

Synthesis of Highly Loaded Gelled Propellants

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Abstract

A new method for preparing gelled propellants containing nano-particles has been demonstrated. Typical propellant fuels, nano-particles and gelling agents were chosen to establish capability of ResonantAcoustic[®] technology to produce viable gelled propellants containing energetic metal and semi-metal nano-particles. A variety of high-solids containing gelled propellants were synthesized. Their rheology, stability and combustion characteristics were measured and compared to gelled propellants produced using conventional propeller mixing techniques.

Keywords: Gelled Propellant, Energetic particles, Mixing, ResonantAcoustics

1. Introduction

The purpose of this project was to demonstrate the feasibility of using ResonantAcoustic[®] mixing technology to synthesis gelled propellant fuels with high energetic-solids content. The formulation of these energetic enhanced fuel gels consisted of a combination of fuel, gellant, surfactant and nano-particle energetics. Two fuels were utilized in this project: kerosene and Dimethylaminoethylazide (DMAZ). The kerosene is representative of a wide variety of propellant fuels currently in use, such as

RP-1 and JP-5. The DMAZ fuel was developed by the Army as a potential replacement for Monomethyl Hydrazine (MMH). The DMAZ is less volatile and less toxic than the MMH fuel. Fumed silica was the gelling agent used throughout. The fumed silica is challenging to use in conventional mixing operations due to its very low bulk density and poor wetting properties, which makes it difficult to mix. A surfactant was used to help wet the nano-particles to aid in the uniform dispersion in the gelled propellant. Two types of nano-



Figure 1. Gelled propellant fuels, both neat and mixed with nano-particles. Pictured from left to right are kerosene, kerosene/nano-aluminum, kerosene/nano-carbon, DMAZ, DMAZ/nano-aluminum, and DMAZ/nano-carbon.

particle were incorporated into the mix formulas; nano-aluminum and nano-carbon. Figure 1 shows examples of some of the gels produced.

Metallized gelled propellants have been studied analytically and experimentally for over forty years. The historical work has focused on the benefits of high specific impulse, high density, and safety [1-5]. Metallized propellants are liquid propellants that contain metal particles. These particles are suspended in a gelled fuel or oxidizer. Aluminum, Boron, Carbon and Magnesium are the most commonly used metals used to synthesize gelled propellants. Typically, metal powder used in making metallized propellants, is in the form of micron-sized particles. The liquid propellant is gelled with an additive that is a very small fraction of the total propellant mass. These metallized propellants have the ability to increase engine specific impulse, increase propellant density and increase system safety [1].

Experimental missiles utilizing gelled fuel and gelled oxidizer combinations have proven to be very advantageous. The Future Missile Technology Integration (FMTI) program demonstrated numerous benefits of gelled propellant including extended range, improved kill power, greater mission flexibility, and lower operational costs [6].

The mixing of gels using conventional technology is problematic. As shown in Figure 2, gelled propellant is stiff, hence difficult to fold into the mixing zone

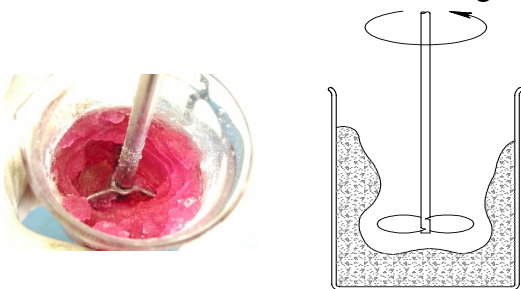


Figure 2. Conventional mixing of fumed silica gelled kerosene.

of conventional mixing system impellers

As shown in Figure 3, state-of-the-art gelled propellant mixing systems are complex. Their limitations include:

- Long total process times
- Poor homogeneity
- Rotating seals
- Product difficult to extract
- Internals (impellers, shafts, etc.) hard to clean
- Hazardous material handling problems
- Product waste



Figure 3. Multi-axis mixer required for non-Newtonian and high density mixtures.

This work presents a novel mixing technology that has been proved to be an advanced technology for mixing gelled propellants that overcomes the issues associated with conventional mixing.

Resodyn Corporation's ResonantAcoustic[®] agitation technology is a new approach to solving mixing and dispersion problems that is uniquely different from either conventional impeller agitation or ultrasonic agitation. Rather than mix by inducing bulk fluid flow, as is the case for impeller agitation, ResonantAcoustic[®] agitation mixes by inducing micro-scale turbulence by the propagation of acoustic waves throughout the medium (see Figure 4). ResonantAcoustics[®] is different from ultrasonic agitation because the frequency of acoustic energy is two orders of magnitude lower and the scale of mixing is two orders of magnitude larger. Another distinct

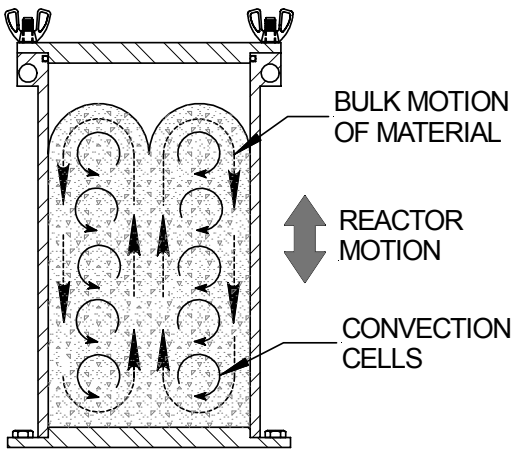


Figure 4. Schematic showing bulk motion of highly loaded mixtures.

difference from ultrasonic technology is that the ResonantAcoustic[®] devices use mechanically driven agitators that can be made large enough to perform industrial scale tasks at reasonable cost.

2. Experimental Procedure

The criteria for selection of liquid propellant fuels considered both how representative the fuel was of the current family of fuels in use and how safe the fuel was to handle.

Two liquid fuels were chosen, kerosene and DMAZ. The fuel used in the initial stage of the project was consumer grade kerosene with a red dye marker. The kerosene was considered a good surrogate for the very

extensively used RP-1, which is basically refined kerosene [7,8]. At a later stage in the project an additional fuel, dimethyl-aminoethylazide (DMAZ) manufactured by 3M, was used as well. The DMAZ was developed by the Army as a substitute for the popular Army propellant, Monomethylhydrazine (MMH). The DMAZ is considerably less volatile and less toxic than the MMH [9,10]. The fuels and mix ratios are listed in Table 1. All gels were created using Cab-O-Sil EH-5 fumed silica as the gelling agent. The Initial mixes were simple gels containing no additives (Mix 1,2 and 10).

A variety of particle additives were incorporated into the project. Early mixing trials (Mix 3 and 4) used powdered aluminum oxide. The alumina had a particle mean diameter of 50 nm. Another mix trial (Mix 5) used course ground aluminum powder with a size distribution of roughly 25 to 250 micron particles. Finally, the materials of primary interest were the high energy density nano-particles of carbon and aluminum (Mix 6 to 9, Mix 11 to 13). The carbon used was Cancarb THERMAX Floform N990 Thermal Carbon. This material has a mean diameter of 240-320 nm and a BET surface area of 7 to 11 m²/g [11]. The majority, roughly 80%, of the particles are spheroidal or ellipsoidal in shape. The

Table 1. Phase I metallized gel propellant mixing trials, weight fractions (wt%) of ingredients.

Mix #	1	2	3	4	5	6	7	8	9	10	11	12	13
Kerosene	91.7	89.0	74.3	74.0	61.7	57.6	64.4				64.8	57.6	
DMAZ								61.8	58.5	89.3			58.5
Alumina			25.7	21.9									
Micro-Aluminum					30.8								
L-Alex nano-Aluminum							29.3	30.9			28.8		
N990 Carbon						36.9			35.1			36.9	35.1
EH-5 Fumed Silica				3.4	6.6	4.6	5.5	6.2	5.4	10.6	5.5	4.6	5.4
PST	8.3	11.0		0.7	0.9	0.8	0.8	1.0	0.9		0.8	0.9	0.9

aluminum used was L-Alex, a passivated nano-aluminum produced by Argonide Corporation. The L-Alex has a mean diameter of 100-200 nm and a BET surface area of 12.3 m²/g [12]. Additionally, a non ionic surfactant Polyoxyethylene sorbitan trioleate (PST) was used in some cases. Surfactants were indicated by the literature to be helpful wetting agents that allowed better dispersion of the metal particles through out the mixture [13].

2.1 Experimental Apparatus

The experimental apparatus utilizing the ResonantAcoustic[®] technology is shown in Figure 5. The apparatus consist of an acoustic energy source, an interface superstructure for attaching to the acoustic driver and the cylindrical reactor vessels. The reactor vessel has no internal moving parts or baffles. Two different mix vessels were used, a nominally 10 cm I.D. by 28 cm



Figure 5. The ResonantAcoustic[®] apparatus shown with a borosilicate glass reactor vessel on the left and a stainless steel reactor vessel on the right.

tall borosilicate glass cylinder, or a 11 cm I.D., 25 cm tall stainless steel cylinder as shown in Figure 5.

2.2 Mix Procedure

A spread sheet was developed based upon the desired mix ratios of the constituent ingredients. The spread sheet, see Table 1, lists the amount by weight of each ingredient required for a specific mix. The ingredients were weighed out to the nearest 0.1 gram and added to the mix vessel. First the fuel was added, then the surfactant, then the metallic particles, and finally the fumed silica. At this point the mix vessel was sealed and the acoustic power source was activated. Mixes were typically run for 10 minutes. Some of the earlier mixes were frequently interrupted to allow for closer examination of the mix and to gage the progress of the gelation reaction. This was especially the case when the stainless steel vessel was used. Visual monitoring of the mix process was possible with the glass mix vessel (Figure 6).

A subset of metallized gelled fuels was produced using a conventional three bladed propeller. The propeller diameter was 6.4 cm and the mixing occurred in a beaker with a 8.4 cm I.D. The beaker contained the fuel, surfactant, energetics and fumed silica in the same proportions as was mixed acoustically. The propeller was operated at 400 RPM for 10 minutes while manually moving the beaker about to ensure

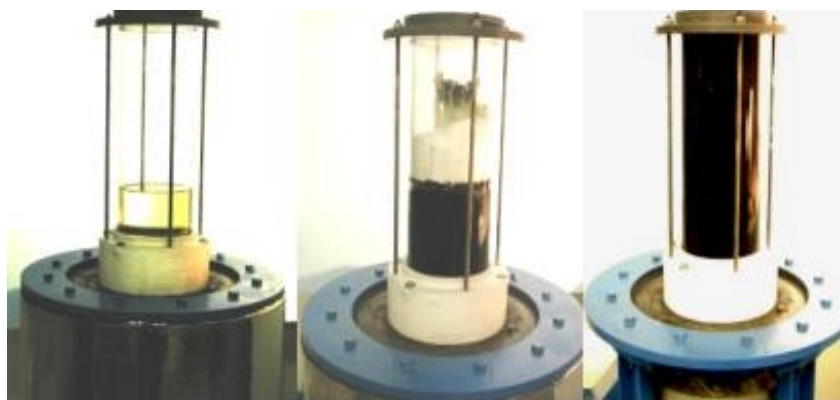


Figure 6. Visual monitoring while mixing DMAZ/nano-Carbon.



Figure 7. Conventional mixing operation using three-bladed propeller.

complete mixing (Figure 7). These conventionally mixed gels were produced and tested for comparison purposes.

2.3 Testing

Three test methods were employed to evaluate the gelled and metallized gelled fuels. First, the viscosity of the gelled mixture was measured at various shear rates. Second, samples of select mixtures were run through an oxygen bomb calorimeter. Finally, a stability test was performed on select mixtures using a centrifuge. Additionally, Scanning Electron Microscopy (SEM) was performed on select gel samples.

2.3.1 Rheology

Gelled propellants typically have viscosity in the range of 20 to 50 mPa.s for shear rates in the range of 10^5 to 10^6 s⁻¹, which are typically encountered in engine injectors [14]. This is within the range of the pumping systems currently used in rocket engines. Since the viscosity of a gelled fuel is a function of variables other than temperature (e.g. – shear rate and temporal rheological effects such as thixotropy and gel relaxation time), the rheology is non-Newtonian, and the measured viscosity is reported as “apparent” viscosity at a specific shear rate.

Due to the viscoelastic nature of metallized gel propellants, a power law rheological model was used to evaluate flow index and consistency factor of the metallized gel propellants. The power law model was represented by the following relation between shear and strain rate:

$$\eta = k\dot{\gamma}^n$$

where:

η = Apparent viscosity at a specific shear rate,

k = Consistency factor of the fluid,

n = Flow index of the fluid, and

$\dot{\gamma}$ = Shear rate

The consistency factor, k and the flow index, n were the primary indicators used to compare flow characteristics.

For this project, the viscosity measurements were performed using a Brookfield Viscometer model number LVDVE. The viscometer used a 3 mm diameter cylindrical spindle (LV4). This viscometer has a spindle speed range of 0.3 to 100 RPM. This unit was capable of measuring viscosities as high as 2,000,000 mPa*s. The test procedure involved preparing a sample by filling a 50 ml beaker with gel. The moving spindle of the viscometer was then lowered into the sample to the level required by the manufacturer’s specification [15]. Next, the spindle was operated at 60 RPM for 30 sec to ensure full contact with the immersed portion of the spindle. The spindle was then operated over a range of speeds from 0.3 to 100 RPM. At each speed setting the spindle was allowed to complete at least one revolution before the viscosity readout was recorded.

2.3.2 Oxygen Bomb Calorimeter

Combustion characteristics of the gelled and energized fuels were investigated using a modified oxygen bomb calorimeter. A standard Parr brand plain bomb calorimeter was modified by the inclusion of a dynamic pressure transducer (Omega DPX101).

Typical bomb calorimeter procedures were used though out this process [16]. A one gram sample of each gel was placed into the sample cup. A measured amount of ignition wire was placed near the sample. The bomb was sealed and pressurized with 1.1 kPa of oxygen. The bomb was then placed in the calorimeter bucket and covered with 2000 ml of water. The bucket was then covered and the stir propeller was connected to the drive motor and the water in the bucket was stirred to achieve a steady state condition. A thermocouple in the bucket water and the

pressure transducer in the bomb were connected to a computer operated data acquisition system using LabView software. After the bomb had sat in the water for five minutes, power was applied to the ignition wire. The initial pressure rise in the bomb was recorded at a rate of 1,000 samples per second. The subsequent temperature rise of the water bath was recorded at a sampling rate of one sample per second. The data collected was reduced and used to calculate certain combustion characteristics of the various fuel samples. These results are presented in a subsequent Section.

2.3.3 Gel Stability

The most common test applied to gelled fuels involves centrifuging a sample at 500 g's for 30 minutes. The amount of liquid separation or syneresis was then measured. The severity of this test is needed to produce an accelerated ageing condition and allow some measurement of the long-term stability of the gel. Stability tests were performed using a General Laboratory centrifuge, model: GLC-2B (Du Pont Instruments).

2.3.4 Scanning Electron Microscopy

Samples of fuels containing nanoparticles were sent to the University of Wyoming. The samples were smeared on a glass slide, vacuum dried and then observed in the SEM. Selections of the resulting photos are shown in the following section.

3. Results and Discussion

The ability to produce gelled and metallized gelled propellants was demonstrated via a variety of different fuel and metal combinations. All the fuels produced used fumed silica as the gelling agent. Rheological, combustion, stability, and microscopy tests were performed on the gelled fuels. For comparison sake, metallized gelled propellants were also

created using conventional mixing techniques. The conventional mix formulations were identical to the formulation of the acoustically mixed gels. Four comparison mix formulations were produced using both kerosene and DMAZ. (Formulation Numbers 8, 11, 12 and 13 previously listed in Table 1.) The results of the comparison tests follow.

The ResonantAcoustic[®] technique for mixing appeared to be insensitive to the order in which mix ingredients were added. Typically, all the ingredients were added at once and then the acoustic mixing was initiated. Unlike problems found in the literature [10] the ResonantAcoustic[®] mixes required no special ingredient handling or separate unit operations when being added to the mix. In general it was observed that the gelation reaction achieved a uniform state much quicker for the ResonantAcoustic[®] mix than for the conventionally mixed gels. While both mixes were run for a total of ten minutes, the acoustic mix appeared to be complete in 30 seconds, yet the impeller mixed samples took at least 3 minutes to achieve the same appearance.

3.1 Rheology Results

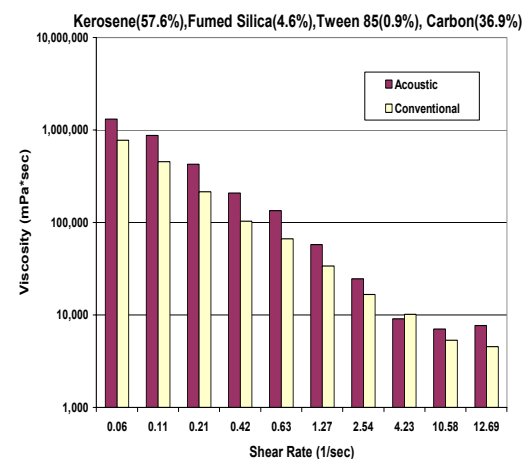


Figure 8. Viscosity versus shear rate for kerosene/nano-carbon.

Figure 8 provides a typical comparison of the results of the viscosity determinations performed on the acoustically mixed gels versus the conventionally mixed gels. The resulting data clearly shows the gels produced by acoustic and conventional methods are shear-thinning. Also, the shape of the viscosity versus shear rate plots for the various mixes indicates the conventional and acoustic mixing yield similar gel characteristics.

Individual plots of viscosity versus shear rate were used to determine the power law indices that were used to extrapolate the gel behavior at higher shear rates, such as those found in the engine flow lines and injector nozzles [14]. The plots in Figure 9 depicts a typical application of a power fit to determine the power law rheological model for each mix. The extrapolated data is summarized in Table 2 and depicted in

Figure 10. This extrapolation illustrates the shear thinning behavior of the gelled fuels. The rheological model in Figure 10 predicts that most of the gels created will have viscosities essentially the same as straight kerosene at shear rates around 50,000 sec^{-1} . Even the higher viscosities predicted for mixes P-4 and Mix#13 are predicted to have viscosities below 30 $\text{mPa}\cdot\text{sec}$ at 1,000,000 sec^{-1} shear rate. This is within the range of the pumping systems currently used in rocket engines [14]. A reference data set was plotted in Figure 10 depicting the results of a gel formed from Alex nano-aluminum and RP-1 fuel. This data was obtained from reference [17]. This served to illustrate the finding observed in that reference, namely that Alex alone acts as a pseudo-gellant while L-Alex without the aid of a gelling agent produces a less viscous gel.

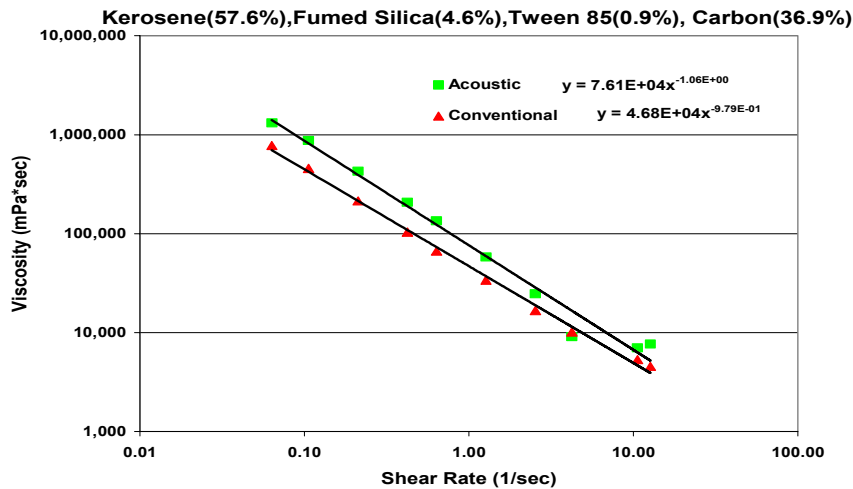


Figure 9. Power law relationship for kerosene/nano-carbon.

Table 2. Power Law Rheological Coefficients

Mix	ID	Type	k ($\times 10^5$)	n
Kerosene/Aluminum	M-11	Acoustic	1.13	-1.05
Kerosene/Carbon	M-12	Acoustic	0.76	-1.06
	P-1	Conventional	0.47	-0.98
DMAZ/Carbon	M-13	Acoustic	0.03	-0.36
	P-4	Conventional	0.12	-0.64

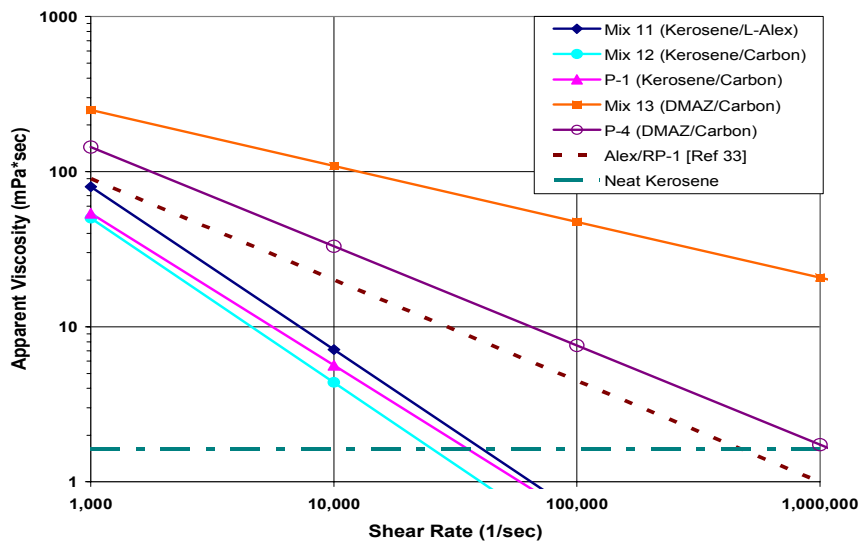


Figure 10. Power law rheological model depicting project results relative to the literature.

3.2 Oxygen Bomb Calorimeter Results

Selected mixes were tested for combustion characteristics. One gram samples from each mix were consumed in the bomb. Five replicate samples were tested for each mix. Additionally, samples of gelled kerosene and gelled DMAZ without any nano-energetics were tested for comparison sake. The calorimeter was calibrated per the manufactures recommendation using benzoic acid [16].

The calorimeter results were used to calculate the heat of combustion for the various samples. The results of those calculations are presented in Figure 11. The Heat of Combustion results are presented in terms of volume of fuel. This form of the data best illustrates the energizing effect produced by the addition of the high density metals.

The data in Figure 11 demonstrates the general similarity of the heat of combustion results between the two types of mixing. The higher calorie output of the carbon filled gels corresponds to the higher weight percentage of particles added (see Table 1).

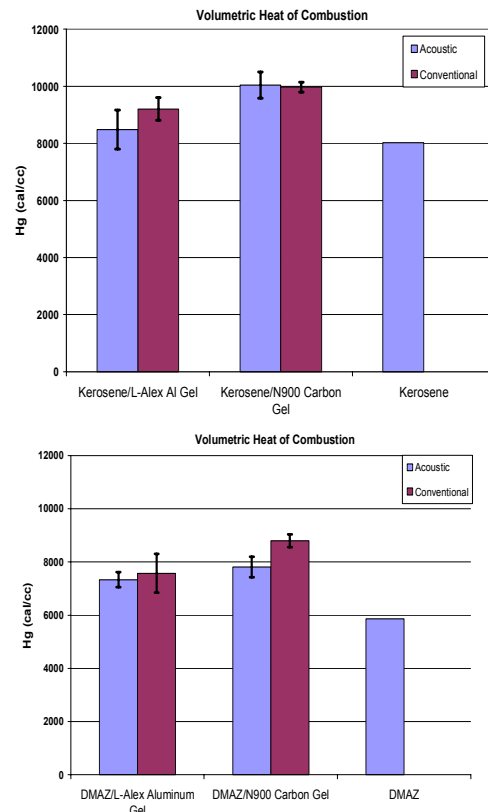


Figure 11. Comparison of the measured heat of combustion between acoustically mixed and conventionally mixed metallized gel propellants. The error bars are for one standard deviation above and below the average.

The pressure rise associated with each calorimeter explosion was also recorded. The time dependent pressure produced by the burning fuel sample, multiplied by the initial fuel sample volume represents an energy release rate. The initial rate change of this data as a function of time represents the preliminary or impulse power of the burning sample. An example plot is shown in Figure 12. The impulse power term was calculated for each test and the results are summarized in Figure 13. The error bars in this figure represent the data scatter.

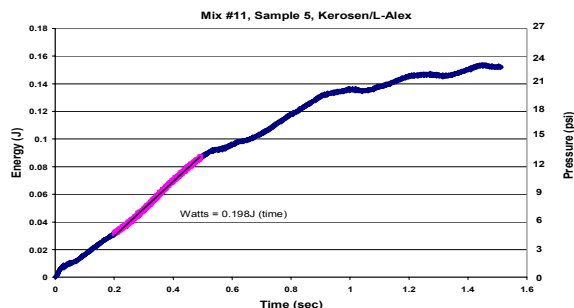


Figure 12. Typical bomb calorimeter pressure transducer data. Energy data is the product of the volume of the fuel sample and the pressure rise created by the burning sample. The slope of the energy plot yields a power term designated as Impulse Power.

The results of this testing show the acoustic and conventional mixing yield

equivalent test results. There is a higher degree of scatter associated with the DMAZ gels. This is probably due to the higher volatility of this fuel [10] relative to kerosene.

3.3 Gel Stability

As mentioned above the most common test for stability involves centrifuging the gel sample to 500 g's for 30 minutes, and then measure the amount of liquid separation or syneresis. For this study the gel samples underwent 60 minutes at 500 g's using a General Laboratory centrifuge, model: GLC-2B (Du Pont Instruments). All the specimens exhibited 7 to 10 percent syneresis. There appeared to be no difference in syneresis due to mix technique.

3.4 Discussion of Results

The primary objective of this project was readily met. The feasibility of using ResonantAcoustic[®] mixing technology to synthesis gelled propellant fuels with high energetic solids content was demonstrated. The results presented above show the ResonantAcoustic[®] technology was capable of creating gelled and metallized gelled propellants. Additionally, when comparison tests were performed between conventionally mixed metallized gels and

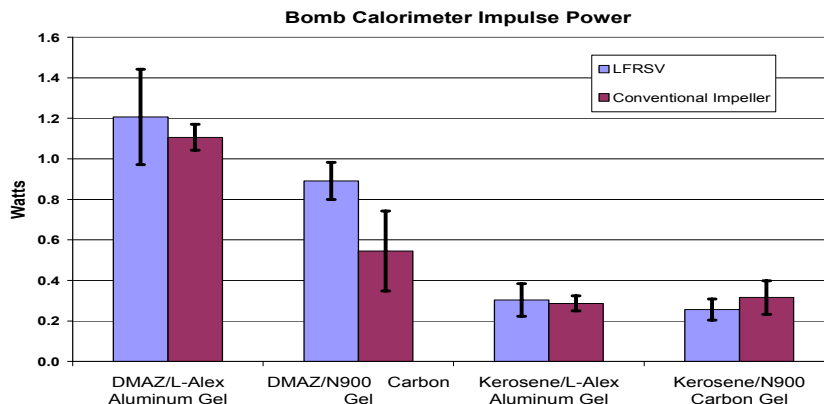


Figure 13. Summary of the bomb calorimeter impulse power calculations.

the ResonantAcoustic[®] gels, the results were essentially identical. The rheology and stability of gelled fuels were similar between conventionally mixed gels and those gels made with un-optimized ResonantAcoustic[®] mix technology.

An additional benefit of this technology concerns the clean-up and waste handling aspects of producing metallized gels. The ResonantAcoustic[®] mix system requires no internal paddles or propellers. This is very significant in terms of clean-up. The only piece of hardware that comes in contact with the propellant is the inside of the mix vessel. There fore only the mix vessel needs cleaned between batches. Due to the pseudoplastic nature of the gels, the bulk of the gel can be removed with a small amount of shear force. The photo in Figure 14 shows one batch of gel that was “poured” from the container. To achieve this result, the top of the vessel was removed; the vessel was then inverted and given a short abrupt shake. This caused a stress in excess of the yield strength of the gel and the “plug” of propellant simply slid out of the vessel. The cleanup potential for this technology is substantial. One operator of a traditional propellant mixer estimated they spend a third of their time during a mix trial on cleaning up the impellers and paddles after a

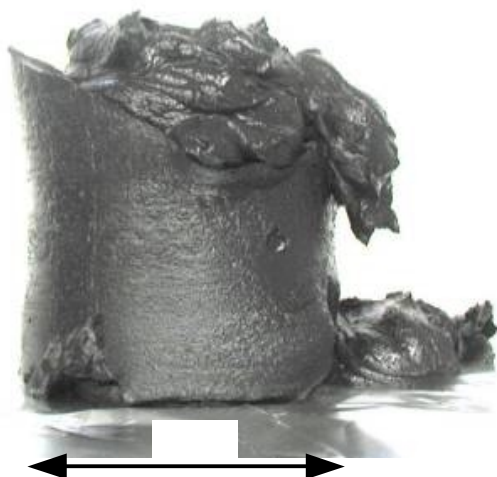


Figure 14. Typical plug of metallized gelled propellant that slid from the reactor vessel.

mix [18]. This in turns creates an increased waste burden that is costly to handle. The simple design of the ResonantAcoustic[®] reactor lends itself to easy and complete removal of the mix product with a low percentage of waste material.

It should be emphasized that the ResonantAcoustic[®] was not optimized for this project. The mixing frequency and reactor vessel geometry were kept constant throughout the test program. It is reasonable to expect an improvement in the operation of the ResonantAcoustic[®] mixer after undertaking an optimization study focused on the specific needs of gelled propellants. The improvements would be measured in terms of decreased time to gelation, increased dispersion of metal particles, decreased shear damage to the gel structure, decreased bubble entrainment, increased efficiency of the mix process, decreased power demands, increased flexibility of the mixing process, decreased clean-up time, and decreased waste products.

4. Conclusions

Based upon the results of this work the ResonantAcoustic[®] technology has been demonstrated as a feasible method for producing metallized and gelled propellants. The use of this technology has several unique advantages over conventional mixing technologies. These advantages are summarized below:

- A single ResonantAcoustic[®] mixer is applicable for low through very high-viscosity systems.
- The results of this study demonstrated the ResonantAcoustic[®] mixer is well-suited for shear-thinning systems.
- The simple and clean design of the ResonantAcoustic[®] reactor requires no internal mixing hardware, such as impellers, axial mixers, baffles, or wipes.

- The design of the ResonantAcoustic[®] reactor reduces cleanup time and the volume of waste material generated.
- No rotating mechanical elements are needed inside the vessel so there are no rotating seals on reactor.
- The clean reactor design creates no practical limitations on equipping the reactor with process temperature and pressure controls.
- The fundamental technology behind the ResonantAcoustic[®] concept does not prohibit scaling the unit from bench to production size.
- The reactor vessel can be multi-functional. It can serve as all of the following:
 - ✓ Primary mixing vessel.
 - ✓ Storage container.
 - ✓ Transportation container -- no need to transfer contents.

Acknowledgements

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