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Removal of Solids from Highly Enriched Uranium Solutions Using the H-Canyon Centrifuge

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Removal of Solids from Highly Enriched Uranium Solutions Using the H-Canyon Centrifuge

T. S. Rudisill and F. F. Fondeur

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Summary

Prior to the dissolution of Pu-containing materials in HB-Line, highly enriched uranium (HEU) solutions stored in Tanks 11.1 and 12.2 of H-Canyon must be transferred to provide storage space. The proposed plan is to centrifuge the solutions to remove solids which may present downstream criticality concerns or cause operational problems with the 1st Cycle solvent extraction due to the formation of stable emulsions. An evaluation of the efficiency of the H-Canyon centrifuge concluded that a sufficient amount (> 90%) of the solids in the Tank 11.1 and 12.2 solutions will be removed to prevent any problems. We based this conclusion on the particle size distribution of the solids isolated from samples of the solutions and the calculation of particle settling times in the centrifuge. The particle size distributions were calculated from images generated by scanning electron microscopy (SEM). The mean particle diameters for the distributions were 1-3 μm. A significant fraction (30-50%) of the particles had diameters which were < 1 μm; however, the mass of these solids is insignificant (< 1% of the total solids mass) when compared to particles with larger diameters. It is also probable that the number of submicron particles was over-estimated by the software used to generate the particle distribution due to the morphology of the filter paper used to isolate the solids.

The settling times calculated for the H-Canyon centrifuge showed that particles with diameters less than 1 to 0.5 μm will not have sufficient time to settle. For this reason, we recommend the use of a gelatin strike to coagulate the submicron particles and facilitate their removal from the solution; although we have no experimental basis to estimate the level of improvement. Incomplete removal of particles with diameters < 1 μm should not cause problems during purification of the HEU in the 1st Cycle solvent extraction. Particles with diameters > 1 μm account for > 99% of the solid mass and will be efficiently removed by the centrifuge; therefore, the formation of emulsions during solvent extraction operations is not an issue. Under the current processing plan, the solutions from Tanks 11.1 and 12.2 will be transferred to the enriched uranium storage (EUS) tank following centrifugation. The solution from Tanks 11.1 and 12.2 may remain in the EUS tank for an extended time prior to purification. The effects of extended storage on the solution were not evaluated as part of this study.

Introduction

Impure materials containing HEU were recently dissolved in Phase I of HB-Line. Some of these materials are known to contain impurities which are difficult to dissolve in nitric solutions containing potassium fluoride. During the development of the dissolution flowsheet for this type of material, the undissolved solids ranged from < 0.5% to approximately 16% of the initial mass. The characterization of the undissolved solids is summarized in Table 1. [1]

Table 1 Undissolved solids from HEU dissolution studies

Sample	Undissolved Mass (wt %)	Major Elements (SEM/EDS)	Minor Elements (SEM/EDS)	Major Compounds (XRD)	Minor Compounds (XRD)
LLNL-1	7.0	U, Cr	Fe, Ni	$UCrO_4$	Nb_2O_5
LLNL-2	2.6	U, W, Fe	Al, Ti, Cr	FeU_3O_{10}	Cr_2WO_6 $UCrO_4$
LLNL-3	2.6	U, Si	Ti, W, Fe, Ni	$U_5W_{13}O_{50}$ FeU_3O_{10}	$UCrO_4$
LLNL-4	16.3	U, Si	Cr, W, Ti	SiC	U_2WO_8 $UCrO_4$ UTi_2O_6
LANL-1	0.22	Pu	Fe, Si, Cr	(1)	(1)
LANL-2	0.18	(2)	(2)	(2)	(2)

(1) Insufficient sample for analysis
(2) Sample not analyzed (duplicate of LANL-1)

Following each dissolution cycle, the contents of the HB-Line dissolver were transferred through a bag filter to the RT-33 product hold tank. The solution was then transferred to Tank 11.1 in H-Canyon. Subsequent analyses of samples removed from Tank 11.1 showed the presence of U-containing solids which indicated that the bag filter may have been penetrated when the solution was transferred to the product hold tank. [2-4] The solution in Tank 11.1 was later transferred to Tank 12.2. The transfer was performed without agitation in Tank 11.1 to minimize the amount of solids transferred to Tank 12.2. However, a significant amount of solids were present in samples removed from Tank 12.2 following the transfer from Tank 11.1. [2,5] Once solution was moved from Tank 11.1 to Tank 12.2, additional transfers of HEU solution were made from HB-Line to Tank 11.1. Analysis of solids present in mid-campaign samples from Tank 11.1 showed a substantial (approximately a factor of four) decrease in the amount of HEU present in these solids. [6]

In preparation for the dissolution of Pu-containing materials in HB-Line, the HEU solutions in Tanks 11.1 and 12.2 must be moved. The proposed plan is to centrifuge the solution using the Head End unit operation in H-Canyon and transfer the solution to the EUS tank. Prior to implementing this plan, H-Canyon Engineering requested SRNL to evaluate the effectiveness of the centrifuge in removing solids from the Tank 11.1 and 12.2 solutions and to determine the effect that any suspended solids not removed by the centrifuge will have on the operation

of the H-Canyon 1st Cycle solvent extraction. [7] To perform the evaluation, particle size distributions for solids in samples from Tanks 11.1 and 12.2 were obtained by analyzing images of the solids obtained by SEM. Calculations were also performed to estimate the settling times in the H-Canyon centrifuge as a function of the particle size and density of the solids. The experimental methods used to obtain the particle size distributions, calculation of the settling times, and a discussion of the results are presented in the following sections.

Experimental

Sample Preparation

A 10-mL sample from Tank 12.2 was received from H-Canyon on December 8, 2008 to measure the particle size distribution for any solids present in the solution. Four additional samples were also received for characterization by another SRNL staff member (and are not a part of this study). These samples were removed from Tank 12.2 several days prior to the removal of the sample for use in the particle size analysis. All of the samples were removed from the tank while the agitator was operating following flushing of the sampler. Photographs of the sample vials are shown in Figure 1. No solids were visually observed in any of the sample vials. The solutions were clear with a light yellow color which is characteristic of uranyl nitrate. The samples which were obtained for characterization were subsequently filtered using a Nalgene® cellulose nitrate membrane filter unit with a 0.45 µm pore size. No solids were visible on the filter papers (Figure 2).

Since no solids were visually observed in any of the Tank 12.2 samples or on the filters used for the four characterization samples, an additional Tank 12.2 sample was requested for use in obtaining a particle size distribution of the solids. The additional Tank 12.2 sample and a sample from Tank 11.1 were delivered to SRNL on December 11, 2008. The samples were removed from the tanks while the agitators were operating following flushing of the samplers. Upon inspection of the sample vials, no solids were visually observed. Photographs of the sample vials are shown in Figures 3 and 4, respectively.

Although no solids were visually observed in either of the Tank 12.2 samples or in the Tank 11.1 sample, particles undetectable by the eye were likely present in the solutions. To obtain a particle size distribution for these solids, a sample from each of the three solutions was prepared for analysis by SEM. Each solution was filtered using a Nalgene® cellulose nitrate membrane filter unit with a 0.45 µm pore size. The solution was transferred to the filter using a slurry pipette taking care to keep the solution in as small an area as possible. This procedure was followed to concentrate any solids in a single spot. The spot on the filter paper containing the solids was then washed with a 20-mL aliquot of 0.5 M nitric acid and allowed to dry. A photograph of the filter papers is shown in Figure 5. After drying, a small piece of the filter paper was cut-out from the area where the solids were concentrated and mounted for SEM analysis.

Particle Size Analysis

Both secondary electron (SE) and back-scattering electron (BSE) images were taken at three magnifications (100x, 500x, and 2000x). The SE images show the sample topography while the BSE images indicate the relative atomic number, where the brighter areas of the solids have a higher atomic number. Energy dispersive spectra were obtained to identify the general elemental composition of the solids and a number of selected particles.

The SEM images were processed using a freeware image analysis program (IMAGEJ 1.37V) from the National Institutes of Health. Both the SE and BSE images were examined. The SE images had better resolution; however, the morphology of the filter paper made it difficult to clearly distinguish the solids from features of the paper. In addition, the nonhomogeneous illumination of the solids in the SE images made them unsuitable for quantifying the surface area of the particles. To minimize the identification of false particles due to the presence of the filter paper, we took advantage of the high signal intensities of the solids compared to the paper in the BSE images. The signal intensities in the BSE images are dominated by the high "Z" material in the solids while the signal from the organic-based filter paper is relatively weak. The detector for the BSE images was also placed nearly normal to the samples, while the detector for the SE images was at a large incidence angle creating shadows. The image magnification also had an effect on the quality of the spatial information on the BSE images. Higher magnifications (2000x) under-represented the population of large particles and lower magnifications (100x) blurred the population of the smaller particles. Therefore, the particle size analyses were performed on the BSE images obtained at 500x (or 45,000 pixels/cm).

Calculated Particle Settling Times

In a settling centrifuge, a particle of given size is removed from the liquid if sufficient time is available for the particle to reach the wall of the centrifuge bowl. A simplified diagram for this type of centrifuge is shown in Figure 6. The feed point is at the bottom (A) and the liquid is discharged at the top (B). For a small particle (Stokes region Re < 2) moving at its terminal velocity, the settling time can be calculated from equation 1: [8]

$$t_f = \frac{18\mu}{(\rho_p - \rho_m)\omega^2 D_p^2} \ln \frac{r_B}{r_A} \qquad (1)$$

where t_f = Settling time
μ = Dynamic viscosity
ρ_p = Density of particle
ρ_m = Density of fluid medium
ω = Angular velocity
D_p = Diameter of particle
r_A = Radial position at t = 0.
r_B = Radial position at t = t_f.

The radial position at t = 0 (r_A) corresponds to the liquid-air interface and the radial position at t = t_f (r_B) corresponds to the wall of the centrifuge bowl. The diameter of the H-Canyon centrifuge bowel is 40-in (1.02 m); [9] therefore, r_A = 20-in (0.51 m). The value of r_B is calculated from the dynamic holdup volume (V) and the height of the centrifuge bowl (L) assuming the slurry occupies an annulus with a thickness of r_B-r_A. The dynamic holdup volume is 60 gal (0.23 m^3) [10] and the height of the centrifuge bowl is 24-in (0.61 m). [9] The calculated radius of the liquid-air interface (14.7-in or 0.37 m) is obtained from equation 2.

$$V = \pi L(r_B^2 - r_A^2) \tag{2}$$

The nitric acid concentration of the solutions in Tanks 11.1 and 12.2 is nominally 5 M. [11] The density and viscosity at this concentration are given in Table 2. [12]

Table 2 Physical properties of 5 M nitric acid

Property	Value	Units
Density	1.1611	g/cm^3
	1,161.1	kg/m^3
Viscosity	1.2540	cp
	0.0012540	kg/m s

The data in Table 1 show that undissolved solids generated during the flowsheet development activities were primarily ternary oxides containing U. The densities of these compounds are not readily available. Therefore, the density of the particle in equation 2 will be assumed to vary between 2 and 4 g/cm^3 (2000-4000 kg/m^3). The theoretical density of uranium oxide particles generally vary between 7 and 11 g/cm^3.[13] Using densities of smaller magnitude will result in the calculation of conservative (i.e., longer) settling times.

The bowl of the H-Canyon centrifuge rotates at 1740 rpm. [10] The angular velocity is given by equation 3.

$$\left(1740\frac{\text{revolutions}}{\text{min}}\right)\left(2\pi\frac{\text{radians}}{\text{revolution}}\right)\left(\frac{1\text{min}}{60\text{s}}\right) = 182.2\frac{\text{radians}}{\text{s}} \tag{3}$$

Using equation 1, the geometry and rotational velocity of the H-Canyon centrifuge, and the physical property data summarized above, the settling times for particles with diameters between 0.1 and 50 µm were calculated. The calculations are summarized in Appendix A and the results are shown in Figure 7. The residence time of the centrifuge is nominally 5 min, based on a dynamic volume of 60 gal [10] and a feed rate of 12 gal/min. [14] This value is also plotted on Figure 7 for comparison.

Results and Discussion

Particle Size Analysis

Using the BSE images (1019 x 765 pixels^2), we performed manual contrast enhancement (to spread the images to the 0-255 gray level scale) and thresholding (two color segmentation of the images) steps to isolate the particles from the filter paper. The resulting binary images contained black-colored particles on a white background. Then particle analysis, with edge detection, allowed measurement of the particle area, perimeter, and diameter. These data were segmented into bins and plotted to generate particle size distribution histograms (number of particles per diameter) from which population averages were computed.

Three images of the solids from each sample were analyzed. The images corresponding to the Tank 11.1, Tank 12.2-1, and Tank 12.2-2 samples are shown in Figures 8, 9, and 10, respectively. The particle size distribution histograms were constructed by binning the number of particles with diameters which fell in the ranges 0-0.5, > 0.5-1, > 1-5, > 5-10, > 10-15, > 15-20, > 20-25, > 25-30, >30-35, > 35-40, >45-50, and > 50-100 μm. The number of particles in each bin was plotted as a function of the maximum particle size of the bin. The histograms for the Tank 11.1, Tank 12.2-1, and Tank 12.2-2 samples are plotted in Figures 11, 12, and 13, respectively.

From the histograms, one can see that the particle size of the solids ranged from many small particles less than 1 μm in diameter to a few particles with diameters in the 50 to 100 μm range. The mean and median particle diameter and total number of particles measured in each image are provided in Table 3 for each histogram. The table also shows the percentage of the particles less than 1 μm in diameter and the percentage of the total volume of the solids which these particles represent. If one assumes the density of the solids is relatively constant, then the volume of the particles is proportional to the mass.

Table 3 Characteristics of solids from Tanks 11.1 and 12.2

Sample ID	Image Number	Mean Particle Diameter (μm)	Median Particle Diameter (μm)	Number of Particles Counted	Particles < 1 μm (%)	Volume of Particles < 1 μm (%)
Tank 11.1	490BS	1.9	1.1	1113	41	0.054
	492BS	1.7	1.1	1111	46	0.31
	494BS	2.6	1.5	961	31	0.011
Tank 12.2-1	496BS	1.6	1.3	1017	30	0.25
	497BS	1.7	1.1	2967	40	0.089
	499BS	1.5	1.2	876	35	0.18
Tank 12.2-2	504BS	1.7	1.2	680	36	0.49
	506BS	2.1	1.3	1852	37	0.039
	508BS	1.5	1.1	1071	48	0.80

Table 3 shows that both the mean and median particle diameters are reasonably consistent within the images obtained from the same samples. The samples also contained a significant fraction of solids with diameters < 1μm; however, the volume (or mass) of these solids is insignificant (< 1% of the total solid volume) when compared to particles with larger diameters.

Effectiveness of Centrifugation

In preparation for the dissolution of Pu-containing materials in HB-Line, the HEU solutions presently stored in Tanks 11.1 and 12.2 must be moved. The proposed plan is to centrifuge the solutions to remove solids and transfer the solution to the EUS tank. A high removal efficiency of the solids by the centrifuge is desired to eliminate any potential downstream criticality concerns and operational problems during the purification of the U by the 1st Cycle solvent extraction. The presence of solids in the feed to a solvent extraction process can produce stable emulsions and "crud" during the mixing of the aqueous and organic phases. [15] The effectiveness of the centrifuge in removing the solids is a function of the physical properties of the solids and the fluid and the operational parameters of the centrifuge. The settling time for particles with diameters between 0.1 and 50 μm was calculated for the H-Canyon centrifuge. Figure 7 shows the settling times for particles with densities between 2 and 4 g/cm^3. Particles with densities greater than 4 g/cm^3 would be more quickly removed. The residence time of solution in the H-Canyon centrifuge is nominally 5 min; therefore, particles with settling times less than 5 min should be removed.

The particle size distributions of the solids in Tank 11.1 and 12.2 are shown in Figures 11-13 and are summarized in Table 3. The distributions show that a significant fraction of the counted particles (30-50%) had diameters < 1 μm. The settling time calculations show that particles of this size may not be effectively removed by the H-Canyon centrifuge. Depending upon the density of the particles, the calculations indicate that particles with diameters less than 1 to 0.5 μm will not have sufficient time to settle. For this reason, we recommend the use of a gelatin strike to maximize the removal of solids by the centrifuge. A gelatin strike has historically been used to enhance the removal of silicon which is present in Al-clad reactor fuels as an impurity in the Al and is formed during irradiation as an activation product. [10] The presence of gelatin during centrifugation should help coagulate the submicron particles and facilitate their removal by the centrifuge; although, we have no experimental basis to estimate the level of improvement. Even if the centrifuge cannot remove a significant fraction of the particles with diameters < 1 μm, these particles do not account for a significant mass of the solids in the tanks. Table 3 shows that > 99% of the volume (or mass) of the solids is accounted for by particles with diameters > 1 μm which the centrifuge should remove. It should also be noted that the number of submicron particles may have been over-estimated by the image analysis software used to generate the distribution histograms. The morphology of the filter paper likely resulted in the identification of false particles which had diameters < 1 μm.

Characterization of Solids

Although the characterization of the solids found in the Tank 11.1 and 12.2 samples was not the primary objective of this work, qualitative information concerning the composition of the solids was obtained during the SEM analyses. Photographs of the solids and raster scans, which show the general elemental composition over a large area of the solids, are shown in Figures 14, 15, and 16 for the Tank 11.1 sample and the two Tank 12.2 samples, respectively. The spectra for the three samples show the presence of the same elements with the exception of Gd and S which were present in the Tank 11.1 sample. Gd was added to Tank 11.1 as a nuclear poison. Sulfur may be present as sulfate from the materials dissolved or the peak in the spectrum may be more appropriately attributed to Mo which occurs at nearly the same energy. Each spectrum showed the presence of fissionable material (U and Pu), several refractory transition metals (Ti, W and Ta), corrosion products (Fe, Cr, and Ni), and the presence of aluminosilicates (Al and Si). BSE images and energy dispersive spectra for areas of the solids from the three samples are also shown on Figures 17, 18, and 19. The composition of the particles examined generally contained the same elements as observed in the raster scans with the addition of a small number of metals such as Mg and K. The presence of Cl was also observed in a number of the particles. Ru was identified in a particle from the 12.2-2 sample. Ru is a fission product from the reprocessing of reactor fuels.

Effect of Solids on 1st Cycle Solvent Extraction

If the solids in the HEU solutions stored in Tanks 11.1 and 12.2 are not removed by the centrifuge, there could be operational problems when the U solution is purified by solvent extraction. The presence of a significant amount of solids may lead to the formation of stable emulsions or "crud" in the contacting equipment. Crud is defined as the material resulting from the agitation of an organic phase, an aqueous phase, and fine solid particles that form a stable mixture. Crud usually collects at the interface between the organic and aqueous phases. [15] Little quantitative information is available on the allowable concentration of solids in the aqueous feed to a solvent extraction process which utilizes tributyl phosphate as the extractant. Generally, clarification equipment for a solvent extraction process should target reducing the solids concentration to about 10 ppm. [15]

The concentration of solids in samples from Tanks 11.1 and 12.2 and Tank RT-33 in HB-Line were reported by Gray on a number of occasions. [2-5] These values are summarized in Table 4.

Table 4 Concentrations of solids in Tanks 11.1 and 12.2

Tank	Sample Date	Concentration (μg/mL)	Concentration(1) (ppm)	Reference
11.1	August 1, 2008	610	530	[2]
RT-33	August 5, 2008	790	680	[4]
11.1	August 14, 2008	110, 54, 110, 160	95, 47, 95,140	[3]
12.2	September 10, 2008	320,370	280, 320	[5]

(1) Calculated using the density of 5 M nitric (1.1611 g/cm^3)

The H-Canyon centrifuge should remove a majority of the solids from the Tank 11.1 and 12.2 solutions which have diameters > 1 μm. The settling times for particles of this size are less than the residence time of the fluid in the centrifuge. The particles which remain will not account for a significant mass of the solids in the tanks. Removal of the particles with diameters > 1 μm should result in the removal of > 99% of the solid mass; therefore, the formation of emulsions during the purification of the HEU using the 1st Cycle solvent extraction should not be a problem.

Under the current processing plan, the solutions from Tanks 11.1 and 12.2 will be transferred to the EUS tank following centrifugation. The solution from Tanks 11.1 and 12.2 may remain in the EUS tank for an extended time prior to purification. The effects of extended storage on the solution were not evaluated as part of this study.

Conclusions and Recommendations

An evaluation of the H-Canyon centrifuge and the 1st Cycle solvent extraction concluded that a sufficient amount (> 90%) of the solids in solutions stored in Tanks 11.1 and 12.2 would be removed to prevent the formation of stable emulsions during purification. We based this conclusion on the particle size distribution of solids isolated from samples of the solutions and the calculation of particle settling times in the centrifuge. Samples of the solids were examined by SEM and the images processed using a freeware image analysis program. The calculated distributions had mean particle diameters of 1-3 μm. A significant fraction (30-50%) of the particles also had diameters < 1 μm; however, the mass of these solids was insignificant (< 1% of the total solids mass) when compared to particles with larger diameters.

Settling times for the H-Canyon centrifuge were calculated as a function of the particle size and density. The calculations showed that particles with diameters less than 1 to 0.5 μm will not have sufficient time to settle. For this reason, we recommend the use of a gelatin strike to maximize the removal of solids. The presence of gelatin during centrifugation should help coagulate the submicron particles and facilitate their removal; although we have no experimental basis to estimate the level of improvement. Incomplete removal of these particles should not have a deleterious effect on the performance of the 1st Cycle solvent extraction. Particles with diameters > 1 μm (which should be removed by the centrifuge) account for > 99% of the solid mass; therefore, the formation of emulsions during the purification of the HEU should not be a problem. The HEU solutions from Tanks 11.1 and 12.2 may be stored for an extended period of time following centrifugation. The effects of extended storage on the solution were not evaluated as part of this study.

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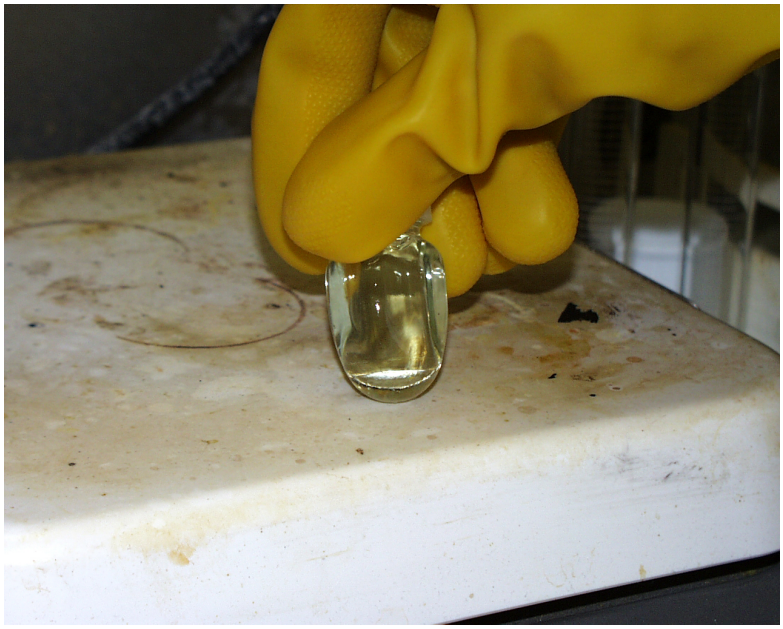
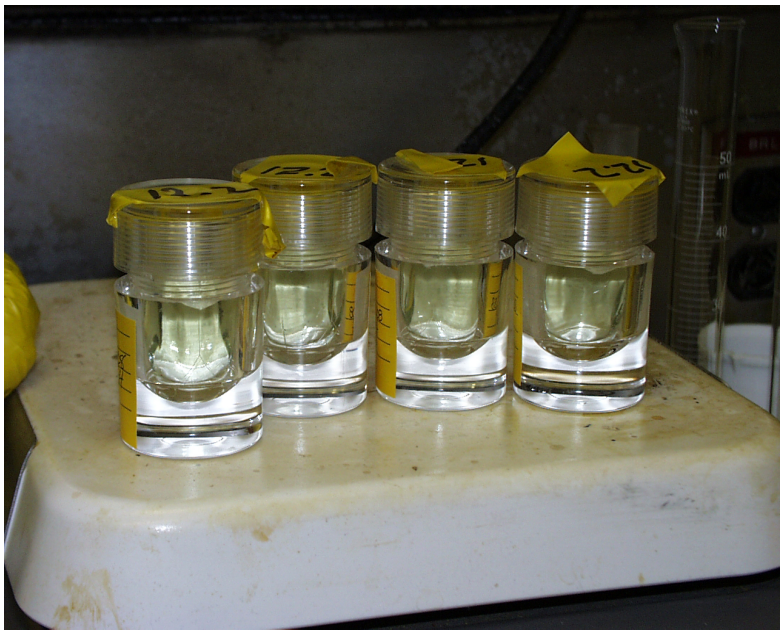
Figure 1 Tank 12.2 Samples



Sample received for particle size analysis



Samples received for characterization

Figure 2 Filter units following filtration of Tank 12.2 characterization samples

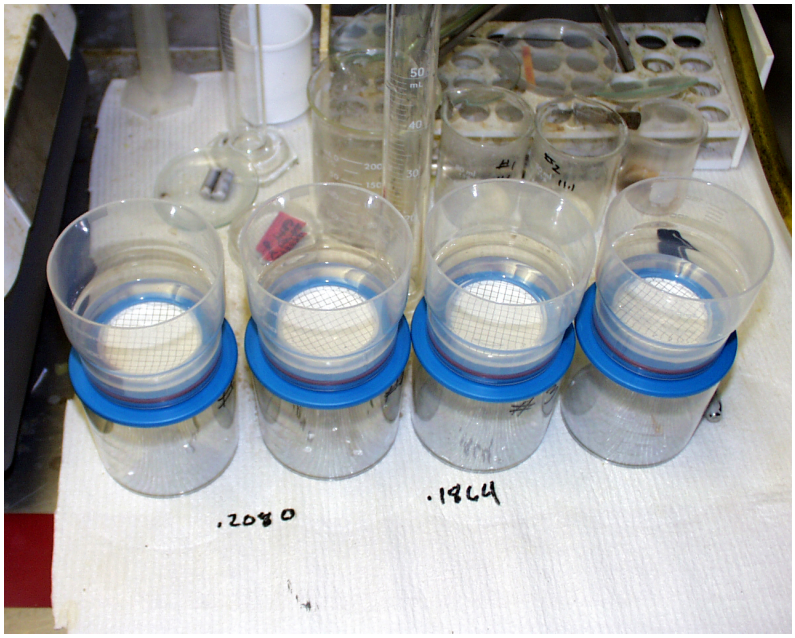


Figure 3 Tank 11.1 sample

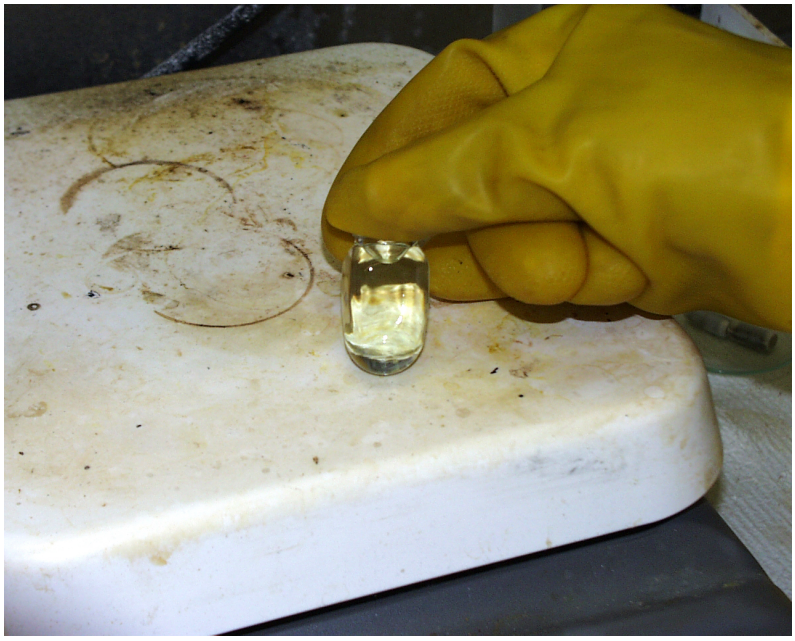


Figure 4 Second Tank 12.2 sample

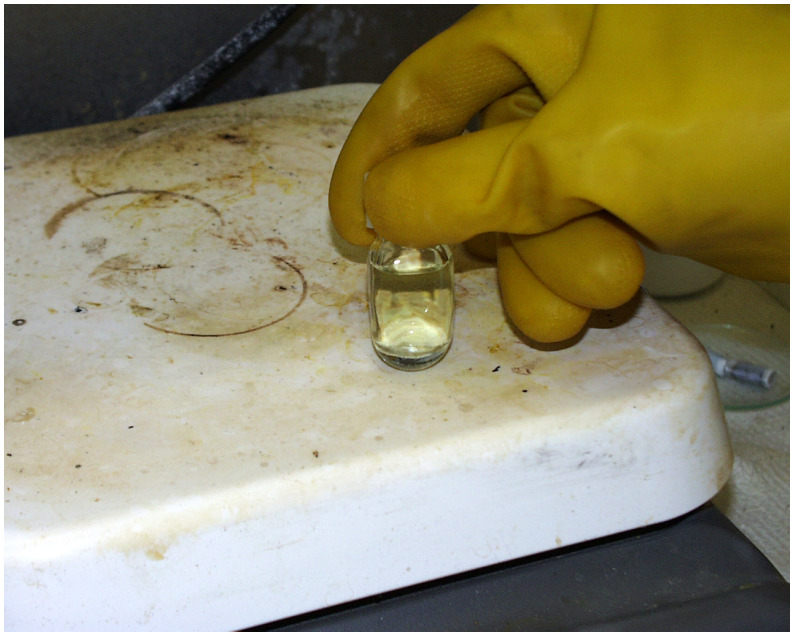


Figure 5 Collection of solids from Tank 11.1 and 12.2 samples

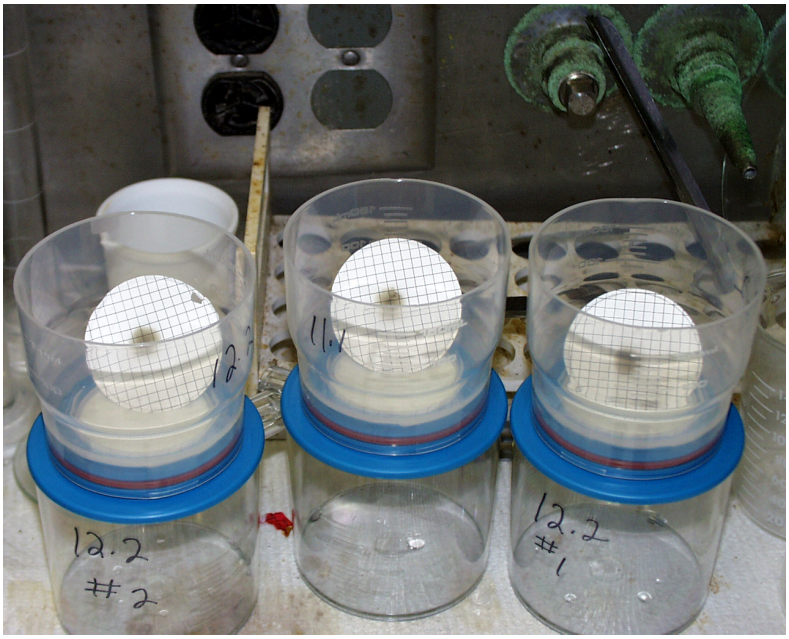


Figure 6 Schematic diagram of a particle-settling centrifuge

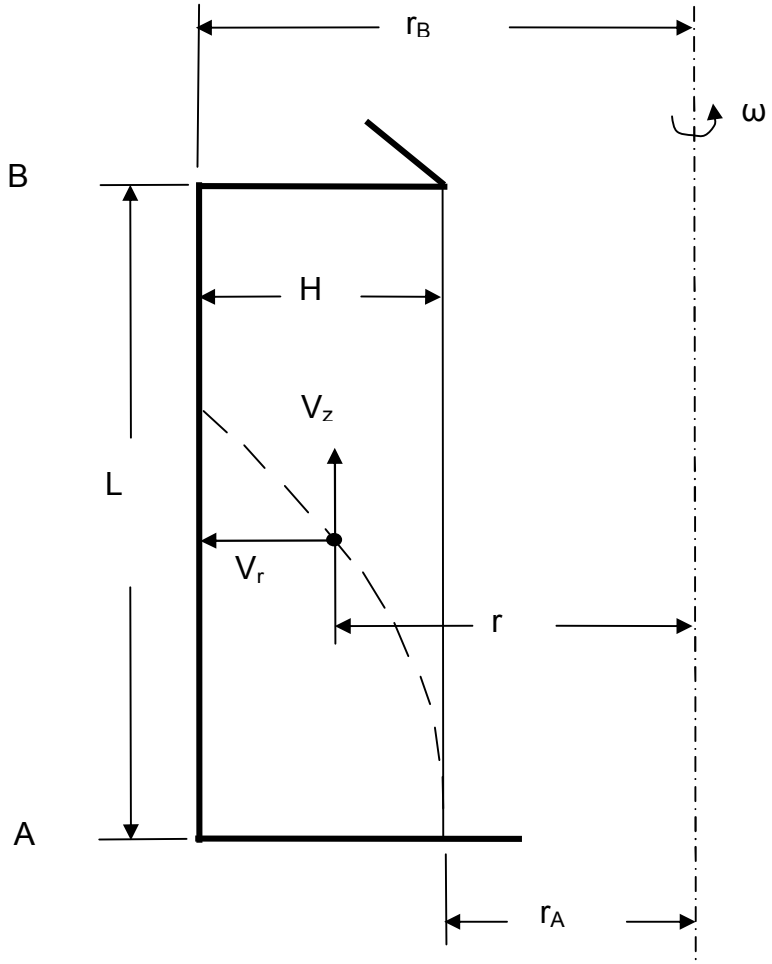


Figure 7 Particle settling times for the H-Canyon centrifuge

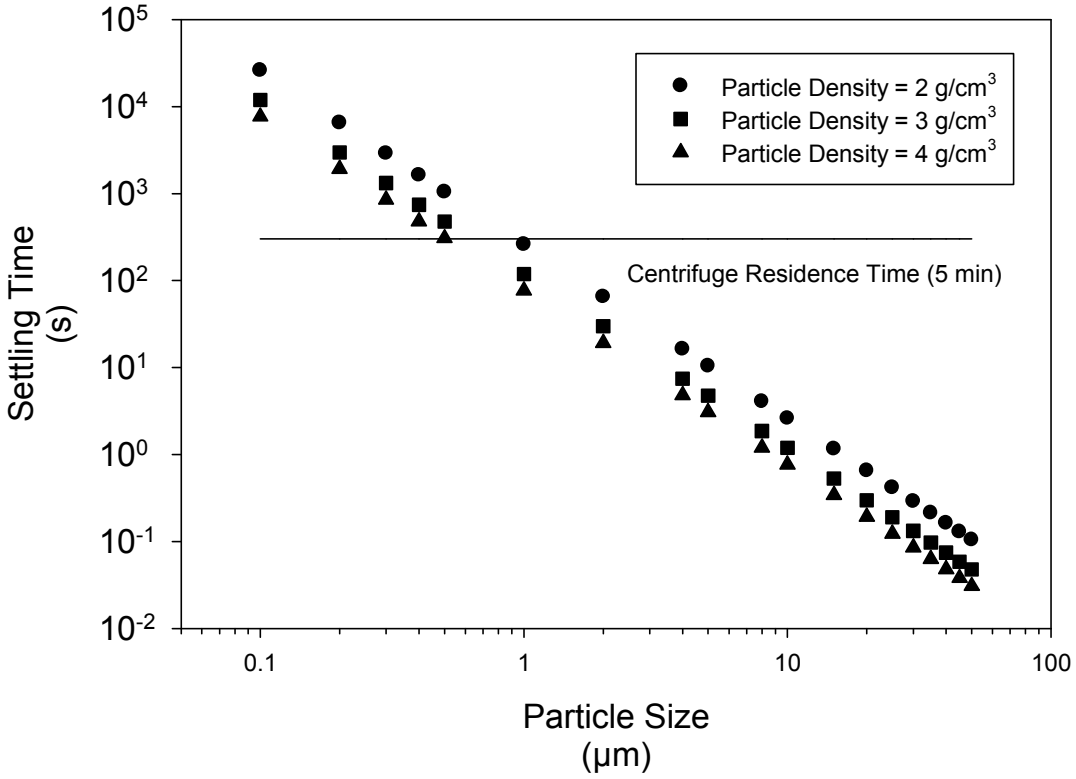


Figure 8 BSE images of solids from Tank 11.1 sample

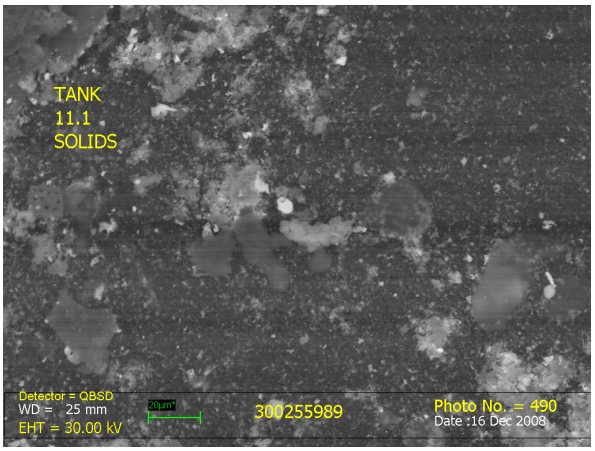


Image 490BS

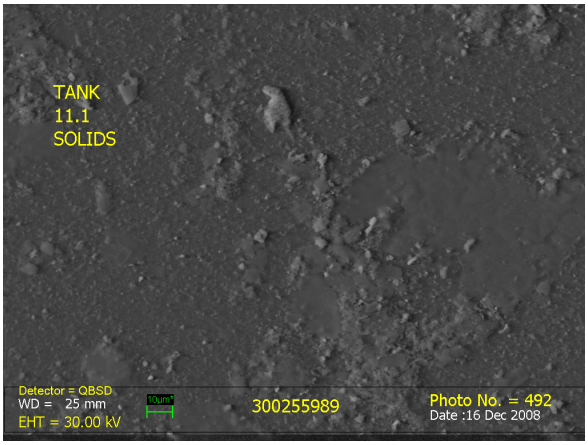


Image 492BS

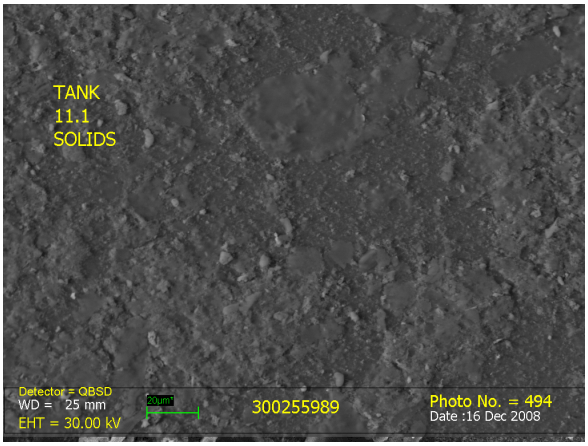


Image 494BS

Figure 9 BSE images of solids from Tank 12.2-1 sample

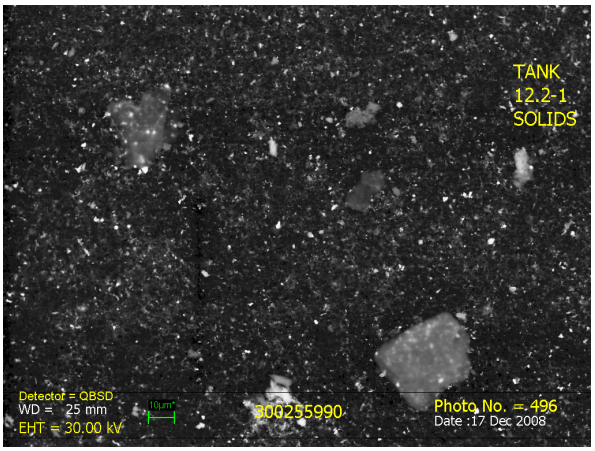


Image 496BS

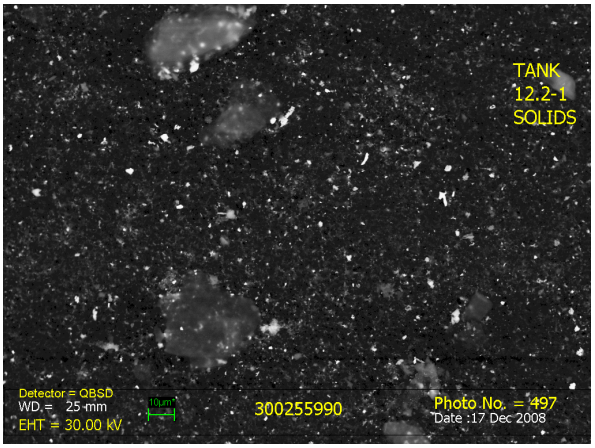


Image 497BS

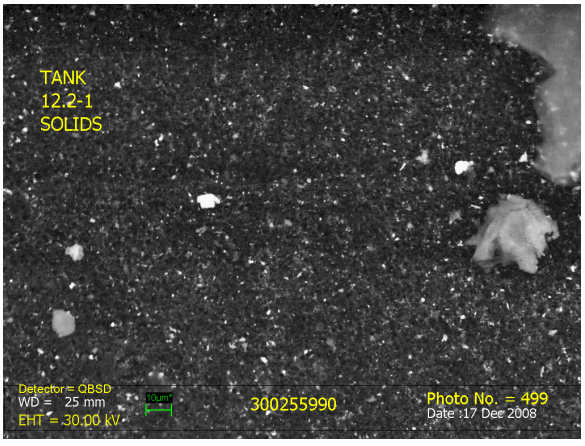


Image 499BS

Figure 10 BSE images of solids from Tank 12.2-2 sample

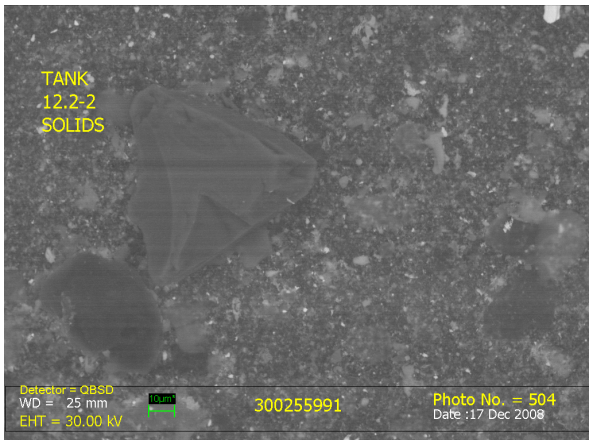


Image 504BS

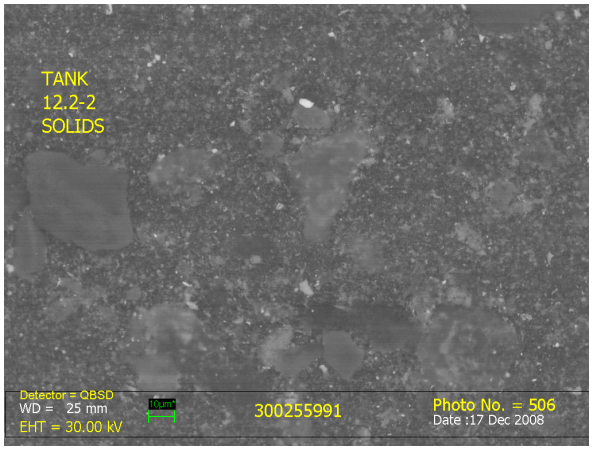


Image 506BS

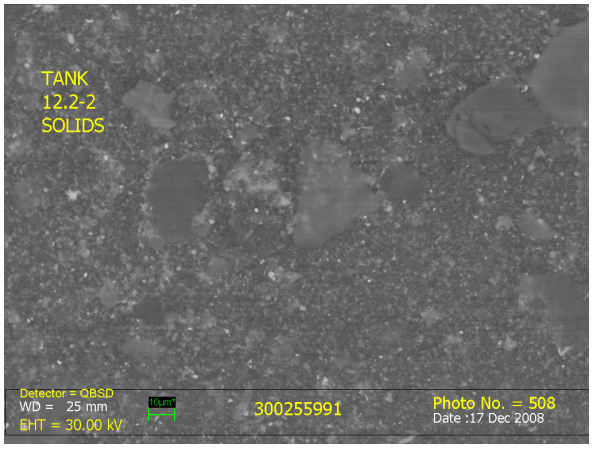
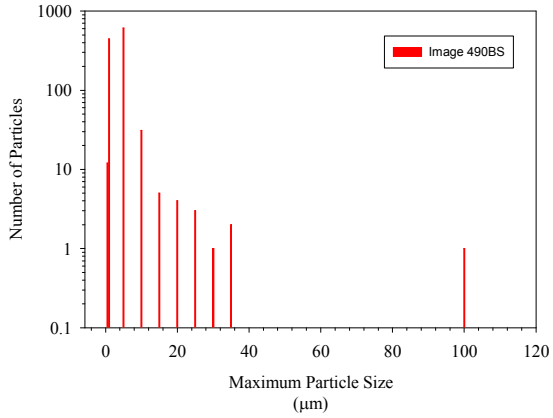
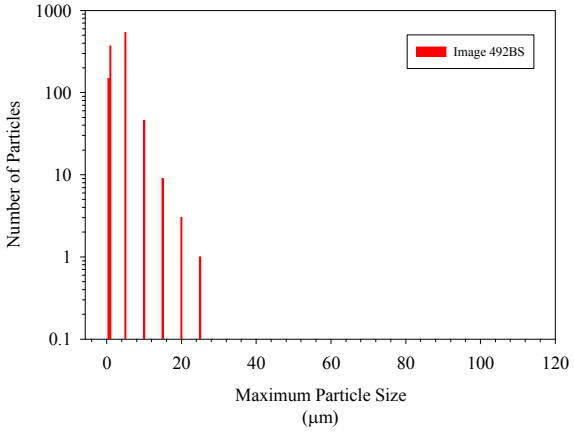


Image 508BS

Figure 11 Particle size distributions for solids from Tank 11.1 sample





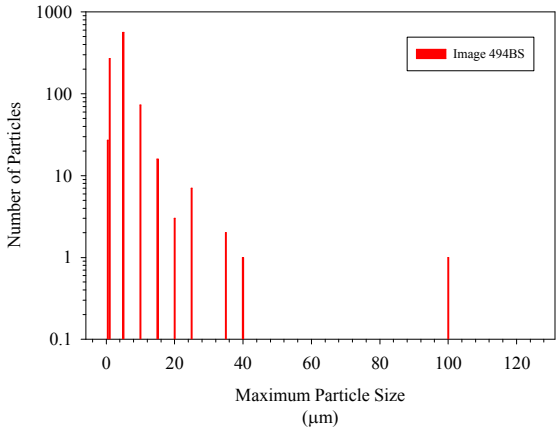
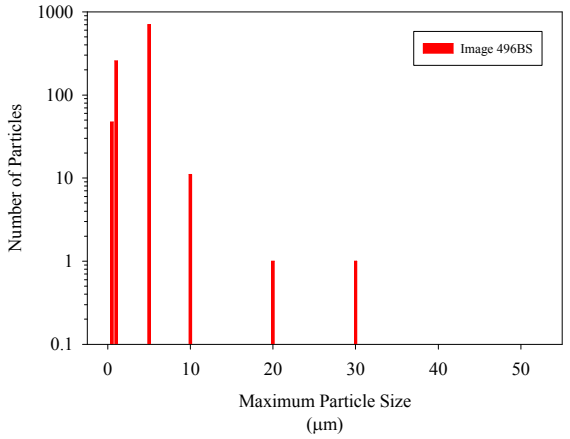
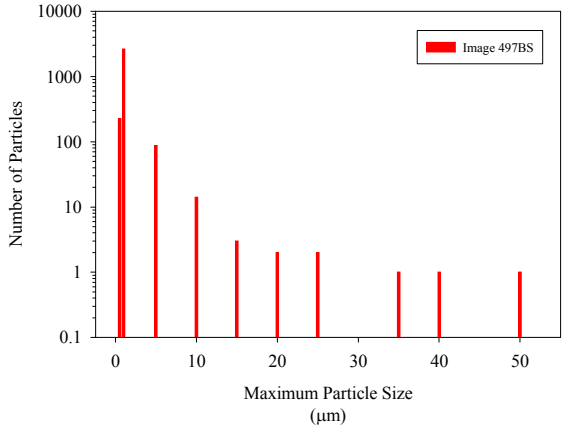


Figure 12 Particle size distributions for solids from Tank 12.2-1 sample





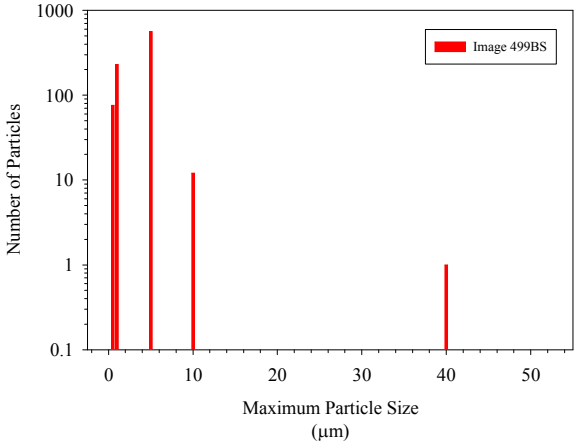
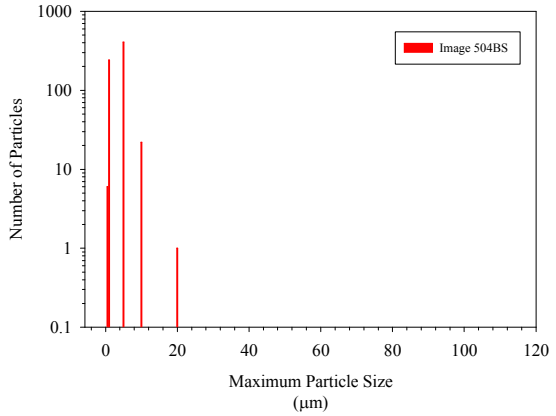
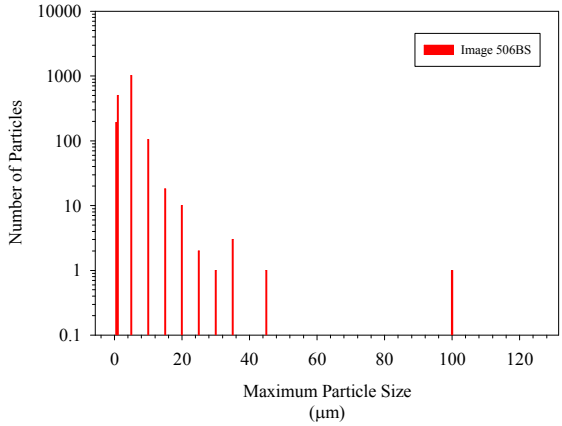


Figure 13 Particle size distributions for solids from Tank 12.2-2 sample





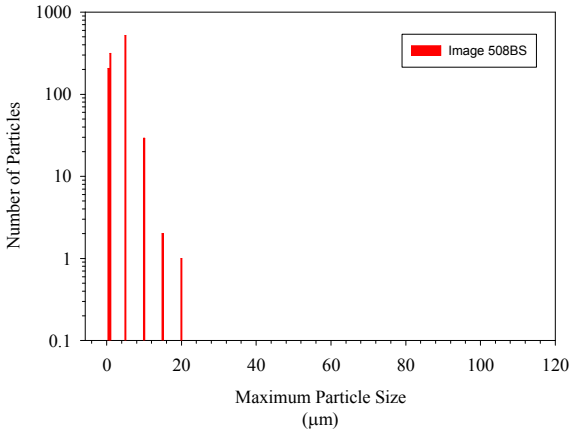
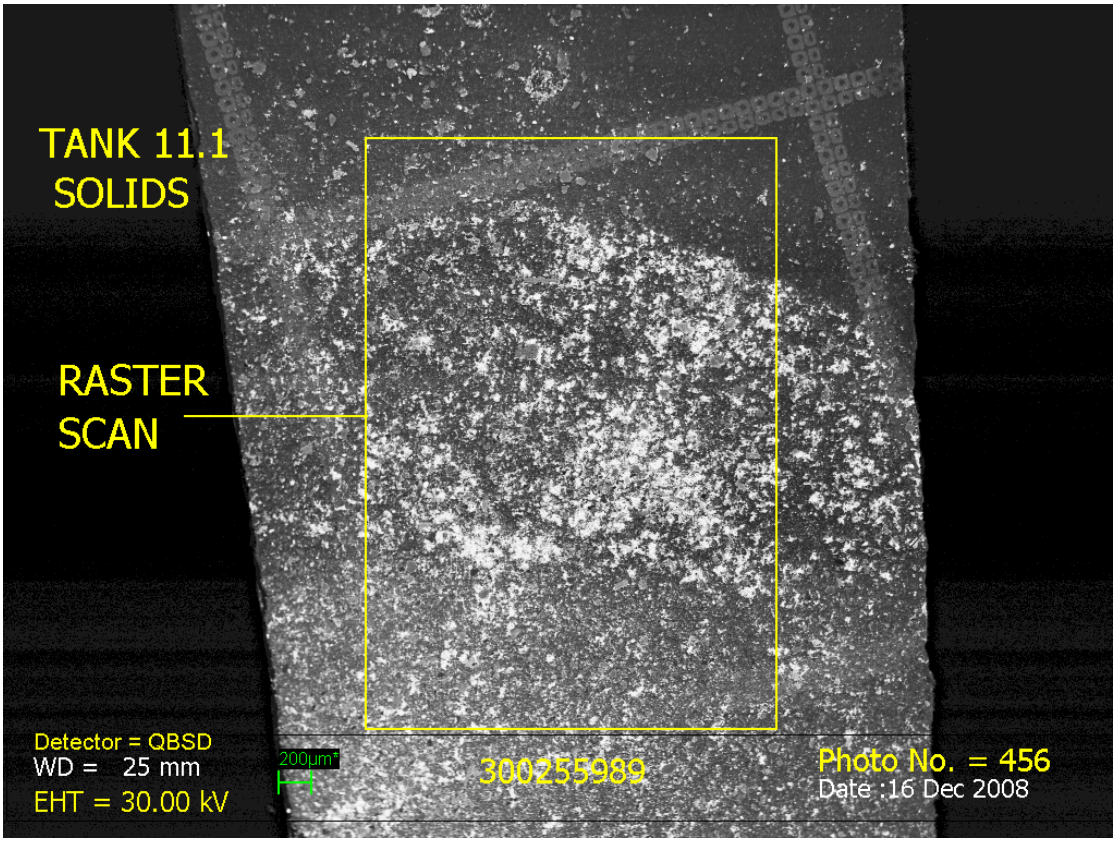


Figure 14 Raster scan and spectrum for Tank 11.1 solids



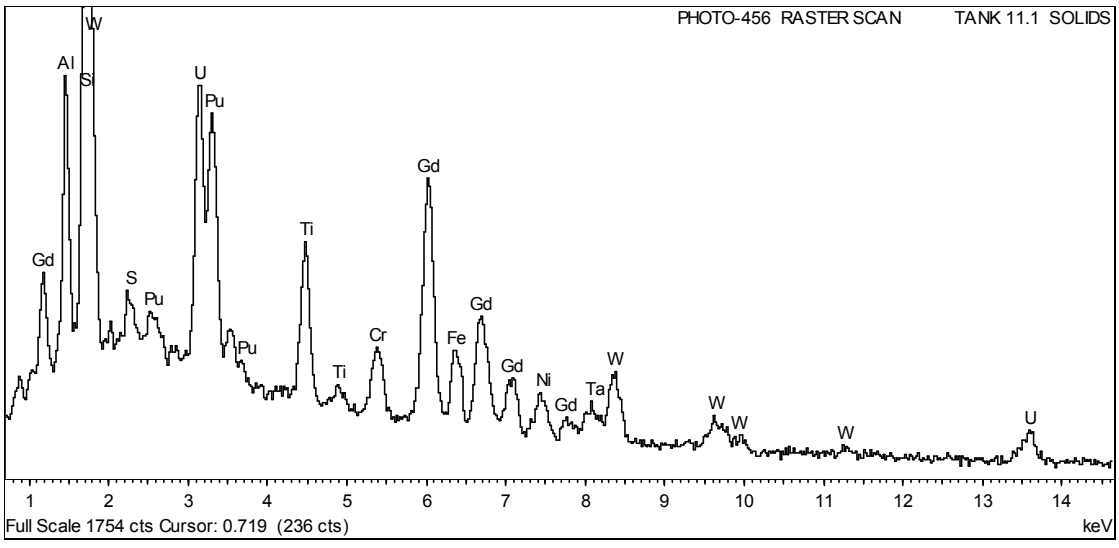
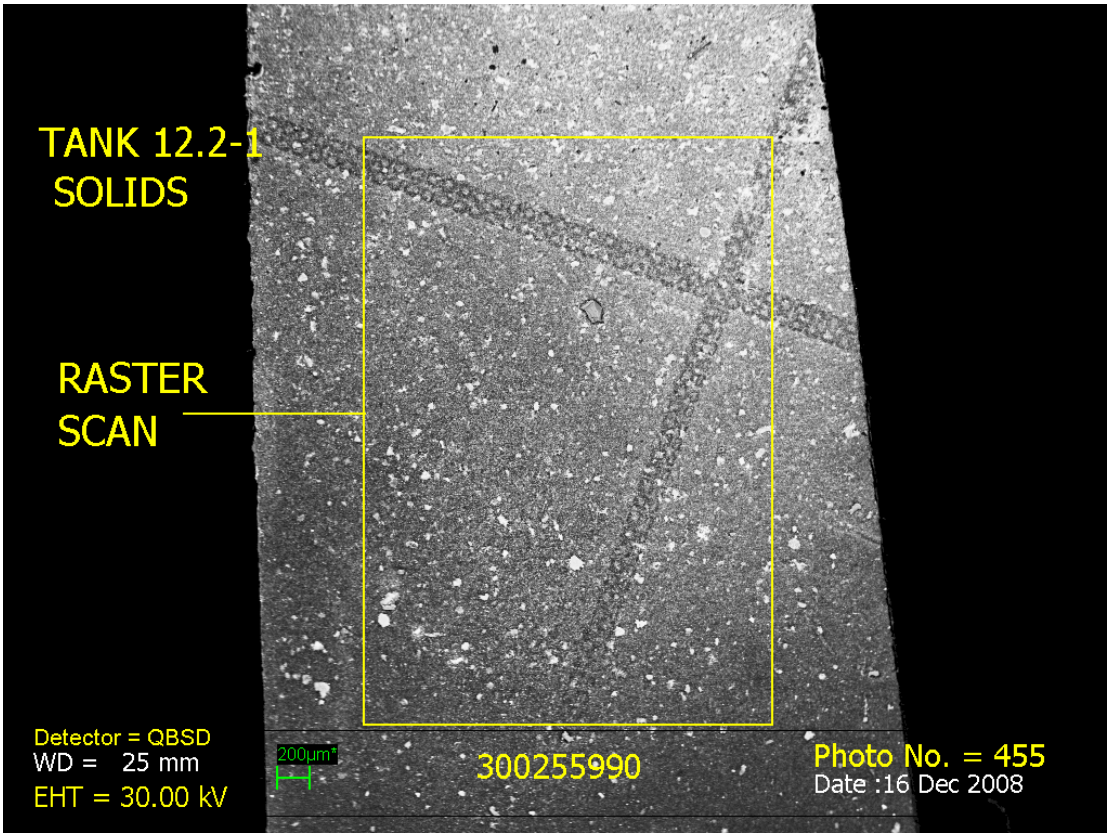


Figure 15 Raster scan and spectrum for Tank 12.2-1 solids



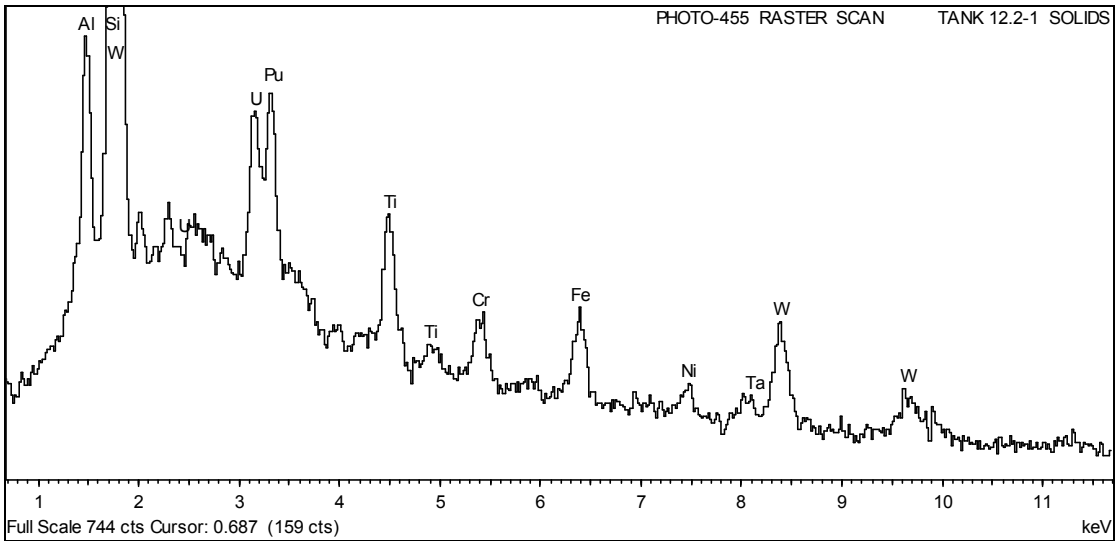
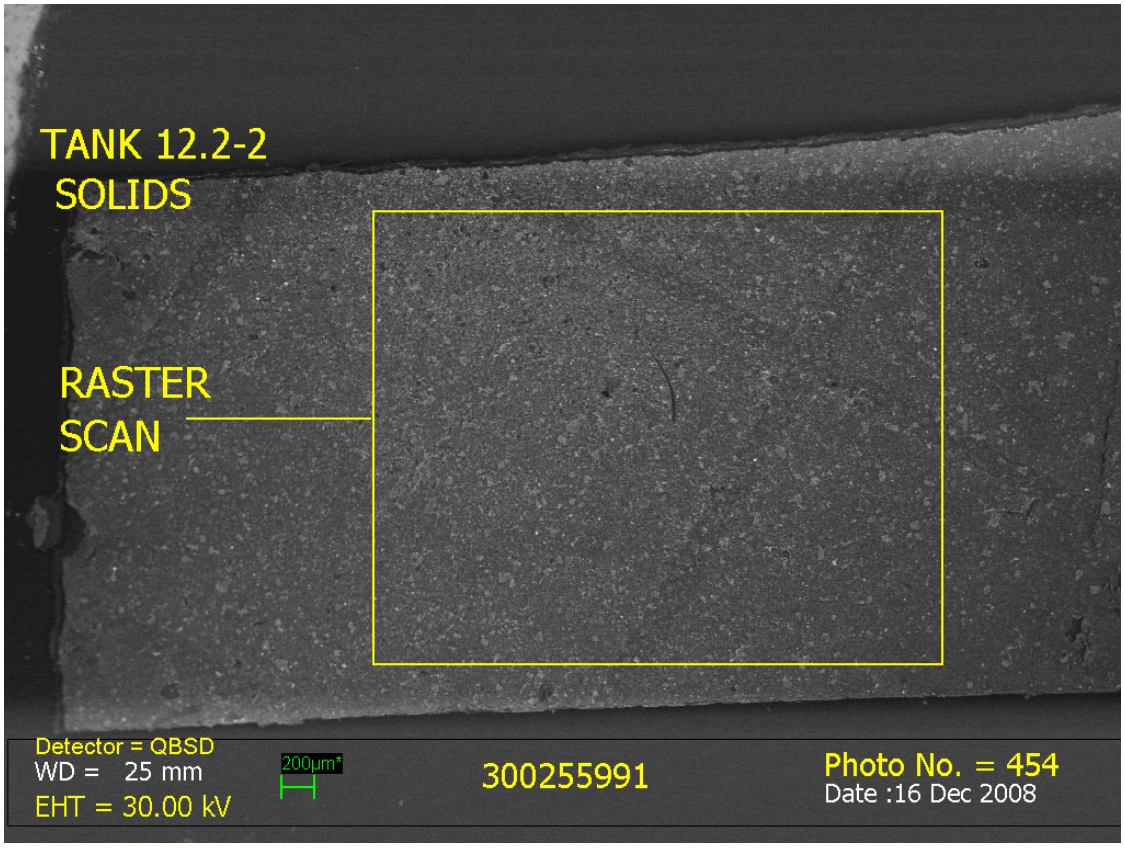


Figure 16 Raster scan and spectrum for Tank 12.2-2 solids



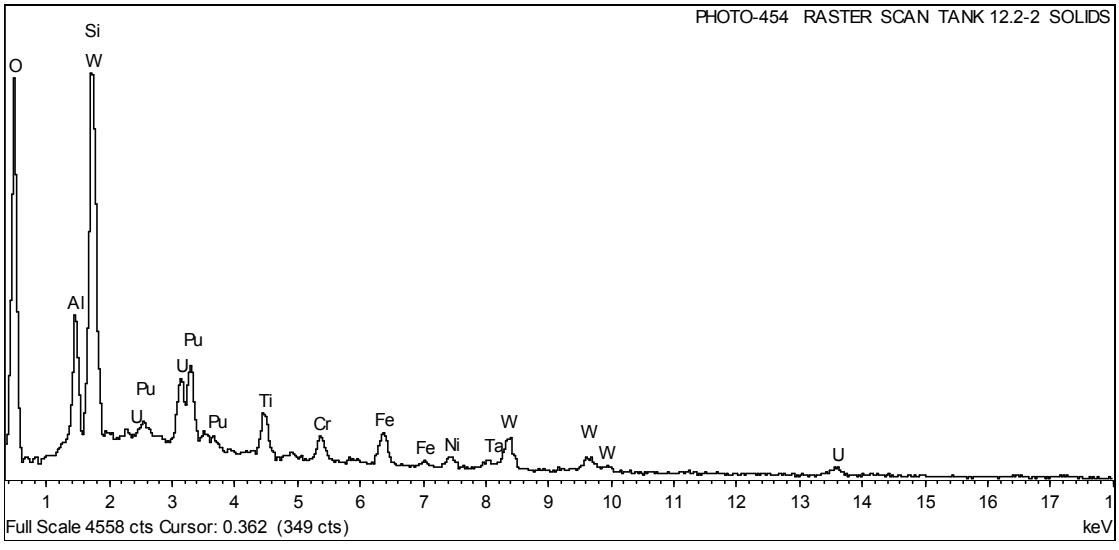
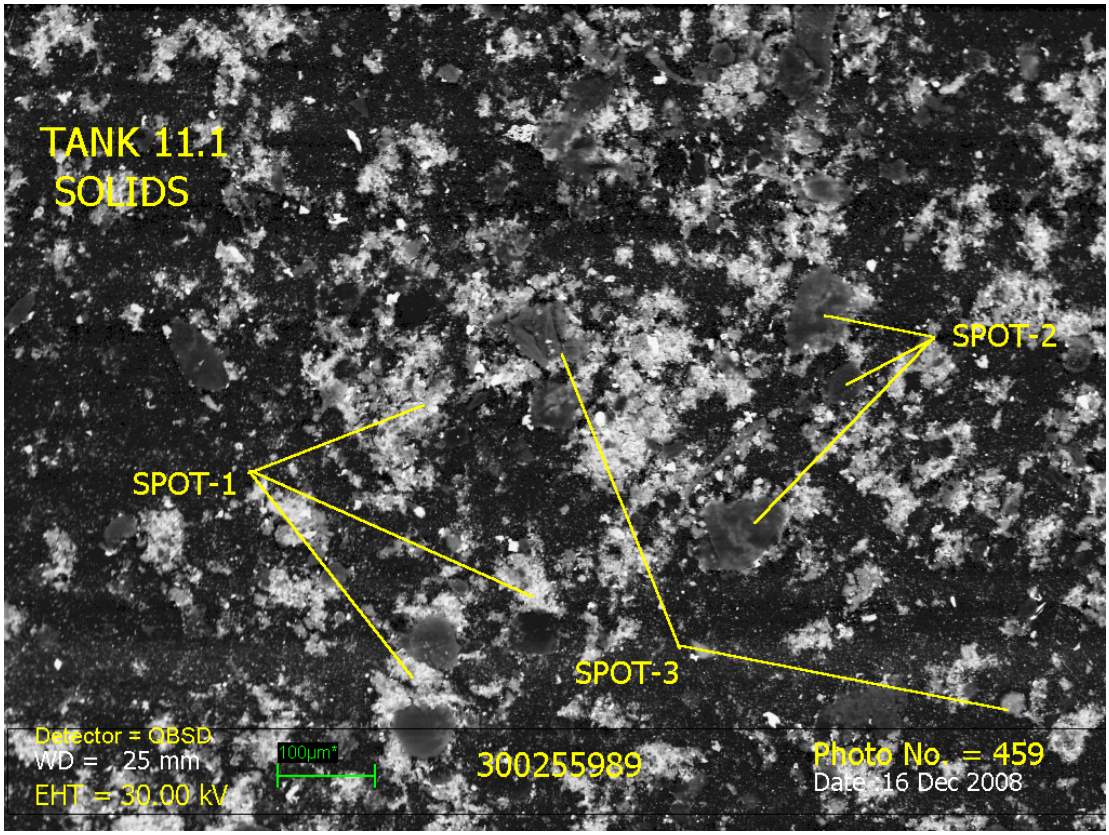
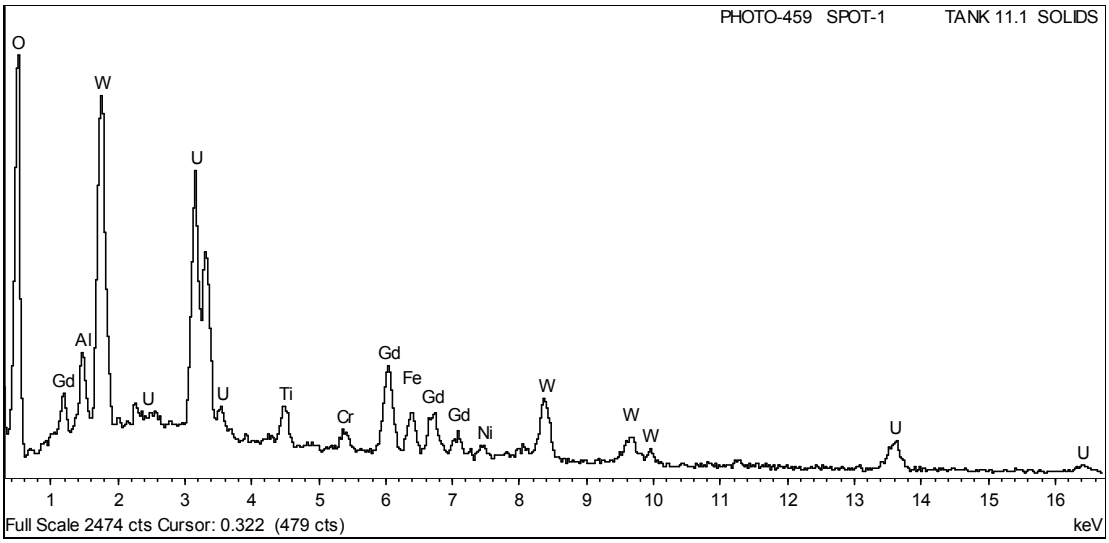


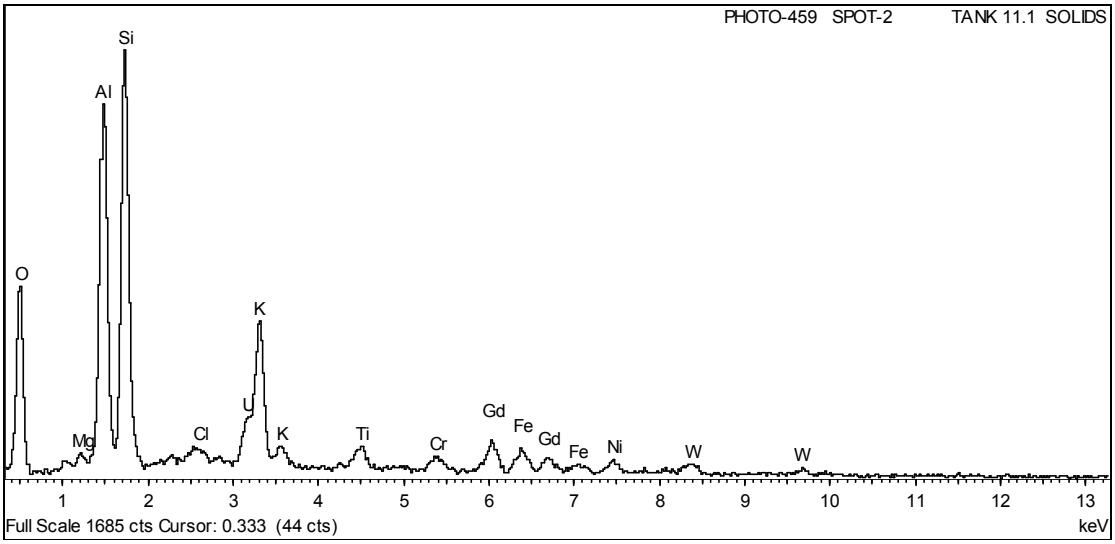
Figure 17 BSE image and spectra for Tank 11.1 solids



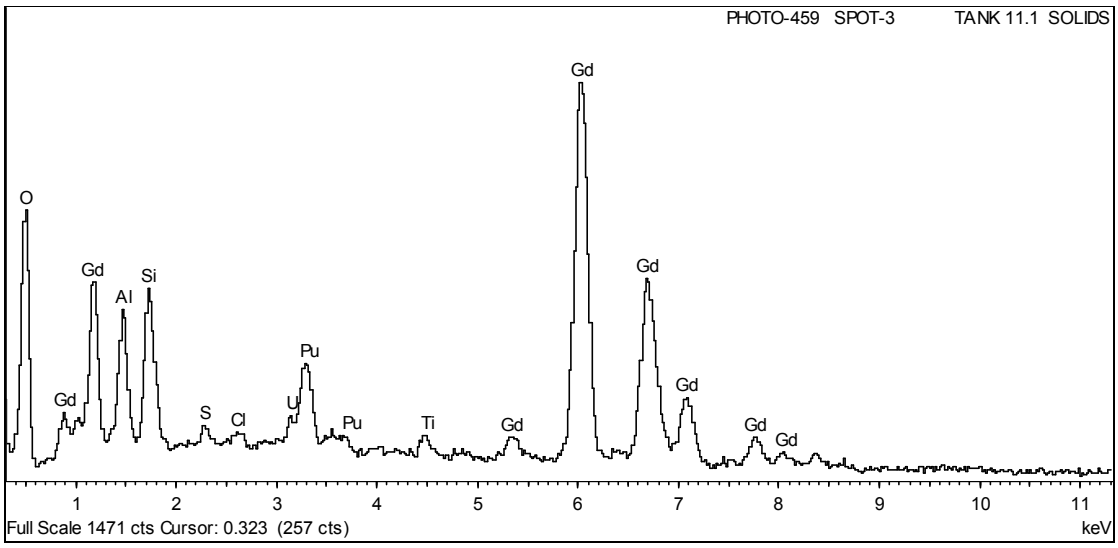


Energy dispersive spectrum for spot 1

Figure 17 continued

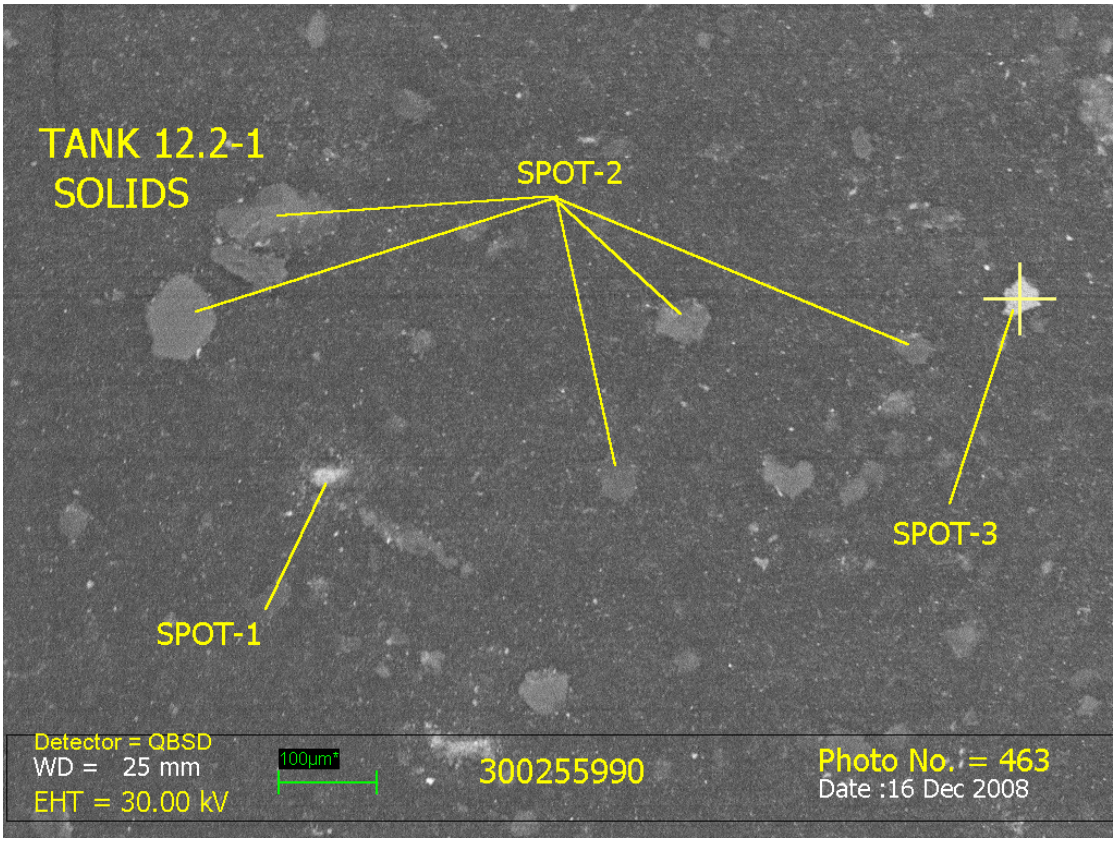


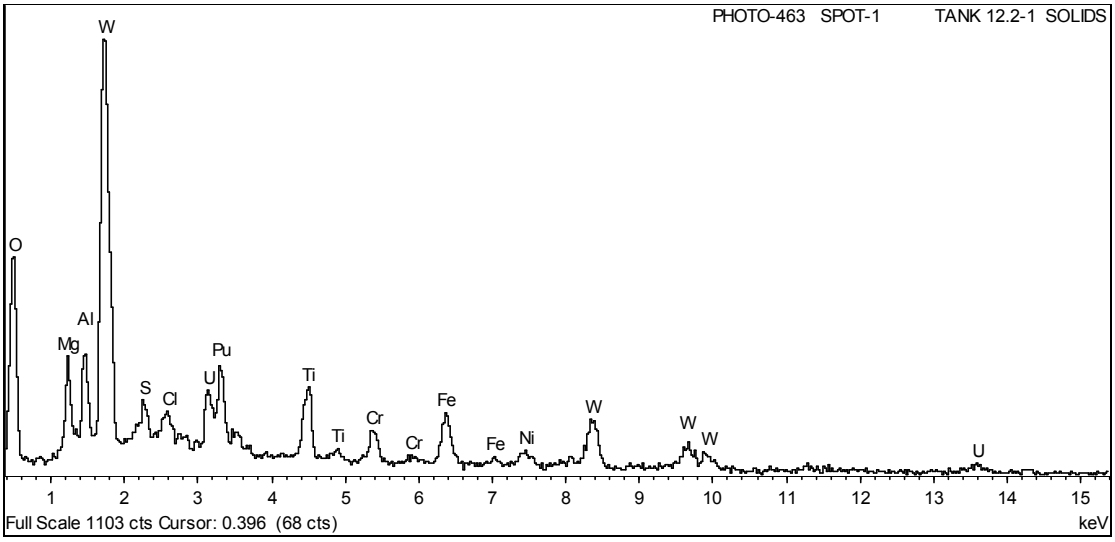
Energy dispersive spectrum for spot 2



Energy dispersive spectrum for spot 3

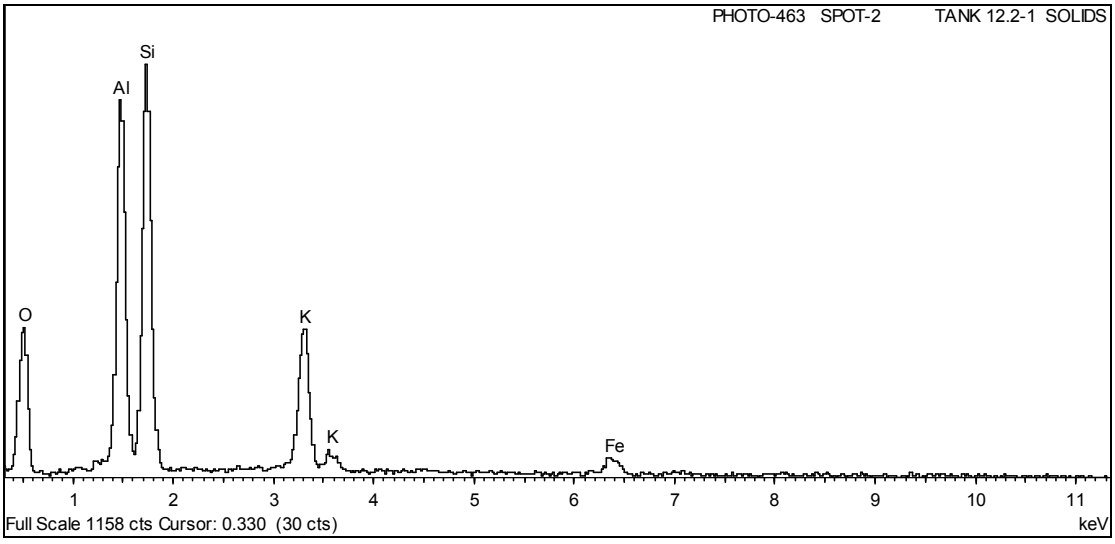
Figure 18 BSE image and spectra for Tank 12.2-1 solids



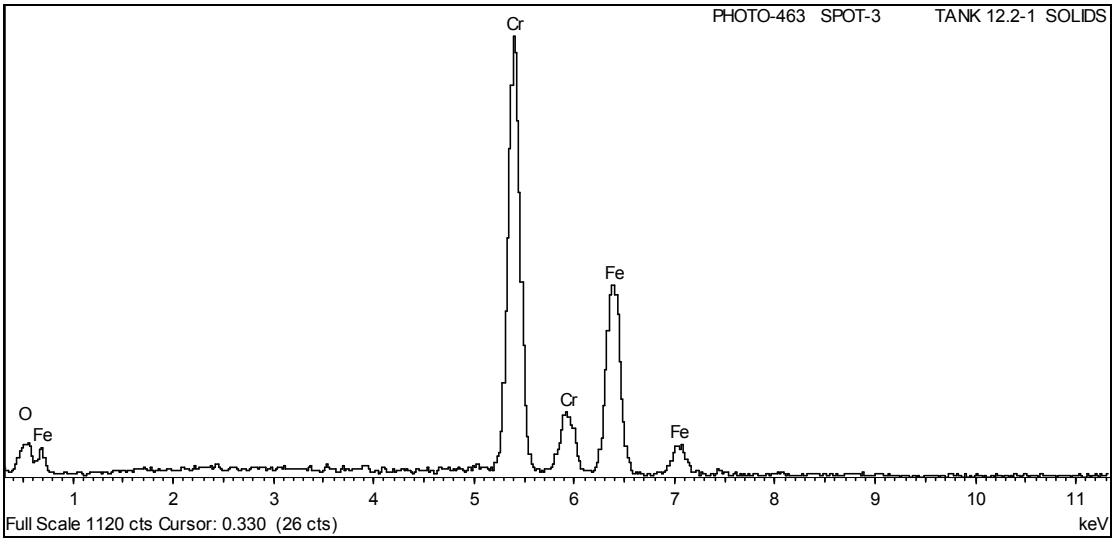


Energy dispersive spectrum for spot 1

Figure 18 continued

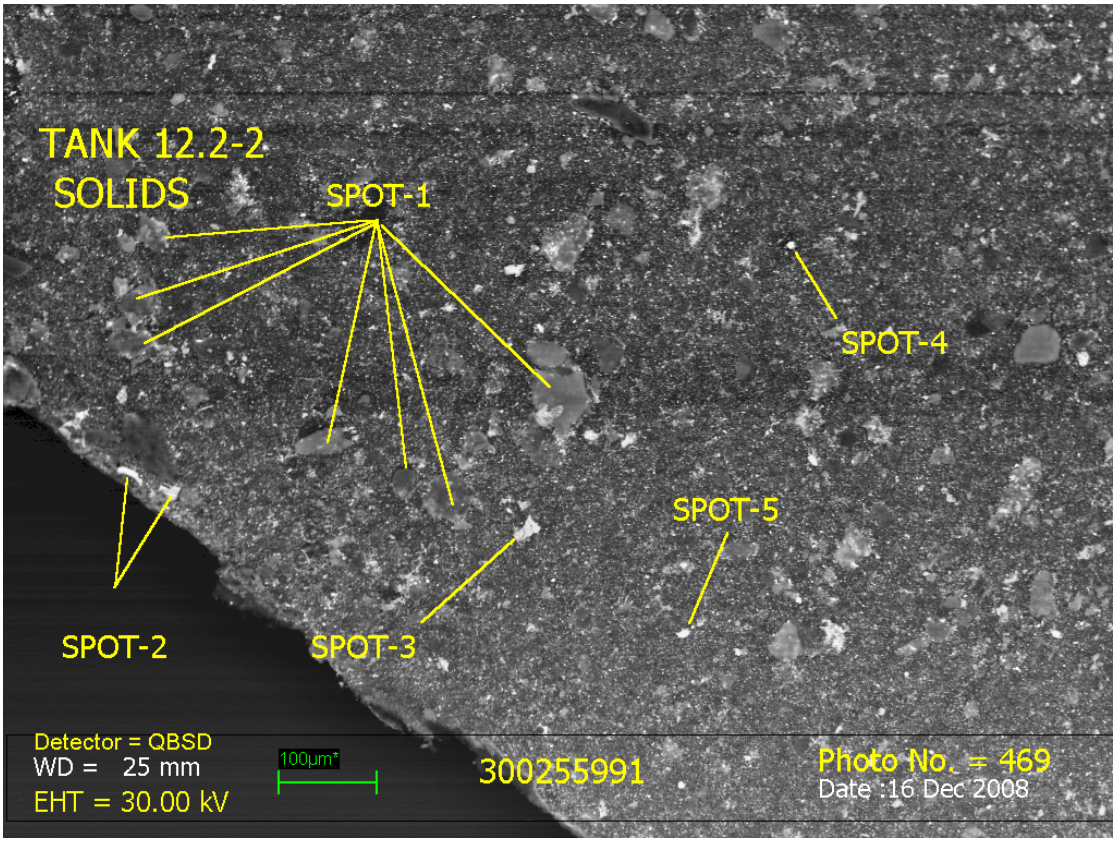


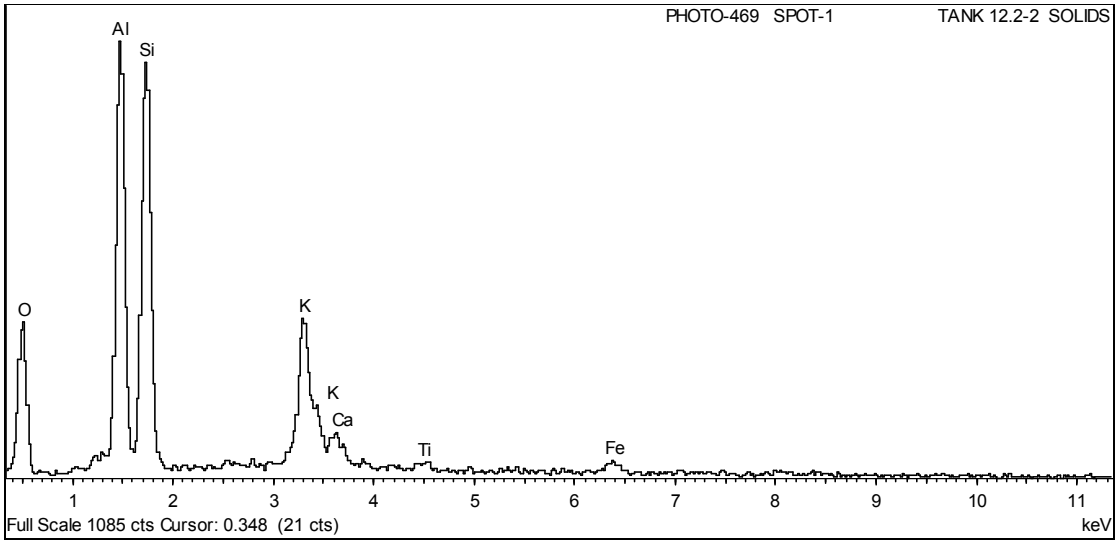
Energy dispersive spectrum for spot 2



Energy dispersive spectrum for spot 3

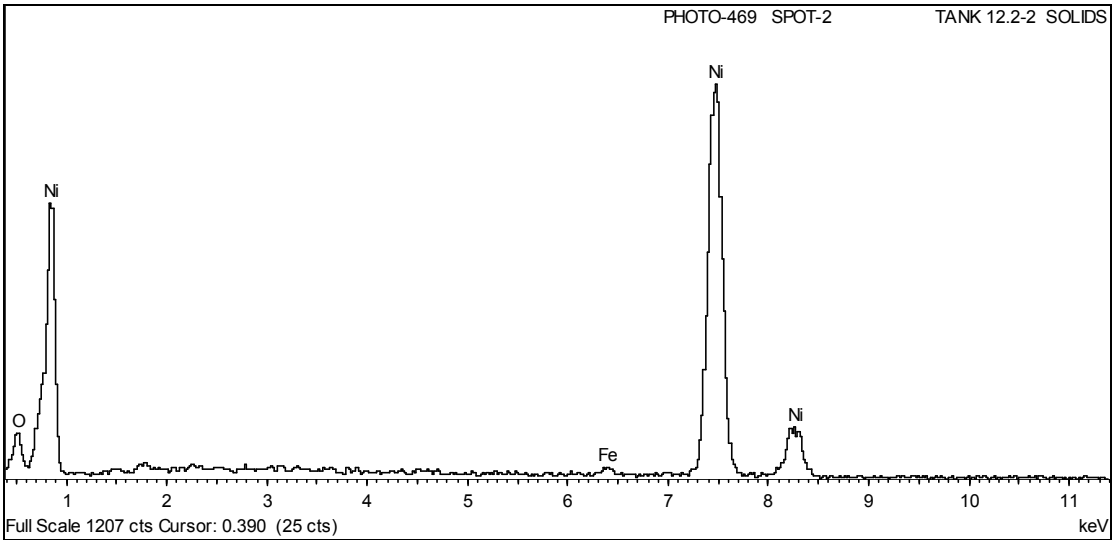
Figure 19 BSE image and spectra for Tank 12.2-2 solids



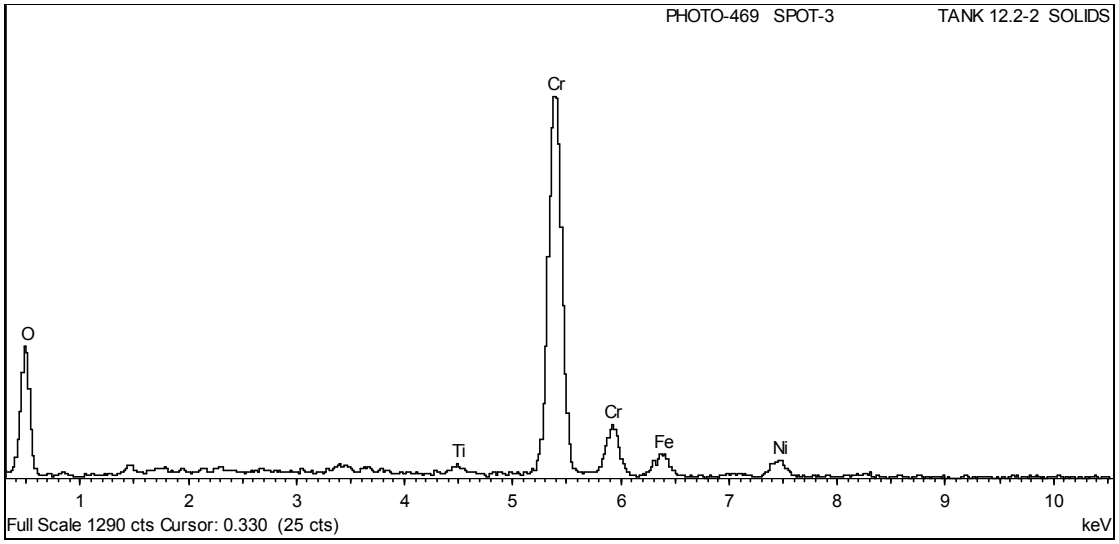


Energy dispersive spectrum for spot 1

Figure 19 continued

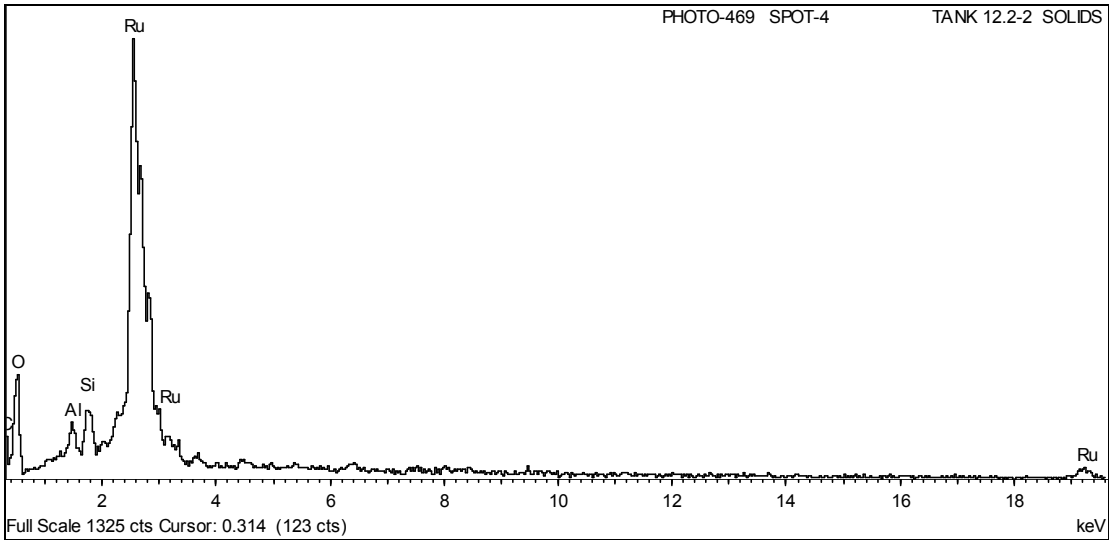


Energy dispersive spectra for spot 2

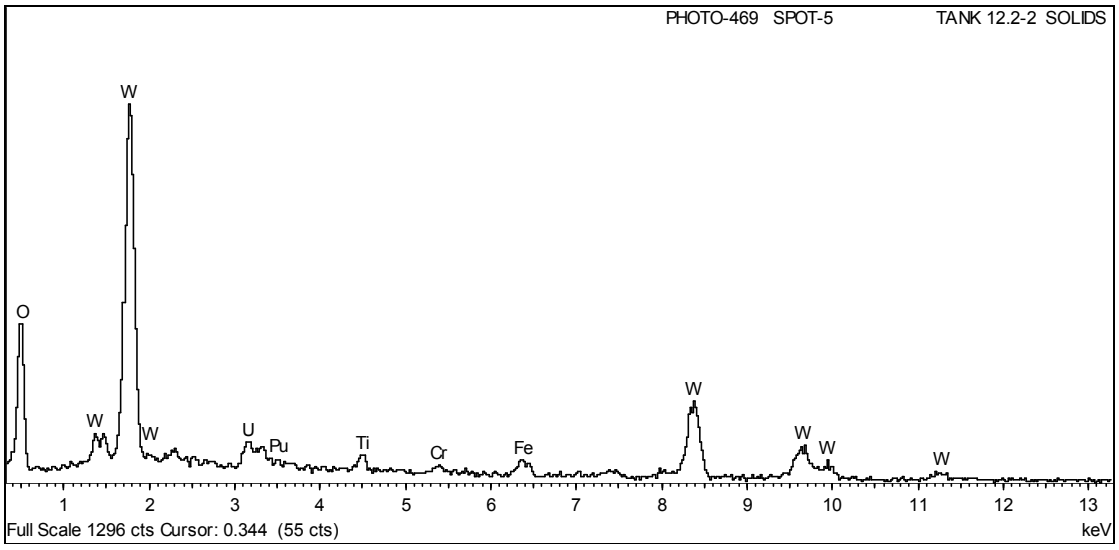


Energy dispersive spectrum for spot 3

Figure 19 continued



Energy dispersive spectrum for spot 4



Energy dispersive spectrum for spot 5

Appendix A Calculation of particle settling times in the H-Canyon centrifuge

The characteristics of the H-Canyon centrifuge used to calculate particle settling times are summarized in Table A.1.

Table A.1 Characteristics of the H-Canyon centrifuge

Parameter	Value	Units	Reference
Bowl Diameter	40	in	[9]
	1.02	m	
Bowl Radius	20	in	
	0.51	m	
Bowl Height	24	in	[9]
	0.61	m	
Dynamic Volume	60	gal	[10]
	0.23	m^3	
Bowl Speed	1740	rpm	[10]
	182.2	rad/s	

The radius of the liquid-air interface (r_A) is obtained from equation 2.

$$V = \pi L(r_B^2 - r_A^2) \tag{2}$$

$$r_A = \sqrt{r_B^2 - \frac{V}{\pi L}}$$

$$r_A = \sqrt{(0.51\ \text{m})^2 - \frac{0.23\ \text{m}^3}{\pi(0.61\ \text{m})}} = 0.37\ \text{m}$$

The particle settling times are now calculated using equation 1. A sample calculation for a spherical particle with a diameter (D_p) of 0.1 μm and a density (ρ_p) of 2 g/cm^3 is shown below.

$$t_f = \frac{18\mu}{(\rho_p - \rho_m)\omega^2 D_p^2} \ln\frac{r_B}{r_A} \tag{1}$$

$$t_f = \frac{(18)\left(\frac{0.0012540\ \text{kg}}{\text{m s}}\right)}{\left(\frac{2000\ \text{kg}}{\text{m}^3} - \frac{1161.1\ \text{kg}}{\text{m}^3}\right)\left(\frac{182.2}{\text{s}}\right)^2 \left(1\text{x}10^{-7}\ \text{m}\right)^2} \ln\left(\frac{0.51\ \text{m}}{0.37\ \text{m}}\right) = 26{,}010\ \text{s}$$

The results of the calculations for the total range of particle sizes and densities are shown in Table A.2.

Table A.2 Settling times for spherical particles in the H-Canyon centrifuge

Density (g/cm^3) Particle Size (μm)	2 Settling Time (s)	3 Settling Time (s)	4 Settling Time (s)
0.1	26010	11866	7686
0.2	6503	2966	1922
0.3	2890	1318	854
0.4	1626	742	480
0.5	1040	475	307
1	260	119	77
2	65	30	19
4	16	7	5
5	10	5	3
8	4	2	1
10	3	1	1
15	1	1	0.3
20	1	0.3	0.2
25	0.4	0.2	0.1
30	0.3	0.1	0.1
35	0.2	0.1	0.1
40	0.2	0.1	0.05
45	0.1	0.1	0.04
50	0.1	0.0	0.03