Acoustic Mixing and Treatment of Organic Waste: Results of Proof of Principle -10168

William Lattin, US DOE Idaho Field Office,
Rodney Arbon, Bechtel BWXT Idaho, LLC, Idaho Falls, Idaho 83401
John Bickley, Resodyn Acoustic Mixers, Inc., Butte, MT 59701
Peter Lucon, Resodyn Corporation, Butte, MT 59701

ABSTRACT

The mission of the Advanced Mixed Waste Treatment Project (AMWTP) is to characterize, treat and ship transuranic (TRU) waste from the Idaho National Laboratory (INL) to the Waste Isolation Pilot Plant (WIPP). One of the waste streams currently being processed is Rocky Flats organic set-ups, Item Description Code (IDC) 003, contaminated with polychlorinated biphenyls (PCBs), which require processing into a non-liquid form in order to meet the WIPP Waste Acceptance Criteria. Laboratory-scale proof-of-principle tests using surrogate waste material have demonstrated that ResonantAcoustic® mixing can efficiently disperse liquid-phase oil into the waste matrix, that absorbent material can be effectively dispersed throughout the matrix treating entrained liquids in-situ, and that the plastic inner bagging can be incorporated within the waste matrix eliminating voids. Additional tests have demonstrated that the technology can mix materials with viscosities larger than those expected to be encountered in the AMWTP waste in 30 gallon containers,, and that it can be readily scaled-up to 55-gallon drums. Because standard mixing conditions would create excessive hydrodynamic pressure for a drum, a custom-designed mixing vessel will be employed with the next phase of testing. These tests should provide confidence that the ultimate 55-gallon application can be successfully deployed.

INTRODUCTION

The mission of the Advanced Mixed Waste Treatment Project (AMWTP) is to characterize, treat and ship transuranic (TRU) waste from the Idaho National Laboratory (INL) to the Waste Isolation Pilot Plant (WIPP). One of the waste streams currently being processed is Rocky Flats organic set-ups, Item Description Code (IDC) 003. Organic set-ups are homogeneous solids contaminated with polychlorinated biphenyls (PCBs), which are regulated under the Toxic Substance Control Act (TSCA). Real-time radiography (RTR) examination of organic set-up drums shows there are approximately 3,142 drums containing PCB-liquids that require processing into a non-liquid form in order to meet the WIPP Waste Acceptance Criteria (WAC). Only one-percent of these drums exhibit excess liquid on the waste surface where it can readily be solidified. The remaining 99-percent of the drums have liquid that is within the inner bag concomitant with the waste matrix, between the inner bag and the drum liner, or between the drum liner and the drum itself.

The AMWTP currently uses mechanical compaction, referred to as a shaker-table, to vibrate and consolidate the organic set-up matrix eliminating the voids which often contain liquids. When the matrix collapses, the liquid is either forced to the surface of the waste where it can be solidified with absorbent, or it is absorbed into the waste matrix. Drums having incidental liquids within the inner bag and entrained with the sludge represent about fifty-percent of the drum population and are excellent candidates for treatment using mechanical vibration. However, that method is slow and currently cannot treat the population of drums in which the liquid is between the liner and the inner drum bag.

Liquid could be removed manually from between the inner bag and liner either by tipping the drum and decanting the liquid, or drilling a hole in the drum and draining the liquid. Both of these methods are manpower intensive and introduce the risk of spread of radioactive contamination. Additionally, direct radiation levels from these drums can range from a few tenths to over one-hundred milli-Roentgens per hour. Excessive handling would result in unnecessary personnel exposure to radiation. A methodology is needed that can readily and quickly mix absorbent with the waste matrix within the drum, treating the liquid in-situ.

BACKGROUND

In accordance with the WIPP WAC, WIPP is not permitted to receive any residual liquids associated with PCB-contaminated waste [1]. Approximately 4,617 drums containing organic set-ups have undergone RTR examination [2]. Based on the RTR examination, 3,142 of the 4,617 drums examined contain residual liquids. TRU waste containing PCB-contaminated residual liquids must be processed into a non-liquid form prior to disposal. The AMWTP has obtained an Environmental Protection Agency Risk-Based Disposal Approval (RBDA) to process Rocky Flats organic set-ups containing residual PCB-liquids into a non-liquid form for disposal at the WIPP facility. Only 1-percent of the organic setup drums with incidental liquids have liquid on the waste surface, where it can be readily solidified. The remaining drums have liquid within the bagging concomitant with the waste matrix, between the bagging and the drum liner, or between the liner and drum. The volume of liquid varies from a quarter-teaspoon to five- to six-gallons per drum. Accessing a quarter-teaspoon of liquid within a drum for absorption is very difficult. One technique that holds significant promise is acoustic-based mixing. Resodyn Acoustic Mixers, Inc., manufactures and sells bench-top and production-scale mixers using the company's patented ResonantAcoustic® Mixing (RAM) technology.

ACOUSTIC MIXING PROCESS

ResonantAcoustic® Mixing (RAM) is a new approach to solving mixing and dispersion problems that is distinct from either conventional impeller agitation, or ultrasonic mixing. This technology relies upon the application of a low-frequency acoustic field to facilitate mixing in an approach that creates micro-mixing zones throughout the system rather than being localized at the tips of the impeller blades, at discrete locations along the baffles, or by co-mingling products induced by tumbling materials. The result is a method that allows faster, more uniform mixing throughout a vessel than can be created by many conventional, state-of-the-art mixing systems.

RAM technology is currently used to mix powders, liquids and highly-viscous pastes using the LabRAM bench-top scale mixer (0.5 kg) and the RAM 5, 5-gallon mixer. The scaled up RAM 55, with a 55-gallon capacity, mixer is currently in design and due to be offered for sale in the second quarter of 2010. A 30-gallon RAM, which was used to conduct this work at Resodyn, is shown in Figure 1.

Figure 1. Resodyn 30-gallon industrial scale ResonantAcoustic® Mixer.

Because the RAM technology does not use impellers, or other intrusive devices, it does not require unique vessel designs, and has the flexibility to mix many types of materials. These may include liquid-liquid, liquid-solid, gas-liquid, and solid-solid systems with viscosities as high as 100 million cP. Each combination of materials can be mixed in the same type of current mixing vessels or application and shipping vessels, without any hardware changes.

The acoustic energy source for the RAM technology is provided by an electro-mechanical system that is operated at resonance. Because the RAM technology operates at resonance, the acoustic field applied to the media can be quite large, by imparting up to 100-*g* of acceleration on the mixing container at a nominal operating frequency of 60 Hz. By harnessing the power of resonance, and using a multiple-mass system design, ResonantAcoustic® Mixers can accelerate a 500 pound mass at 100-g of acceleration using only 30 kW of power, with minimal forces imparted to the frame mountings. This can be contrasted with a non-resonant system that would require over 560 kW to achieve the same mixing conditions. The RAM technology can also operate at resonance at very low acceleration levels. Operating conditions of the RAM mixer are automatically modulated by a closed loop feedback control system that allows the operator to preset the operating condition to the mixing process requirements.

The mixing principal is illustrated in Figure 2, which shows a vessel being subjected to a low-frequency acoustic field in the axial direction; resulting in bulk motion of the fluid, known as acoustic streaming. The acoustic streaming in turn creates micro-mixing cells throughout the mixing vessel as depicted in Figure 2. The characteristic mixing length for the RAM technology, operating at 60 Hz is nominally 50 microns. Moreover, the strength of the pressure waves associated with the acoustic streaming flow is strongly correlated to the displacement of the acoustic source, e.g. the base of the vessel [3].

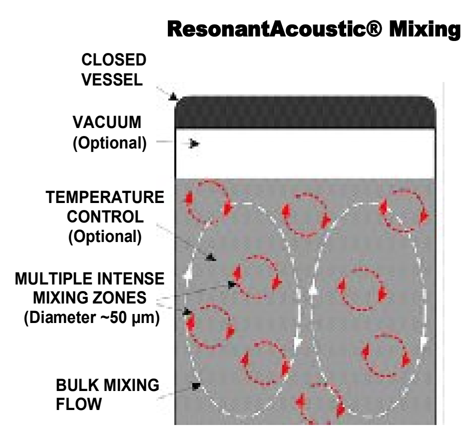

Figure 2. Mix material motion schematic.

PROOF-OF-PRINCIPAL TESTING

The evaluation of RAM technology for the treatment of AMWTP organic sludge waste can be accomplished in three phases:

1) Prepare surrogate material with a range of viscosity and free liquids that bound the viscosity and free liquids of the AMWTP organic sludges.
2) Demonstrate that RAM is a feasible technology capable of mixing absorbents into surrogate material and investigate mixing complications presented by the plastic bag, drum liner, and inner containers.
3) Analyze and estimate the forces that will be applied to the 55-gallon drum during the RAM mixing process in order to determine whether the unsupported drums will be able to withstand the resultant stresses.

Proof-of-principal testing also involved investigation of mixing-induced temperature increases, and determination of the vessels to be used for the organic waste treatment.

Surrogate Development

Performing proof-of-principle testing on TRU waste creates a number of challenges. To simplify these tests, surrogate sludge was utilized in proof-of-principle testing. The goal was to generate a surrogate that simulates the organic sludge with respect to specific gravity, viscosity, thixotropic behavior and stickiness. The organic sludge was generated at the Rocky Flats Plant and consists of various organic liquids that were mixed with MicroCel E®, a synthetic calcium silicate, to form a grease-like paste. The organic liquids were primarily oil and chlorinated solvents generated from machining and degreasing of plutonium metal. The amounts of material added to a drum were not metered. Rocky Flats operators would adjust the composition until a paste like consistency was achieved. Typically, the composition of organic set-ups was nominally 30-gallons of liquid organic waste to 100-pounds of MicroCel E®.

Table I. Surrogate Formula.

	% by Weight
Texaco Regal Oil A	46%
Carbon tetrachloride	9%
1,1,2 Tricholoroethane	7%
1,1,2 Trichloro-1,2,2-trifluoroethane	5%
Trichloroethylene	2%
Calcium silicate – MicroCel E®	31%

The surrogate formula depicted in Table I was the base material and was used in the testing to simulate the organic sludge.

As determined by RTR, up to 6-gallons of free liquid may be expected in each 55-gallon drum. In order to simulate the worst-case, 11-percent by volume liquid (either Texaco Regal Oil, or carbon tetrachloride) was added in some mixing tests.

Viscosities

Initial experiments were run using surrogate waste materials at three different viscosities: 200,000 cP, 400,000 cP and 600,000 cP. While the 400,000 cP surrogate is representative of the majority of the drums, the lower and higher viscosities were tested to determine absorbent performance under worst-case liquid conditions, and to investigate if excessive viscosity would degrade the mixing capabilities, respectively.

Early testing of the ResonantAcoustic® mixing process confirmed compatibility of the mixer with all viscosities of the surrogate material. Additional testing performed by Resodyn demonstrated rapid mixing of a simulated material comprising Dow 200 polymer with 77-percent solids loading of drywall compound. Colored chalk was added as a mixing indicator. The mixing was performed in a 30-gallon vessel, and resulted in a post-mix compound that was fully uniform in color, and having viscosity exceeding 1 million cP. The ResonantAcoustic® mixing time was 2 minutes.

Sorbent Selection

If liquids are not completely absorbed during acoustic mixing, the technique is of no value. A key component to success is the use of the proper sorbents. Clay-based absorbents, i.e. Petroset-II® and Clayton-AF®, can be effective for absorbing combinations of free oil, volatile organic compounds and water. When this study commenced, the AMWTP-permitted sorbent was Petroset II®. Petroset II® functions best when used with a chemical activator. The manufacturer recommends methanol. Methanol, however, is flammable. Flammable volatiles in the headspace of drums to be shipped to the WIPP can complicate, and in some cases prevent, shipment of the drum.

A search was conducted for a nonflammable activator that produces acceptable absorption results. Multiple chemicals were tested. It was ultimately shown that both propylene glycol and tetra-glycol are effective activators that significantly enhance the absorbent capabilities of Petroset II® and are nonflammable. In addition to Petroset II®, two additional sorbents were investigated; MicroCel E® (synthetic calcium silicate) and Clayton-AF®. Since Clayton-AF® and MicroCel E® do not require activators, they present a simpler process and require approximately 25-percent less material than Petroset-II® to produce the same results.

Laboratory-scale testing of the different absorbents (Petroset II® plus activator, MicroCel E® and Clayton-AF®) were performed using 250ml mixing samples and the LabRAM bench-top ResonantAcoustic® mixer. A 194-gram sample of 200,000cP surrogate was used to represent the most-liquid set-ups, and “worst case” concentrations of free oil, VOCs and water were added in combinations to produce a total free-liquid concentration of 11-percent by volume. For each absorbent, initial process optimization work was undertaken to identify the approximate concentration required.

Representative results of these tests are given in Table II. The ranking system given in Table II was based on visual inspection and assessment of “dryness” of the treated material. A treatment rank with a dryness of 6, or greater, yielded acceptably dry results.

Table II. Representative Solidification Results for Petroset II® + Propylene glycol activator, MicroCel E®, and Clayton Activator Free absorbents.

Base material: 194 grams 200,000 cP surrogate

Free Liquid Component			Absorbant Petroset II®	Activator Propylene glycol	Dryness	Absorbant Microcel E	Dryness	Absorbant Clayton AF®	Dryness
Carbon Tetrachloride	Free Oil	Water							
13.07 grams	7.35 grams	0.82 grams	27 grams	0.75 grams	7+	20 grams	7	20 grams	7
22.57 grams	1.41 grams	0.78 grams	27 grams	0.75 grams	7+	20 grams	7+	20 grams	7+
2.73 grams	13.81 grams	0.85 grams	27 grams	0.75 grams	7+	20 grams	7+	20 grams	7+
13.07 grams	7.35 grams		27 grams	0.75 grams	7	20 grams	7+	20 grams	8
13.07 grams		8.99 grams	27 grams	0.75 grams	8	20 grams	6	20 grams	8

ResonantAcoustic® Mixing of Surrogate Waste Material

Characterization tests had shown that the surrogate material had a relative density of approximately 1.3, and that mixing conditions of 40- to 50-g acceleration for 4 minutes yielded uniform blending. The high system efficiency resulted in an average material temperature increase of only 2.2°C under these operating conditions.

The test plan for this series of experiments involved mixing on the largest ResonantAcoustic® system available at this time, which was configured at a maximum capacity compatible with 30-gallon drums. (Figure 1, above.) In order to investigate the feasibility of mixing the waste directly in the metal 30-gallon drums, the forces produced by the mixing process were calculated. Mixing with an acceleration of 40-g would create a hydrodynamic pressure of 386 kPa at the bottom of the drums used in these tests during each mixing cycle. Modeling that was conducted showed that 30-gallon drums constructed from 1.2mm steel can survive an acceleration of 56-g's. However, previous experimentation with metal drums by Resodyn has shown that the folded seams that are used to connect the drum cylinder wall to the base and lid are a weak link in their construction and required a de-rating factor of 0.3 to be applied. This led to the conclusion that mixing in 30-gallon metal drums was not feasible for the proof-of-principal testing, and that the planned treatment of waste by mixing directly in unsupported metal drums that may be rusted and damaged is also not feasible.

Since the waste is contained within rigid HDPE liners inside the drums, a custom steel mixing vessel was constructed into which the HDPE liners holding the waste could be loaded for mixing. By carefully matching the dimensions of the liner to the steel holder, and vacuum clamping the liner within the holder, mixing of the waste was achieved without removing it from the storage liner. Further experimentation showed that by using a properly designed holder, and a vacuum system to hold the liners in close contact with the vessel walls, liners of varying thickness down to 90-mil can be used for mixing the organic waste. This mixing technique could, therefore, be used for all of the organic set-up liquid configurations in the AMWTP inventory.

30-Gallon (30-gallon) Drum Testing

The effectiveness of this process is illustrated in the following photographs.

The starting condition, Figure 3, is a base matrix of 200,000 cP surrogate. An additional 11-percent by volume liquid oil and volatile organics was split between the bag, liner, and the surface of the sludge. This mix of ingredients simulates a 55-gallon drum containing over 4.8-gallons of free liquids and, like the drum, has approximately 20-percent headspace.

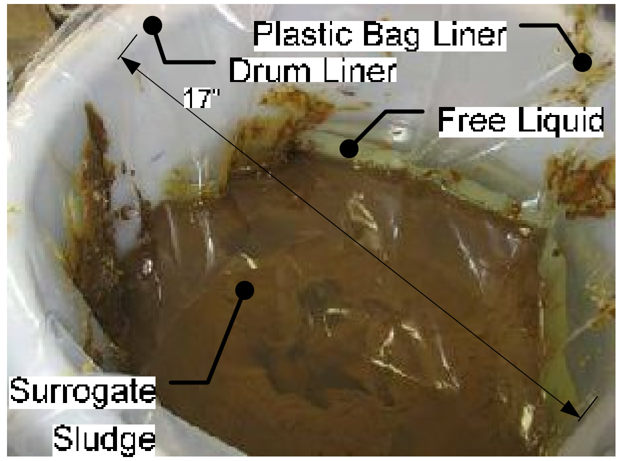

Figure 3. 200,000 cP surrogate sludge plus 2.6-gallons of free liquid.

MicroCel E® was then added at a concentration of 10.3-percent by weight of the surrogate sludge content. Because the required MicroCel E® powder occupied a volume of 14.7-gallons, approximately one-third of the MicroCel E® was added and mixed for one minute at a nominal 40-g acceleration. A porthole in the steel vessel lid was removed, and more MicroCel E® was added until the vessel was full. The contents were mixed for one minute at 40-g acceleration and the process repeated until a total of four material additions had delivered all the required MicroCel E® into the mix. A final mix of two minutes ensured uniform blending of all the contents, with no significant increase in temperature and pressure.

As shown in Figure 4, the treated material is dry and clumped. The HDPE liner was loaded into a 30-gallon drum and shipped to the AMWTP. A detailed real-time radiography examination was performed. No liquids were detected.

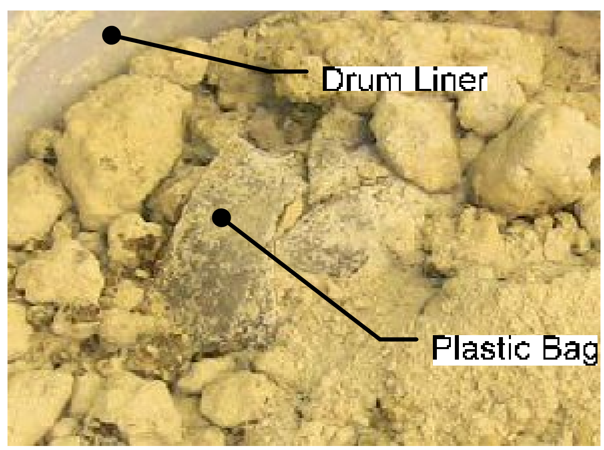

Figure 4. Surrogate sludge after the addition of MicroCel E® and ResonantAcoustic® mixing.

Sealed Containers

A 1-gallon plastic jug containing a quart of Regal oil was placed in the bottom of the bag, inside a 30-gallon (HDPE) drum liner. 24-gallons of a sludge-surrogate material was added on top of the jug. The configuration was installed on the RAM 30 and mixed at nominally 40- to 50-g acceleration for four minutes. Inspection at the end of mixing showed the plastic jug to be on the top of the sludge. Based upon Resodyn experience with the ResonantAcoustic® mixing process, this ejection occurs whenever a solid object is included in a viscous material mix. As illustrated in Figure 2, above, the mixing process results in a bulk motion of the mixer vessel contents. This bulk motion, caused the plastic jug to be moved to the surface. Once it reaches the surface, the stickiness of the sludge is not sufficient to overcome the buoyancy of the jug, and it remains spinning on the surface until the mixing is finished. Hence, the jug can be easily and safely extracted.

Drum Bagging Entrainment

A surrogate test sample was prepared in a 30-gallon HDPE liner as described earlier, and shown in Figure 3. Immediately prior to adding the absorbent, the bag was slit in four quadrants, each 90-degrees apart using a metal lance.

In the series of vessel qualification tests it had been determined that the plastic bag became fully entrained into the sludge when mixed at the nominal 40- to 50-g of acceleration for four minutes. This bag entrainment under these conditions was repeated in ten consecutive tests with no failures. It was therefore concluded that the nominal 40- to 50-g acceleration for four minutes was the mixing condition that could be used to result in bag entrainment, and was the mixing condition used in these tests.

Upon inspection after mixing, the 10-mil bag was visible at the surface of the mixture. (Figure 3, above.) Examination concluded that the portion of the bag that was exposed at the mixture surface was the "seamed" section of the bag originally placed at the junction between the walls and bottom of the liner. It was determined that the bag had been entrained in the matrix, and had been close to the material surface when the mixing cycle was ended.

Implementing 55-Gallon Drum Mixing

This project demonstrated the feasibility of using the ResonantAcoustic® mixing technology to successfully mix organic wastes containing viscous sludge material and free liquids. Based on the results of testing performed on 30-gallon drums, this process can be scaled to 55-gallon drums. A 55-gallon capacity version of the ResonantAcoustic® mixer will be required. However, in order to provide a 55-gallon industrial solution for the organic waste mixing, further development work will be required to establish the specifications necessary for Resodyn Corporation to design, fabricate, and operate such a robust waste treatment process.

This project has established that the mixing of the organic waste cannot be performed in the metal drums in which it is stored, without reinforcing the drums. This work also has demonstrated that mixing in the HDPE drum liners is possible. Three different HDPE drum liners have been used for the waste storage at AMWTP. Custom engineering will be necessary to design a holding vessel that is compatible with each of these three types of drum liners that have been used in packing the organic waste. The work will also be used to validate that the drum liners perform well in the mixing process over the complete range of waste material types that will be mixed. Some of the factors relating to these liners that must be determined prior to the start of actual design include:

- Method of lifting the liners out of the current drums and into the mixing vessel.
- Method of lid removal.
- Fabrication of liner supporting boot and attachment to a custom mixing vessel.
- Compatibility of liner shape with vacuum clamping.
- Interchangeability between different drum liner types.
- Ability of final vessel design to support mixing at high g-levels for each drum liner type.
- Method of material containment if a vessel should split due to age or damage prior to the mixing process.
- Unloading of the drum liner and subsequent packaging for transportation.

In order to answer these questions and to validate the design of a system that can be used to successfully mix the organic wastes in-situ, it will be necessary to conduct mixing tests in 55-gallon drum liners. The 55-gallon system, essentially a scale-up of the 30-gallon system used to conduct this work at Resodyn, is currently under design and will be launched by Resodyn Acoustic Mixers as a standard product offering the second quarter of 2010.

SUMMARY

Proof-of-principal testing has demonstrated that ResonantAcoustic® mixing, with the proper application of absorbent material, eliminates free liquids contained on the surface, between the drum liner and bag, or trapped within the matrix, in drums containing surrogate organic sludge and excess liquid. Testing also demonstrated that the ResonantAcoustic® mixing process can bring submerged bottles containing liquid to the surface of the sludge, allowing for the ready removal and treatment of this prohibited item. In addition, polythene liner bags, which often times isolate fluids between the bag and the high-density polyethylene (HDPE) rigid drum liner, can be fully entrained within the sludge matrix, resulting in the successful remediation of all liquids present.

The suite of mixing viscosities performed to date has provided a clear demonstration that the RAM technology can efficiently disperse liquid phase oil into the waste matrix. The testing also demonstrated that absorbent material can be effectively dispersed throughout the material volume.

References

1. U.S. Department of Energy. (2009). Contact-Handled Transuranic Waste Acceptance Criteria for the Waste Isolation Pilot Plant, DOE/WIPP-02-3122. Carlsbad Field Office, Carlsbad, New Mexico.

2. RTR is a nondestructive examination technique, similar to an x-ray, which is routinely used to identify liquid within TRU waste.

3. M. K. Aktas, B. Farouk, Numerical simulation of acoustic streaming generated by finite amplitude resonant oscillations in an enclosure. *Journal of the Acoustical Society of America*, 116 (2004) pp. 2822-2831.