Experimental Evaluation of Solids Suspension in Canyon Process Vessels (U)

by

N. M. Hassas

Westinghouse Savannah River Company
Savannah River Site
Aiken, South Carolina 29808

DOE Contract No. DE-AC09-89SR18035

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Keywords: Uniformity
Canyon vessels
Solids suspension
Mixing

Retention : Permanent

Classification: U

James E. Landon
Authorized Derivative Classifier

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N. M. Hassan

June 25, 1996

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Westinghouse Savannah River Company
P. O. Box 616
Aiken, SC 29802

Prepared by the U. S. Department of Energy under Contract ICO 229486JL

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N. M. Hassan

Issued: June 25, 1996

Approvals

N. M. Hassan, Author

6-25-1996

Date

J. E. Laurinat, Technical Reviewer

6-25-96

Date

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SUMMARY AND CONCLUSION

Experimental evaluation of solids suspension in canyon process vessels was performed at several paddle agitator speeds and different volume levels in a geometrically similar vessel. The paddle agitator speeds examined were 280, 370, 528, and 686 rpm and volume levels were 30%, 50%, and 70% fill capacity. Experiments were conducted with simulated solid particles that have particle size range and density similar to plutonium particles and corrosion products typically seen in canyon vessels. Solids suspension took place in baffled cylindrical vessel equipped with two flat-blade agitators and cooling helices.

The data collected from model equipment were analyzed for nickel content to simulate plutonium particles seen in canyon vessels. The analytical results were employed to determine the degree of suspension of nickel in slurry. Experimental results indicate that, at a minimum agitator speed of 280 rpm, up to 95% suspension was achieved for nickel particles. Scale-up of model tests using an equal power per unit volume relationship between the model and plant equipment show that a plant agitator speed of 68 rpm provides nearly complete uniformity of suspension of solids in canyon vessels. The results also indicate that the degree of suspension of solid particles decreases with increasing volume levels, as expected. The data collected from vertical sampling heights directly above the regular sampling location revealed no apparent difference in the degree of

suspension of solid particles in the axial direction. Therefore, it is concluded that current agitator speed in process vessels (i.e. 68 rpm) is sufficient to provide nearly complete uniformity of plutonium particles in canyon vessels.

INTRODUCTION:

A previous study performed to document the quality of solids suspension in canyon process vessels, WSRC-TR-95-0258, "Mixing Quality Characterization in Separations Process Tanks Part II: Solid-Liquid Suspension (U) ", suggested a 1.7-fold increase of the plant agitator speed to at least 120 rpm for the attainable condition of complete, but non-uniform, suspension of solids in separations process vessels. The study, which was conducted with simulated material (sea sand), identified appropriate total liquid levels, minimum agitation speed for off the bottom suspension for two geometrically similar scales of operation, and the conditions under which the regular plant sampling point gives results comparable to the average of variously placed measurements around the vessel. Since the simulated material that was previously used (sand) had larger particle size range and smaller average density than heaviest particles (Plutonium particles) found in canyon vessels, further experimental work was needed to be conducted with simulated solids (surrogate) in slurry that had physical properties (e.g. average density and particle size range) similar to the solids found in canyon vessels.

This series of experiments was aimed at evaluating the performance of current agitation in canyon process vessels to provide nearly complete uniformity of solid particles in suspension. The experiments were conducted in model equipment to determine the agitator speed that would provide complete off-bottom suspension and/or nearly complete uniformity of solid particles with average density and particle size range similar to that of solids typically seen in canyon vessels. Near complete uniformity is defined hereafter as the degree of suspension in which the ratio of the weight percentage of solids in suspension to the weight percentage of solids in the agitated vessel is up to 95% and nearly equal at all

points in the vessel. Complete uniformity, which is defined as 100% suspended solids, can only be approached but never really achieved in practice.

An important variable in assessing the degree of suspension of solid particles is the settling velocity of particles being suspended, which is a function of particle size and density. Since plutonium particles typically seen in canyon vessels have average settling velocity of less than 0.5 ft/min in the Stokes Law region (Table 2), they settle so slowly that they are relatively easy to disperse and complete uniformity is relatively easy to obtain. In the presence of corrosion products and silica, however, the solids may only be lifted off the bottom by a slow moving paddle agitator and not uniformly suspended. Thus, experiments were conducted at several different volume levels and paddle agitator speeds, including one which reflects the normal canyon operation, to determine the agitator speed for complete suspension of solid particles with appropriate size and average density relative to solids in canyon vessels.

EXPERIMENTAL

Test Equipment: The details of the model equipment was given in WSRC-TR--0258, "Mixing Quality Characterization in Separations Process Tanks, Part II: Solids Suspension". It consists of a cylindrical, flat-bottom, Plexiglas tank with an inside diameter of 18 inches and an overall height of 36 inches. The tank is equipped with six radial baffles of width equal 1.25 inches, three banks of coil helices made of 1/2-inch diameter stainless steel tubing, and two shear-type radial flow, four-bladed paddle agitators. The flat blades were 4.5-inch diameter and were mounted on a centrally located shaft. All of the model equipment dimensions (Table I) were chosen such that the ratio of the length scale of the plant equipment to lab equipment was 5.33:1.

Test Procedure: Known amounts of simulated solids in slurry with density and particle sizes similar to solids in canyon vessels were charged to the model vessel. The volume level was adjusted to the desired fill capacity and the vessel was agitated at various agitator speeds, including but not limited to the scaled plant speed of 68 rpm - different

agitator speeds were necessary in order to determine the experimental minimum speed for complete particle suspension (N_{js}). After agitation was fully developed, after about 30 minutes, samples were withdrawn from the regular sampling location 1 inch from vessel floor. Five replicate samples were obtained for each agitator speed and volume level. The withdrawal velocity was maintained above the settling velocity of the heaviest particles. The weight of total solids (i.e. weight of sand, iron, and nickel) was determined by drying and weighing. The nickel content of the samples was determined by neutron activation analysis performed by the Analytical Development Section (ADS) of SRTC.

The standard test controls employed in this work were found to be adequate and met QA requirements. The transparent lab vessel was marked with a graduated scale to allow easy reading and adjustment of volume levels. The volume level readings were taken by more than one observer to minimize possible observer bias. The estimated errors in volume level readings were ± 0.5 inches. A hand-held phototachometer was used to measure and control agitator rotational speeds. The phototachometer was calibrated using a fluorescent light bulb which gave a reading of 7200 rps.

Simulated Solids (Surrogate) in Slurry: There is no completely reliable substitute for using actual process slurry containing solids that are typically seen in canyon vessel. In the absence of process slurry, however, a simulated solids with average density and particle sizes similar to solids found in process vessels can obviate correction factors for property differences upon scale-up. Portions of the samples obtained from the process vessels were analyzed by inductively coupled plasma/mass spectrometry (ICP/MS) after complete dissolution of the samples in 8M nitric acid. After filtration, the ICP/MS analysis of the solution filtrate was used to determine the concentration of metal ions in samples. The undissolved solid residues remaining on filter paper were analyzed by scanning electron microscope (SEM). The scans and pictures were used to determine the particle size range of solids found in the samples.

The samples obtained from Tank 18.3 over an eight month period were analyzed in SRTC and the data were recorded as shown in Tables 2 through 5. The metal ions identified in the samples from Tank 18.3 were divided into three major groups: Group I consisted of oxides of Si (sand), Na, Al, Ca, B, and P. The average density and particle size of this group were 2.6 g/cm^3 and $243 \text{ }\mu\text{m}$, respectively. Group II solids were comprised of oxides of Fe, Cr, Cu, Mn, Zr, and Zn (corrosion products) with an average density of about 5 g/cm^3 and a mean particle size of $93 \text{ }\mu\text{m}$. Group III solids consisted of the oxides of Ni, Mo, and PuO_2 with an average density 10.7 g/cm^3 and mean particle size of about $13 \text{ }\mu\text{m}$. The mean concentration of total solids in slurry was limited to 1% by weight in the lab tests. The simulated solids slurry was made of 0.6% sand, 0.3% iron powder, and 0.1% by weight of nickel powder. The physical properties of the simulated solids slurry are shown in Table 2.

RESULTS AND DISCUSSION

The minimum agitator speed (N_{js}) for off-bottom suspension was predicted from Zweitering correlation using the dimensions of the model equipment, property of solids slurry, and different weight percent ratios of solids in slurry. The Zweitering equation was given as

$$N_{js} = \frac{S v^{0.1} d_p^{0.15} \left(\frac{g \Delta \rho}{\rho_L} \right)^{0.45} \bar{\phi}^{0.13}}{D^{0.85}} \quad (1)$$

where S is a dimensional constant, v is the kinematic viscosity, d_p is the suspended particle diameter, g is the gravitational acceleration constant, $\Delta \rho$ is the density difference between

the solid particles and liquid phase, D is the impeller diameter, ρ_L is the density of liquid phase, and $\bar{\phi}$ is mean solids concentration. The agitator speeds (N_{js}) predicted to suspend solid particles in lab model and plant equipment are shown in Table 6 (columns 5 and 6, respectively). The minimum agitator speed for suspension of solid particles in canyon

vessels was calculated from a scale-up relationship developed by Chapman³ for agitated vessels without coil helices. The scale up relationship was given as

$$\frac{N_{js-2}}{N_{js-1}} = \left(\frac{\bar{\phi}_2(1-\bar{\phi}_1)}{\bar{\phi}_1(1-\bar{\phi}_2)} \right)^{0.12} \left(\frac{d_{p2}}{d_{p1}} \right)^{0.15} \left(\frac{\Delta\rho_2}{\Delta\rho_1} \right)^{0.4} \left(\frac{D_1}{D_2} \right)^{0.8} \quad (2)$$

The subscripts 1 and 2 refer to the model and the plant equipments, respectively. Since the simulated solids slurry has the same physical properties as that of solids in a plant-size vessel, we use $\bar{\phi}_1 = \bar{\phi}_2$, $d_{p1} = d_{p2}$, and $\Delta\rho_1 = \Delta\rho_2$, in which case equation (2) reduces to a simple equal power per unit volume relationship on two scales of operation. Equation (2) becomes

$$\frac{N_{js-2}}{N_{js-1}} = \left(\frac{D_1}{D_2} \right)^{0.8} \quad (3)$$

The data in Table 6 show the minimum agitator speed for complete off the bottom suspension of solid particles as a function of the weight percentage of nickel particles in the simulated solids slurry. It can be seen that a plant agitator speed of about 77 rpm is capable of providing complete off the bottom suspension of solids slurry containing of up to 0.05% of simulated plutonium bearing solid particles. A 10-fold increase of the amount of nickel or simulated Pu bearing solid particles in the simulated solids slurry (i.e. from 0.05 to 0.5 wt.%) would increase the plant agitator speed by 37%, thus requiring an agitator speed of 106 rpm to achieve complete off the bottom suspension. Since Pu bearing solids are not expected to exceed 0.05 wt.% of total solids in canyon vessels, the current plant agitator speed is sufficient for complete off the bottom solid suspension.

Confirmation of the correlation results was made by conducting experiments at agitator speeds of 280, 370, 528, and 686 rpm. The agitator speed in the plant (68 rpm) corresponds to a lab agitator speed of 280 rpm. Plutonium bearing solid particles in

canyon vessels were simulated by the nickel content in the surrogate material, which consists of 0.6% sand, 0.3% iron, and 0.1% by weight nickel. The nickel content in samples taken from agitated vessel was determined by neutron activation analysis. The analytical results were used to determine the degree of suspension of nickel in the tank. The average, standard deviation, lower 95% confidence limit, and upper 95% confidence limit for the degree of suspension of nickel was determined from a set of 5 replicate samples shown in Table 7. Figure 1 shows the degree of suspension of nickel as a function of agitator speed for 30%, 50%, and 70% volume levels. As shown in Figure 1, all the agitator speeds 280, 370, 528, and 686 rpm achieved nearly complete uniformity of nickel particles in the vessel. It should be noted that the degree of suspension of solid particles decreases with increasing volume levels, as expected.

Figure 2 shows the degree of suspension of nickel, sand, and total solids as a function of agitator speed. The vessel was filled up to 70% volume capacity and charged with simulated solids slurry containing up to 0.5% by weight of nickel in sea sand; the mean solids concentration was 1% by weight. The weight percentage of solids in samples taken from agitated vessel was determined by drying and weighing and the weight percent of nickel was determined by neutron activation analysis. It can be seen in Figure 2 that the degree of suspension of nickel and sand/total was more than 95%, thus indicating nearly complete uniformity of solids in the agitated vessel. Figure 3 shows the degree of suspension of surrogate material containing 0.05% of nickel as a function of different volume levels. It may be noted that the degree of suspension of solid particles decreases with increasing volume levels and agitator speed. The results indicate that the solid particles were suspended off the bottom, but complete uniformity was not achieved throughout the vessel. It can be seen that as the agitator speed was increased more solids were suspended off the bottom and then dispersed into the upper section of the vessel. As a result, the concentration of solid particles at the sampling location (i.e. near the vessel floor) decreased with increasing agitator speed.

Solid particle concentration uniformity in the axial direction (i.e. with vertical sampling height above the vessel floor) was examined using simulated solids slurry containing 0.25 % nickel and 0.75% sand; the mean concentration of solids in the slurry was 1% by weight. The vertical sampling heights were 1", 3", 6", and 9" above the vessel floor. At each sampling height, 5 (replicate) samples were taken and analyzed for nickel content by neutron activation analysis. The uncertainty associated with the analytical method of neutron activation analysis for each sample was determined at the 1 sigma level. The average values from each set of 5 samples were calculated to be 1.31, 1.38, 1.28, and 1.43% at 1", 3", 6", and 9" above the vessel floor, respectively. The degree of suspension of nickel was calculated in the same manner as previously described and, the results were analyzed statistically to determine the 95% confidence intervals. The average, standard deviation, lower 95% confidence limit (LCL), and upper 95% confidence limit (UCL) are given in Table 8. The data reveal no apparent difference in degree of suspension in the axial direction.

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Table I. Dimensions of Lab Model and Plant Equipment Vessels.

	<u>Model (Lab) equipment</u>	<u>Plant equipment</u>
Tank, diameter (inches)	18	96
<u>Impellers</u>		
diameter, inches	4.5	24
width, inches	1	5
thickness, inches	0.125	0.75
<u>Shaft</u>		
diameter, inches	0.625	3.375
length, inches	42	226
<u>Baffles</u>		
width, inches	1.25	7
Clearance (Off the bottom),	1	6
<u>Coils (1/2 inch SS tubing)</u>		
Inner diameter, inches	8	45
Middle diameter, inches	10.6	60
Outer diameter, inches	13.3	75

Table 2. Physical Properties of Simulated Solids Slurry

<u>Sea sand</u>		<u>Iron powder</u>		<u>Nickel powder</u>	
<u>size (μm)</u>	<u>vol. %</u>	<u>size (μm)</u>	<u>vol. %</u>	<u>size (μm)</u>	<u>vol. %</u>
> 704.00	2.69	> 248.90	8.81	> 62.23	3.53
497.80	12.32	176.00	20.33	44.00	8.36
352.00	32.09	124.45	24.07	31.11	13.59
248.90	35.40	88.00	19.09	22.00	16.66
176.00	13.59	62.23	12.59	15.56	14.74
124.45	2.17	44.00	7.44	11.00	12.67
88.00	1.33	31.11	3.89	7.78	10.61
< 62.23	0.43	22.00	2.17	5.50	7.92
		15.56	1.30	3.89	6.93
		< 11.00	0.32	2.75	4.16
				1.94	0.71
				< 1.38	0.13
Average particle diameter (m)	243×10^{-3}	93×10^{-3}		13×10^{-3}	
Average particle density (kg/m ³)	2650	5210		8980	
Settling velocity, U _t (m/sec)	0.0306	0.0196		8×10^{-4}	
(ft/min)	(6.03)	(3.89)		(0.16)	

Table 3. Metal Ion Concentrations ($\mu\text{g/l}$) in Tank 18.3.

ions \ month	January	February	March	April	June	July	August	October
Fe	24	8	30.6	1530	8	303	179	3040
Si	701	527	227	232	1310	1320	212	56
Cu	1	0	0	0	0	0	0	11
Zn	0	0	0	0	1	2	23	0
Na	127	159	141	127	239	283	66	65
Ni	6	18	6	221	1	43	12	469
B	75	60	3	45	149	153	41	44
Ca	24	57	72	47	45	21	694	0
Cr	3	0	7	431	0	78	1	831
Al	59	47	21	24	119	148	2510	42
P	0	0	0	0	0	0	45	13
Pb	0	0	0	0	0	0	0	0
Ba	11	9	6	0	216	0	0	0
Mn	0	0	0	31	0	6	2	57
Mo	0	0	0	0	0	1	7	12
Sn	0	0	0	0	0	4	11	20
Zr	6	3	18	291	6	281	0	1100
Total	1037	888	559	2980	2094	2643	3803	5700
vol., ml	0.01	0.037	0.028	0.026	0.030	0.021	0.017	0.018

Table 4. Weight of Oxides (mg) of Metal Ions in Tank 18.3.

month oxides	January	February	March	April	June	July	August	October
Fe ₂ O ₃	0.69	0.85	2.45	113.75	0.69	18.19	8.70	156
SiO ₂	15.00	41.72	13.60	12.90	84.1	59.30	7.71	2.16
CuO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
ZnO	0.00	0.00	0.00	0.00	0.04	0.05	0.49	0.00
Na ₂ O	3.42	15.86	10.64	8.90	19.33	16.02	3.02	3.15
NiO	0.08	0.85	0.21	7.31	0.04	1.15	0.26	10.74
B ₂ O ₃	4.83	14.30	5.41	7.54	28.79	20.69	4.49	5.10
CaO	0.34	2.95	2.82	1.71	1.89	0.62	16.51	0.00
Cr ₂ O ₃	0.09	0.00	0.57	32.76	0.00	4.79	0.05	43.72
Al ₂ O ₃	2.23	6.57	2.22	2.36	13.49	11.75	161	2.86
P ₂ O ₅	0.00	0.00	0.00	0.00	0.00	0.00	2.72	0.83
PbO	0.00	0.00	0.00	0.00	0.00	0.00	0.00	0.00
BaO	0.12	0.37	0.19	0.00	7.23	0.00	0.00	0.00
MnO ₂	0.00	0.00	0.00	1.28	0.00	0.20	0.05	1.62
MoO ₂	0.00	0.00	0.00	0.00	0.00	0.03	0.16	0.29
SnO	0.00	0.00	0.00	0.00	0.00	0.10	0.21	0.41
ZrO ₂	0.08	0.15	0.68	10.22	0.24	7.97	0.00	26.75
Total	27	84	39	199	156	141	205.62	254

Table 5. Weight Percent Oxides of Metal Ions in Tank 18.3.

month Oxides	January	February	March	April	June	July	August	October
B ₂ O ₃	18.02	17.05	13.88	3.77	19.19	10.86	2.19	0.76
SiO ₂	55.86	49.88	35.04	6.48	56.39	26.77	3.78	0.97
Na ₂ O	12.74	18.92	27.45	4.48	12.92	38.71	1.49	1.06
P ₂ O ₅	0.00	0.00	0.00	0.00	0.00	0.00	0.4	0.22
CaO	1.11	3.53	7.29	0.85	1.27	0.32	8.09	0.00
Al ₂ O ₃	8.28	7.87	5.76	1.19	9.04	6.19	79.17	0.73
MnO ₂	0.00	0.00	0.00	0.65	0.00	0.11	0.02	0.98
Fe ₂ O ₃	2.56	1.01	6.31	57.28	0.44	9.68	4.25	52.64
Cr ₂ O ₃	0.35	0.10	1.49	16.44	0.05	2.53	0.03	14.41
ZrO ₂	0.29	0.17	1.79	5.13	0.16	4.2	0.00	19.30
ZnO	0.00	0.00	0.00	0.00	0.02	0.02	0.24	0.00
BaO	0.44	0.46	0.44	0.00	0.48	0.00	0.00	0.00
CuO	0.06	0.00	0.00	0.00	0.00	0.00	0.00	0.20
SnO	0.00	0.00	0.00	0.00	0.00	0.05	0.11	0.34
NiO	0.28	0.99	0.53	3.68	0.03	0.6	0.13	8.12
MoO ₂	0.00	0.00	0.00	0.00	0.00	0.02	0.08	0.2
PuO ₂	0.02	0.01	0.01	0.06	0.01	0.02	0.01	0.07
Total	100	100	100	100	100	100	100	100

Table 6. Minimum Agitator Speed (N_{js}) Predicted from Zweitering Correlation

Weight % ratio (Si:Fe:Ni)	Avg. density of <u>slurry</u> (kg/m^3)	Avg. particle size (m)	N_{js} , model (rps)	N_{js} , model (rpm)	N_{js} , Plant (rpm)
1 : 0.0 : 0.0	2650	243×10^{-3}	4.71	282.8	74.1
0.98 : 0 : 0.02	2777	238×10^{-3}	4.87	292.4	76.6
0.95 : 0 : 0.05	2967	231×10^{-3}	5.07	304.3	76.7
0.9 : 0 : 0.1	3283	220×10^{-3}	5.37	322.1	79.7
0.75 : 0 : 0.25	4233	186×10^{-3}	6.07	364.1	84.4
0.5 : 0 : 0.5	5815	128×10^{-3}	6.74	404.4	106
0.6 : 0.3 : 0.1	3777	190×10^{-3}	5.69	341.6	89.5

Table 7. Degree of Suspension of Nickel Particles in Model Vessel.

Agitation	283 rpm			370 rpm			528 rpm			686 rpm		
(% vol.)	30 %	50 %	70 %	30 %	50 %	70 %	30 %	50 %	70 %	30 %	50 %	70 %
sample #												
1	114	107	95	111	118	95	121	110	90	109	111	107
2	118	103	108	120	110	129	121	110	100	117	138	90
3	117	114	93	112	104	108	117	102	107	132	107	99
4	89	102	111	122	102	94	129	105	88	111	113	103
5	107	95	88	102	106	85	88	107	85	89	98	94
Average	109	104	99	113	108	102	115	107	94	112	113	99
Std. Dev.	11.9	7.1	10	8.0	6.0	17.1	15.8	3.4	9.2	15.7	14.9	6.9
LCL	94	96	87	104	100	81	96	103	83	92	95	90
UCL	124	96	111	123	116	123	135	111	105	131	132	107

Simulated solids: 0.1% Ni, 0.3% Fe, and 0.6% sand by weight

LCL = Lower @ 95% confidence limit

UCL = Upper @ 95% confidence limit

Table 8. Degree of Suspension of Nickel Particles at Vertical Sampling Heights.

<u>Sample #</u>	<u>1" Off-bottom</u>	<u>3" Off - bottom</u>	<u>6" Off - bottom</u>	<u>9" Off- bottom</u>
1	98.6	90.7	95.9	98
2	93.4	94.37	99.5	96.5
3	94.8	96.6	91.2	94.7
4	94.9	98.4	97.5	95.9
5	93.4	93.1	96.6	115.4
Average	95.1	94.6	96.1	100.1
Std. deviation	2.1	3.0	3.1	8.7
LCL	92.5	90.9	92.3	89.4
UCL	97.7	98.4	100	111

LCL = lower 95% confidence limit

UCL = Upper 95% confidence limit

Fill capacity = 70% (v/v)

Simulated solids slurry:

nickel=0.1% (w)

iron=0.3% (w)

sand=0.6% (w).

total= 1.0% (w)

Figure 1. The degree of suspension of nickel in agitated small vessel.

Figure 2. The degree of suspension of solid particles as a function of agitation speed..

Figure 3 .The degree of suspension of solid particles as a function of tank fill capacity.

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C. E. Pickett, 221-H
C. R. Wolfe, 773-A
D. L. Bleile, 704-30F
E. A. Kyser, 773-A
F. R. Graham, 773-A
J. R. Knight, 773-A
J. S. Evans, 703-F
T. A. Reily, 707-F
T. C. Robinson, 221-F
S. J. Brown, 221-14F
D. A. Burge, 773-A
STI, 703-43A.