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Characterization of Solids Deposited on the Modular Caustic-Side Solvent Extraction Unit (MCU) Coalescer Media Removed in May and October 2014

F. F. Fondeur

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Savannah River National Laboratory™
OPERATED BY SAVANNAH RIVER NUCLEAR SOLUTIONS

Prepared for the U.S. Department of Energy under contract number DE-AC09-08SR22470.

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EXECUTIVE SUMMARY

During routine maintenance, the coalescers utilized in the Modular Caustic-Side Solvent Extraction Unit (MCU) processing of Salt Batch 6 and a portion of Salt Batch 7 were sampled and submitted to the Savannah River National Laboratory (SRNL) for characterization, for the purpose of identifying solid phase constituents that may be accumulating in these coalescers. Specifically, two samples were received and characterized: A decontaminated salt solution (DSS) coalescer sample and a strip effluent (SE) coalescer sample. Aliquots of the samples were analyzed by XRD, Fourier Transform Infrared (FTIR) Spectroscopy, SEM, and EDS. Other aliquots of the samples were leached in acid solution, and the leachates were analyzed by ICP-AES. In addition, modeling was performed to provide a basis for comparison of the analytical results.

Two constituents were identified as being present in the samples and being likely contributors to the coalescer fouling: aluminum hydroxide and amorphous titanium dioxide.

Other constituents identified through laboratory analysis were thought to be artifacts of the rinse solution used to flush the media, rather than constituents present during processing. Other constituents expected to deposit during processing were apparently removed during flushing of the media that the facility performs prior to coalescer removal and /or remained present following flushing, but found to be undetectable by the analytical methods utilized.

The primary constituents that deposited in the DSS were aluminum hydroxide (or oxyhydroxide), amorphous titanium dioxide, and multi-cation oxides (particles with elevated concentration of iron, silicon, aluminum, manganese and nickel), and stainless steel fines. It is believed that soluble titanium and aluminum hydroxide passed through the primary filter and precipitated at the extraction stages (where they experienced a possible pH and dilution change when the salt solution contacts the scrubbing solution). The multi-cation oxides are believed to have passed through the filter and the stainless steel fines are generated from moving parts in the MCU. The data suggest that “pore blocking” played a significant roll in the plugging of the DSS coalescer.

The constituents that deposited in the SE coalescer were Bayerite (aluminum hydroxide), silica, multi-cation oxide solids and stainless steel fines. It is believed that silica particles passed through the filter rather than precipitating from an oversaturated solution where in the presence of aluminum, sodium aluminosilicate (NAS) would have precipitated. Mercury was detected in one location of the SE coalescer. The excess aluminum and titanium found in these coalescers may have entered the salt solution at the Actinide Removal Process (ARP), where a washing with dilute caustic is applied to reduce the sodium concentration-(also to prevent sodium aluminosilicate precipitation) prior to transferring the solids to the Defense Waste Processing Facility (DWPF), with the end result of increasing the amount of aluminum and titanium dissolved in the salt solution. MCU modifier was found in both coalescers. No oxalates were found. The porosities of both coalescer samples were lower than those of unused coalescer media. Because the same two compounds (aluminum hydroxide and amorphous titanium dioxide) were found in both coalescers, a (pulsating) rinse of (possibly warmed) caustic solution may remove them from these coalescers. Such treatment options will require demonstration/optimization prior to implementation.

In the case of the SE coalescer, the data indicate that the deposited inorganic solids were not responsible for the increase in the SE pressure drop. The data appears to indicate that organics (a double emulsion) were likely the cause of the reduced permeability in the SE coalescer. Both amides and modifier were observed on the SE coalescer fibers.

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LIST OF ABBREVIATIONS

ARP	Actinide Removal Process
CSSX	Caustic-Side Solvent Extraction
DI-DD	De-ionized Doubled-distilled
DSS	Dissolved salt solution
DWPF	Defense Waste Processing Facility
EDS	Energy dispersive spectroscopy
FTIR	Fourier transform infrared
IC	Ion chromatography
ICP-AES	Inductively coupled plasma atomic emission spectroscopy
MCU	Modular caustic side solvent extraction unit
MST	Monosodium titanate
NAS	Sodium aluminosilicate
NGS	Next Generation Solvent
PPS	Polyphenylene sulfide
SB	Salt batch
SE	Strip effluent
SEM	Scanning electron microscopy
SRNL	Savannah River National Laboratory
SSFT	Salt solution feed tank
XRD	X-ray diffraction

1.0 Introduction

During operations of the Modular Caustic-Side Solvent Extraction [CSSX] Unit (MCU), pressure drops across the MCU coalescers have been found to increase over time as the volume of solution processed through the coalescer increases. Such pressure drop increases have been attributed to pluggage and /or fouling of the coalescers associated with accumulation of inorganic /organic compounds and /or changes in the characteristics of the coalescer fibers. The net result is reduction in coalescer porosity.

When pressure drops reach a level impeding normal operations, the coalescers are replaced so that effective processing operations can resume. Alternatively, the coalescers are also replaced opportunistically as a routine maintenance practice when other facility outages occur. This applies to the DSS coalescers, the SE coalescers, and the DSS coalescer prefilters. Another potential alternative to replacement of spent coalescers is a treatment that results in the dissolution of accumulated compounds and/or restoration of the original coalescer fiber characteristics by pulsing the appropriate dissolving solution through the coalescer. However, this method may require additional piping and tanks to effect continuous rinsing, recycling and pulsating flow at the coalescer. Furthermore, several dissolving solutions may be required when a wide chemical spectrum of solids are present in the coalescer. The potential to identify such a treatment requires a sound understanding of the compounds accumulated within the coalescers and the nature of any such coalescer fiber changes; however, the economics of in-situ coalescer cleaning preclude this idea from implementation (versus simply replacing with new coalescers).

In this document, characterization of spent DSS coalescer and SE coalescer samples is reported. Another objective is to determine any spatial variation in the solid deposition within a coalescer.

2.0 Quality Assurance

Requirements for performing reviews of technical reports and the extent of review are established in manual E7 2.60. SRNL documents the extent and type of review using the SRNL Technical Report Design Checklist contained in WSRC-IM-2002-00011, Rev. 2.[2] This report was developed in accordance with the protocols identified in Task Technical and Quality Assurance Plan SRNL-RP-2013-00536.[1]

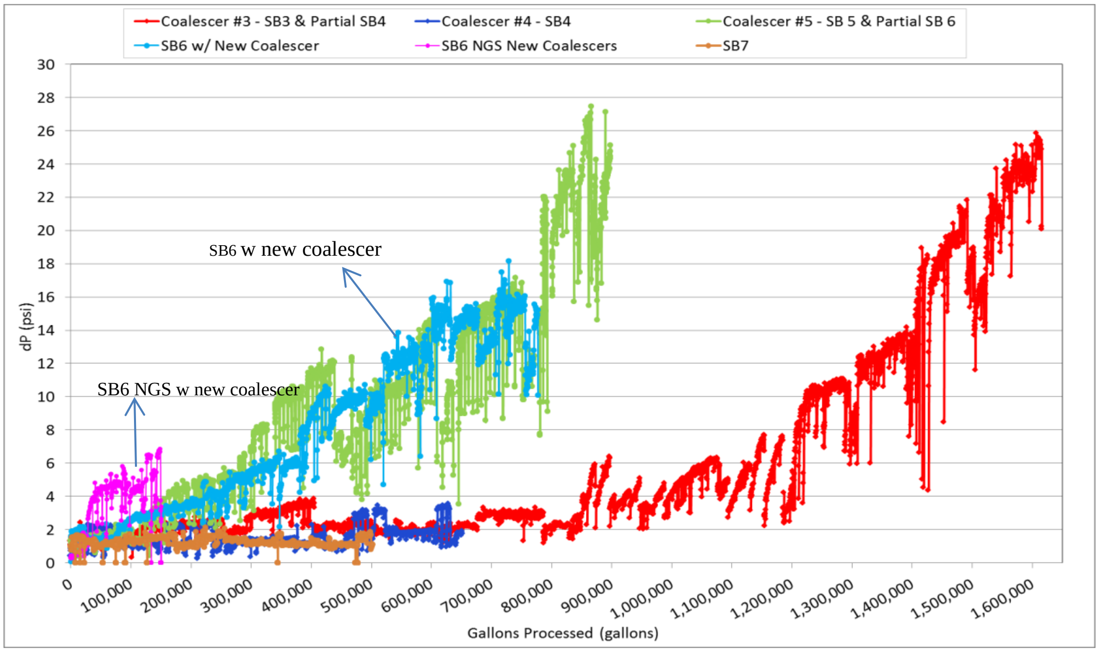
3.0 Background Information

The coalescer samples addressed in this report are those associated with MCU operations during part of Salt Batch 6D (SB6D) and a few batches of Salt Batch 7 (SB7). Salt batches are blended in Tank 21, sampled, and analyzed for qualification. This designates the Salt Batch number. Once qualified the batch (or partial batch) is moved from Tank 21 to Tank 49. If partial batches are sent from Tank 21 to Tank 41, then an alphabetical designation is attached to the number. Salt batches may be adjusted with 50 wt% NaOH solution to reach a free hydroxide concentration of 2.0 ± 0.2 M.[3,4] A batch number is assigned to the content of Tank 49H when its compositional chemistry changes slightly due to the content transfer from Tank 21H. In the amount transferred along with cold chemical additions, the concentration of major chemical components of concern such as hydroxyl, sodium, carbonates, aluminates, phosphate, nitrate, nitrite, and sulfate are typically similar from batch to batch. Batches with slight change in chemical composition are identified with an alphabet letter (for example A, B, C, and D). Specifics of the coalescer media from which the samples were collected are summarized below.

The DSS coalescer was installed on April 2013 and remained in operations until May 2014. A total of 137.4K gallons of salt solution (mostly Salt Batch 6D) were processed through the DSS coalescer and the corresponding maximum pressure drop across the coalescer was approximately 7 psi (see Fig. 1) before it was removed. The pressure drop increase rate was larger when the working solvent was 50:50

NGS:CSSX (see Fig. 1 Magenta line) than it was with the previous solvent (pure CSSX). Note that all pressure drop increases (16x in the blue line curve and 25x in the green and red line in Fig. 1) in Fig. 1 appeared to correlate with the deposition of solids on these coalescers. Solids may deposit at the entrance or throughout the coalescer.[5] The rate of pressure drop rise is typical of pore blocking or deep bed filter plugging at the entrance of these coalescers (as noted by the nonlinear rise in pressure rise with time). Cake build-up did not play a significant role in the pressure rise of these coalescers (typically, pressure rise increases linearly with process time during cake build-up). Prior to sampling (but following completion of the normal DSS operations), the DSS was rinsed with approximately 100 gallons of a solution containing 28.5 to 31.5 weight percent sodium hydroxide solution (~10 M sodium hydroxide) to reduce the gamma emitting radionuclide concentration in the media.

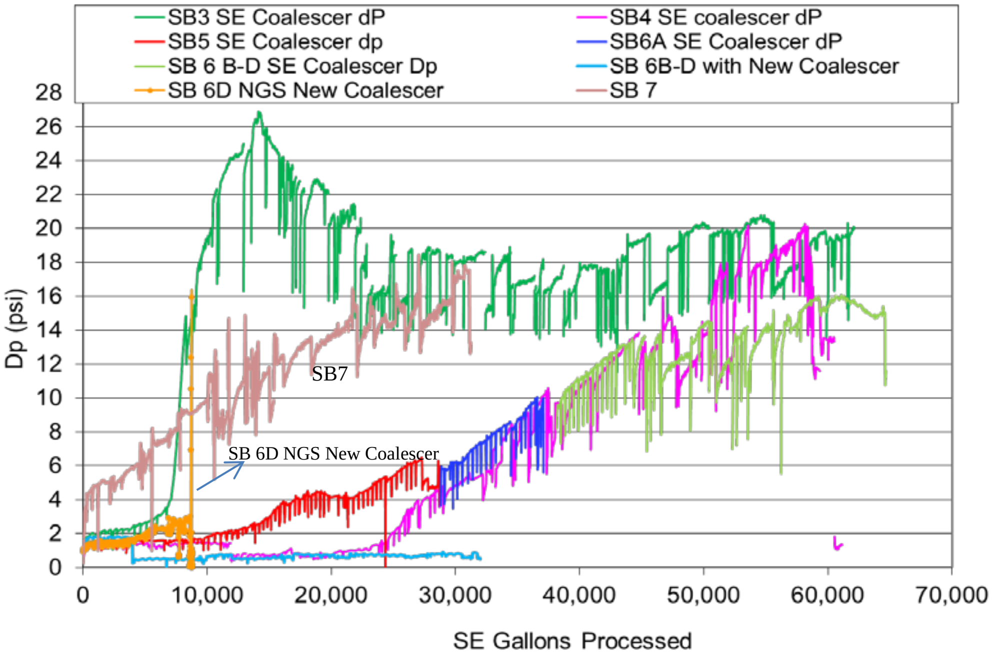
Figure 1 Pressure Drop Across the DSS Coalescer



The SE coalescer was installed on May, 2013 and remained in operations until October 2014. A total of 31.4K gallons of SE solution (Salt Batch 6D and part of Salt Batch 7) was processed through the coalescer and the maximum pressure drop rose almost instantaneously from 3 to 16 psi (an unexpected high rate of increase as seen in the Gold line in Fig. 2). Although, the data in Fig. 2 can be explained by assuming a clogging film at the entrance of the coalescer, the rate of pressure drop rise in Fig. 2 is very large suggesting a different clogging mechanism. Prior to sampling (but following completion of the normal coalescer operations), the SE coalescer was rinsed with 500 gallons of 0.001 M nitric acid solution. Curiously, after the SE coalescer was replaced, the vacuum breakers on the cold feed lines leaked and an inspection of the cold feed lines found aged modifier and algae.

The coalescer characterization activities at SRNL began in late January 2014. Given this time frame, it is clear that the lag times between the sample collection dates and sample analysis dates were on the order of 8 months for the DSS coalescer sample and 3 months for the SE coalescer sample. The lag times provide an indication of the extent that aging could possibly have caused potential crystallization and /or decomposition of compounds.

Figure 2 Pressure Drop Across the SE Coalescer



4.0 Experimental Procedure

4.1 Sample Preparation and Laboratory Analyses

Upon arrival at SRNL, the samples were placed in the Shielded Cells, where they were visually inspected for “gross” flaws and /or defects. No such abnormalities were found (See Fig. 3). The coalescers were then prepared for leaching and chemical analysis. Six one-inch wide “ring” segments of each sample (coalescer) were removed from each respective solid core. Three rings were cut from the closed-end of the coalescer and the other three from the middle. A picture of a cut ring from the SE October 2014 coalescer is shown in Figure 4. The rings inner surfaces appeared loaded with material while inside the ring, the media looked clean. A set of two “ring” segments from each sample (one from the closed-end and one from the middle) were submitted to the laboratory “as is,” for solids characterization by X-ray Diffraction (XRD), Fourier Transform Infrared (FTIR) spectroscopy, Scanning Electron Microscopy (SEM), and Energy Dispersive Spectroscopy (EDS). A second set of two “ring” segments from each sample was placed in its own “leaching bottle” and immersed in 400 mL of 3 M nitric acid, at ambient temperature and under quiescent conditions. The remaining set of two “ring” segments from each coalescer was placed in 400 mL of deionized and double-distilled (DI-DD) water. The weights of each coalescer piece leached in water and nitric acid are listed in Table 1. Leachate aliquots (about 6 mL each) were removed from each bottle (both the nitric acid and DI-DD water) after periods of 1, 7, 14, and 28 days. The respective leachate aliquots from the nitric acid and DI water were submitted to the laboratory for elemental analysis by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP-AES) and for anions by ion chromatography (IC).

Figure 3. A picture of the October 2014 DSS coalescer sent to SRNL

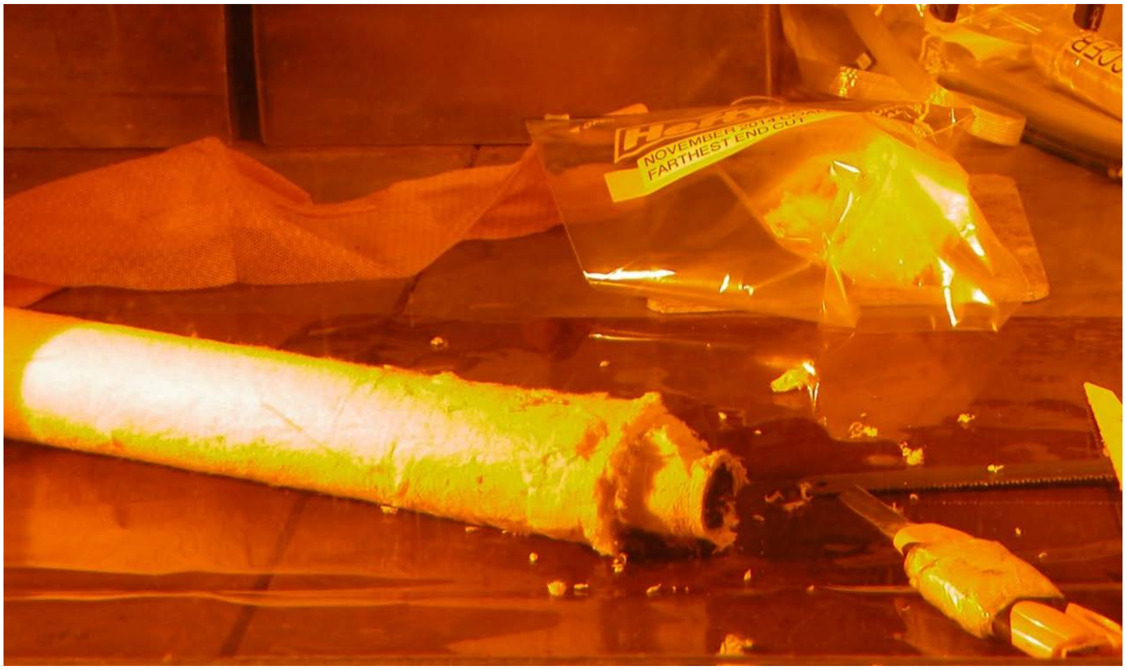
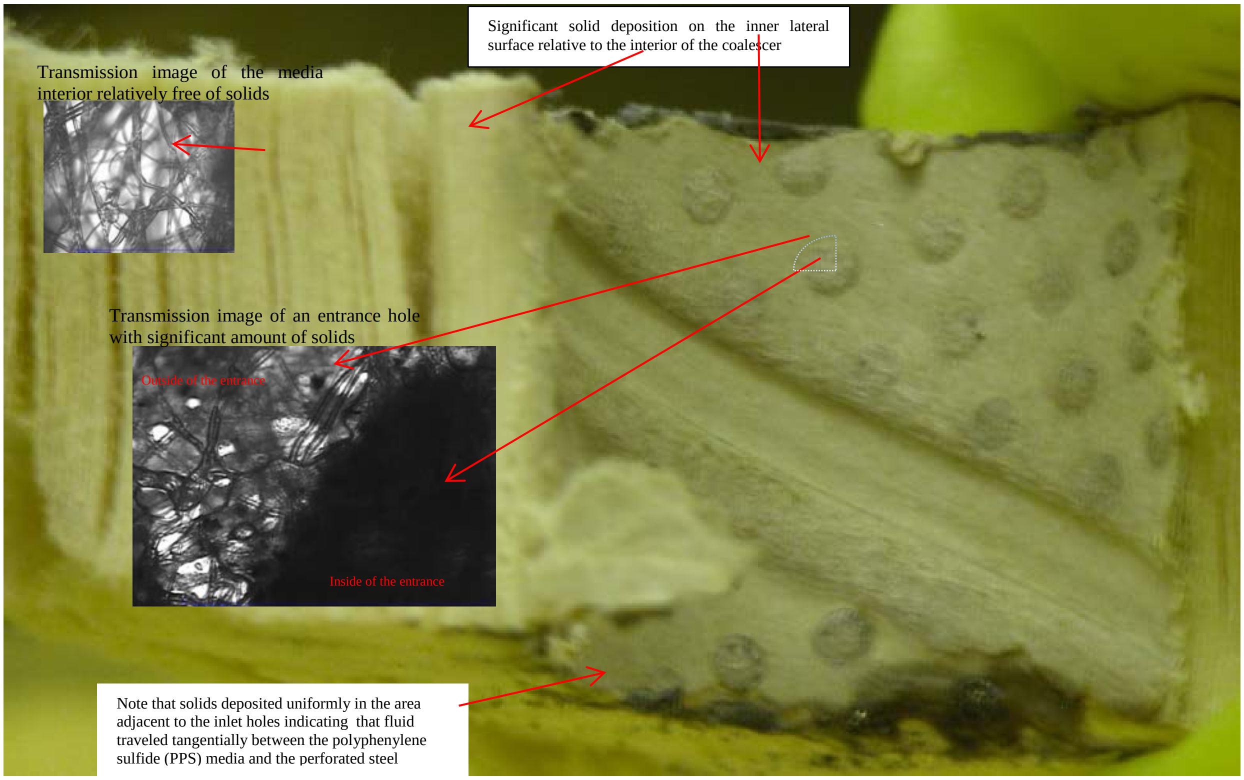


Figure 4. A picture of a cut ring from the SE October 2014 coalescer


Significant solid deposition on the inner lateral surface relative to the interior of the coalescer

Transmission image of the media interior relatively free of solids

Transmission image of an entrance hole with significant amount of solids

Note that solids deposited uniformly in the area adjacent to the inlet holes indicating that fluid traveled tangentially between the polyphenylene sulfide (PPS) media and the perforated steel

Table 1. Gravimetric weights of the cut samples used in the nitric acid and water leaching tests

Coalescer and cut piece location	Donut weight (g) Nitric acid leaching	Donut weight (g) Water leaching
DSS middle piece	9.494	13.982
DSS closed-end piece	13.966	11.569
SE middle piece	7.279	10.399
SE closed-end piece	9.565	7.747

5.0 Results and Discussion

5.1 X-ray Diffraction (XRD)

A summary of the solid-phase constituents identified by XRD is given in Table 5-1, and the corresponding XRD spectra are given in Figures 5 and 6. Note that the polyphenylene sulfide compound identified in each XRD spectrum is the primary structural component of the coalescer media and it has a diffraction peak at 20.5 ° 2θ. As such, it should not be considered a solid-phase compound accumulating during processing and or contributing to coalescer fouling. For this reason, consideration of the existence of polyphenylene sulfide is removed from further discussion.

Table 5-1. Summary of Constituents Identified by XRD

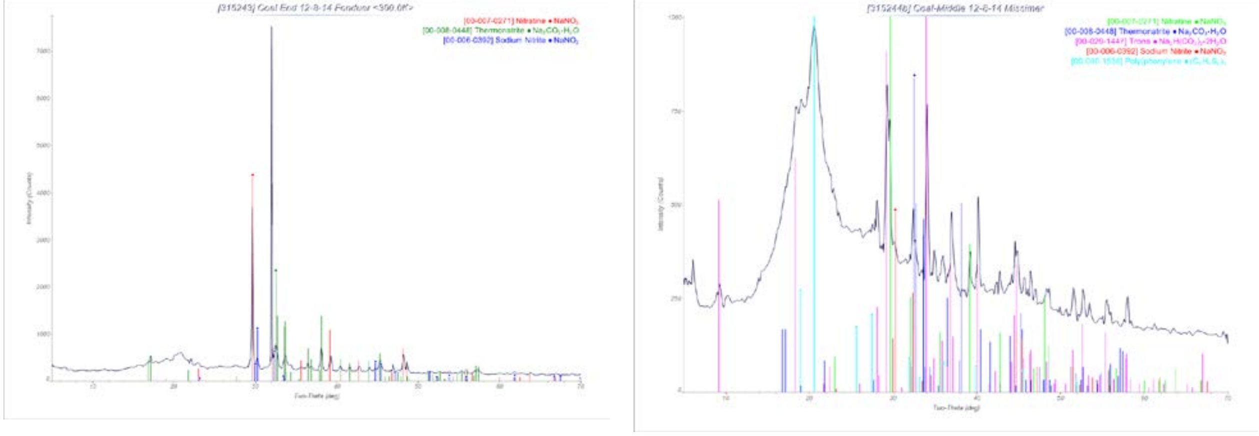
Sample Descriptor	$NaNO_3$ (Nitratine)	$NaNO_2$ (Sodium Nitrite)	$Na_2CO_3 \cdot H_2O$ (Thermonatrite)	$Na_3H(CO_3)_2 \cdot 2H_2O$ (Trona)	SiO_2
DSS Coalescer	X	X	X	X	
SE Coalescer					X

Four solid-phase constituents were detected in the DSS coalescer sample: sodium nitrate (nitratine); sodium nitrite; sodium carbonate monohydrate (thermonatrite) and sodium bicarbonate (Trona). This last compound was not observed in the characterization of the DSS coalescer removed in November 2013 from MCU.[6] These compounds are consistent with the solids predicted by the OLI software modeling conducted in Ref. 6 for SB5 and part of SB6. These salt batches are chemically similar and differ only in the concentrations of minor components that do not impact the assumptions at MCU. In this case, SB5, SB6, and SB7 are chemically similar (hydroxides, nitrates, nitrites, sulfates, carbonates, phosphates, sodium, aluminum, and other major species of concerns). Thus, the predicted solid precipitations in Ref. 6 still apply for the salt batches processed by the coalescers studied in this paper. Appendix A shows a table from Ref. 6 summarizing the most likely solids that may precipitate at MCU under various processing conditions. Note this table only indicates possible compounds that may form assuming no kinetic barriers and that they are derived from a limited database. The predicted compounds are consistent with the October 2014 ARP sample analysis and the recent characterization of the 512-S guard filter analysis.[7a]

No detectable sodium aluminosilicate (NAS) solids were observed on these coalescers. NAS (as well as aluminum compounds and sodium oxalate) has been observed in the Salt Solution Feed Tank (SSFT) (Tank 102).[7b] Aluminosilicates were also observed in previous coalescer characterization efforts and their absence may be due to new mixing practices (less agitation energies in Tank 102) or the increase in hydroxide concentration in the macrobatches to minimize NAS formation.

The existence of sodium bicarbonate was not predicted (based on OLI modeling) to exist in supernatant or in the conditions of the high hydroxide post-processing rinse. Given the relatively long storage period of the DSS coalescer (~ 7 month), a possible explanation for the bicarbonate is the absorption of carbon dioxide from the air into the (neutral pH) residual solution of the coalescer. However, no Trona compound was detected in the middle section of the DSS coalescer which also experienced the same storage time and conditions as the closed-end piece. Therefore, the spatial variation of Trona deposition in the DSS coalescer is possibly due to the hydrodynamics inside a closed-end perforated tube like the coalescer. As mentioned in a previous study, crystalline sodium nitrite may have originated from the decomposition of sodium nitrate.

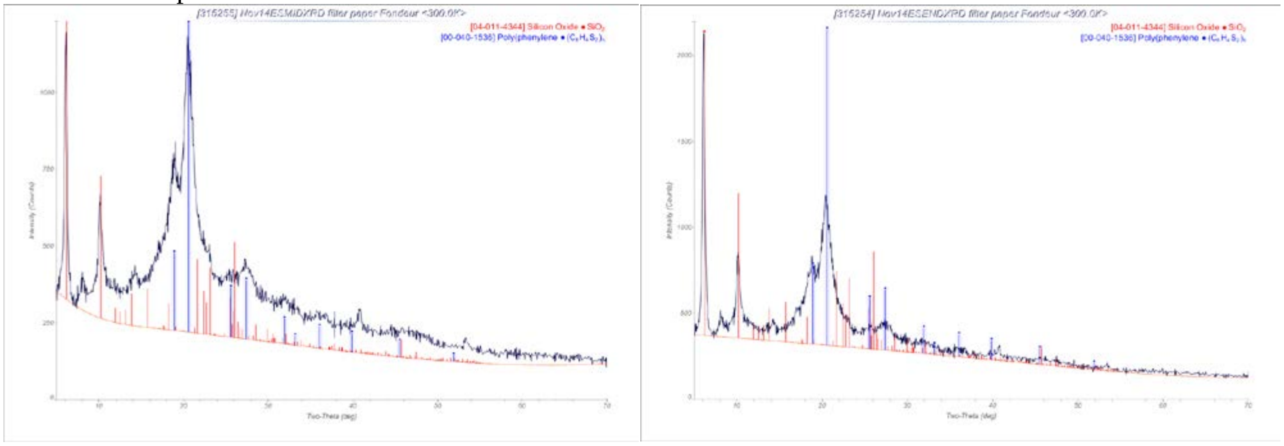
Figure 5. XRD spectra of the mid and end-section of the DSS (May 2014) Coalescer



May 2014 Mid-point DSS

May 2014 Closed-End DSS

Figure 6. XRD spectra of the mid and end-section of the SE (October 2014) coalescer



Nov 2014 Midpoint SE

Nov 2014 Closed-end SE

Non-detection of the other solid-phase constituents predicted for the DSS stream (sodium oxalate, hexasodium carbonate bisulfate, and hydroxysodalite dihydrate) does not necessarily mean that these constituents were absent from the samples, but it indicates that such constituents did not dominate the solids and or were not present in crystalline form. Also, given the likely spatial variations associated with solids precipitating in the coalescer media, there is the understanding that concentrations of deposited solids in one segment of a given sample can be markedly different from those in another segment.

For the SE coalescer sample, a low concentration of crystalline silicon dioxide particles was detected at the middle and at the closed end of the SE coalescer. In previous studies, aluminosilicates or pure aluminum hydroxide compounds have been observed in the SE coalescer.[5,1] Silica has been observed in the pre-filter element (coalescer), used for filtering the macrobatches of salt solution from Tank 49H, and in the extraction contactors (SEP-401)[7]. Carry-over salt solution from centrifugal contactor 501 (heated to 33ºC) may be the source of silicon to the SE coalescer. In a low dielectric media, such as the MCU solvent, silica (as well as other elements capable of forming oxygen polyhedral) readily precipitates as a crystalline oxide. As discussed above, the few peaks in the XRD spectrum do not assure the absence of solids – it merely indicates that solid-phase constituents were not present in high concentrations and /or that existing solids were non-crystalline. Moreover, the pore network in the coalescer is radially directional and the deposited solids in this network may appear unidirectional to the X-ray beam from the XRD. Thereby, XRD peaks may not be proportional to the solid concentration (bias low relative to solid concentration) on the coalescer. As can be seen in Figures 5 and 6, except for one plot, the amorphous PPS peak is as large as the other peaks assigned to salts and glass giving the impression of a coalescer packed or saturated with lots of crystalline solids.

5.2 Fourier Transform Infrared (FTIR) Spectroscopy

A summary of the constituents identified by FTIR is given in Table 5-2, with the supporting data presented in Figures 7, 8, and 9.

Table 5-2. Summary of Constituents Identified by FTIR

Sample Descriptor	$NaNO_3$ (Nitratine)	$NaNO_2$ (Sodium Nitrite)	CO_3	PseudoBoehmite	Bayerite Gibbsite	Modifier*	Amide
DSS Coalescer	X	X	X	X		X	X
SE Coalescer					X	X	X

*1-(2,2,3,3-Tetrafluoropropoxy)-3-(4-*sec*-Butylphenoxy)-2-Propanol
Note: Isopar™L was also observed in both coalescer

Four primary constituents were detected in the DSS sample: sodium nitrate (nitratine); sodium nitrite; carbonate; and pseudoboehmite (see Fig. 6.3 and 6.4). Pseudoboehmite is an aluminum oxyhydroxide (AlO(OH)) that typically results from overheated aluminum hydroxide or from precipitation of a highly caustic supersaturated aluminum solution. Detectable quantities of the MCU modifier[♣] were also observed. The FTIR also detected an amide-containing substance in one location of the DSS sample (middle portion). Given the known process chemistry and the anticipated precipitation tendencies, all of the constituents identified by FTIR are consistent with expectations. The source of the amide could be a decomposition product from the MCU suppressor *N, N',N"*-Tris(3,7-dimethyloxtyl)guanidine (TiDG) or bacteria from groundwater (or rain water run-off). The scrub and Strip solutions are made by a vendor and the purchase requirements call for the use of de-ionized water in the solutions make-up.

The nitrate, nitrite, carbonate and aluminum hydroxide compounds were predicted to precipitate (see Ref. 5). In this case, pseudoboehmite was observed indicating a fast precipitation of aluminum from a solution containing high caustic and nitrate concentration.

[♣] Modifier is 1-(2,2,3,3-Tetrafluoropropoxy)-3-(4-*sec*-Butylphenoxy)-2-Propanol

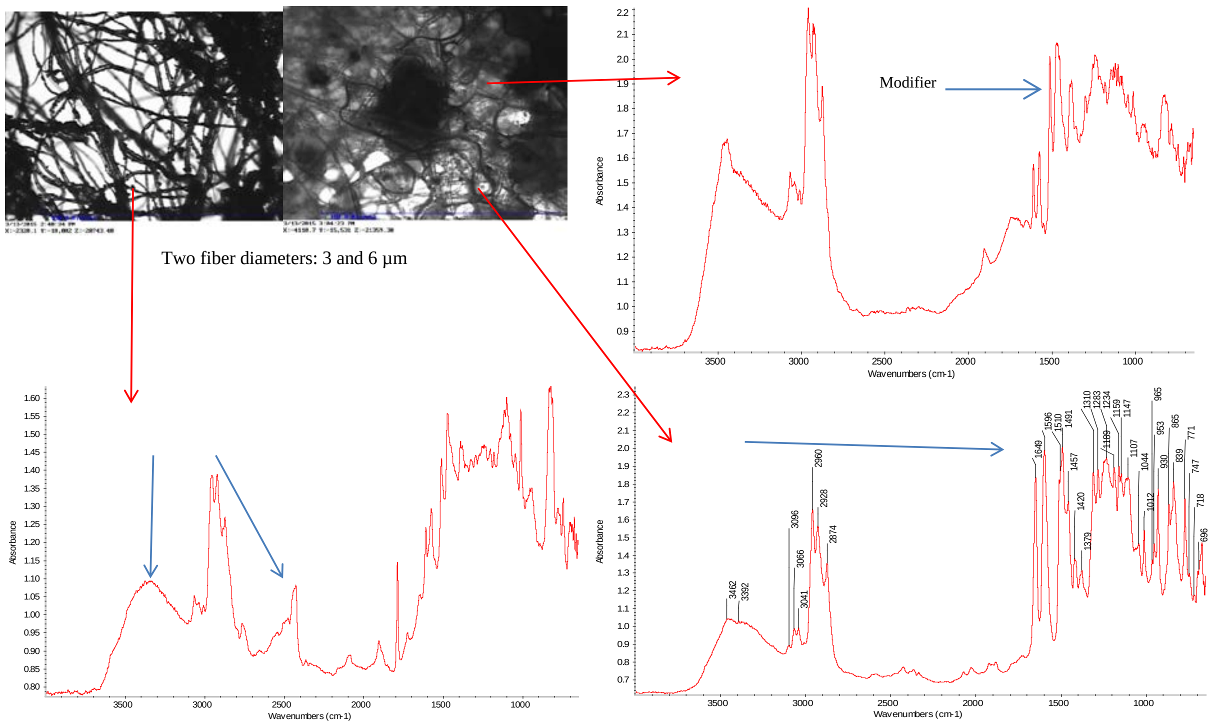
In the case of the SE coalescer, two constituents were found (Gibbsite and Bayerite) along with detectable quantities of the MCU modifier (see Fig. 7). The aluminum hydroxide, Bayerite, forms under fast crystallization conditions (or high aluminum saturation) while Gibbsite forms under slower crystallization conditions[8] and the fact that both were observed suggest an aging process where the Bayerite is transforming into Gibbsite (Gibbsite is thermodynamically more stable).

The absence of NAS is proof of the additional step personnel has taken to prevent its precipitation given the past widespread presence of NAS in MCU operations; this includes increasing the hydroxyl concentration of the supernatant to enhance the NAS solubility.[9,10]

The presence of MCU modifier in the coalescer is reasonably consistent with past observations of the modifier sorbing on different surfaces, as seen in previous cold simulant testing.

The inner most surface of the media that touches the perforated tube has substantial solid loading in both coalescers. The lateral porosity (the available empty space between the fibers when viewing the inner surface in a normal direction) at the hole entries was reduced from 60% to 42%. That reduction coupled with an increased pressure forced the aqueous solution to push the media away from the perforated tube and permeate the media in areas adjacent to the entrance holes on the perforated tube. The coalescer which is meant to capture material throughout its medium, instead acted as a dead-end filter with particles blocking the pores (or reducing the porosity) of its inner-most surface (for which it is not designed for). The caking began at the closed end of the coalescer, forcing the permeate to exit at the entrance of the coalescer (a shorter working coalescer). The fiber thickness in these media ranged from 3 to 6 microns. Thus, these media were less efficient coalescing oil droplets less than 3 microns.

Figure 7. FTIR Results of the middle portion of the DSS coalescer May 2014



Two fiber diameters: 3 and 6 µm

Figure 8. FTIR Results of the Closed-end of the DSS coalescer May 2014

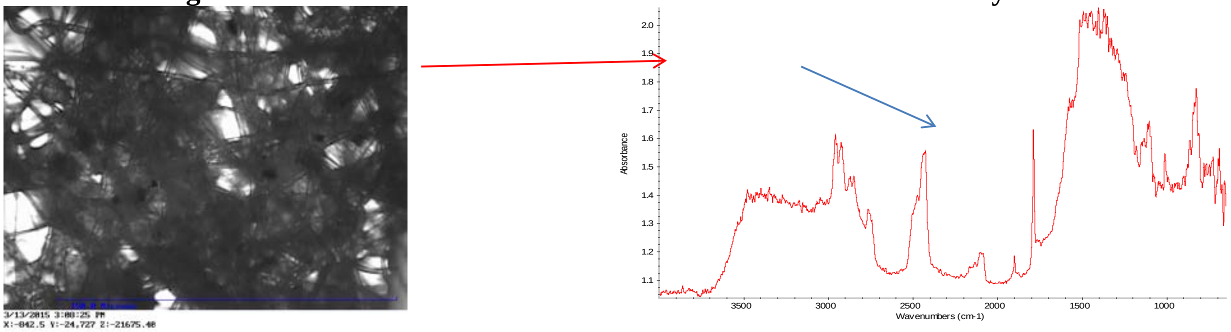
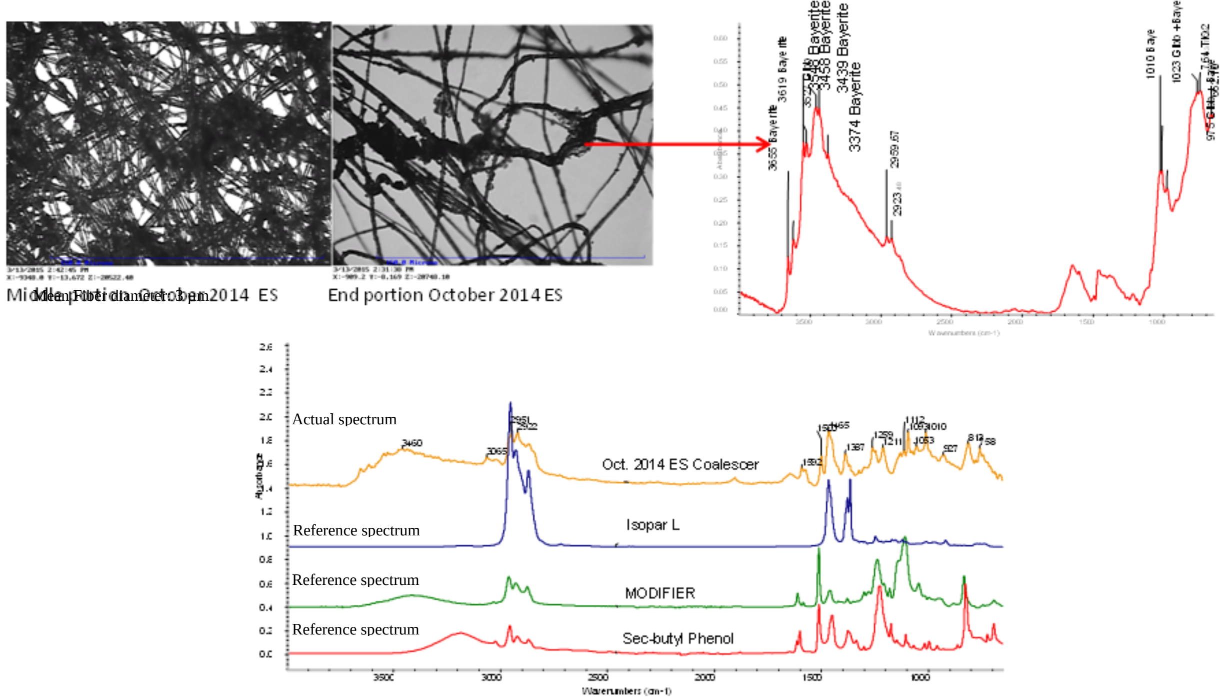


Figure 9. FTIR Results for the SE coalescer (Oct. 2014) middle portion (left) and closed-end (right)

Scanning Electron Microscopy (SEM) Energy Dispersive Spectroscopy (EDS)



5.3 Scanning Electron Microscopy (SEM) and Energy Dispersive Spectroscopy (EDS)

A summary of the elemental constituents identified by EDS is given in Table 5-3, with the individual SEM images and EDS results presented in figures 10, 11, 12, and 13.

Table 5-3. Summary of Elements Identified by EDS

Sample Descriptor	Al	Si	U	Fe	K	Hg	Ni Cr	P	Ti
DSS Coalescer Middle	X	X		X	X		X		X
DSS Coalescer Closed-End	X	X			X			X	
SE Coalescer Middle	X		X	X			X		X
SE Coalescer Closed-End	X		X	X		X			X

Based on results of the EDS analyses, a relatively broad suite of elemental constituents was identified. Several elements were detected in all two samples, including Al, Fe, K, U, P, Si, and Ti, all normal constituents of salt waste (with the exception of Ti). This data also appears to suggest the presence of amorphous aluminosilicates since XRD only detected aluminum and silica compounds. Aluminosilicates have a strong IR adsorption band at 1000 cm^{-1} and none were observed. But it is possible that both silicates and aluminates were precipitated independently on the coalescers. Sodium aluminate solids were observed in the Feed tank in 2014. Additional elements that were detected in the DSS sample included Cr, Ni, and Fe, all from stainless steel debris, and Na from the DSS stream. The Fe could possibly be from sludge (sludge particles that passed through the 0.1 and 0.5 microns at the Actinide Removal Process [ARP]) or from the iron sulfamate precipitation, however it is most likely that, due to the accompanying co-presence of chromium and nickel which are typical, the source of the Fe is from the stainless steel debris. In contrast, the only additional element detected in the SE coalescer sample was mercury.

Elements such as Cr and Ni may be introduced into DSS and SE solution through introduction of stainless steel fines. Phosphorous is typically undetectable in the SE stream, but may participate in reactions that generate measurable quantities of insoluble phosphate compounds over time. Potassium, a potential competitor to cesium for MaxCalix in NGS, is possibly an impurity from the cold chemical additions to the tanks for corrosion control or from radioactive decay processes in the Tank Farm. Titanium derives from the unintended caustic leaching of MST used at ARP for the removal of actinides and strontium [Ref. 14].

Based on the SE coalescer SEM images (Fig. 12 and 13), a high concentration and non-uniform deposition of solids occurred. Where the solid concentration is highest, the lateral porosity is low (~42%) and the solids appear to cover most of the free space. In contrast, in the cases where the solids accumulations are minor, the solids can be seen as individual particles clinging to fibers, with only slight reductions in porosity.

Figure 5. SEM and EDS spectra of the middle portion from the DSS coalescer (May 2014)

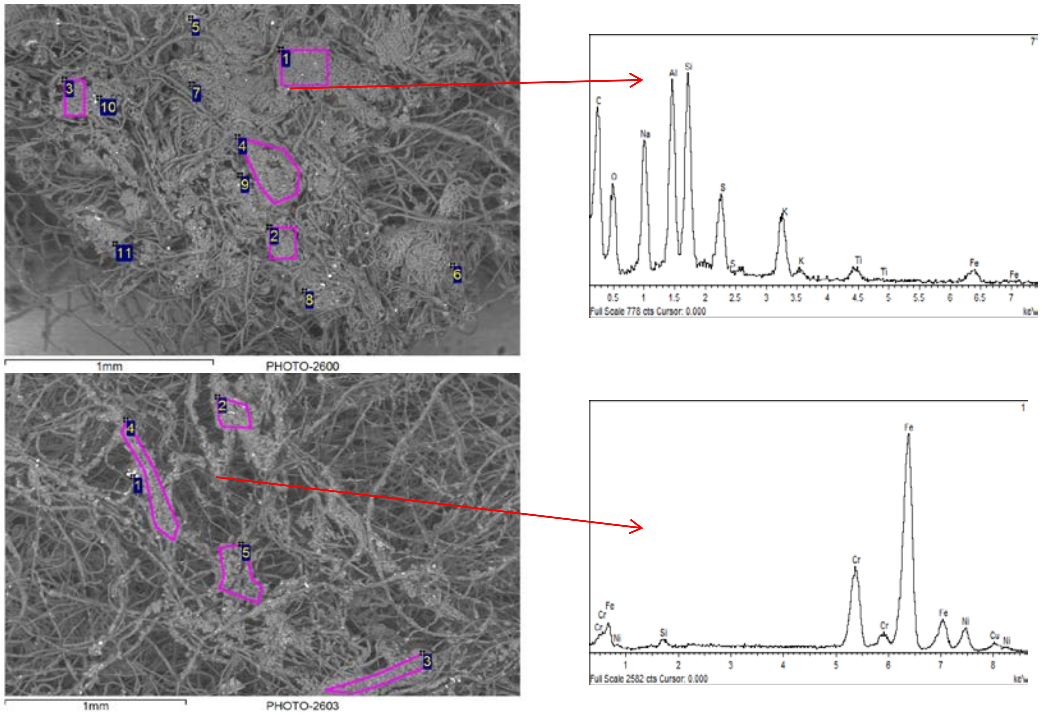


Figure 6 SEM and selective EDS spectra of the closed-end portion of the DSS coalescer

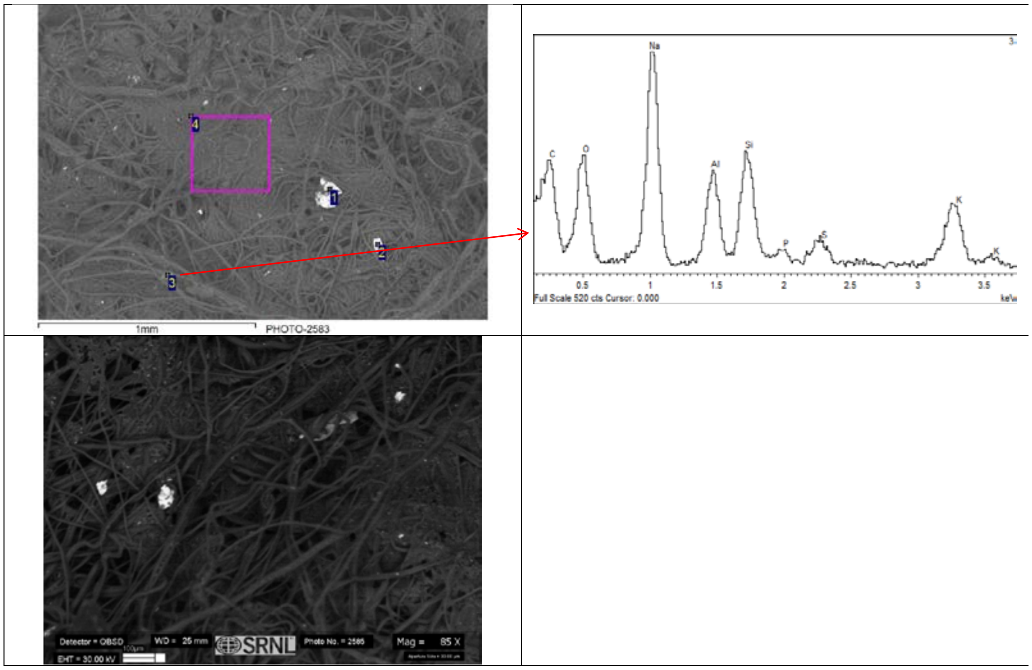


Figure 7 SEM and selective EDS spectra of the closed-end portion of the SE coalescer (October 2014

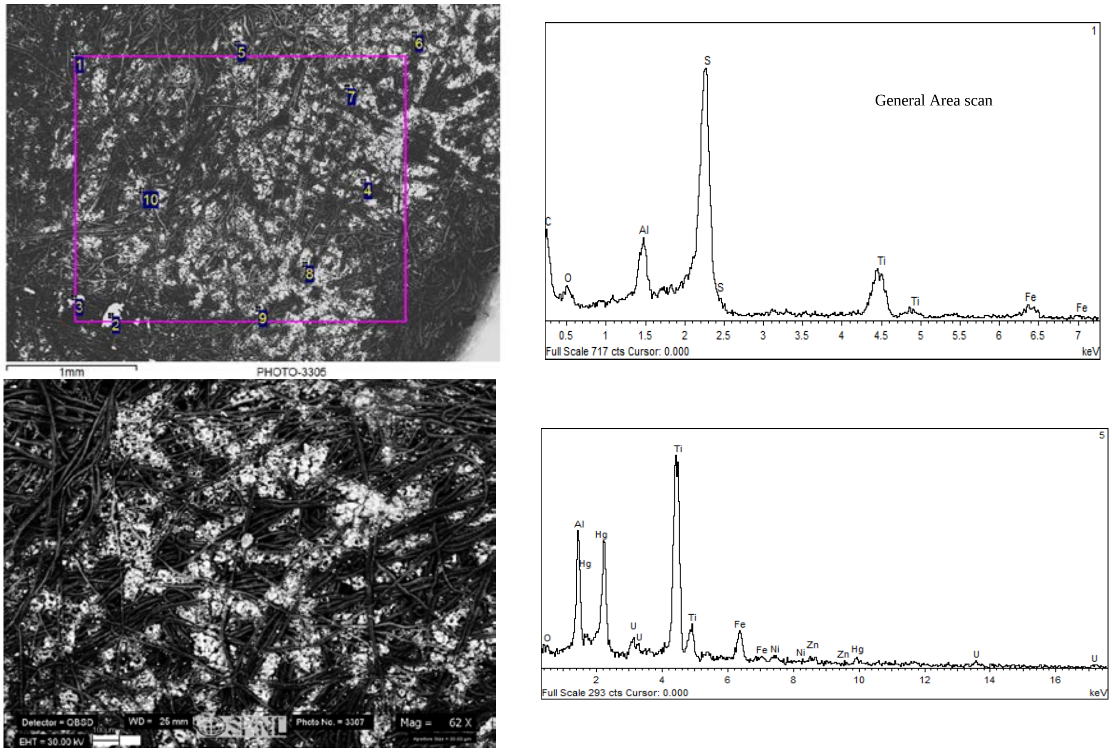
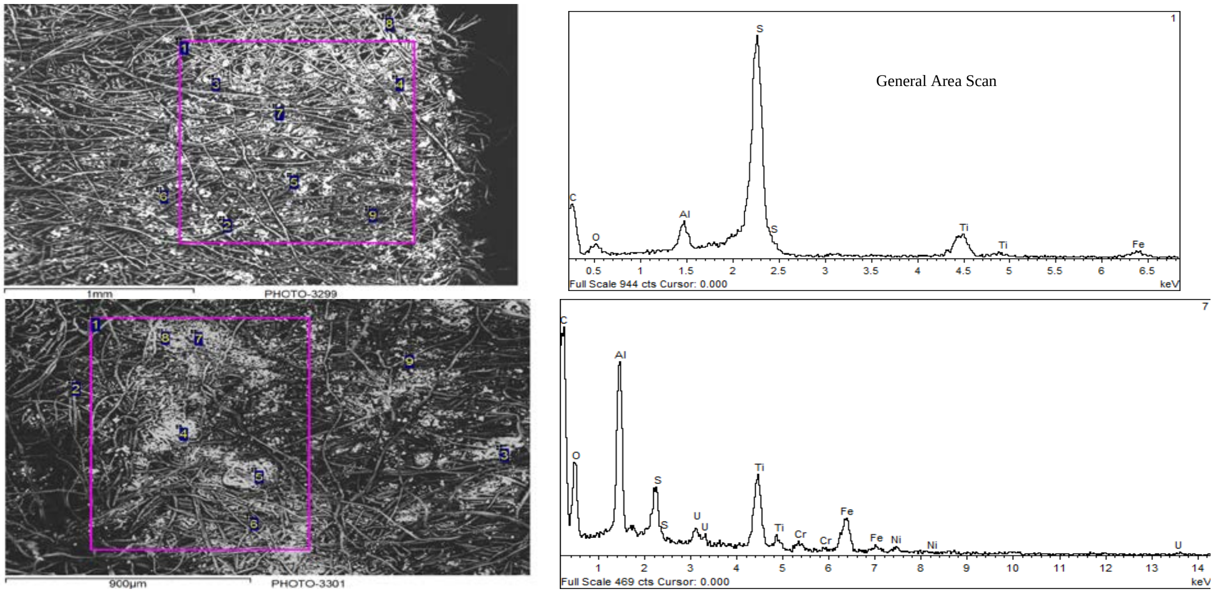


Figure 8 SEM and selective EDS spectra of the middle portion of the SE coalescer



5.4 Inductively Coupled Atomic Emission Spectroscopy (ICP-AES) of Coalescer Leachates

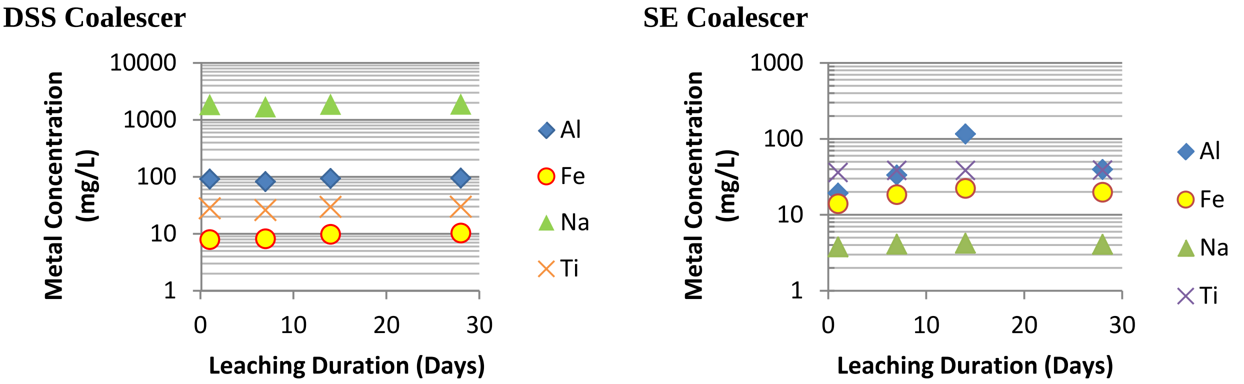
A summary of the most dominant metals found in the coalescer leachate solutions at the end of the leaching period (t = 28 days) is given in Table 5-4. Twenty eight days were sufficient for the dominant metals in the leachates to reach steady state (see Fig. 14). For the DSS coalescer leachates, the relative concentrations of the dominating metals were: Na > Al > Ti > Fe. This ranking, on a mass basis, differs slightly from that in Ref. 7 (for DSS Na > Al > Ti > Fe, SRNL-STI-2011-00513) (SE: Al > Ti > Fe > Na >Cr = Si, SRNL-STI-2010-00088) but it is consistent with previous DSS coalescer characterization reports. The concentration level of each metal is much lower than in previous DSS coalescer reports. In contrast, for the SE coalescer leachate, the relative concentrations of the dominating metals were: Ti ≈ Al > Fe > Na. This ranking differs slightly from that listed in Ref. 7 but it is consistent with prior SE coalescer reports. Comparing the two coalescer leachates (DSS vs. SE) reveals their differences in relative constituent abundances which are due to a combination of the pH effects (high pH for the DSS and a pH of 8-10 for the SE stream) and the compositional differences between the DSS and the SE solution. However, the leachate data clearly indicates that both coalescers accumulated substantial concentrations of aluminum hydroxides (and oxyhydroxides) and amorphous titanium. Regardless, the dominance of these four metals reflects the contributions of salt waste (Na and Al), monosodium titanate (Ti), sludge (Fe and Al), and /or steel fines (Fe). Given that noticeable levels of mercury have recently been observed at MCU, it is important that this data has not shown (yet) any evidence of enhanced corrosion of the steel components (especially 316 SS).

A complete listing of the ICP-AES results, for all of the elemental constituents measured, is given as a function of the leaching time in Table 5-5 and Table 5-6.

Table 5-4. Dominant Elements in Leachates from the closed-end of each coalescer

Sample Descriptor	Metal Concentration, mg/L (t = 28 days)						
	Na	Ti	Al	Fe	Si	P	K
DSS Coalescer	1890	29.8	94.7	10.3	8.1	30.1	10.5
SE Coalescer	4.1	38.7	39.4	19.8	1.1	2.8	<0.78

Figure 9. Dominant metal concentration from the closed-end of the DSS and SE coalescer leachates



DSS Coalescer

SE Coalescer

Table 5-5. Elemental Constituent Concentrations in DSS Coalescer

Element	**DSS Coalescer (May 2014) Concentration, mg/L**							
	Closed End				**Middle Portion**			
	t = 1 day	**t = 7 days**	**t = 14 days**	**t = 28 days**	**t = 1 day**	**t = 7 days**	**t = 14 days**	**t = 28 days**
Ag	< 0.388	< 0.388	< 0.388	< 0.388	< 0.388	< 0.388	< 0.388	< 0.388
Al	91	82.3	93.7	94.7	34.8	37.3	37.2	37.6
B	< 4.35	< 4.35	< 4.35	< 4.35	< 4.35	< 4.35	< 4.35	< 4.35
Ba	0.261	0.237	0.275	0.281	0.239	0.273	0.277	0.281
Be	< 0.028	< 0.028	< 0.028	< 0.028	< 0.028	< 0.028	< 0.028	< 0.028
Ca	1.41	1.3	1.73	1.5	1.16	1.24	1.22	1.24
Cd	< 0.254	< 0.254	< 0.254	< 0.254	< 0.254	< 0.254	< 0.254	< 0.254
Ce	< 2.24	< 2.24	< 2.24	< 2.24	< 2.24	< 2.24	< 2.24	< 2.24
Co	< 0.35	< 0.35	< 0.35	< 0.35	< 0.35	< 0.35	< 0.35	< 0.35
Cr	1.27	1.24	1.45	1.53	0.411	0.496	0.512	0.696
Cu	< 0.708	< 0.708	< 0.708	< 0.708	< 0.708	< 0.708	< 0.708	< 0.708
Fe	7.92	8.18	9.78	10.3	2.61	3.18	3.32	4.77
Gd	< 0.888	< 0.888	< 0.888	< 0.888	< 0.888	< 0.888	< 0.888	< 0.888
K	9.79	9.31	11.3	10.5	< 7.82	< 7.82	< 7.82	< 7.82
La	< 0.394	< 0.394	< 0.394	< 0.394	< 0.394	< 0.394	< 0.394	< 0.394
Li	2.47	< 2.54	2.63	< 2.54	< 2.54	< 2.54	< 2.54	< 2.54
Mg	< 1.72	< 1.72	< 1.72	< 1.72	< 1.72	< 1.72	< 1.72	< 1.72
Mn	1.06	0.973	1.1	1.12	< 0.16	< 0.16	< 0.16	< 0.16
Mo	< 3.59	< 3.59	< 3.59	< 3.59	< 3.59	< 3.59	< 3.59	< 3.59
Na	1860	1690	1880	1890	820	837	834	837
Ni	1.03	0.95	1.07	1.05	< 0.91	< 0.91	< 0.91	< 0.91
P	26.7	27.1	29.8	30.1	< 9.46	< 9.46	< 9.46	< 9.46
Pb	< 26	< 26	< 26	< 26	< 26	< 26	< 26	< 26
S	< 240	< 240	< 240	< 240	< 240	< 240	< 240	< 240
Sb	< 8.22	< 8.22	< 8.22	< 8.22	< 8.22	< 8.22	< 8.22	< 8.22
Si	7.28	6.75	7.63	8.1	3.77	4.19	4.35	4.32
Sn	< 18.6	< 18.6	< 18.6	< 18.6	< 18.6	< 18.6	< 18.6	< 18.6
Sr	< 2.55	< 2.55	< 2.55	< 2.55	< 2.55	< 2.55	< 2.55	< 2.55
Th	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10
Ti	27.9	25.9	29.6	29.8	6.06	6.36	6.35	6.39
U	< 14	< 14	< 14	< 14	< 14	< 14	< 14	< 14
V	< 0.138	< 0.138	< 0.138	< 0.138	< 0.138	< 0.138	< 0.138	< 0.138
Zn	1.49	1.39	1.55	1.54	0.396	0.417	0.408	0.43
Zr	< 0.124	< 0.124	< 0.124	< 0.124	< 0.124	< 0.124	< 0.124	< 0.124

Al, B, K, Li, Na, Si, Ti, Sn, and Zr are expected to be soluble in salt solution
Ba, Ca, Co, Cr, Mg, Mn, Ni, Th, and Zn are expected to be insoluble in salt solution

Table 5-6. Elemental Constituent Concentrations in SE Coalescer

Element	ES October 2014 Concentration, mg/L							
	Closed End				Middle Portion			
	t = 1 day	t = 7 days	t = 14 days	t = 28 days	t = 1 day	t = 7 days	t = 14 days	t = 28 days
Ag	< 0.039	< 0.039	0.0472*	< 0.039	< 0.039	< 0.039	0.0424	< 0.039
Al	19.2	33.3	116*	39.4	4.63	8.3	30.1	10
B	4.02	4.66	4.65	4.75	1.03	1.28	1.33	1.35
Ba	0.32	0.362	0.364	0.371	0.0784	0.0971	0.102	0.107
Be	< 0.003	< 0.003	< 0.003	0.0028	< 0.003	< 0.003	< 0.003	< 0.003
Ca	1.15	1.21	1.41	1.25	0.674	0.602	0.638	0.617
Cd	0.317	0.333	0.329	0.337	0.0611	0.0699	0.071	0.0771
Ce	< 0.224	< 0.224	< 0.224	< 0.224	< 0.224	< 0.224	< 0.224	< 0.224
Co	0.0374	0.0386	0.0396	0.0394	0.0406	0.048	0.0454	0.047
Cr	1.27	1.48	2.6*	1.6	0.463	0.575	1.2	0.686
Cu	0.318	0.343	0.422*	0.353	0.108	0.0971	0.12	0.112
Fe	14	18.4	22.2*	19.8	5.58	7.88	10.2	9.23
Gd	< 0.089	< 0.089	< 0.089	< 0.089	< 0.089	< 0.089	< 0.089	< 0.089
K	< 0.782	< 0.782	< 0.782	< 0.782	< 0.782	< 0.782	< 0.782	< 0.782
La	< 0.039	< 0.039	< 0.039	< 0.039	< 0.039	< 0.039	< 0.039	< 0.039
Li	< 0.254	< 0.254	< 0.254	< 0.254	< 0.254	< 0.254	< 0.254	< 0.254
Mg	0.557	0.606	1.59*	0.65	0.27	0.273	0.553	0.287
Mn	0.234	0.268	1.07*	0.291	0.245	0.0934	0.305	0.109
Mo	< 0.359	< 0.359	< 0.359	< 0.359	< 0.359	< 0.359	< 0.359	< 0.359
Na	3.77	4.1	4.21	4.08	2.8	2.73	2.74	2.71
Ni	2.09	2.35	2.79	2.49	0.887	0.974	1.15	1.05
P	2.58	2.7	2.68	2.83	0.788	0.91	0.953	1.15
Pb	< 2.6	< 2.6	< 2.6	< 2.6	< 2.6	< 2.6	< 2.6	< 2.6
S	< 24	< 24	< 24	< 24	< 24	< 24	< 24	< 24
Sb	< 0.822	< 0.822	< 0.822	< 0.822	< 0.822	< 0.822	< 0.822	< 0.822
Si	0.494	0.812	1.61*	1.1	0.569	0.484	0.796	0.648
Sn	< 1.86	< 1.86	< 1.86	< 1.86	< 1.86	< 1.86	< 1.86	< 1.86
Sr	< 0.255	< 0.255	< 0.255	< 0.255	< 0.255	< 0.255	< 0.255	< 0.255
Th	< 0.151	0.182	0.17	0.157	< 0.151	< 0.151	< 0.151	< 0.151
Ti	36	38.3	38.4	38.7	5.64	7.25	7.6	7.96
U	7.67	8.05	8.87	8.08	1.58	1.78	1.92	1.81
V	< 0.014	< 0.014	< 0.014	< 0.014	< 0.014	< 0.014	< 0.014	< 0.014
Zn	2.25	2.4	2.55	2.44	0.594	0.613	0.671	0.667
Zr	< 0.578	< 0.578	< 0.578	< 0.578	< 0.578	< 0.578	< 0.578	< 0.578

*Anomaly high values were verified with a second measurement on the same liquid sample indicating contamination. These values (at 14 days) were not used in any calculation.

An inspection of Table 5-5 and 5-6 shows that the concentration levels of the elements in these tables are less than those reported in Ref. 7. The differences are attributed to the lower volume of salt solution processed by the DSS coalescer and correspondingly lower volume of stripping solution used. Despite using a lower density and lower viscosity solvent (NGS vs. CSSX), both coalescers reached noticeable pressure drops (closer to the administrative limit of 25 psi) at much lower salt solution volumes. In addition to a slightly compositional difference between batches, we believe that there is an effect of the new solvent NGS (including its caustic scrub solution and stripping boric acid solution) that negatively impacts the performance of the coalescers, namely, it might be inducing faster heterogeneous nucleation and precipitation of solids (pseudoBoehmite, Bayerite, and amorphous titanium oxide) on the coalescer and it may be causing the formation of water-in-oil emulsion inside the coalescer as well. Oil-in-water emulsion droplets on PPS fiber are possibly repelling each other and that reduces their removal from the coalescer. The accumulation of oil-in-water emulsion on the coalescer reduces the available porosity.

For example, mixing with 25 mM NaOH (15:1 salt solution to scrub solution) scrub solution may precipitate aluminum compounds.
The sources of the detectable metals listed in Table 5-5 and 5-6 have origins from site processes. The Ca, Zn, and P might originate from the additives in degraded oil (oil used at the centrifuges). Phosphate may also originate from the phosphate-based extractant used at the solvent extraction process at the HB-line. Boron is believed to come from the strip solution (boric acid) used at MCU.

The aluminum, iron, manganese, nickel, chromium, uranium, and silicon are prevalent in the SRS supernatant from different processes (spent fuel dissolution H-Canyons) at the site. The titanium is from the known solubility of titanium (in Monosodium Titanate (MST)) in caustic solution). Potassium, calcium and barium may originate from both nuclear decay processes of the nuclear waste as well as trapped chemicals in the cold additions to the Tanks for corrosion control and batch qualification. Boron is believed to come from the strip solution (boric acid) used at MCU.

An estimate of the amount of solids deposited on the coalescer is shown in Table 5-7. This estimate is based on the following expectation:
Excluding P, Ba, Ca, and Na, all detectable metals listed in Tables 5-5 and 5-6 are assumed to be non-hydrated oxides. Examples include AlOOH for aluminum, Fe_2O_3 for iron (instead of ferric hydride), SiO_2 for silicon, TiO_2 for titanium, ZnO_2 for zinc, and UO_2 for uranium. Also shown in Table 5-7 is the estimate of the solids on the coalescer by subtracting the weight of an "as received" coalescer of the same fiber diameter from the gravimetry data shown in Table 1.

Table 5-6. Estimation of the solid loading on the coalescer by gravimetric and ICP-AES data

Source	Ratio	DSS	SE
Table 1 (gravimetric)*	g of Solids / g of Coalescer	1.13	0.46
	mg of solids / mL of Coalescer	123	50
Table 5-5 and Table 5-6 (ICP-AES)#	g of Solids / g of Coalescer	0.44	0.01
	mg of solids / mL of Coalescer	48	1.5

* Assumes that the grams per length of the coalescer (without the metal mandrel core) is 0.258 g/mm. It also assumes that the volume of a one inch tall piece of coalescer is 25.78 mL. These values were subtracted from the gravimetric values in Table 1 to obtain an estimate of the material loaded on the coalescer. These numbers captures organic, dried salts and other hydrated solids on the coalescers.
# Assumes all metals are oxides (excluding Na, Ba, Ca and the nonmetals). Numbers from the acid leaching ICP-AES data of each coalescer's end were used for estimating their solids loading.

As can be seen in Table 5-7, a large discrepancy was observed between the gravimetric and the ICP-AES data of the DSS coalescer. The large difference is due to the larger set of hydrated solids and organics detected in the gravimetric measurement and to a larger extent, to a larger error in matching the weight of the leached coalescer piece to a blank coalescer. Therefore, the amount of solids loaded determined from the ICP-AES analysis of the leachate appears to be a more reliable estimate. If the estimates from the ICP-AES are correct, a larger amount of solids loaded on the DSS coalescers consistent with the visual inspection of the optical pictures from each coalescer (more solids were in the DSS relative to the ES coalescer). The estimated solid loadings on the SE coalescer is approximately half the concentration observed in the pluggage of other packed bed systems[9] where a 50% increase in the pressure drop was observed with just 5 mg of carbon particles per mL of packed bed. With 1.5 mg of solids per mL of coalescer, the pressure drop increased 16x in the October 2014 SE coalescer. The solids loading on the DSS were 32x larger than in the SE coalescer. This supports the view that pore blocking and an organic material deposition played a significant role in the pluggage of the coalescers (rather that pore reduction by inorganic solid deposition). In the case of the SE coalescer, the numbers obtained by gravimetric measurements differed by order of magnitudes from the ICP-AES estimates. It is believed that a significant portion of the SE coalescer gravimetric data is due to water. Nonetheless, the ICP-AES data indicates that the deposited solids can't account for the increase in the pressure drop (16x) observed with this coalescer in November 2014. In order to have a 16x increase in the pressure drop, the coalescer porosity must be reduced from 0.9 to 0.6 and based on the optical pictures where the PPS fibers are nearly pristine (with an occasional coverage of solids in some places), the solids could not have reduced the porosity of the SE coalescer. It is more likely that if the deposited solids had any effect, it would be at the closed-end of the SE coalescer. There is no evidence that precipitated solids caused the pressure drop increase. Instead, this data seems to indicate that possibly another plugging mechanism may have reduced the SE coalescer permeability. Although no evidences have been obtained, it is believed that secondary emulsion formation or sorption may be occurring at the coalescer fibers (illustrated in Fig. 10). Droplets of secondary emulsions (aqueous droplets inside NGS droplets) have less buoyancy than pure NGS droplets and it is expected that these type of droplets will grow to bigger sizes before they detach from the coalescer fibers. Therefore, a significant reduction in porosity may occur (these structures are reversible and can be removed in higher flow regimes).

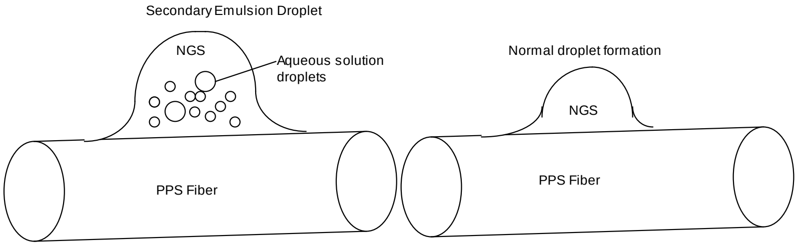


Figure 10. A pictorial description of a secondary emulsion droplet on a PPS fiber

The preferential solid loading at the closed end of the coalescer is best observed from the ratio of the ICP-AES data of the different locations in the coalescers. The ratios of the elemental analysis of the leachate from the closed-end to the middle portion of each coalescer (DSS and SE) were calculated and are shown in Table 5-8. These ratios demonstrate there are more (table 5-8) solids loaded on the closed-end of each coalescer than in the middle portion as noted by the large ratio values. The elements known in the past to deposit on the coalescer are highlighted in bold type font. This observation is consistent with the fluid hydrodynamics of perforated closed-end tubes.[11] Note that in the case of the DSS coalescer, a ratio value could not be calculated for many of the elements since many of them were not detected.

Table 5-8. Elemental concentration ratio of the leachate from the closed-end to the middle portion of the DSS and SE coalescer

Element	Concentration Ratio at 28 Leaching Days (DSS)	Conc. Ratio at 28 Leaching Days (SE)
Al	2.5	3.9
B	LOD	3.5
Ba	1.0	3.5
Ca	1.2	2.0
Cd	LOD	4.4
Cr	2.2	2.3
Cu	LOD	3.2
Fe	2.2	2.1
Mg	LOD	2.3
Mn	>7.0	2.7
Na	2.3	1.5
Ni	>1.2	2.4
P	>3.2	2.5
Si	1.9	1.7
Ti	4.7	4.9
U	LOD	4.5
Zn	3.6	3.7

LOD = Limit of Detection

The higher concentration of deposited solids at the closed-end of both coalescers is consistent with the expected hydrodynamics of a closed-end perforated tube.[10] Since the coalescers are approximately one meter long, the pressure drop along the perforated tube (mandrel) is insufficient to slow the axial flow down the coalescer and the highest discharge flow out of the coalescer (or out of the perforated tube) then occurs at the closed-end of the tube. Thus, if a suspension of solids enters the coalescer, it will flow down the coalescer and exit at the closed-end first. Once the discharge friction at the closed-end of the coalescer increases, the discharge flow out of the coalescer shifts toward the inlet of the coalescer. In the case of a supersaturated solution, a high liquid flow over the polyphenylene sulfide fibers may cause heterogeneous precipitation over these fibers. Longer coalescers or increased axial flow friction (possibly from solid deposition along the wall of the coalescer) may cause significant discharge flow at the middle and inlet region of a coalescer.

In a previous report (Ref. 7), it was recommended to look for oxalates in the leachate of the coalescers. Along this line, Ion Chromatography analysis of the water leachate from each coalescer (DSS and SE) showed nitrates and nitrites (see table 5-9). Both nitrates and nitrites are typically found in the salt solution. The absence of phosphate in the water leachate indicates the phosphorus containing solids are more soluble in nitric acid. No detectable oxalate anions were observed (oxalates were observed in Ref. 5) despite oxalates being observed in salt solution feed tanks and centrifuge contactors.

Table 5-9. IC-Anion analysis of the water leachates from the closed-end of the DSS and SE coalescers

IC-Anions	DSS Coalescer, µg L				SE Coalescer, µg L			
Component	1 day	7 days	14 days	28 days	1 day	7 days	14 days	28 days
Fluoride	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10
Formate	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10
Chloride	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10
Nitrite	310	366	372	378	< 10	< 10	< 10	< 10
Bromide	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10
Nitrate	1990	2360	2140	2150	< 10	15	< 10	< 10
Phosphate	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10
Sulfate	< 100	< 100	< 100	< 100	< 10	< 10	< 10	< 10
Oxalate	< 10	< 10	< 10	< 10	< 10	< 10	< 10	< 10

Given that both coalescers have aluminum and titanium compounds, these compounds are soluble in low ionic strength caustic media but their rate of dissolution may be different (particle size and shape) and a once-pass through rinsing of the coalescer may not remove sufficient amount of solids to restore the permeation through a bed of nonwoven fibers. Given that it is far easier to trim the salt solution to prevent NAS precipitation and replace the coalescers with new elements rather than clean a plugged coalescer, it is recommended that this practice should be continued.

6.0 Conclusions

1) Pluggage of the DSS was most likely due to the accumulation of :

   - Precipitation of Multi-cation oxide solids downstream from the filter that included iron, silicon, titanium, nickel, manganese, and aluminum took place at MCU (past the prefilter) and some of these particles were captured by the coalescer. A significant portion of the solids found in the coalescer were PseudoBoehmite (aluminum oxyhydroxide), amorphous titanium dioxide compounds and carried over stainless steel fines. A significant portion of the solids preferentially deposited at the closed end of the coalescer where the fluid velocity is negligible but the pressure is significant for outflow. Evidence of algae deposition was observed but their concentration was not as high as that of the deposited solids. The data suggest that "pore blocking" plays a role.

   Sodium nitrate, sodium nitrite, and hexasodium carbonate bisulfate, although present in the DSS sample, were not responsible for pluggage during operations, as they were generated due to the post-processing high hydroxide rinse. MCU modifier was also found on these fibers as expected since the modifier has a tendency to sorb on most materials (Isopar™L has a tendency to evaporate).

2) Pluggage of the SE coalescer was most likely due to an organic liquid. The accumulation of solids (aluminum hydroxide and amorphous titanium dioxide) was not sufficient to cause a 16 times pressure drop increase. Some organic amides were observed on the coalescer fibers indicating organics played a role in the reduced permeability of the SE coalescer in November 2014. MCU modifier was found on these fibers as expected. One location of the analyzed SE sample contained mercury by SEM-EDS.

3) Given the common chemistry of the solids in both coalescers, treatment options for reducing coalescer pluggage will likely require use of high caustic cleaning solutions to dissolve the solids. Such potential treatment options will require demonstration/optimization through testing prior to implementation.

7.0 Recommendation

The recommendations listed in Ref. 6 are presented again for future DSS coalescer sample handling, it is recommended that:

- Flushing of the coalescer media should be performed using solutions with the same approximate hydroxide concentration as the feed solution. In the case of the SB6D and SB7 samples, a rinse solution containing approximately 2 M sodium hydroxide would be best (although not hard-piped to the coalescer plumbing, the scrubbing solution can be used to rinse the DSS coalescer). Maintaining a consistent hydroxide concentration will maximize the chance that accumulated solids remain unchanged during rinsing. The SE coalescer can be rinsed with the stripping solution (boric acid).

- In the case that organic sorb on the coalescer, replacing the coalescer is a better option than rinsing the plugged coalescer.

8.0 References

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[14] C. L. Crawford, “Actinide Removal Process, October 2014 Sample Analysis”, SRNL-STI-2014-00609, April 2015.

[15] K. M. L. Taylor-Pashow, F. F. Fondeur, and S. D. Fink, “Leaching of Titanium from Monosodium Titanate (MST) and Modified MST (mMST)” SRNL-STI-2012-00237, August 2012.

[16] L. H. Delmau and B. A. Moyer, “S*olvent Blending Strategy to Upgrade MCU CSSX Solvent to Equivalent Next-Generation CSSX Solvent*”, ORNL-LTR-NGCSSX-010, July 22, 2011

Appendix A. Solid Phase Compounds Expected to Precipitate Out of Solution

Waste Stream	Processing Conditions	Solids Expected to Precipitate Out of Solution*
DSS	Routine processing ([OH$^-$] ≈ 2 M)	- Aluminum hydroxide - Sodium oxalate - Cancrinite monohydrate
	Post-processing ([OH$^-$] ≈ 10 M)	- Sodium nitrate - Sodium oxalate - Sodium carbonate monohydrate - Hexasodium carbonate bisulfate - Hydroxysodalite dihydrate
SE solution	Low constituent concentrations Iron present as Fe(II)	- No solids expected to precipitate
	High constituent concentrations Iron present as Fe(II)	- Silica
	Low constituent concentrations Iron present as Fe(III)	- Iron oxide
	High constituent concentrations Iron present as Fe(III)	- Iron oxide - Silica

Inputs for Equilibrium Modeling of Solids in the DSS Coalescer*

Species	Concentration, M	Quantification Method
Sodium nitrate	2.82	Ion chromatography
Sodium hydroxide	2.08 (processing conditions)	Base titration
	10.0 (post-processing conditions)	Rinsate concentration
Sodium nitrite	0.560	Ion chromatography
Sodium aluminate	0.264	ICP-AES (for Al)
Sodium carbonate	0.238	Total inorganic carbon
Sodium sulfate	0.0917	ICP-AES (for S)
Sodium orthophosphate	0.00542	ICP-AES (for P)
Sodium oxalate	0.00275	Ion chromatography
Sodium metasilicate	0.00162	ICP-AES (for Si)

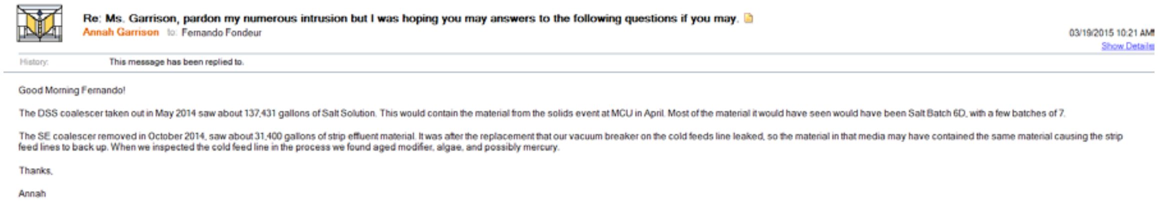
Inputs for Equilibrium Modeling of Solids in the SE Coalescer*

Species	Concentration, M	
	Low	High
Nitric acid	1.00E-03	1.00E-03
Sodium nitrate	6.72E-04	2.92E-03
Zinc nitrate	1.93E-04	2.65E-04
Ferrous or ferric nitrate	1.88E-04	2.78E-04
Aluminum nitrate	1.46E-04	9.37E-04
Calcium nitrate	1.40E-04	1.95E-04
Sodium metasilicate	8.37E-05	2.54E-04

*OLI Analyzer Studio version 9.0, with the MSE (H_3O^+ ion) databank selected, the redox option selected, the Helgeson direct equilibrium constant selected

[$] S. H. Reboul, T. B. Peters, and F. F. Fondeur, “Characterization of Solids in Fouled Modular Caustic Side Solvent Extraction Unit (MCU) Pre Coalescer Media” SRNL-STI-2014-00097, July 2014.

Appendix B. Correspondence between MCU Personnel and the author



Re: Ms. Garrison, pardon my numerous intrusion but I was hoping you may answers to the following questions if you may.

Annah Garrison to: Fernando Fondeur

03/19/2015 10:21 AM

History: This message has been replied to.

Good Morning Fernando!

The DSS coalescer taken out in May 2014 saw about 137,431 gallons of Salt Solution. This would contain the material from the solids event at MCU in April. Most of the material it would have seen would have been Salt Batch 6D, with a few batches of 7.

The SE coalescer removed in October 2014, saw about 31,400 gallons of strip effluent material. It was after the replacement that our vacuum breaker on the cold feeds line leaked, so the material in that media may have contained the same material causing the strip feed lines to back up. When we inspected the cold feed line in the process we found aged modifier, algae, and possibly mercury.

Thanks,

Annah

Distribution:

T. B. Brown, 773-A
D. E. Dooley, 999-W
A. P. Fellinger. 773-42A
S. D. Fink, 773-A
C. C. Herman, 773-A
E. N. Hoffman, 999-W
F. M. Pennebaker, 773-42A
M. E. Stone, 999-W
W. R. Wilmarth, 773-A
Records Administration (EDWS)

P. R. Jackson, DOE-SR, 703-46A

E. A. Brass, 241-121H
C. K. Chiu, 704-30S
J. S. Contardi, 704-56H
E. J. Freed, 704-S
A. G. Garrison, 241-121H
B. A. Gifford, 704-56H
V. Jain, 766-H
R. T. McNew, 766-H
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A. R. Shafer, 766-H