

**Final Report: Illinois Institute of Technology Antifoam  
Recommendation to SRTC**

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by

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SAVANNAH RIVER SITE

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**D. P. Lambert**

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## EXECUTIVE SUMMARY

The attached report is a summary of the work performed by Dr. Darsh Wasan, Dr. Alex Nikolov, and their researchers at the Illinois Institute of Technology (IIT). IIT is under subcontract to do the basic research necessary to understand the cause of foaming in the Small Tank Tetraphenylborate Process (STTP) and to develop a short list of antifoaming and defoaming agents. IIT recommended the use of three antifoam agents for use in more prototypic testing by SRTC:

1. IITB52 – This antifoam agent was developed for the STTP by IIT. It was the best antifoaming agent and best defoamer tested by IIT.
2. IITPM - This antifoam agent was developed for the STTP by IIT. It was the second best antifoaming agent tested by IIT.
3. Surfynol® DF-110L - This is a commercially available defoamer manufactured by Air Products. It was the second best defoamer tested by IIT.

The text of the report is attached to this cover letter starting on page 3. In addition, IIT videotaped the experiments they performed to demonstrate the defoaming and antifoaming properties of the agents they recommended. This information is included as digital video files on a compact disc that can be viewed using Microsoft's Windows Media Player and Apple's Quicktime viewer. The following files summarize their testing:

File Name	Description	Viewer/Time
Part1.avi	Testing of unwashed 10 wt % KTPB precipitate in foam column without antifoam, also clip of water in foam column	Windows Media Player/1:13
Part2.avi	Formation of KTPB Particle Stabilization Barrier on the air/liquid surface	Windows Media Player/0:32
Part3.avi	Testing of unwashed and washed 10 wt % KTPB precipitate in foam column with 1500 ppmv Surfynol® DF-110L defoamer and IITPM antifoamer	Windows Media Player/8:07
Part4.avi	Demonstration of dancing effect of Surfynol® 104 at air aqueous surface	Windows Media Player/1:26
iitb52.mov	Testing of unwashed 10 wt % KTPB precipitate in foam column with 1000 IITB52 antifoamer	Apple Quicktime 4/2:25

In addition, the following files of interest to the antifoam testing have also been included on the CD:

File Name	Description	Program
Acceptance Letter Issuing IIT final antifoam report TPB.doc	This document including the IIT final report	Microsoft Word
Movie description of B52 tests.doc	Document describing the video (iitb52.mov) of the IITB52 antifoam tests	Microsoft Word
Sr.ppt	Summary of testing to chose the first two antifoam agents with links to the media player four movies (part1.avi, part2.avi, part3.avi, part4.avi).	Microsoft PowerPoint
Iitfinal.doc	IIT's final report (incorporate in this document)	Microsoft Word
Srfigs.ppt	The three figures attached to IIT's final report.	Microsoft PowerPoint
Alex_srs_photo.ppt	Photos of the testing of unwashed 10 wt % KTPB precipitate in foam column with 1000 IITB52 antifoamer	Microsoft PowerPoint

**SUBCONTRACT # AE- 14869S  
ALTERNATIVE SALT PROCESSING  
(New TPB Process)**

*FINAL REPORT*

By

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Date: July 4, 2000

### EXECUTIVE SUMMARY

- The foaminess and foam stability of KTPB 10 wt% unwashed slurry were studied. Foam was generated by aeration of the KTPB slurry by passing air through a sintered glass filter at 150 cm<sup>3</sup>/sec at room temperature (22-24°C). Under these conditions, the foaminess of KTPB is about 450-500 vol% and the foam remains stable for more than an hour.
- The effect of KTPB particles on foam lamella stability was studied. During aeration, the KTPB particles are attached to the air/liquid surface of the floating bubbles and form a particle structural stabilization barrier (PSSB), thereby lending stability to the bubbles.
- Three antifoamers/defoamers with 1500 ppm concentration were tested: Air Products' Surfynol<sup>®</sup> DF 110L, IIT Particle Modifier (IITPM), and IIT B52. The antifoamers were added to the slurry by mixing (subsurface) before commencing the foaming experiments.
- It was observed that IITPM has a better antifoaming performance than DF 110L, however DF 110L has a better defoaming performance than IITPM. IIT B52 has the best antifoaming and defoaming performances even at 1000 ppm.
- The antifoaming/defoaming performance of IIT B52 was tested in two ways: mixing or sub-subsurface (the antifoamer was added to the slurry before foam generation), and by adding the antifoamer over the foam surface (i.e. on the top) during the foam generation. In both cases, IIT B52 showed excellent antifoaming and defoaming performances.
- The antifoaming/defoaming performance of the three antifoamers were studied after 24-hour pre-equilibration of the antifoamers with the KTPB slurry. It was observed that the antifoaming performance of both the DF 110L and IITPM decreased by 30 to 40 vol%. The antifoaming performance of IIT B52 decreased by only 15-20 vol%. However, when the antifoamer was pre-equilibrated with the KTPB slurry (for 24 hours), an additional concentration of only 100 ppm of IIT B52 was needed to eliminate foaminess.
- The antifoaming/defoaming mechanisms of DF 110L, IITPM and IIT B52 were studied. It was shown that the three tested antifoamers/defoamers disintegrate the KTPB particle structure at the liquid-gas surface by generating a surface tension gradient. IIT B52 also is a good wetting agent, causing the KTPB particles to become wetted and settle to the bottom of the vessel.
- The effect of temperature on the foaminess of KTPB slurry was studied. At 40C°, the KTPB slurry is much more viscous than that at room temperature. Due to the increased viscosity, both foaminess and foam stability increased. However, at a concentration of 1000 ppm, IIT B52 is found to be efficient in eliminating foaminess and foam stability.

## INTRODUCTION

The Defense Waste Processing Facility (DWPF) at SRTC is developing alternative salt processing for the immobilization of high level nuclear waste materials such as cesium. The immobilization process includes the formation of KTPB slurry. The process flow diagram of TPB precipitation is shown in (Figure 1). In reactors # 1 and #2 (Figure 1), the TPB precipitation takes place. The precipitation starts in reactor #1 where the input components NaTPB, MST, and water are mixed. During mixing, air bubbles are incorporated into the slurry and foaming is observed. The precipitation processes continue in reactor #2 where the slurry concentration is increased up to 10 wt%. The aqueous solution of the slurry contains 5 mol/l electrolyte and the pH is 16-17 (highly alkaline). It was observed that the foaming is very high (several hundred volume percent) and the foam is very stable. All of these findings for foaming and foam stability during the TPB precipitation were discussed during the technical meeting held at SRTC in February of this year. At this meeting, it was proposed that IIT test the antifoaming efficiency of Surfynol® DF 110L using KTBP slurry. It was suggested that IIT propose two or more antifoamer that will be tested at the SRTC facilities.

## FOAM TESTING

Both foaming and foam stability of KTPB 10 wt% unwashed slurry were studied using the experimental set-up shown in Figure 2. The bottom of a glass cylindrical tube, 3 feet long and 2.5 inches in diameter, is sealed with a sintered glass filter (pore size 10  $\mu\text{m}$ ). Foam was generated by aeration of KTPB slurry by passing air through the sintered glass filter at 150  $\text{cm}^3/\text{sec}$ . Under these conditions, the foaming of KTPB is about 450-500 vol%. The foaming was defined based on the total amount of air incorporated into the slurry. The studies were conducted at room temperature (22-24  $^{\circ}\text{C}$ ). In one case, the slurry was heated to 40  $^{\circ}\text{C}$  and both foaming and foam stability were monitored. A high-resolution optical bench was used to monitor foam bubble dynamics and the foam lamella size distribution (Figure 2).

### **Mechanisms of Foaming in the Presence of KTPB Particles**

We have studied the interactions of KTPB particles and their structure formation at the air/KTPB slurry surface using various optical techniques. It was shown that KTPB particles have a biphilic nature (see our Second Report on Subcontract # AE-14869S, submitted May 10, 2000). Due to their biphilic nature, the KTPB particles are adsorbed at the air bubble air/slurry surface and form a structural stabilization barrier. Figure 3 schematically presents the adsorption of KTPB particles at the air/slurry surface and the formation of a particle structural stabilization barrier (PSSB) inside the foam lamella. To eliminate foaming, it is necessary to disintegrate the PSSB. There are two possible ways to disintegrate the PSSB: one way is to modify the surface of biphilic particles to become hydrophilic (water wetted), and the second method is to break the PSSB by generating a surface tension gradient at the air/slurry surface.

We studied the Surfynol<sup>®</sup> DF 110L surface tension activity at the air/slurry surface. It was observed that the oil droplets of Surfynol<sup>®</sup> DF 110L, by attaching to the air/slurry surface, create a strong surface tension gradient which disintegrates the KTPB particle structure (see the movie clip, part 4, Second Report of Subcontract # AE-14869S, submitted May 10, 2000). The observations reveal that the waxy-oil droplets of nonionic tertiary acetylenic diol (Surfynol<sup>®</sup> DF 110L) attach to the air/slurry bubble surface and spread at the surface, thereby generating a surface tension gradient which disintegrates the KTPB particle structure by adsorbing on the hydrophobic surface. The waxy nature of oil droplets of Surfynol<sup>®</sup> DF 110L definitely makes the KTPB particles more hydrophobic. The defoaming efficiency of Surfynol<sup>®</sup> depends on the oil droplet size. The smaller the droplet size, the better the defoaming efficiency. The droplet size of the Surfynol<sup>®</sup> depends on the type of solvent and the presence of emulsifier. The Surfynol<sup>®</sup> DF 110L will have a better antifoamer performance if the size of the waxy-droplet is controlled. Due to its lower water solubility, most of the Surfynol<sup>®</sup> is not well dispersed into the KTPB slurry and flows as large oily lenses at the air/liquid surface.

Another way to destabilize the PSSB is to modify the surface of KTPB particles, so that they become hydrophilic and cannot attach to the hydrophobic bubble surface (air by nature is hydrophobic). To make the KTPB particle surface hydrophilic, a wetting agent is added to the slurry. The IITPM contains a wetting agent, which modifies the surface of the KTPB particles so that they become saturated and attach to the air bubble surface. However, the most effective way to eliminate foaminess would be to combine the two approaches discussed above: an antifoamer which is a very good wetting agent and also very surface active so that it can create sufficient surface tension gradient to break the PSSB. The formula of IIT B52 is based on this concept. The antifoamer B52 has two features: is a good wetting agent and is very surface active at the air/slurry surface so that it is able to create a surface tension gradient. Proof that IIT B52 is a good wetting agent is the observation that the KTPB particles settle, under gravity, to the bottom of the vessel. Surface tension activity of IIT B52 can be demonstrated by the spreading of a pure water droplet on a hydrophobic surface when IIT B52 is added to it.

#### **Antifoamer/Defoamer Performance**

Using the experimental set-up shown in Figure 2, the foaminess and foam stability of KTPB, 10 wt% unwashed slurry were studied. The KTPB slurry was prepared at SRTC and shipped to IIT. The foam was generated by aeration of KTPB slurry by passing air through a sintered glass filter at 150 cm<sup>3</sup>/sec at room temperature (22-24°C). Under these conditions, the foaminess of KTPB was about 450 -500 vol%, and the foam remains stable for more than an hour.

The antifoaming/defoaming performance of Surfynol<sup>®</sup> DF 110L, IITPM, and IIT B52 was tested with a concentration of 1500 ppm. The antifoamer was added to the slurry by mixing before the foam test. It was observed that IITPM has a better antifoaming performance than DF 110L (e.g. the foaminess with DF 110L is 120 vol% while foaminess in the presence of IIPM is 70-80 vol% ). However, DF 110L has a better defoaming performance than IITPM (e.g., 5 minutes after stopping aeration, the foam level with DF 110L dropped to 30 vol% , while the foaminess with

IITPM dropped to 40-50 vol% ). IIT B52 showed excellent antifoaming and defoaming performance even at a concentration of 1000 ppm. The foaminess in the presence of IIT B52 was the same as the foaminess of aerated water (15-20 vol%).

The antifoaming/defoaming performance of IIT B52 was also tested in two ways: mixing (subsurface adding before foam generation) and by adding over the surface (from the top) during the foam generation. In both cases, IIT B52 showed excellent antifoaming and defoaming performance; the foaminess was similar to the foaminess of aerated water.

#### **Effect on Antifoamer Performance**

The antifoaming/defoaming performance of the three antifoamers was tested after pre-equilibration with KTPB slurry for 24 hours. It was observed that the antifoaming performance of the DF 110L and IITPM decreased by 30-35 %, while the antifoaming performance of IIT B52 decreased by less than 15-20 %. For example, when the KTPB slurry was pre-equilibrated with the antifoamer for 24 hours, an additional concentration of only 100 ppm of IIT B52 was needed to eliminate foaminess. (See the appended movie clip.)

#### **Effect of Temperature on Foaminess**

At 40C°, KTPB slurry is much more viscous than at room temperature. Our preliminary observations show that due to the increased viscosity of the KTPB slurry, foaminess increases (e.g., foaminess at 40C° is 500-550 vol% , while at room temperature it is 450-500 vol% ), and foam stability is increased (to several hours). Only 1000 ppm of IIT B52 added to the foam surface is sufficient to eliminate foaminess.

### **RECOMMENDATIONS**

In the KTPB precipitation process under development at SRTC, the antifoamer is present during the particle precipitation. In order to understand the effect of antifoamer on KTPB particle precipitation, particle size, particle interactions and morphology need to be monitored. This information will help in optimizing the precipitation process and reducing foaminess using low concentrations of antifoamer. For example, [if present] at the beginning of precipitation, the concentration of antifoamer doesn't need to be as high as 1000 ppm. Also, based on information for the particle size interactions and morphology, the KTPB precipitation process can be controlled and foaminess can be reduced and eliminated using less antifoamer.

Foam is formed due to gas bubbles dispersed into the KTBP slurry

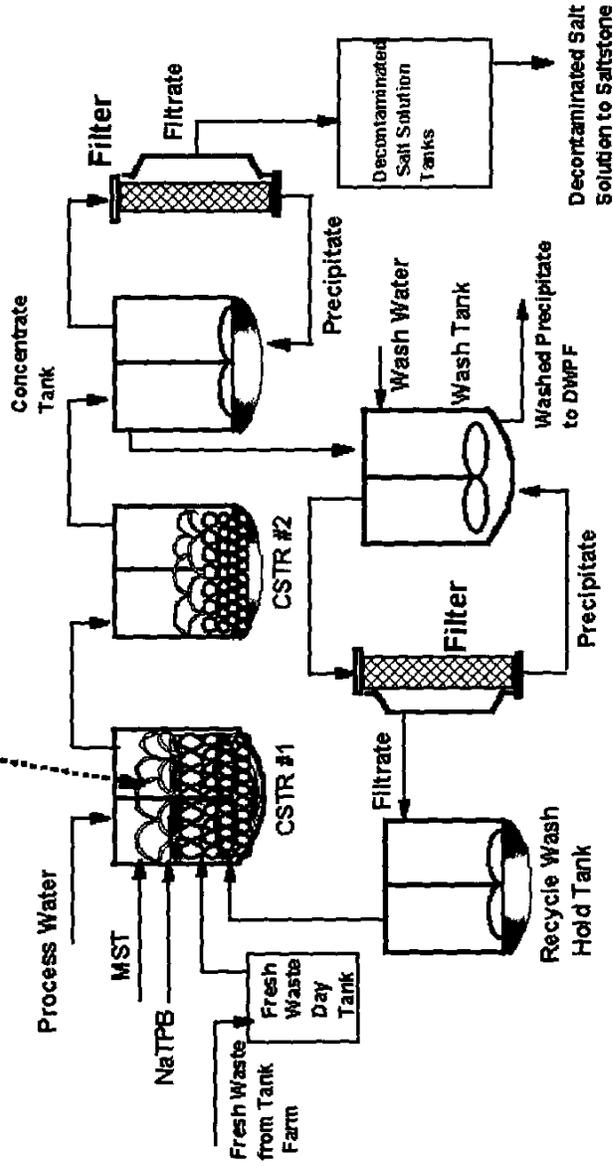


Figure 1  
Process Flow Diagram of TPB Precipitation  
High Level Waste Salt Processing

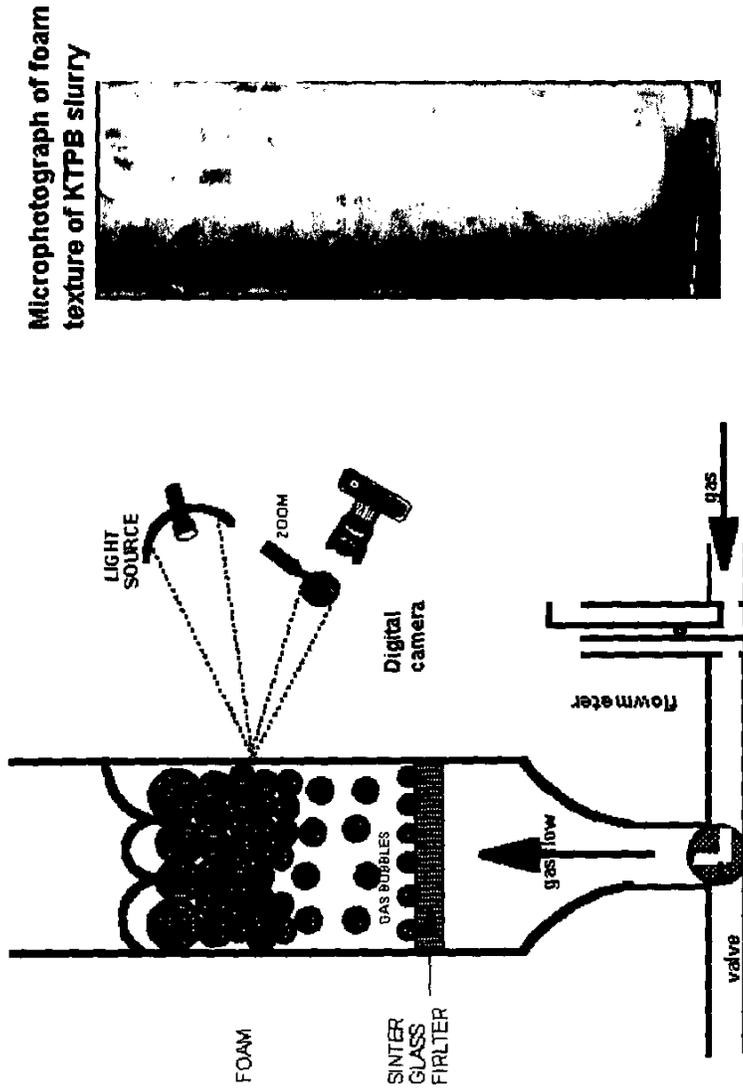


Figure 2  
Sketch of experimental set-up for foam generation  
and observation of foam lamella texture

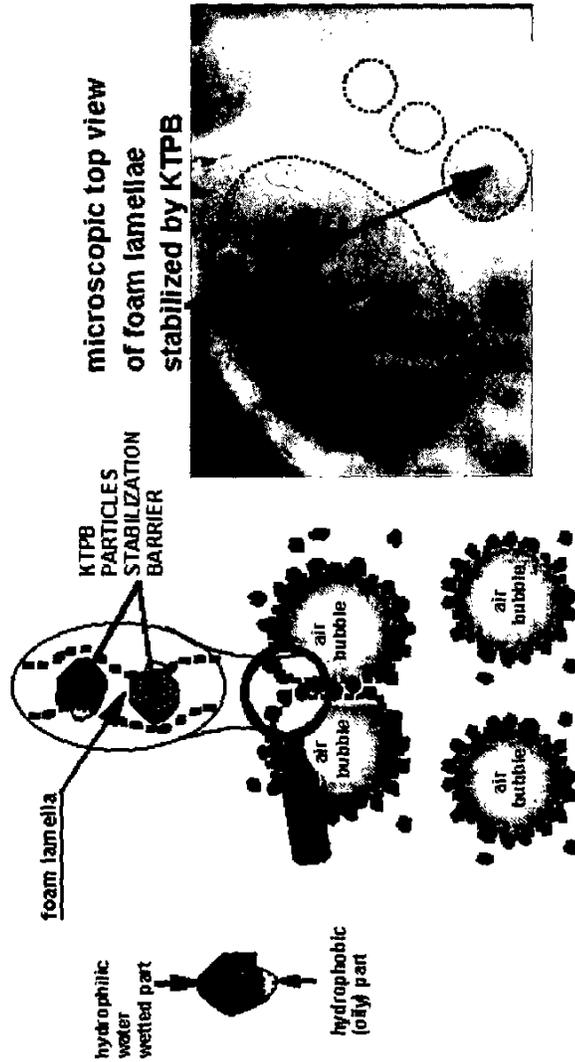


Figure 3

FOAMINESS AND FOAM STABILITY IN THE PRESENCE OF KTPB PARTICLES ARE DUE TO BI-PHILIC NATURE OF THE PARTICLES. PARTICLE BI-PHILICITY CAUSES THE FORMATION OF STRUCTURAL STABILIZATION BARRIER AT AIR BUBBLE SURFACE.

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