

Leaching of Titanium from Monosodium Titanate (MST) and Modified MST (mMST)

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August 2012

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Savannah River Nuclear Solutions, LLC
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Prepared for the U.S. Department of Energy under
contract number DE-AC09-08SR22470.



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Printed in the United States of America

**Prepared for
U.S. Department of Energy**

Keywords: *MST, mMST,
titanium, leaching*

Retention: *Permanent*

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EXECUTIVE SUMMARY

Analysis of a fouled coalescer and pre-filters from Actinide Removal Process/Modular Caustic Side Solvent Extraction Unit (ARP/MCU) operations showed evidence of Ti containing solids. Based on these results a series of tests were planned to examine the extent of Ti leaching from monosodium titanate (MST) and modified monosodium titanate (mMST) in various solutions. The solutions tested included a series of salt solutions with varying free hydroxide concentrations, two sodium hydroxide concentrations, 9 wt % and 15 wt %, nitric and oxalic acid solutions.

Overall, the amount of Ti leached from the MST and mMST was much greater in the acid solutions compared to the sodium hydroxide or salt solutions, which is consistent with the expected trend. The leaching data also showed that increasing hydroxide concentration, whether pure NaOH solution used for filter cleaning in ARP or the waste salt solution, increased the amount of Ti leached from both the MST and mMST. For the respective nominal contact times with the MST solids – for filter cleaning or the normal filter operation, the dissolved Ti concentrations are comparable suggesting either cause may contribute to the increased Ti fouling on the MCU coalescers.

Tests showed that Ti containing solids could be precipitated from solution after the addition of scrub acid and a decrease in temperature similar to expected in MCU operations. FTIR analysis of these solids showed some similarity to the solids observed on the fouled coalescer and pre-filters.

Although only a cursory study, this information suggests that the practice of increasing free hydroxide in feed solutions to MCU as a mitigation to aluminosilicate formation may be offset by the impact of formation of Ti solids in the overall process. Additional consideration of this finding from MCU and SWPF operation is warranted.

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LIST OF ABBREVIATIONS

| | |
|---------|---|
| ARP | Actinide Removal Process |
| DSS | Decontaminated Salt Solution |
| FTIR | Fourier transform infrared spectroscopy |
| ICP-ES | inductively coupled plasma – emission spectroscopy |
| MCU | Modular Caustic Side Solvent Extraction Unit |
| MST | monosodium titanate |
| mMST | modified monosodium titanate |
| PVDF | polyvinylidene fluoride |
| SEM-EDS | scanning electron microscopy-energy dispersive spectroscopy |
| SRNL | Savannah River National Laboratory |

1.0 Introduction

During Actinide Removal Process/Modular Caustic Side Solvent Extraction Unit (ARP/MCU) operations, fouling of both the decontaminated salt solution (DSS) coalescer and subsequently the pre-filters was observed.¹ The coalescer and pre-filters were replaced, and the fouled materials were sent to SRNL for analysis.² Analysis of the fouled coalescer and pre-filters showed evidence of Ti containing solids. Based on these results a series of tests were planned to examine the extent of Ti leaching from monosodium titanate (MST) and modified monosodium titanate (mMST) in various solutions. The solutions tested included a series of salt solutions with varying free hydroxide concentrations to determine if the shift to higher free hydroxide solutions in ARP/MCU was resulting in higher Ti solution concentrations. The concentrations selected represent the range of increase in free hydroxide for ARP/MCU feed solutions since initial operations through current practice. Two sodium hydroxide concentrations, 9 wt % and 15 wt %, were also tested to examine the effect of using a higher sodium hydroxide concentration for cleaning in ARP. The normal cleaning solution is the 9 wt % hydroxide but a recent cleaning operating in ARP used the more concentrated (15 wt %) solution. In addition, nitric and oxalic acid solutions were tested to examine the potential ability of these acids to dissolve Ti containing solids on the filters and coalescers. Nitric acid is available in MCU to clean the contactors or coalescers. Oxalic acid is used in ARP to clean the filters.

2.0 Experimental Procedure

2.1 Leaching Tests

Eight different solutions were prepared to examine the extent of Ti leaching from monosodium titanate (MST) and modified monosodium titanate (mMST) under various conditions. Solutions tested included 1 and 3 M nitric acid, 0.5 M oxalic acid, 9 and 15 wt % sodium hydroxide, along with three salt solutions with free hydroxide concentrations of 1, 2, and 3 M. The target compositions of the salt solutions are provided in Table 2-1.

Table 2-1. Composition of Salt Solutions.

| Component | Salt A Conc. | Salt B Conc. | Salt C Conc. |
|-------------------|--------------|--------------|--------------|
| Free NaOH | 1.00 M | 2.00 M | 3.00 M |
| NaNO ₃ | 3.80 M | 3.00 M | 2.20 M |
| NaNO ₂ | 0.75 M | 0.60 M | 0.43 M |
| Total Na | 5.6 M | 5.6 M | 5.6 M |

The MST used in this testing was prepared by Harrell Industries, Lot # 121010, and the mMST was prepared by Optima Chemical Group, Lot # 06-QAB-0139. The target MST/mMST concentrations for the leaching tests were 5 g per 100 mL of solution. Both the MST and mMST are provided as 15 wt % slurries. Aliquots of the slurry providing the desired mass of MST or mMST were centrifuged at 4400 rpm for 15 minutes to isolate the solids. The supernate was decanted, and the solids were then transferred to the test bottle. A small amount of the decanted supernate (1-2 mL) was used to rinse any remaining solids from the centrifuge tube into the test bottle. The desired leaching solution was then added to the test bottles, and the start time was recorded. The bottles were shaken vigorously to disperse the solids, and were then transferred to a 25 °C shaker oven and agitated at 175 rpm. Samples were removed periodically by pulling a few mL of the suspension into a syringe, and filtering the contents through a 0.1-µm PVDF syringe filter. These samples were then submitted for inductively coupled plasma – emission spectroscopy (ICP-ES) to determine the Ti concentration.

A white precipitate was observed to form in the samples from the 1 and 3 M HNO₃ tests with MST. Attempts to redissolve the solid by increasing the acid concentration proved unsuccessful. These two tests were restarted, and the samples pulled were immediately acidified with the addition of concentrated nitric acid, bringing the final HNO₃ concentration of the samples to 9.4 M to prevent the precipitate from forming.

2.2 Precipitation Tests

In an attempt to reproduce solids found on the MCU coalescers, precipitation tests were performed with the leachate solutions from the Salt B and Salt C MST tests. The mixtures containing the MST solids were placed in a 40 °C shaker oven in an attempt to increase the Ti concentration to maximize the solids obtained. The Salt B mixture was removed after 6 days, and was filtered through a 0.45-μm filter to remove the MST solids. A sample of the filtrate was acidified with concentrated nitric acid and submitted for ICP-ES analysis to determine the Ti concentration. The remaining filtrate (160 mL) was combined with 11.4 mL of 0.05 M nitric acid and allowed to stand at room temperature. After 5 days, the solution was centrifuged at 4400 rpm for 20 minutes. A small amount of white solid was observed at the bottom of the centrifuge tubes. These solids were suspended in a small amount of the supernate, and were collected by filtration on a piece of pre-weighed 0.2-μm Nylon filter paper. The filter paper and solids were then allowed to air dry for several days before being analyzed using FTIR and SEM-EDS analyses.

The Salt C mixture was removed from the oven after 14 days, and was filtered through a 0.45-μm filter to remove the MST solids. A sample of the filtrate was acidified and submitted for ICP-ES analysis, and the remaining filtrate (156 mL) was combined with 11.1 mL of 0.05 M nitric acid. After standing for 5 days, the solution was centrifuged to collect the solids. The isolated solids were transferred to a piece of 0.2-μm Nylon filter paper using a spatula, and were left to air dry for several days. The solids were then analyzed using FTIR and SEM-EDS analyses.

3.0 Results and Discussion

3.1 Leaching Tests

The measured Ti concentrations in the filtrates as a function of time are presented in Table 3-1. In addition, the percentage of MST or mMST dissolved to provide those concentrations is also provided.

Table 3-1. Results of Leaching Tests

| 9 wt % NaOH | | | | | |
|--------------------------|------------------|--------------------|-----------------|------------------|--------------------|
| MST | | | mMST | | |
| Time (h) | Ti (mg/L) | % Dissolved | Time (h) | Ti (mg/L) | % Dissolved |
| 0.27 | 1.64 | 0.01% | 0.28 | 256 | 1.11% |
| 1.48 | 1.62 | 0.01% | 1.50 | 400 | 1.73% |
| 2.98 | 1.84 | 0.01% | 3.00 | 508 | 2.19% |
| 7.98 | < 1.48 | < 0.01% | 7.98 | 502 | 2.17% |
| 24.1 | 2.3 | 0.01% | 24.1 | 545 | 2.35% |
| 48.5 | 3.21 | 0.01% | 48.5 | 545 | 2.35% |
| 97.3 | 3.6 | 0.02% | 97.3 | 533 | 2.30% |
| 168.5 | 3.74 | 0.02% | 168.5 | 499 | 2.16% |
| 507.2 | 6.98 | 0.03% | 507.2 | 371 | 1.60% |
| 840.0 | 6.31 | 0.03% | 840.0 | 272 | 1.17% |
| 15 wt % NaOH | | | | | |
| MST | | | mMST | | |
| Time (h) | Ti (mg/L) | % Dissolved | Time (h) | Ti (mg/L) | % Dissolved |
| 0.28 | 9.52 | 0.04% | 0.28 | 428 | 1.85% |
| 1.50 | 18.10 | 0.08% | 1.50 | 628 | 2.71% |
| 3.00 | 24.20 | 0.10% | 3.00 | 649 | 2.80% |
| 7.98 | 50.00 | 0.21% | 7.97 | 678 | 2.93% |
| 24.1 | 63.80 | 0.27% | 24.0 | 786 | 3.40% |
| 48.5 | 57.70 | 0.24% | 48.5 | 794 | 3.43% |
| 97.3 | 53.10 | 0.22% | 97.3 | 785 | 3.39% |
| 168.5 | 31.60 | 0.13% | 168.4 | 788 | 3.40% |
| 507.2 | 32.00 | 0.13% | 507.3 | 668 | 2.89% |
| 840.0 | 27.10 | 0.11% | 840.0 | 556 | 2.40% |
| 0.5 M Oxalic Acid | | | | | |
| MST | | | mMST | | |
| Time (h) | Ti (mg/L) | % Dissolved | Time (h) | Ti (mg/L) | % Dissolved |
| 0.28 | 12700 | 53.03% | 0.28 | 4640 | 20.04% |
| 1.48 | 13800 | 57.62% | 1.50 | 6220 | 26.87% |
| 2.98 | 14400 | 60.13% | 3.00 | 6620 | 28.60% |
| 8.00 | 15000 | 62.63% | 8.00 | 6780 | 29.29% |
| 24.0 | 15500 | 64.72% | 24.0 | 7200 | 31.10% |
| 48.5 | 15700 | 65.55% | 48.5 | 7350 | 31.75% |
| 97.3 | 15900 | 66.39% | 97.3 | 7330 | 31.66% |
| 168.4 | 16100 | 67.22% | 168.5 | 7600 | 32.83% |
| 1 M Nitric Acid | | | | | |
| MST | | | mMST | | |
| Time (h) | Ti (mg/L) | % Dissolved | Time (h) | Ti (mg/L) | % Dissolved |
| 0.25 | 3493 | 14.59% | 0.23 | 1060 | 4.58% |
| 1.55 | 4951 | 20.67% | 1.52 | 1650 | 7.13% |
| 3.00 | 5089 | 21.25% | 3.03 | 1820 | 7.86% |
| 8.07 | 4997 | 20.86% | 8.05 | 1980 | 8.55% |
| 24.0 | 4557 | 19.03% | 24.1 | 2060 | 8.90% |
| 48.0 | 3956 | 16.52% | 48.1 | 2110 | 9.11% |
| 98.3 | 2168 | 9.05% | 96.9 | 2090 | 9.03% |
| 167.9 | 1249 | 5.22% | 168.1 | 2050 | 8.86% |

Table 3-1. Results of Leaching Tests (Continued)

| 3 M Nitric Acid | | | | | |
|------------------------------------|------------------|--------------------|-----------------|------------------|--------------------|
| MST | | | mMST | | |
| Time (h) | Ti (mg/L) | % Dissolved | Time (h) | Ti (mg/L) | % Dissolved |
| 0.25 | 10380 | 43.34% | 0.23 | 2170 | 9.37% |
| 1.60 | 12200 | 50.94% | 1.53 | 3560 | 15.38% |
| 3.00 | 12860 | 53.70% | 3.03 | 3930 | 16.98% |
| 8.02 | 13920 | 58.12% | 8.07 | 4500 | 19.44% |
| 24.0 | 15080 | 62.96% | 24.1 | 4990 | 21.56% |
| 48.0 | 13620 | 56.87% | 48.1 | 5230 | 22.59% |
| 98.2 | 4660 | 19.46% | 97.0 | 5360 | 23.15% |
| 167.9 | 1156 | 4.83% | 168.1 | 5350 | 23.11% |
| Salt A – 1 M OH⁻ | | | | | |
| MST | | | mMST | | |
| Time (h) | Ti (mg/L) | % Dissolved | Time (h) | Ti (mg/L) | % Dissolved |
| 1.97 | 3.82 | 0.02% | 1.97 | 161 | 0.70% |
| 4.05 | 4.76 | 0.02% | 4.03 | 184 | 0.79% |
| 8.00 | 3.06 | 0.01% | 8.00 | 202 | 0.87% |
| 24.2 | 4.27 | 0.02% | 24.2 | 238 | 1.03% |
| 48.6 | 4.8 | 0.02% | 48.6 | 220 | 0.95% |
| 97.1 | 5.63 | 0.02% | 97.4 | 214 | 0.92% |
| 168.5 | 9.1 | 0.04% | 168.5 | 182 | 0.76% |
| 504.2 | 12.1 | 0.05% | 504.2 | 95.2 | 0.40% |
| 840.1 | 13.8 | 0.06% | 840.1 | 51.8 | 0.22% |
| Salt B – 2 M OH⁻ | | | | | |
| MST | | | mMST | | |
| Time (h) | Ti (mg/L) | % Dissolved | Time (h) | Ti (mg/L) | % Dissolved |
| 1.98 | 6.62 | 0.03% | 1.98 | 285 | 1.23% |
| 4.03 | 7.22 | 0.03% | 4.02 | 321 | 1.39% |
| 8.00 | 8.9 | 0.04% | 8.00 | 358 | 1.55% |
| 24.1 | 10.7 | 0.04% | 24.1 | 403 | 1.74% |
| 48.6 | 12.9 | 0.05% | 48.6 | 415 | 1.79% |
| 97.3 | 16.5 | 0.07% | 97.4 | 400 | 1.73% |
| 168.5 | 24.2 | 0.10% | 168.5 | 369 | 1.54% |
| 504.2 | 30.2 | 0.13% | 504.2 | 245 | 1.02% |
| 840.1 | 30.2 | 0.13% | 840.1 | 159 | 0.69% |
| Salt C – 3 M OH⁻ | | | | | |
| MST | | | mMST | | |
| Time (h) | Ti (mg/L) | % Dissolved | Time (h) | Ti (mg/L) | % Dissolved |
| 2.00 | 7.72 | 0.03% | 2.00 | 367 | 1.59% |
| 4.03 | 7.12 | 0.03% | 4.02 | 427 | 1.84% |
| 8.02 | 17.2 | 0.07% | 8.02 | 474 | 2.05% |
| 24.1 | 27.9 | 0.12% | 24.1 | 525 | 2.27% |
| 48.6 | 25.1 | 0.10% | 48.6 | 560 | 2.42% |
| 97.3 | 31.1 | 0.13% | 97.3 | 554 | 2.39% |
| 168.5 | 42.1 | 0.18% | 168.5 | 521 | 2.18% |
| 504.2 | 37 | 0.15% | 504.2 | 399 | 1.67% |
| 840.1 | 30.9 | 0.13% | 840.1 | 291 | 1.26% |

Results showed that increasing hydroxide concentration, whether pure NaOH solution or salt solution, increased the amount of Ti leached from both the MST and mMST. In the sodium hydroxide and salt solutions, mMST appeared to leach more Ti than the MST. For MST, Figure

3-1 shows the dissolved Ti concentration as a function of exposure time and free hydroxide concentration. As the free hydroxide concentration increases from 1 to 2 M, the dissolved Ti roughly doubles and approaches ~30 mg/L at ~33 days (~800 h). At 3 M free hydroxide, the dissolved concentration at short times increases nearly proportionally again, but at longer exposures the concentration declines, suggestive of slow precipitation. The Ti concentrations leached from mMST are an order of magnitude higher with more obvious signs suggesting delayed precipitation at all hydroxide concentrations (Figure 3-2).

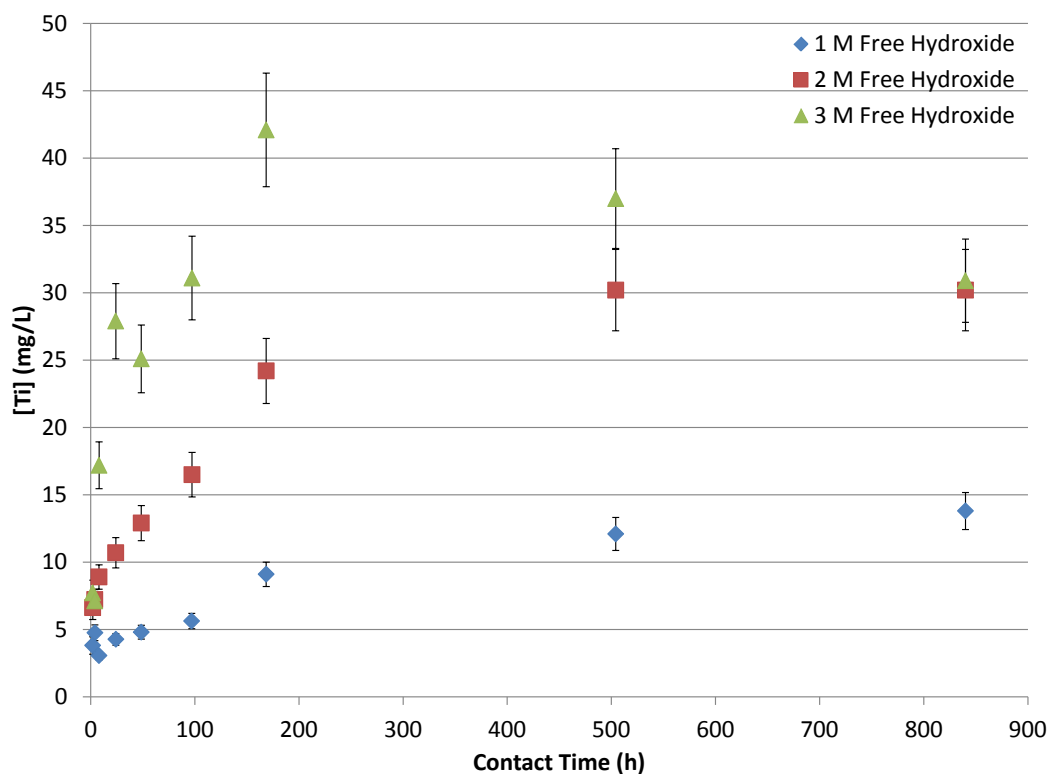


Figure 3-1. Dissolved Ti from MST as function of free hydroxide concentration.

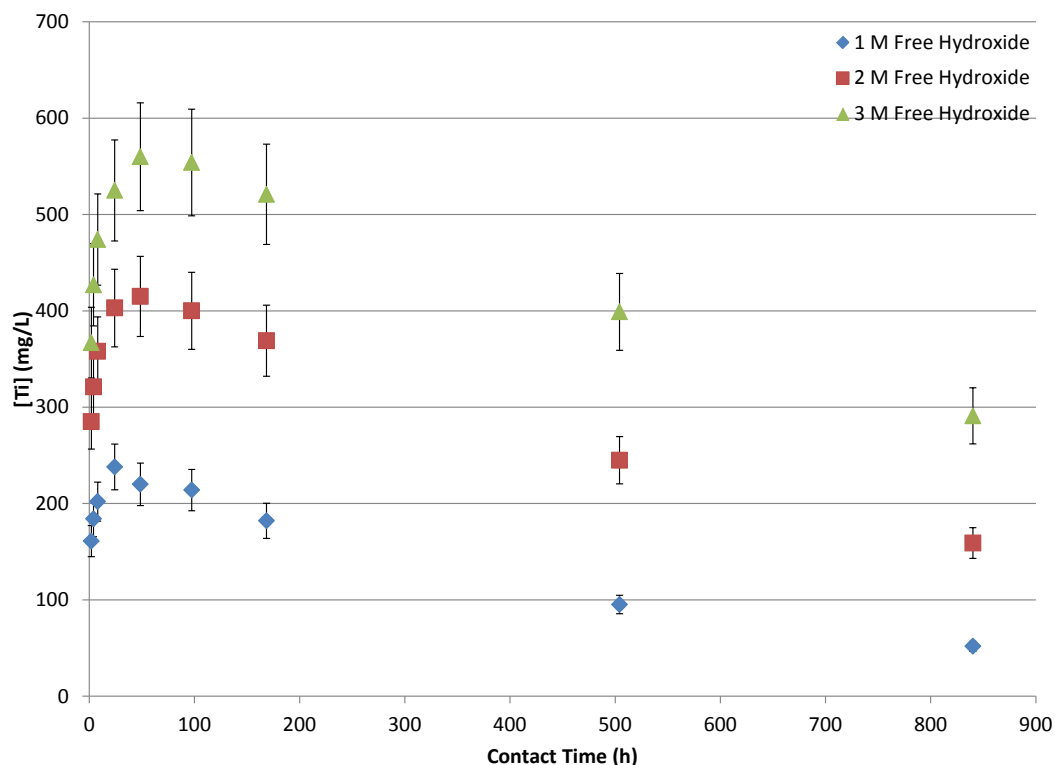


Figure 3-2. Dissolved Ti from mMST as function of free hydroxide concentration.

The process flowsheet adopted higher hydroxide concentrations for the feed solution in ARP/MCU to mitigate precipitation concerns of aluminosilicates and fouling of the coalescers. However, reducing solids formation for aluminosilicate is apparently counteracted by potential risk of higher Ti leaching and post precipitation. With the current MCU design modifications to facilitate more rapid replacement of the coalescers, some thought may be warranted on reducing the free hydroxide (and the added chemical and disposal costs). For the Salt Waste Processing Facility, additional study of the optimal hydroxide concentration is warranted.

Short duration (e.g., 24 hour) exposure of MST to 15 wt % NaOH solution produced comparable dissolved Ti concentrations as the month long exposure to the salt solutions. In contrast, the normal 9 wt % NaOH solution limited dissolved Ti concentration to an order of magnitude lesser values. Hence, this data suggests that the inadvertent use of more concentrated filter cleaning solutions may have contributed to the fouling of coalescers.

Of the acid solutions tested, 0.5 M oxalic acid appeared to dissolve the MST and mMST to the greatest extent, as expected.³ As shown in Figure 3-3, in the 1 and 3 M nitric acid solutions with MST, the Ti concentration appeared to reach a maximum value, and then decrease at the later time points, indicating precipitation. This is consistent with the observed precipitate that formed in the samples after filtration. In the repeat tests, the acid concentration of the samples was increased after filtering to prevent the formation of this precipitate; however, the precipitate could still form in the test bottles causing a decrease in the soluble Ti concentration measured in the samples. This was not observed in the mMST tests (Figure 3-4). There was some indication of precipitation of Ti at later time points for both MST and mMST in the hydroxide solutions.

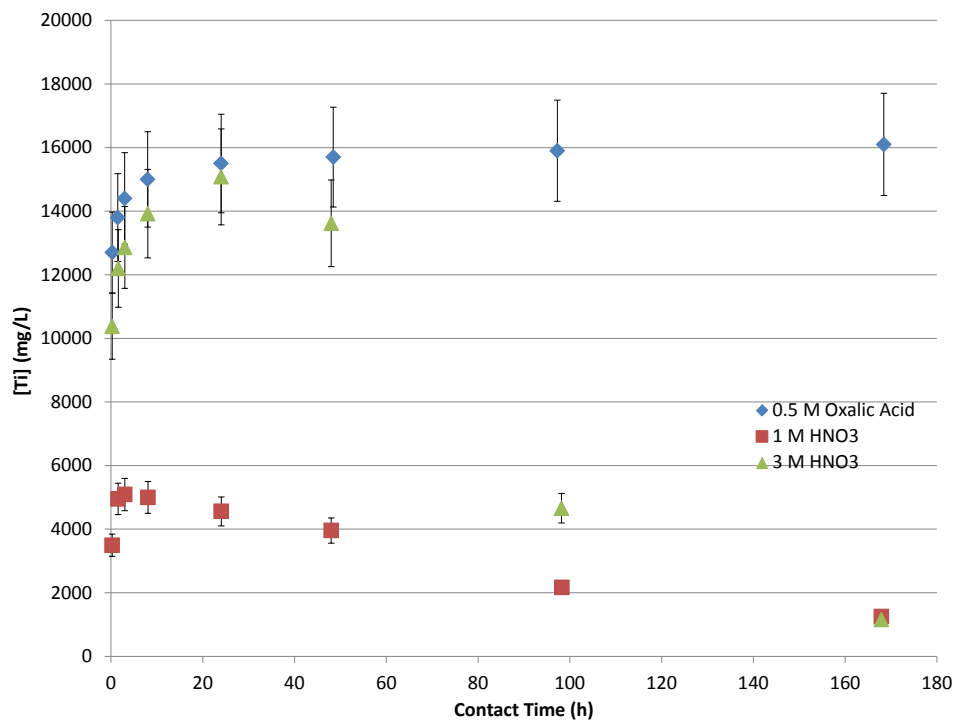


Figure 3-3. Dissolved Ti from MST in various acid solutions.

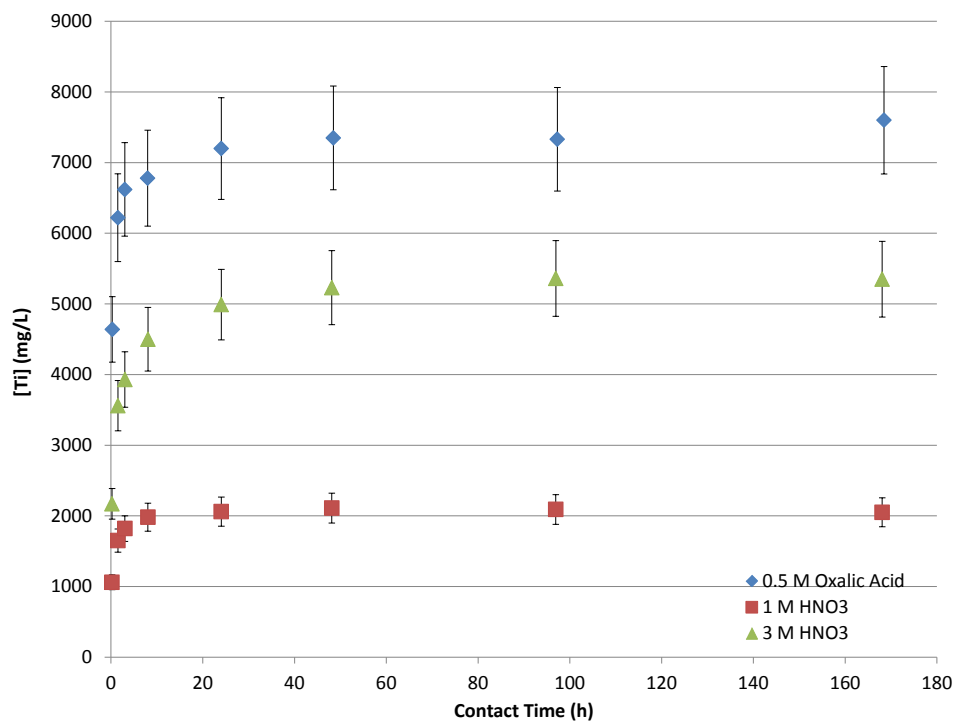


Figure 3-4. Dissolved Ti from MST in various acid solutions.

The acid leaching data suggests that nitric acid cleaning of the coalescers could be viable if needed; however, the evolution would be non-trivial given the lack of an active pumping loop in the design to circulate cleaning fluids.

3.2 Precipitation Tests

Approximately 4.66 mg of solids were obtained from the Salt B solution after adding 0.05 M nitric acid to emulate scrub acid contact with the salt solution at in MCU operations. These solids were analyzed using FTIR and SEM-EDS analyses. The results of the FTIR analysis showed the presence of carbonate and nitrate salts (Spectra 3 & 4) along with some evidence of hydrated titanate material (Spectra 1 & 2). See Figure 3-5 for FTIR spectra of solids from Salt B.

SEM-EDS analysis showed evidence of Ti containing solids along with some sodium salts. See Figure 3-6 for SEM images and Figure 3-7 for EDS spectra.

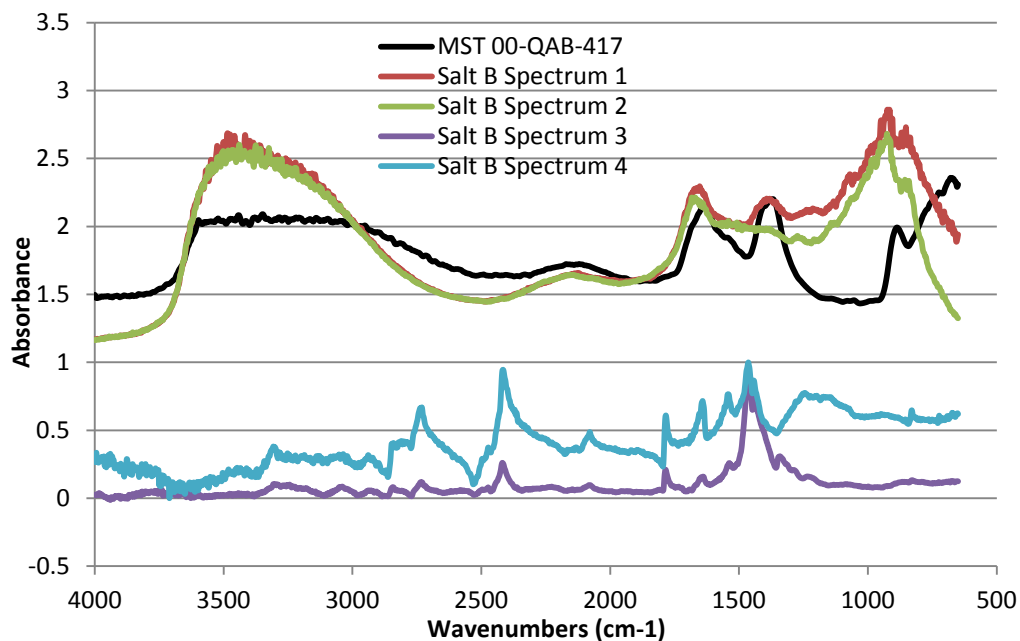


Figure 3-5. FTIR Spectra of solids precipitated from Salt B leachate solution compared to FTIR spectrum of MST (black).

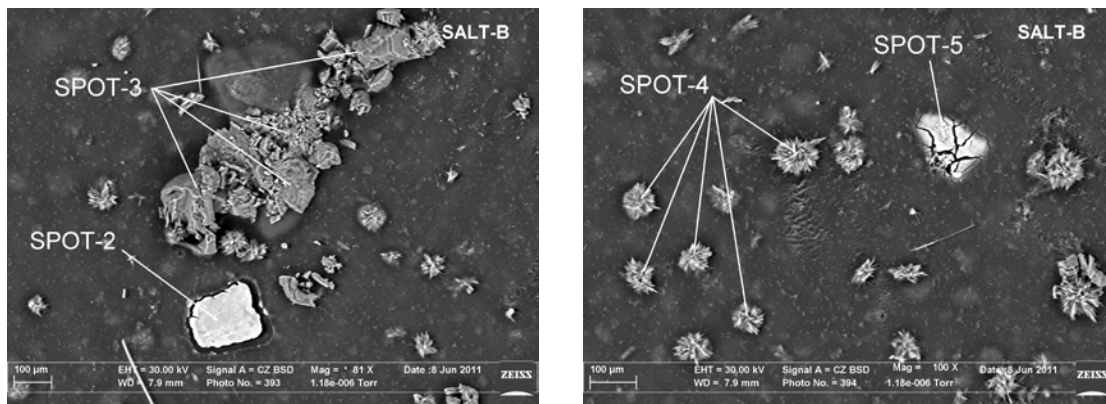


Figure 3-6. SEM images of solids precipitated from Salt B leachate solution.

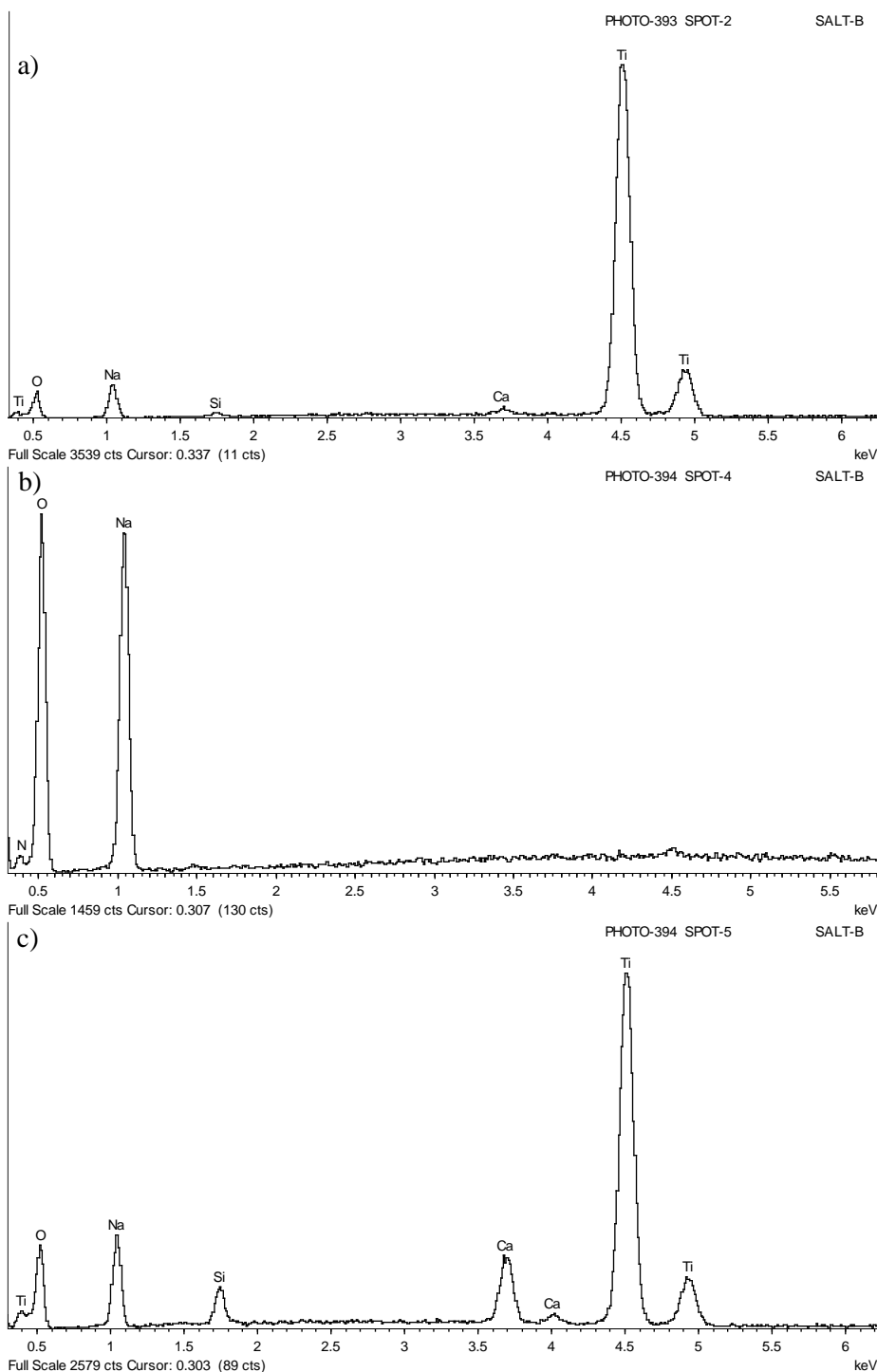


Figure 3-7. EDS spectra of solids precipitated from Salt B leachate solution. The spectra correspond to spots labeled in the images shown in Figure 3-6: a) Spot-2, b) Spot-4, and c) Spot-5.

Approximately 12.03 mg of solids were obtained from the Salt C solution after adding 0.05 M nitric acid to emulate scrub acid contact with the salt solution at in MCU operations. These solids were analyzed using FTIR and SEM-EDS analyses. The results of the FTIR analysis showed

similarities between these solids and those found on the MCU coalescers and filters (Spectra 1 & 2). See Figure 3-8 for FTIR spectra of solids from Salt C.

Although the spectra show variances, the information is roughly consistent. Hence the data is consistent with a higher free hydroxide causing increased dissolved Ti concentrations in the feed solution to the solvent extraction process in MCU. When the scrub solution is carried into the extraction contactors of MCU, the nitric acid causes precipitation and the solids eventually are trapped on the DSS coalescer.

SEM-EDS analysis showed evidence of Ti containing solids along with some sodium salts. See Figure 3-9 for SEM images and Figure 3-10 for EDS spectra.

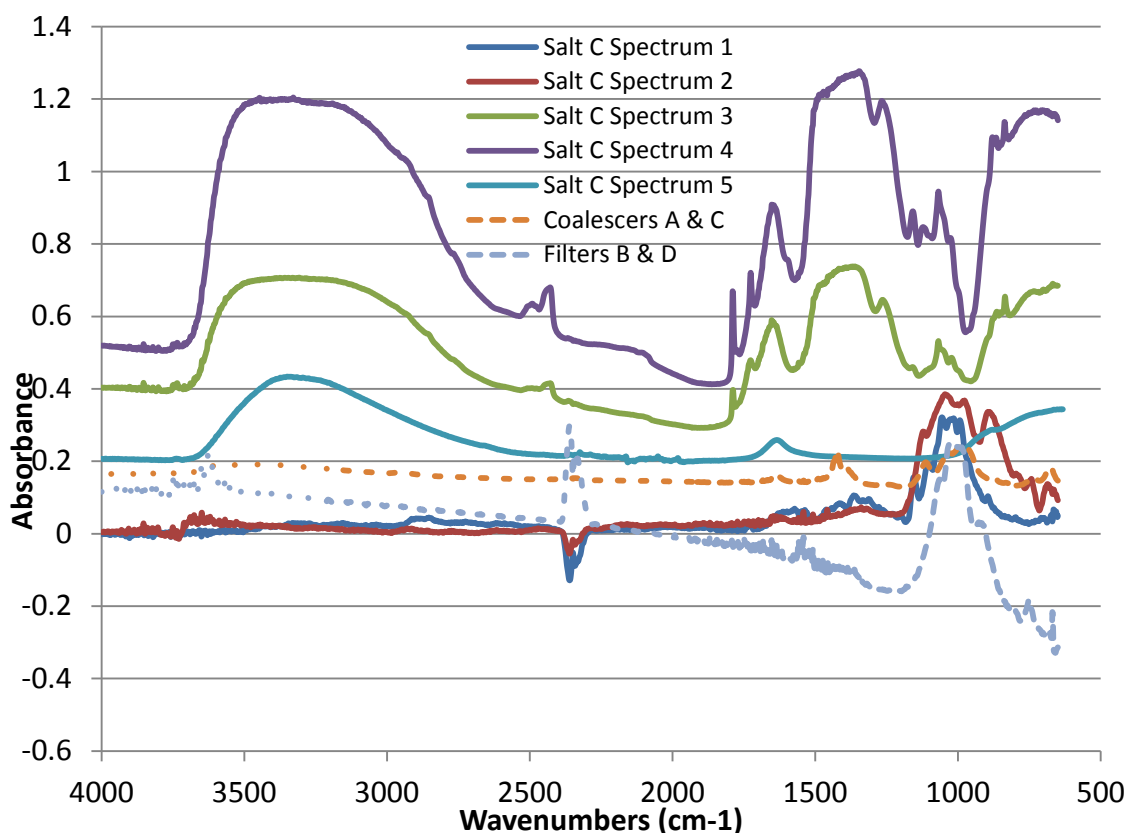


Figure 3-8. FTIR Spectra of solids precipitated from Salt C leachate solution compared to FTIR spectra of solids on MCU coalescers and filters (dashed lines).

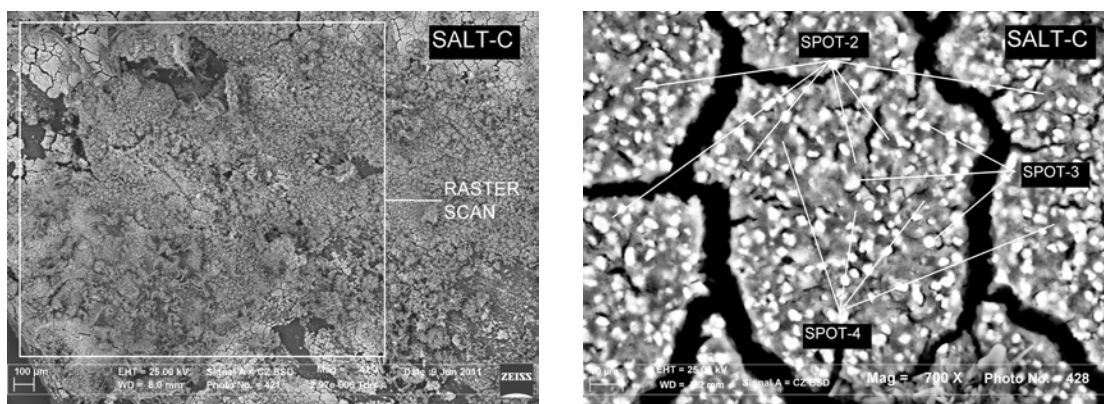


Figure 3-9. SEM images of solids precipitated from Salt C leachate solution.

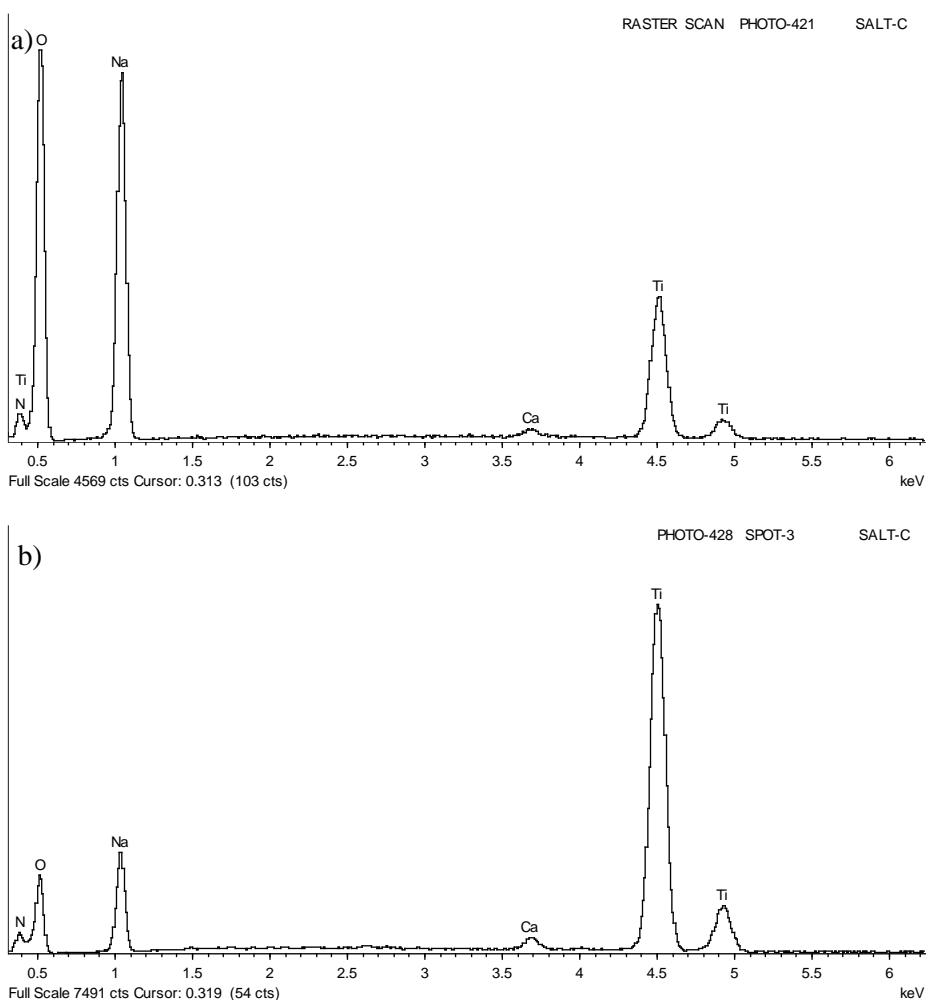


Figure 3-10. EDS spectra of solids precipitated from Salt C leachate solution. The spectra correspond to areas labeled in the images shown in Figure 3-9: a) Raster Scan and b) Spot-3.

4.0 Conclusions

Overall, the amount of Ti leached from the MST and mMST was much greater in the acid solutions compared to the sodium hydroxide or salt solutions, which is consistent with the expected trend. The leaching data also showed that increasing hydroxide concentration, whether pure NaOH solution used for filter cleaning in ARP or the waste salt solution, increased the amount of Ti leached from both the MST and mMST. For the respective nominal contact times with the MST solids – for the filter cleaning or the normal filter operation, the dissolved Ti concentrations are comparable suggesting either cause may contribute to the increased Ti fouling on the MCU coalescers.

Tests showed that Ti containing solids could be precipitated from solution after the addition of scrub acid and a decrease in temperature similar to expected in MCU operations. FTIR analysis of these solids showed some similarity to the solids observed on the fouled coalescer and pre-filters.

Although only a cursory study, this information suggests that the practice of increasing free hydroxide in feed solutions to MCU as a mitigation to aluminosilicate formation may be offset by the impact of formation of Ti solids in the overall process. Additional consideration of this finding for MCU and SWPF operation is warranted.

5.0 References

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B. J. Giddings, 786-5A
C. C. Herman, 999-W
S. L. Marra, 773-A
F. M. Pennebaker, 773-42A
W. R. Wilmarth, 773-A
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K. M. L. Taylor-Pashow, 773-A
D. T. Hobbs, 773-A
T. B. Peters, 773-42A
F. F. Fondeur, 773-A
T. C. Shehee, 773-A

E. J. Freed, 704-56H
D. J. Martin, 241-152H
M. W. Geeting, 241-152H
T. A. Le, 766-H
A. R. Shafer, 704-27S
C. K. Chiu, 704-27S
S. E. Campbell, 241-152H
S. P. Mcleskey, 241-152H
B. A. Gifford, 704-56H
R. M. Wolfenden, 704-56 H
K. L. Lang, 704-27S
C. E. Duffey, 704-61H
A. W. Wiggins, 704-60H

P. R. Jackson, DOE-SR, 703-46A
K. H. Subramanian, 766-H

W. B. Brasel, 992-2W
C. Conner, Parsons
R. D. Lentsch, Parsons
M. S. Brugh, Parsons
T. D. Burns, Parsons
A. N. Singer, Parsons
M. J. Thomas, Parsons
R. J. Schepens, Parsons
R. K. Leugemors, 992-5W
P. G. Suggs, 704-S