defining the energy stimuli for normal and abnormal scenarios and conditions. Key parameters for assessment included explosive composition,

Table 1. National Explosives Regulations.

Country	Regulation/Guidance	Reference
Australia	Dangerous Substances (Explosives) Regulations 2004	[21]
France	Référentiel Des Textes Intéressant La Sécurité Pyrotechnique Des Munitions or Guide 17500	[22]
Germany	Explosives Act; Sprengstoffgesetz – SprenG	[23]
United Kingdom	Explosives Regulations 2014	[24]
	Approved Code of Practice (ACOP) to the Manufacture and Storage of Explosives Regulations 2005	[25]
United States	Safe Explosives Act of 2002	[26]

physical states, confinement, and process construction materials. In order to carry out the analysis, data was required on the sensitiveness of the energetic material to the defined hazards, such as friction, impact, electrostatic discharge. The sensitivity and/or reactivity of the energetic materials was also required to understand their propensity to propagate; these tests include pressure/time test, Koenen, Gap test and critical diameter.

Hazards analysis or Process Failure Mode Effects Analysis (PFMEA) can be used to identify and evaluate the potential failures of a **process** [28]. It requires a group approach and an understanding of the equipment/process to identify what might go wrong at the subsequent effect to the process.

Using all three parts, Regulation, Process and Energetics Response, one can assess the suitability of the system for processing energetic materials safely.

3 RAM Safety Assurance

The key aspects for any new process is to understand the critical pathways and where there are gaps in knowledge and understanding. The MSIAC survey was designed to understand how the RAM users approached the safety and control measures to ensure the safe operation of the RAM with energetic material [2].The survey was conducted over a two year period (2015–2017) and had a total of nine respondents. A full analysis can be found within Limited Report L-243 [2].

It should also be noted that the technology has evolved since this work was completed as Resodyn has developed the LabRAM IIH which has integrated engineering solutions to reduce the hazard of mixing energetic materials. This paper and associated reports serve as a reference for new users of the technology. Resodyn supply a range of mixers, from the LabRAM (0.5 kg capacity) to the RAM 55 (420 kg), but only the LabRAM will be discussed within this section.

3.1 Experience and Assessment

All respondents to the survey had experience mixing and processing of energetic materials. The information is summarised in Table 2.

Each facility used different types of mixers, which was dependent upon the type of material being processed or the rheological properties of that formulation. The most common type of mixer being used was the high shear type for processing propellant and high explosive formulations.

Experience gained using traditional mixers is essential for knowing how to handle the energetic materials, the hazards of mixing and what the 'end of mix' looks like. However, the use of new technologies, like RAM, require different methods to assess when a material is completely mixed.

Table 2. RAM Users experience with processes for energetic materials.

Mixer Material	HS[a]	Horiz.[b]	TSE[c]	Anch.[d]	Impell.[e]	Dual Con.[f]	Other[g]
Propellant	6	4	2	–	–	1	–
Pyro.	1	1	2	–	–	–	2
High Explosive	6	5	2	2	2	1	3
Inert	2	2	3	–	3	–	4

[a] High Shear; [b] Horizontal [c] Twin Screw Extrusion; [d] Anchor; [e] Impeller; [f] Dual Conical; [g] E.g. hand mixing

What is the community doing to ensure that the processing technology is suitable and safe for using with energetic materials?

There are two viewpoints here, one from the perspective of the apparatus and the second from the energetic material. For the former, one has to assess whether the apparatus is suitable for handling and processing energetic materials. For the latter, one has to assess if there is an increased risk of initiation of the energetic material.

In the survey we asked the community what the national guidelines were and how they aided in generating a safe system of work for using the LabRAM. Of the respondents, a number highlighted national safety policy (similar to Table 1) whilst others focussed on local policy. A number of respondents quoted defence publications which are equivalent to, or more stringent than, civil legislation. European respondents also pointed to the use of Equipment for Potentially Explosive Atmosphere (ATEX) directive, and the Provision and Use of Work Equipment Regulations (PUWER) for the use of the RAM. For energetics and other chemicals, the Control of Substances Hazardous to Health (COSHH) regulations was used. General risk assessments for workflows were used to identify residual risks and determine whether they were acceptable.

In each case there was a requirement to show that the use of energetic materials within the RAM would not cause harm to the operators. Section 2 discussed the hazards associated with a given process; the following sections demonstrate the output from the assessment against each hazard and the physical methods that the users put in place to reduce risk. Only the LabRAM will be discussed in this section.

Fault tree analysis for the LabRAM was assessed by McPherson [28]. Of the main potential processes that could have led to a failure in RAM the following modes were postulated:

- Electrostatic Build-up/Spark (ESD Hazard),
- Energy Input via Mix – possible initiation of propellant,
- Spill; Container Failure,
- Dust or Explosive Vapor,
- Detonation via Inter-particle Impact.

Although the analysis was performed specifically for propellants, the same hazards can be relevant for all energetic materials. A number of these failure modes will be addressed below. Information in this section references both the MSIAC survey and available information in the open literature.

3.2 Energetic Materials

The overall trend for processing energetic materials using RAM has started with those that present the lowest hazard. This is reflected in the literature with the earliest publications for RAM starting with rocket propellant formulations that are without secondary explosives [28], have low friction and impact sensitiveness, and have high binder/liquid content [$>$16% (w/w)]. Those users that have processed secondary energetic materials have used RDX in mouldable formulations, such as ARX-2045 [29] with a 12% (w/w) Polydimethylsiloxane (PDMS) binder, or castable materials such as thermally-cured RDX/Al polymer bonded explosive [30]. Most recently, non-aqueous slurry mixing to form moulding powders based on HMX and 10% (w/w) Viton have also been trialled [31].

Several pyrotechnic formulations have been reported starting with thermites [4]. More recent reports have described work on flare compositions [8]. Primary explosives have been trialled but again this is fairly recent [32]. The most difficult of material is traditional gun propellant, mixing nitrocellulose with nitroglycerine (NG).

In all but the thermite materials mixing has been carried out in the presence of a phlegmatiser, be that water, another solvent or a viscous polymer. Best practice from conventional mixing has been applied by the users to the RAM.

3.3 Electrostatic Discharge

The largest concern with using the LabRAM was to ensure that there was no build up of electrostatic charge that could cause ignition of the energetic materials. Work was undertaken to understand the potential for RAM to generate static charge [33] in inert but relevant materials. Of the methods trialled to measure static charge generation, the field meter measurements taken above the vessel after a period of mixing were able to detect changes to the electric field. They also showed that charge could be dissipated through the addition of water or routes to ground i.e. earth-bonding to the conductive mixing vessel. The field meter method has previously been used to determine electric fields in high speed slurry mixers [34].

Most RAM formulation work reported in the literature uses a liquid phase that can provide a route for charge dissipation. An exception is thermite mixing which tends not to use a liquid phase; therefore, consideration is needed on their ability to build up charge.

Every respondent had carried out a modification of the apparatus to provide a continuous path from pot to ground. This was achieved through numerous ways including using diagnostics (thermocouples) to act as pathways back to ground. It was noted that several attempts were needed to find the correct solution due to the high G environment and also the large displacement of the vessel (at 60 Hz). In one case bespoke ground-break detection equipment enabled a fail safe (disconnected power to the mixer) should the ground connector break.

Pot/vessel design for most respondents was primarily made from a conducting material e.g. stainless steel, titanium. However, these vessels are not optically transparent making it difficult to observe the process.

3.4 Thermal

Temperature and pressure are parameters that respondents thought required both monitoring and controlling. Mix durations on the LabRAM is much shorter than on conventional mixers. Not all users implemented temperature control due to short mix times. Creating bespoke vessels to control temperature, such as heated/cooling jackets, were only considered by four respondents. Consideration was required for the load/mass of the jacket as this had to be kept to a minimum to maximise mixing load; this being 500 g for the LabRAM.

Most respondents wanted the ability to measure the temperature within the vessel and considered it a high priority for safety diagnostics. Multiple respondents reported using one or more thermocouples that were part of the vessel assembly. These were bespoke vessel assemblies. Some respondents had separate software to record the temperature profile during mixing. One respondent used bespoke software to collate all data output from the LabRAM and ancillary diagnostics.

Pressure considerations included the ability to reduce pressure (vacuum) for the removal of solvents but also to understand the effect on mixing. Vacuum controllers were installed by some respondents. Over-pressure considerations were considered in case of burn/deflagration events; some vessels were designed with weak points to rapidly reduce confinement.

3.5 Impact and Pressure

The potential for the RAM, during operation, to cause ignition or initiation of the energetic materials has been analysed by several users. Work presented by Daniel determined the energy of a single particle of RDX at 100 G to be 2.8×10^{-8} J [35]. This was based on transfer of energy from the RAM, operating at 100 G and 60 Hz. This energy was compared to the 50% initiation threshold value for RDX on the Rotter. The impact energy, based on a single crystal, was shown to be more than 200,000 times that delivered by the RAM. A similar calculation using the BAM data, $H_{50}=7.5$ J for RDX [36], resulted in more than 18,000 times the energy delivered by the RAM.

For the chosen energetic material there appears to be a large safety factor involved which helps to provide confidence in its operation. It must be highlighted that this is only one energetic material, at a given particle size, with assumed high purity (low internal defects). For each material mixed consideration must be given to all ingredients (purity, size and morphology) and their potential interactions, vessel design (material and dimensions), and RAM operating conditions.

Users also considered accidental energetic material release into the RAM. Users covered exposed areas or locations where material could enter the vessel. Some users redesigned their vessel clamping system to minimise the probability of spillage.

Adiabatic compression of a gaseous bubble within the liquid phase is another potential hazard. Specifically for the RAM there has been little reported in the literature on this mechanism, and neither was it mentioned within the survey responses. Looking to the work of Bowden and Yoffe they determined the effect of a 1 mm diameter bubble on the initiation of NG. The bubble, undergoing a minimum compression ratio of 20:1, generates a temperature of 450–480 °C, which is sufficient to initiate an explosion [37]. The time from compression to initiation, caused by a 75 g hammer falling 7 cm, was recorded at 150 µs.

Returning to the RAM, the pressure being generated by the mixing mass, within an operational RAM, changes depending upon the point in the cycle (60 Hz) and the acceleration being applied.

In an attempt to determine whether adiabatic compression of a gaseous bubble could lead to ignition temperatures we have chosen to bound the problem. At maximum acceleration the pressure being applied at the bottom of a vessel can be calculated using hydrostatic pressure $p=\rho.g.h$; where p is pressure (Pa), ρ is density (kg m^{-3}), g is acceleration (m s^{-2}) and h is column height (m). Setting the temperature to 25 °C (298 K) and a mixing vessel (height = 0.2 m) containing a liquid ($\rho_{water}=997$ kg m^{-3}) that is experiencing 100 G, the hydrostatic pressure at the base of the vessel would equate to 195 kPa (1.93 atm). We can approximate the pressure ratio (P_2/P_1) of the gas bubble as 3:1 and applying this pressure to a bubble of an ideal gas, the temperature can be found from $T_2=T_1.(P_2/P_1)^{(\gamma\ -1)/\gamma}$. T_2 is 401 K or 128 °C; where T_1 is the initial temperature (K), T_2 is the final temperature (K), P_1 is the initial pressure (Pa), P_2 is the final pressure (Pa) and γ is the specific heat ratio for an ideal gas (1.4).

For the RAM, operating at 60 Hz, the vessel completes a cycle every 16.6 ms, therefore the calculated temperature would only be experienced for a shorter duration taken to be $^1/_4$ of the cycle, or 4 ms, before the acceleration would decrease, pressure would drop, and the bubble then would perform expansion work and cool. Although this is a rough calculation, it would be expected that the highest cyclic pressures to be experienced by compressible bubbles are roughly an order of magnitude less than the case for NG initiation.

3.6 Facility

Installation of power interlocks or power kill switches was considered normal for these types of operation. These were installed by the users of each facility. The interlocks are there to prevent access to the mixer during its operation. Standard practice for energetics processing is to remotely operate the equipment so that in the unlikely event of an initiation only the equipment is lost. Remote facilities were generally designed to withstand explosive events or have frangible walls that relieve overpressures.

Electrical regulations for explosive facilities prevent the use of standard apparatus (EU-240 V; standard plug) due to conductive conditions and/or specific power requirements. Several survey respondents had to separate loading of energetic materials in a suitable facility for handing explosives from mixing with the LabRAM. Within the time frame of the survey, Resodyn released a hazardous areas approved version, LabRAM IIH, which meets the specification for use with energetic materials.

3.7 System

As part of assurance, many of the respondents carried out daily checks on the LabRAM prior to mixing. This included the grounding (visual or via software) set up, wear of the vessel and its holder. It was mentioned that the vessel clamping system had been re-designed by some users to reduce the probability of the vessel coming off the mixer during operation. Some respondents carried out an empty run of the system before any inert/live work.

3.8 Trials

The use of inert trials was mentioned by all respondents as part of their risk assessment process. Transparent vessels were initially used to understand how the mixer mixed and to observe any potential reactions.

Live trials are always needed to proof the apparatus. Minimising the quantity of energetics is the primary mitigation. Information disclosed in the survey showed starting quantities at 100–1000 mg for were used for evaluation of energetic materials; up to 100 g for formulation development; and up to 500 g for material characterisation.

Development of small mixing vessels (<5 g) were shown to effectively mix materials that would otherwise have been done by hand. Apparent homogeneity of these mixes was better than those done by hand; thermal analysis and SEM was closer to that of the large scale mix [38]. Removing handing mixing is a big step in improving safety to explosive researchers.

One nation carries out development and proof testing using a LabRAM on a range. It is accepted that the LabRAM will be lost should a high order event occur. The results from the successful tests provide confidence in continued use of RAM for those tested energetic materials.

One nation has instituted a safe system of work by generating a living document with the safety authority that allows updates when new information is discovered or there is a change to a process. Flexibility is the key with technology that is new and evolves very quickly so as to be safe but yet not limit the type of work that can be achieved.

3.9 Survey Summary

Research across the respondents is based on the LabRAM. There was a range of processing techniques being using by all respondents on all energetic materials. However castable materials (e.g. rocket propellant formulations) was of greatest interest due to the lower risk of ingredients, high binder percentage and methods to rapidly compare batches. All facilities performed safety assessments with some using national guidelines whilst others use the local or defence equivalent.

Modifications of the LabRAM was universal across respondents, which were necessary to meet safety requirements and/or for specific applications. The major focus was on reducing or eliminating electrostatic charge generation which was achieved through grounding of the vessel/mixer. Other modifications included vessel design, diagnostics and thermal regulation.

Sharing of safety information was considered important by all users.

4 RAM Fundamental and Applied Research

In modern science, all the aspects of a problem can be summarized in three branches: experiment, simulation and theory; Figure 4.

This process for understanding RAM has been reviewed by Wolff [39], which is summarised herein. Experiments, theoretical analysis and computational simulations have been conducted around the LabRAM in order to optimize the material's performance.

4.1 Experimentation

A lot of experimentation has been focused on determining the end of mix and homogeneity of the final product. Near infra-red chemical imaging (NIR-CI) is one of the technique

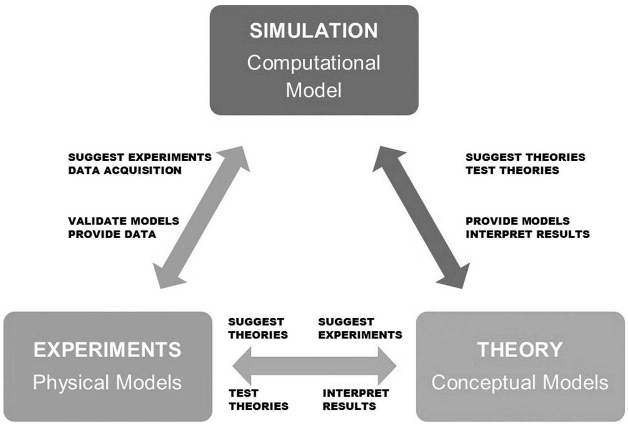

Figure 4. Model of modern science.

that can be used to see the degree of agglomeration but requires direct access to the material. Ultrasound is being tested and correlated against known completely mixed materials [40]. This has led to the development of embedded sensors, external sensor, and sampling/compositional analysis. Other experiments included beam line radiography/tomography, X-ray diffraction, NIR-CI and other imagery techniques [40].

Most of the experiments that were conducted by the organizations tend to compare the performance of conventionally mixed with Resonant Acoustic mixed energetic materials. Organisations tend to agree on several aspects:

- RAM provides a more homogeneous mix [31,41],
- RAM is faster than conventional mixing, yet producing a similar product [2],
- Similar density can be found [42] and less voids are observed with the RAM,
- Similar safety properties are observed in terms of impact, friction and ESD [43],
- Similar performance can be seen with RAM [40],
- Similar sensitivity of the final product [44].

4.2 Theory

The mixing behaviour depends on the design and process parameters, the material properties of the raw materials and the main mixing mechanism of the blender. So comparing conventionally mixed materials with RAM produced materials does not provide useful information for the optimization of RAM as the operation and design are drastically different. Experiments only show how promising RAM could be as it shows similar results with conventionally mixed energetic materials without having been optimised.

Understanding the theory is a necessary step in order to provide better properties. In a review of Resonant Acoustic Mixing by Wolff [45], the different mixing regimes were described in detail. Mixing regimes affect macromixing characteristics and their parameters are summarized in Table 3.

There are then many important process parameters that affect macromixing states which have to be taken into account in the mixing. Some experimental approaches have tended to describe the interactions and influence of each parameter. There are multiple methods like analysis of variance (ANOVA) for example that measure the influence of a set of chosen parameters [46].

The wetting step in a solid-liquid mixing is crucial for the final product. It impacts agglomeration and homogeneity of the mix. Micromixing characteristics, such as cohesion, flowability, or shape of the particle, have a great influence on the wetting. Incomplete wetting could lead to voids, cracks and inhomogeneities.

4.3 Simulation – Computational/Numerical

Simulations can be used to understand and visualise the effect of macromixing and micromixing parameters. First, a turbulence model has to be chosen depending on the value of the Reynold's number. This model is based on continuity, momentum, and heat transfer equations. Once the

Table 3. Summary table on mixing regime parameters [45].

Mixing	Solid-Solid	Solid-Liquid	Liquid-Liquid/Gas
Dominant phenomenon	Particle-particle collisions	Acoustic stream	Faraday instabilities
Important parameters to establish a mixing regime	Acceleration Fill ratio Aspect ratio Vacuum Pressure	Acceleration Fill ratio Viscosity Order of ingredients Aspect ratio Vacuum	Acceleration Fill ratio
Scale-up parameters	Height of bed Transducer area Number of transducers Density Elasticity Pressure	Partial vacuum	No data

turbulence model is chosen, some auxiliary equations can be added to the model.

In all cases, the mixing entails multiphase flow. So each phase is assigned a separate set of properties. Gravitational and centrifugal forces can cause separation between the different flows. Several multiphase models exist such as Dispersed Phase Model, VOF Model, Eulerian Multiphase Model, Eulerian Granular Multiphase Model and Algebraic Slip Mixture Model.

There are many numerical methods that can be applied: continuous media models (Finite Volume Method, Finite Element Method…) and discrete particles models (Molecular Dynamic, Discrete Element Method…) [47]. Le Bouchet Research Centre (ArianeGroup/CRB) for example uses Finite Volume and finite difference to model the inside of a RAM (M. Plaud and A. Aumelas, Personal Communication). Similarly, Nance [48] details a second step made toward a high fidelity, first principles numerical simulation of the mixing for the resonant acoustic mixer. The computer code was LESLIE3D (Large Eddy Simulation with LInear Eddy modelling in 3 Dimensions) which was developed by Suresh Menon at the Georgia Institute of Technology. LESLIE3D has been modified to produce the acoustic excitation required for the mixer. More interestingly, some organisations have started making RAM simulations using a discrete particle model [49]. This method allows the interpenetration of a liquid phase with a solid phase.

Although a lot of work has been done, all these simulations and experiments still have to be improved and scaled up for larger units such as RAM 5 or RAM 55. Organizations are still at the beginning of understanding the RAM process.

5 Conclusion

By classifying existing processing technologies for energetic materials and comparing the hazards for each we were able to show how translational – vessels had advantages through the absence of blades or mixing media to transfer energy to the mix. The method of mixing differs across the techniques but the hazards for the energetic materials are similar. Some methods, such as TSE, have proven to be difficult to use and to process materials safely. However TSE has been an example whereby users coming together have benefited from shared experiences. These lessons learnt are invaluable for RAM and their users. Sharing of experience, especially those relating to safety, are vital so that the same mistakes are not made by different groups.

The summary of the MSIAC survey on how RAM users provide safety assurance in using the technology with energetic materials has highlighted areas for consideration by other users of the equipment. The generation of electrostatic charge and the potential subsequent discharge was the biggest concern for users and great efforts have been made to ensure the system has an available route to safely discharge. This is very much material specific and a full assessment should be carried out on a user's specific application.

Although experiments showed satisfying results in terms of performance, the community must use fundamental and applied research to continue to understand the technology. Indeed, macromixing parameters that impact mixing regimes play a large role in designing an adequate processing cycle. Micromixing parameters, on the other hand, are key to the quality of the final product. The global aim will then be to move from a trial and error process to a scientific-based assessment. Work on computational simulations is being conducted to meet this goal and to be able to optimise the technology.

This paper outlines the development of the safe use of RAM technology. The same necessary steps can be applied to any other innovative processing technology to manufacture novel and safe energetic formulations. Indeed, the radical changes in our way of designing new energetics must not be done at the expense of safety.

Symbols and Abbreviations

ANOVA	Analysis of Variance
ATEX	Explosive Atmosphere
CDB	Cast Double Base
CMEUG	Continuous Mixer & Extruder Users Group
COSHH	Control of Substances Hazardous to Health
EM	Energetic Materials
ESD	Electrostatic Discharge
NIR-CI	Near Infra-Red Chemical Imaging
NG	Nitroglycerine
PDMS	Polydimethylsiloxane
PFMEA	Process Failure Mode Effects Analysis
PUWER	Provision and Use of Work Equipment Regulations
RAM	Resonant Acoustic® Mixing
RDX	1,3,5-Trinitro-1,3,5-tetraazacyclohexane
SEM	Scanning Electron Microscopy
TSE	Twin Screw Extrusion.

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References

[1] AMFG, *The Additive Manufacturing Landscape 2019*, White Paper, AMFG, London, UK, **2019**. https://amfg.ai/whitepaper-the-additive-manufacturing-landscape-2019/ (Accessed 09 May 2019)

[2] M. Andrews, *ResonantAcoustic(R) Mixing – Survey*, Report L-243, MSIAC, NATO HQ, Brussels, BEL, **2019**.

[3] A. Wolff, A. Andrews, C. Collet, Additive Manufacturing Processes Applied to Energetic Materials, *16th Rapid Design Prototyping & Manufacturing Conference*, London, UK, April 4–5, **2019**.
[4] R. Nellums, B. Terry, B. Tappan, S. Son, L. Groven, Effect of Solids Loading on Resonant Mixed Al–Bi2O3 Nanothermite Powders, *Propellants, Explos., Pyrotech.* **2013**, *38*, 605–610.
[5] D. am Ende, S. Anderson, J. Salan, Development and Scale-Up of Cocrystals Using Resonant Acoustic Mixing, *Org. Process Res. Dev.* **2014**, *18(2)*, 331–341.
[6] K. Hope, H. Lloyd, S. Konar, C. Bull, C. Pulham, Putting the squeeze on energetic co-crystals: High-pressure studies of 2 (CL-20):HMX and CL-20:TNT, *New Trends Res. Energ. Mater., Proc. Semin., 20th*, Pardubice, CZE, April 26–28, **2017**, 184–192.
[7] A. Nelson, The Advantages of ResonantAcoustic Mixing (RAM) For Making Novel High-Energy Composite Materials, *RAM Technical InterChange*, Butte, MT, USA, August 3–6, **2015**.
[8] C. Yamamoto, E. Miklaszewski, J. Dunham, A. Shaw, Issues and Observations During Scale Up of Pyrotechnic Flare Compositions in Resonant Acoustic Mixers, *43rd International Pyrotechnic Society Seminar*, Denver, CO, July 8–13, **2018**.
[9] T. Pourpoint, T. Wood, M. Pfeil, J. Tsohas, S. Son, Feasibility Study and Demonstration of an Aluminum and Ice Solid Propellant, *International Journal of Aerospace Engineering*, **2012**, *2012*, 1–11.
[10] A. Wolff, *Common Processes Used for Explosives and Propellants*, Report L-240, MSIAC, NATO HQ, Brussels, BEL, **2019**.
[11] J. M. Tauzia, *À propos des matériaux énergétiques*, Report SNPE Matériaux Énergétiques, Sourges, FRA, **2006**.
[12] A. Fadeyibi, Z. Osunde, G. Evans, E. Evans, Mixing Index of a Starch Composite Extruder for Food Packaging Application, in: *Green Polymer Composites Technology: Properties and Applications*, (Ed: Inamuddin), CRC Press, **2016**, p. 591.
[13] M. Andrews, C. Collet, A. Wolff, C. Hollands, *ResonantAcoustic (R) Mixing: Processing and Safety*, Report L-244, MSIAC, NATO HQ, Brussels, BEL, **2019**.
[14] P. Pelletier, J. Drolet, *Curable Plastic Bonded Explosive (CX-84A) – Production Scale-Up*, Report M-2673/84, DREV, Quebec, CAN, **1984**.
[15] W. Haite, The Slurry Mix Process for Nitroplasticol Propellant, *5th Annual Explosives Safety Seminar on High Energy Solid Propellants*, Santa Monica, CA, USA, August 20–22, **1963**, 174–185
[16] M. Zebregs, C. Schoolderman, C. van Driel, Scaling Up of Gun Propellant Co-Extrusion Using Simulation Software, *Int. Annu. Conf. ICT, 38th*, Karlsruhe, DEU, June 26–29, **2007**, P113.
[17] J.-M. Tauzia, P. Penel, G. Maller, Continuous Twin Screw Processes: Evolution of the Design of the Extruders in Regards to Safety Concerns, *12th Continuous Mixer & Extruder Users Group*, Indian Head, MD, October 29, **2002**.
[18] E. Gray, H. Marsh, M. McLaren, Review: A Short History of Gunpowder and the Role of Charcoal in its Manufacture, *J. Mater. Sci.* **1982**, *17*, 3385–3400.
[19] P. Deacon, Nitrocellulose Processing in a Flammable Solvent: Designing a Safer Process, *7th Nitrocellulose Symposium*, Montreal, CAN, May 31–June 2, **2016**.
[20] B. Seaholm, RAM Mixer Technology Controls Introduction and Control at Resonance, *6th Resodyn Technical Interchange*, Butte, MT, USA, September 19–23, **2016**.
[21] ACT, Dangerous Substances (Explosives) Regulations 2004, *Australian Capital Territory*, **2004**.
[22] DGA, Référentiel Des Textes Intéressant La Sécurité Pyrotechnique Des Munitions, *Ministère Des Armées*, **2019**.
[23] German Government, Sprengstoffgesetz – SprengG, *German Government*, **1976**.
[24] HSE, Explosives Regulations 2014: Safety Provision, *Health & Safety Executive*, UK, **2014**.
[25] UK Government, The Manufacture and Storage of Explosives Regulations 2005, UK, **2005**.
[26] USA, Safe Explosives Act of 2002, United States Code, **2002**.
[27] C. Guymon, In-Process Classification of Explosives, *Improved Explosives & Munitions Risk Management (IEMRM) MSIAC Workshop*, Granada, ESP, September 10–14, **2018**.
[28] M. McPherson, Resonant Mix Process Development for Castable Propellants and Related Energetics, *4th Resodyn Technical Interchange*, Butte, MT, USA, September 17–21, **2014**.
[29] A. Provatas, C. Wall, Development, Characterisation & Ageing of an Alternative Plastic Explosive, *Int. Annu. Conf. ICT, 48th*, Karlsruhe, DEU, June 27–30, **2017**, P108.
[30] R. Davey, J. Wilgeroth, A. Burn, New Age of PBX Manufacturing: Optimisation of RAM, *Int. Annu. Conf. ICT, 49th*, Karlsruhe, DEU, June 26–29, **2018**, P123.
[31] E. Beckel, Environmentally Friendly Energetic Processing Via Resonant Acoustic(R) Mixing, SERDP & ESTCP Webinar Series, **2018**.
[32] M. Puszynski, R. Cook, D. Perkins, G. Cheng, N. Mehta, Using RAM Technology to Mix Primary Explosive Based Composites for Additive Manufacturing, *43rd International Pyrotechnic Society Seminar*, Denver, CO, July 8–13, **2018**.
[33] I. Pavey, *Preliminary Electrostatic Investigations: Measurement and Monitoring*, Report Dekra Insight, **2017**.
[34] I. Pavey, Hazard Assessment of High Speed Slurry Blending Using Computer Modelling of Electric Fields and Potentials, *J. Phys.: Conf. Ser.* **2015**, *646*, 012023.
[35] M. Daniel, Dry Polymer Coating of Energetic Materials: Feasible or Futile?, *5th Resodyn Technical Interchange*, Butte, MT, USA, August 4–6, **2015**.
[36] M. S. Gruhne, T. M. Klapötke, M. Lommel, M. H. H. Wurzenberger, N. Szimhardt, J. Stierstorfer, Impact Sensitivities of Explosives Explored by the OZM Ball Drop Tester (BIT-132), *New Trends Res. Energ. Mater., Proc. Semin., 22nd*, Pardubice, CZE, April 10–12, **2019**, 410–417.
[37] F. P. Bowden, A. D. Yoffe, Chapter III: Initiation by Impact of Explosion in Liquids, in: *Initiation and Growth of Explosion in Liquids and Solids*, Cambridge University Press, Cambridge, UK, **1952**, p28–55.
[38] A. Nelson, M. Celestine, M. Bird, W. Merriman, D. Turnbaugh, M. Siirila, K. Shermer, Resonant Acoustic Mixing (RAM) of High Energy Composite Materials, *6th Resodyn Technical Interchange*, Butte, MT, USA, September 19–23, **2016**.
[39] A. Wolff, M. Andrews, C. Collet, *Resonant Acoustic Mixing Performance and Optimization for Energetic Materials*, Report L-246, MSIAC, NATO HQ, Brussels, BEL, **2019**.
[40] D. Jubb, The Falcon Project, *1st MSIAC Technical Meeting: Impact of Resonant Acoustic Mixing (RAM) on Munitions Safety and Suitability for Service*, Portland, OR, USA, April 17, **2018**.
[41] A. Nelson, "Mix-in-Case Green Processing With Resonant Acoustic Mixing", SERDP & ESTCP Webinar Series, **2018**. https://www.serdp-estcp.org/Tools-and-Training/Webinar-Series/05-31-2018 (Accessed 09 May 2019)
[42] M. Zebreg, RAM work at TNO, *2nd MSIAC Technical Meeting: Impact of Resonant Acoustic Mixing (RAM) on Munition Safety and Suitability for Service*, Shrivenham, UK, July 4–5, **2018**.
[43] E. Beckel, K. Lee, J. Marin, A. Shah, Processing of Explosives at ARDEC Using the LabRAM, *6th Resodyn Technical Interchange*, Butte, MT, USA, September 19–23, **2016**.
[44] V. Komanschek, Comparison of Gap Test Results of PBX KS 33: RAM vs. Planetary Mixer, *2nd MSIAC Technical Meeting: Impact*

of Resonant Acoustic Mixing (RAM) on Munition Safety and Suitability for Service, Shrivenham, UK, July 4–5, **2018**.

[45] A. Wolff, M. Andrews, C. Collet, *Resonant Acoustic® Mixing Applied to Energetic Materials*, Report L-245, MSIAC, NATO HQ, Brussels, BEL, **2019**.

[46] J. Osorio, *Macro and Micro Characterization of Powder Mixing Processes*, PhD Thesis, Rutgers, The State University of New Jersey, NJ, USA **2014**. Retrieved from https://doi.org/doi:10.7282/T3NK3CB1

[47] S. Zhu, *Particle Dynamics Simulation toward High-Shear Mixing Process in Many Particle Systems*, PhD Thesis, Columbia University, NY, USA, **2018**. Retrieved from https://doi.org/10.7916/D89G74QC

[48] D. Nance, *An Examination of the Resonant Acoustic Mixer's Flow Field*, Report, AFRL-RW-EG-TR-2013-108, AFRL, Eglin Air Force Base, FL, USA, **2013**.

[49] J. Osorio, F. Muzzio, LabRAM Characterization Studies, *RAM Energetics Conference and Workshop*, Butte, MT, USA, July 14, **2011**.

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