FINAL REPORT

U.S. Department of Energy

**“Foaming and Antifoaming in Radioactive Waste Pretreatment and
Immobilization Processes”**

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EXECUTIVE SUMMARY

Savannah River National Laboratory (SRNL) has reported severe foaminess in the bench scale evaporation of the Hanford River Protection - Waste Treatment Plant (RPP-WPT) envelope C waste. Excessive foaming in waste evaporators can cause carryover of radionuclides and non-radioactive waste to the condensate system. The antifoams used at Hanford and tested by SRNL are believed to degrade and become inactive in high pH solutions. Hanford wastes have been known to foam during evaporation causing excessive down time and processing delays.

Experimental and theoretical investigations of the surface phenomena, suspension rheology, and bubble generation and interactions that lead to the formation of foam during waste processing were pursued under this EMSP project. The first major task accomplished in the grant period involved establishment of the main mechanisms of formation and stabilization of foams containing very small insoluble particles, since the radioactive waste foams are typically stabilized by such particles. The results of this research were published in a series of papers (See Publications 1-12). Fundamental research into the particle structuring phenomena in the confined boundaries of a film also lead unexpectedly to a new mechanism for soil remediation (See Publication 7).

The second major task involved an investigation of the major mechanisms of antifoaming of foams stabilized by solid particles. Based on the mechanistic understanding of the antifoaming action in solid particle-stabilized foams, and working closely with SRNL, we have successfully developed and tested antifoam agent IIT-B52 to mitigate foaming in a process originally developed for the

disposition of Savannah River Site (SRS) high level radioactive (HLW) salt solutions. This new and advanced antifoam agent was found to be more effective than those commercially available. Process demonstration studies using advanced antifoam agent IIT-B52 reveals that the antifoam agent also acts as a powerful rheological aid that can dramatically reduce the rheology of SRS HLW potassium tetraphenylborate slurries and can de-entrain air from the slurry.

Therefore, the results of this research have led to the successful development of a new antifoam agent which can also act as a rheology modifier, enhance the processing throughput of the HLW slurries, and accelerate the DOE mission.

RESEARCH OBJECTIVE

The objective of this research is to develop a fundamental understanding of the physico-chemical mechanisms that cause foaminess in the DOE High Level (HLW) and Low Activity radioactive waste separation processes and to develop and test advanced antifoam/defoaming agents. Antifoams developed for this research are tested using simulated defense HLW radioactive wastes obtained from the Hanford and Savannah River sites.

METHODS AND RESULTS

Experimental and theoretical investigations of the surface phenomena, suspensions rheology, and bubble generation interactions that lead to the formation of foams during waste processing were pursued. During the grant period, the following four objectives were accomplished (I) elucidating the key mechanisms that produce foaming in the presence of small solid particles present in radioactive wastes, (II) development of advanced confocal microscopic technique to observe particle-gas-liquid interactions in simulated DOE wastes (III) determination of the effects of soluble and insoluble inorganic components and organic complexants on foam formation and stability in defense waste matrices, and (IV) development and testing of advanced antifoam/defoamer agents.

I. Major Mechanisms of Foam Formation and Stabilization

The first task accomplished in the grant period involved establishment of the major mechanisms of formation and stabilization of foams containing very small insoluble particles since the radioactive waste foams are typically stabilized by such particles. The solid particles stabilize foams in two ways: by adsorption of biphilic or amphiphilic (i.e., containing both hydrophilic and hydrophobic parts) particles at the surface of the foam lamella (liquid-gas interfaces) and by layering of the particles trapped inside the foam lamella or film. During bubble generation and rise, hydrophilic solid particles organize themselves into a layered structure due to confinement between neighboring bubbles, and this structure provides a barrier against the coalescence of the bubbles, thereby causing foaming and foam

stability. This novel stabilization mechanism involving particle structuring phenomenon in macrodispersions such as foams opened new vistas in dispersion science and engineering. The results of this research were published in a series of papers (see publications below under this project). **This research was recognized by the American Institute of Chemical Engineers with its national award in fluid-particle systems presented to the principal investigator, Wasan. Also, he was elected to the National Academy of Engineering in 2004 for his accomplishments in interfacial engineering and colloidal processing.**

Fundamental research into the particle structuring phenomenon in the confined boundaries of a film also led unexpectedly to a new mechanism for soil remediation. This work is described in our paper published in the journal Nature in 2003 (see publications listed under this project).

II. Particle-Gas-Liquid Interactions Using Advanced Microscopy

Under task two, an advanced technique using a new laser-scanning confocal microscope was developed to understand the effect of particle-particle interactions on the gas-liquid-solid interface structure. This technique allows the slurry to be analyzed in an as-made condition. The microscope, which is located at SRNL, has the ability to make both two-dimensional pictures and three dimensional representations of a sample. Figure 1 shows a two-dimensional color image of the Savannah River HLW tetraphenylborate slurry (TPB). Three-dimensional representations were made by scanning two-dimensional images at 1-micron increments. Image analysis software provided by Carl Zeiss, Inc. was used to

stack the images together in a two-dimensional image that provides a color gradient corresponding to the depth of the sample. These three-dimensional representations were used to understand the actual physical structure of Hanford slurries. Figure 2 shows a three-dimensional representation of the (RPP-WTP) AZ102 slurry. The scale in the upper left-hand corner shows the depth of the image and the scale in the lower portions of the image shows the horizontal scale. The red, green and blue colors correspond to a depth of 0.75, and 15 microns in the slurry sample. The slurry particle shown in this color image in Figure 2 appear to be flocculated into larger (75 microns) sized flocs. These flocs are suspended by smaller particles.

Figure 1. Two-dimensional laser scanning confocal microscope image of SRS HLW TPB Slurry.

**Figure 2. Three-dimensional Representation of Simulated AZ102 Envelope D Slurry
- Dashed Circle Shows Examples of Large Flocculated Particles.**

Figure 3 shows a three-dimensional representation of the AZ102 slurry with 1000 ppm of CTAB (cationic surfactant) modifier added. The image clearly shows that CTAB dispersed the RPP-WPT AZ102 particles into fine particles which appear to be smaller than 1 micron. Analysis of Figure 2 and 3 together appears to indicate that AZ102 slurry is actually composed of particles that are much smaller than 0.5 microns. The smaller particles (< 1 micron) shown in Figure 3 are dispersed and not flocculated like the particles shown in Figure 2.

Figure 3. Three-dimensional Representation of Simulated AZ102 Envelope D Slurry with 1000 ppm CTA - The micrograph shows dispersal of AZ102 sludge into fine particle suspension.

This three-dimensional confocal microscopic technique will allow researchers to understand the impact of various rheology modifiers and antifoam agents on the surface chemistry of high level radioactive waste and increase the waste content of the DOE slurries. This research was selected by the Journal of Visualization as the cover for the April 2004 issue (See Publication 12).

III. Effects of Soluble and Insoluble Components on Foaming

Under task three, foaming experiments employing our high-resolution optical bench to monitor bubble dynamics and foam lamella texture were carried out using several Hanford simulants. Foaminess and foam textures during boiling of the simulants at both atmospheric pressure (700 mm Hg) and at low pressure (110 mm Hg) were monitored. The effects of various parameters such as heating flux, organic components such as paraffin hydrocarbon, tributyl phosphate and surfactants on foaminess during boiling of the simulants were investigated.

During boiling, bubbles are generated which collide and coalesce. The size of the polyhedral foam cells are typically a few millimeters in size and are shown in the inset in Figure 4. A sharp maximum in foaminess of the order of 450 vol % was observed at a total solid concentration of 53 wt % (Figure 4). No foaminess was observed in a system without insoluble particles. Foaminess is defined as the ratio of total volume of the gas incorporated into the liquid during boiling to the liquid volume without boiling. In the previous studies conducted by IIT [1] the maximum in foaminess occurred at around 22 wt %. The shift in the maximum of foaminess (53 wt %) when compared with the previous work done in our laboratory is due to

chemical composition of the simulated waste. Hanford AN102 waste, commonly referred to as “supernate”, is primarily composed of soluble sodium salts (hydroxide, nitrate, carbonate, etc.) and soluble transition metals (e.g., Fe) complexed by citric acid, EDTA and gluconic acid. Previous work (1) was conducted with insoluble transition metal nuclear waste, commonly referred to as “sludge” that is precipitated from acidic nuclear waste with NaOH. The resulting “supernate” and “sludges” are separated and managed in separate waste tanks. The AN102 waste is a special subtype of “supernate” that was derived from acidifying “sludge” and processing the acidic waste with complexants (e.g. EDTA) to remove Cs and Sr in Hanford B plant.

Figure 4. Foaming during boiling of the sludge AN-102

Figure 4 shows that at the onset of boiling, foaminess was less than 30 vol %, but foaminess increased as the simulant was concentrated. During boiling, nuclei (particles) are precipitated out. Additional particles are formed as the solids content is increased. These particles are of crystalline material and have different surface energies, that is, they have different wettabilities. The Janus (or amphiphilic) particles attach themselves at the air-water interface, prevent the coalescence of bubbles, and enhance foaminess. The attachment of the particles at the bubble surface can be seen in Figure 5. With an increase in the concentration of the amphiphilic particles, foaminess increases. Also, the interparticle distance decreases upon increasing concentration. The hydrophobic interactions between the particles promote flocculation of the particles. This flocculation then facilitates clustering of the particles which results in a decrease in foaminess. The photographs showing the clustering of particles are shown in Figure 6. A typical cluster size was $30 \pm 10 \text{ }\mu\text{m}$. Thus two competing effects take place during boiling of the simulated waste: attachment of the particles at the air-water interface, which promotes foaminess, and clustering of particles, which decreases the foam. The foam collapses immediately as soon as the heating is stopped. This shows that the foam is unstable, dynamic in nature, and forms only during boiling.

Figure 5. Coverage of the air bubble by nuclei (particles) at various stages of dynamic foam formation

Figure 6. Clustering of nuclei (particles) after the maximum in foaminess

The increase in water evaporation flux rate enhances foaminess. Less foaminess was observed at low pressure (110mm Hg) and low water evaporation flux (0.05 ml/min sq.cm). However, at low pressure and high evaporation flux rate (0.09 ml/min sq.cm), the maximum foaminess was about 1200%. This shows that foaminess increases with increasing water evaporation flux. Extrapolated data for water evaporation flux (0.56 ml/min sq. cm) simulated at the Hanford plant show severe foaminess (>1500%) at low solid concentration (<40%).

The addition of 25 ppm of normal paraffin hydrocarbon (NPH) and tributyl phosphate (TBP) to the sludge samples with severe foaminess did not show a noticeable change in foaminess. However, addition of 100 ppm of NPH increases foaminess at maximum by 25% and addition of 100 ppm TBP reduces foaminess by a factor of three at maximum. **NPH and TBP are also encountered in the SRS HLW salt solutions, therefore these results will also provide a better understanding of how these compounds affect the foaminess of SRS HLW salt solutions.**

The Savannah River Technology Center has reported that during the sludge processing some surfactants could be generated or formed inside the sludge. During the boiling of the alkaline sludge, some of the hydrocarbon derivatives present in the sludge decompose and become surface active. Therefore, we carried out a systematic study to understand the effect of the type and concentration of the surfactants on the foaminess. For this study, we used two commercial surfactants, hexadecyl trimethyl ammonium bromide (a cationic surfactant) and sodium salt of dodecane sulphonic acid (an anionic surfactant) and the simulant AN-107.

Different concentrations of the surfactants were tested, varying from 1000 to 3300 ppm. Due to the presence of the surfactants, the surface tension decreases and small bubbles are formed during boiling. The gas solubility is high at low temperatures, but with an increase in temperature, the gas solubility decreases and more gas is released. This leads to the generation of more bubbles, as a result of which the foaminess increases. As time progresses, the temperature increases and the gas solubility achieves a steady state, and the foaminess decreases. Nuclei or fine crystals are formed when the solution becomes over-saturated. The crystals attach to the foam lamella surfaces and provide a structural stabilization barrier, which makes the foam lamella stable.

IV. Antifoaming

Under task four, based on the mechanistic understanding of the antifoaming action in solid particle-stabilized foams, and working closely with the Savannah River National Laboratory, we have successfully developed and tested antifoam agent IIT-B52 to mitigate foaming in the process being developed for the disposition of SRS HLW radioactive salt solutions. This new and advanced antifoam agent was found to be more effective than those commercially available. Process demonstration studies using advanced antifoam agent IIT-B52 revealed that the antifoam agent also acts as a powerful rheological aid that can dramatically reduce the rheology of SRS HLW potassium tetraphenylborate slurries and also can act to de-entrain air from the slurry. Thus, fundamental understanding of the physico-chemical factors that affect foaming and air entrainment can lead to the development of antifoam and rheological agents that

can enhance the processing characteristics of DOE HLW slurries. When the yield stress of these wastes is reduced, the glass forming solids content of the waste can be increased. If properly developed and deployed, these antifoam agents and rheology modifiers can increase the solids content of the DOE slurries that are immobilized. Increasing the solids content that is immobilized can increase the waste processing throughput which will accelerate the DOE mission.

The antifoaming efficiency of two commercially available antifoamers (DOW 1520 and DOW Q2-3183A) using the pretreated Hanford simulant AN-107 at higher evaporation flux was also tested. The antifoamer DOW 1520 was found to be ineffective. However, antifoamer DOW Q2-3183A reduced the foaminess at low pressure from 275 vol% to 180 vol%. Fundamental research into the mechanisms and advanced antifoam development for radioactive wastes is currently in progress.

RELEVANCE, IMPACT AND TECHNOLOGY TRANSFER

One of the greatest challenges facing the DOE is the remediation of 1×10^8 gallons of high-level and low-level waste in 334 underground storage tanks (UST) at its Hanford, Savannah River, Oak Ridge, Idaho, West Valley and Fernald sites [2]. Additionally, the DOE Office of the Environmental Management has launched an effort to accelerate the closure of the DOE waste sites [3,4]. With the exception of Fernald and Oak Ridge, the waste was generated primarily from plutonium production processes. The waste in a typical UST is partitioned into three phases: saltcake, an aqueous supernatant, and sludge. The radioactivity of the waste, 1×10^{11} Bq/L, is primarily due to Cs-137, Sr-90, and Pu-238. Major inorganic species result from removal of cladding, and from chemicals added in the fuel element dissolution or radiochemical separations. Inorganic and organic chemicals that were used for radiochemical separations include iron and aluminum, complexing agents and chelating agents, and other hydrocarbons.

These inorganic and organic chemicals can cause foaming and gas entrainment problems in HLW/LAW treatment and immobilization systems that ultimately result in excessive shutdowns and loss of attainment [5,6].

In most cases, the high level (HLW) and low activity (LAW) nuclear wastes will be immobilized into glass or, alternatively, grout waste forms prior to permanent disposal [7-9]. The baseline processing technology includes pretreatment processes that separate water and non-radioactive components from the radioactive waste by evaporation, filtration, precipitation and ion exchange. These pretreatment processes are mixed with agitators, pulse jet systems and air spargers.

The concentrated radioactive liquids and solids are ultimately vitrified in joule heated ceramic melters. The DOE wastes are also known to excessively entrain air, which becomes incorporated as a result of the mixing systems employed at the Defense Waste Processing Facility (DWPF) and the Hanford Waste Treatment Plant (WTP).

The Department of Energy, like commercial companies, is faced with the challenges of determining the most cost effective and profitable technologies for their businesses. Uncontrollable foaming and air entrainment can severely impact the production rate and ultimately the cost effectiveness of a chemical process. Consequences of foaming and air entrainment, other than the obvious impact on attainment, are the potential for contamination of evaporator condensates, overflow of the pretreatment waste storage tank systems where air sparging is used to mix the waste, pumping problems due to loss of prime, and plugging of the process vessel vent system. Further, for example, foam carryover actually rendered the melter feed unacceptable for vitrification due to preferential carryover of aluminum [10,11]. Excessive foaming has been also observed in the Hanford waste filtration studies [12-15]. Past operations of the Hanford HLW 242-A tank farm have resulted in severe foaming and numerous evaporator shutdowns during the evaporation of waste [5]. The Savannah River National Laboratory (SRNL) found that severe foaming occurred during the continuous bench scale evaporation of a radioactive alkaline waste from Hanford tank 241-AN102 [16].

Research to understand the basic mechanisms controlling both the foaminess and foam stability, and gas entrainment action in the three-phase gas/liquid/solids systems continues to be a high priority need for the DOE [17]. Furthermore, the knowledge and understanding developed by this research directly benefits the commercial chemical industry [18]. Long-term pretreatment and immobilization of high-level and low activity wastes in glass media will require control and mitigation of foaming and gas entrainment during operation of the baseline waste treatment processes.

PROJECT PRODUCTIVITY

The project accomplished all of the proposed goals. We have successfully developed and tested antifoam agent IIT-B52 to mitigate foaming SRS tetraphenylborate salt solutions. In addition, a number of commercially available antifoamers for mitigating foams in Hanford wastes were tested and the antifoamer DOW Q2-3183A was recommended for use in testing waste simulants.

PERSONNEL SUPPORT

The project team supported by this grant included Professors Wasan and Nikolov, two graduate students (A. Shah and K. Vijayaraghavan) in addition to a postdoctoral fellow (Dr. A. Trokhymchuk). One master's student graduated and the doctoral student is presently continuing his thesis work under a continuing grant from the EMSP program.

Publications Resulting from DOE Grant (EMSP Project ER14828)

This research has resulted in nine papers published in peer-reviewed international journals including one in the journal *Nature*, in addition to an article in the *Encyclopedia of Surface and Colloid Science*, and two papers in conference proceedings as listed below.

1. “Stability of Thin Liquid Films Containing Polydisperse Particles,” G. Sethumadhavan, S. Bindal, A. Nikolov and D.T. Wasan, Colloids and Surfaces 204, 51-62 (2002).
2. “Confinement-Induced Structural Forces in Colloidal Systems,” D.T. Wasan, A.D. Nikolov, A. Trokhymchuk and D. Henderson, in *Encyclopedia of Surface and Colloid Science*, A. Hubbard, Ed., Marcel Dekker, Inc., NY, pp 1181-1192, (2002).
3. “Foaming Mechanisms in Surfactant Free Particle Suspensions” S.K., Bindal, G. Sethumadhavan, A.D. Nikolov and D.T. Wasan, AIChE J., 48, 10, 2307-2314 (2002).
4. “Effect of Film Curvature on Drainage of Thin Liquid Films,” K. Kumar, A.D. Nikolov and D.T. Wasan, J. Colloid Interface Sci., 256, 194-200 (2003).
5. “Computer Modeling of Ionic Micelle Structuring in Thin Films,” A. Trokhymchuk, D. Henderson, A. D. Nikolov and D.T. Wasan, J. Phys. Chem. B, 107, 3927-3937 (2003)

6. “New Vistas in Dispersion Science and Engineering”, D.T. Wasan, A.D. Nikolov, D. Henderson, AICHE Journal, 49, No. 3, 550-556 (2003).
7. “Spreading of Nanofluids on Solids” D.T. Wasan and A.D. Nikolov, Nature 423, 156-159 (2003).
8. “Rheology Modifiers for Radioactive Waste Slurries,” E.D. Kay, T. Bond Calloway, D.C. Koopman, R.L. Brigman and R.E. Eibling, Proceedings of ASME Fluid Engineering Division Summer meeting, July 6-10-2003.
9. “Radioactive Waste Evaporation: Current Methodologies Employed for the Development, Design and Operation of Waste Evaporators at the Savannah River Site and Hanford Waste Treatment Plant,” T.B. Calloway, W.R. Wilmarth. J.E. Josephs, R.E. Eibling, D.A. Crowley, C. J. Martino, M.E. Stone, C.D. Barnes, A..S. Choi, M.A. Baich, C.M. Jantzen, R.A. Pierce. W.E. Daniel, T.L. White, J.D. Johnson, K. Vijayaraghavan, A. Nikolov and D.T. Wasan. Proceedings of ICEM ‘03: The 9th International Conference on Radioactive Waste Management and Environmental Remediation. September 21-25, 2003, Examination School, Oxford, England.
10. “Stability of Films with Nanoparticles,” G. Sethumadhavan, A. Nikolov, D. Wasan, J. Colloid Interface Sci. 272, 167-171 (2004).
11. “Foaming and Antifoaming in Boiling Suspensions,” D. Wasan, A. Nikolov, A. Shah, Ind & Eng Chem. Res. 43, 3812-3816 (2004).

12. “Laser Scanning Confocal Microscopic Investigations of Simulated Nuclear Waste Structures,” T. B. Calloway, R. L. Brigmon, W.E. Daniel and R. E. Eibling, Journal of Visualization, Vol. 7 No. 2 (2004) 108.

INTERACTIONS

The results of this research have been presented at the following conferences:

- a. American Institute of Chemical Engineers, Indianapolis, November 2002.
- b. EMSP Conference at PNNL Richmond, May 2003.
- c. American Society of Mechanical Engineers meeting, July 2003.
- d. International Conference on Radioactive Waste Management and Environmental Remediation, Oxford, England, September 2003.
- e. American Institute of Chemical Engineers, San Francisco, November 2003.
- f. Meeting coordinating all DOE projects related to foaming, Savannah River, December 2003.
- g. Meeting coordinating all DOE projects related to foaming, Savannah River, December 2004.
- h. EMSP Conference, Savannah River, January 2005.

TRANSITIONS

The IIT researchers have developed two new antifoaming agents to support processing at the Savannah River National Laboratory (SRNL). The first, antifoam IIT-747, has been implemented in the Defense Waste Processing Facility (DWPF) and is currently being used to accelerate the radioactive sludge processing at SRNL. IIT researchers worked closely with SRNL researchers to develop and test antifoam agent IIT-B52 to mitigate foaming in tetraphenylborate

salt solutions. The new antifoam agent was developed for the Small Tank Tetraphenylborate Process to minimize foam produced during precipitation, washing and concentration of high level radioactive waste at the Savannah River Site. This enabled efficient filtration of the slurry without severe foaming experienced using existing commercial antifoam agents. IIT antifoam developers continue to collaborate with SRNL to develop better antifoam agents and modifying the rheology of radioactive slurries. Also, we are currently working with SRNL researchers on antifoam development for the Hanford River Protection Project Waste Plant as outlined in the Future Work section below.

PATENTS

No patents or disclosures have been submitted to date.

FUTURE WORK

IIT has received a continuing grant (DE-FG02-05ER64004) from the EMSP program to further develop effective antifoam agents/defoamers/rheology modifiers that will, when successfully implemented, increase the throughput of the various DOE radioactive waste pretreatment and immobilization processes. Methods developed by IIT under the previous EMSP project and advanced instrumental techniques will be used to determine the effects of soluble and insoluble inorganic components and ionic strength on foam formation and gas entrainment in alkaline and acidic defense waste matrixes. The interaction of insoluble solids components with soluble waste components will be investigated

to support the understanding of the surface interaction between the solid-liquid-gas phases and the bulk solution physical properties (e.g., rheology), which are important in the design and operation of DOE radioactive processes. Once a relationship is established between the key parameters that aggravate foaming and gas entrainment, development of more effective rheological modifiers and antifoams will be initiated and tested using waste matrixes that are specific to the Hanford River Protection Project Waste Treatment Plant (RPP-WTP) LAW/HLW Evaporators and the Savannah River Site (SRS) Defense Waste Processing and Extended Sludge Processing Facilities. The rheological modifiers and antifoams will be tested in simulated and radioactive waste matrixes and compared against existing commercial surfactant technology. These goals are consistent and additive to the DOE site needs (17,19,20).

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FEEDBACK

This project is a collaboration between IIT and SRNL. Every year, we held a coordination meeting of all DOE projects related to foaming at Savannah River and Hanford sites. The reason for the success of this project can be attributed to the fact that the IIT researchers sought to understand the science and limitations in the customers' waste processing facilities through close working relationships with the customers at both SRNL and Hanford.