WASTE SLURRY PARTICLE PROPERTIES
FOR USE IN SLURRY FLOW MODELING [a]

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ABSTRACT

Hanford's tank farm piping system must be substantially modified to deliver high-level wastes from the underground storage tanks to the Waste Treatment Plant now under construction. Improved knowledge of the physical properties of the solids was required to support the design of the modified system.

To provide this additional knowledge, particle size distributions for composite samples from seven high-level waste feed tanks were measured using two different laser light-scattering particle size analyzers. These measurements were made under a variety of instrumental conditions, including various flow rates through the sample loop, various stirring rates in the sample reservoir, and before and after subjecting the particles to ultrasonic energy. A mean value over all the tanks of 4.2 μm was obtained for the volume-based median particle size. Additional particle size information was obtained from sieving tests, settling tests and microscopic observations.

BACKGROUND

The Department of Energy's River Protection Project is building a Waste Treatment Plant (WTP) to convert the nuclear waste stored in the Hanford Site's underground storage tank system into final waste forms. One responsibility of the Tank Farm Contractor, currently CH2M HILL Hanford Group, Inc., is to prepare piping systems to transport the wastes from the storage tanks to the WTP.

Although much of the tank waste will be dissolved and delivered as liquids, a sizable fraction of the waste is relatively insoluble, and must be transported as aqueous slurries. As part of the design basis for the piping system, semi-empirical calculations have been developed (1) to estimate pipeline pressure drops (head losses) to be expected during transfers of these slurries. After reviewing the literature (2), the method of Oroskar and Turian (3) was selected to determine the minimum flow velocity required to prevent the settling of the insoluble solids (the so-called "critical velocity"), and the method of Wasp (4) was selected to predict the pressure drop that will develop as a result of the slurry flow through the pipeline.

Inputs for these models include parameters regarding the characteristics of the piping system (effective length, diameter, roughness, and slope) and characteristics of the waste slurry to be transported. Waste characteristics include density and viscosity of the liquid portion of the slurry, density and size distribution of the solid particles, fraction of solids in the slurry, and viscosity of the slurry. This paper will focus on the characterization of the Hanford tank waste physical properties important to slurry transport design.

INTRODUCTION

Particle Size Data Review

The first attempt at the modeling of the Hanford waste slurry flows (5), released in the year 2000, was hampered by a lack of information concerning the size distribution and density of the solid particulates in the waste. Since an increase in either of these quantities would result in higher pressure drops, the search for adequate particle data intensified. A review of particle size data obtained for Hanford tank wastes (6) was prepared. This report showed that measurements up to that time, mostly dated in the 1980s and 1990s, had been obtained with a number of different instruments having various measurement technologies. The measurement results varied widely from one report to another, but no study addressing the suitability of the instruments for analysis of tank waste had been performed, and no effort had been made to evaluate the differences of results obtained by various instruments.

Moreover, the most recent measurements yielded generally higher particle sizes than those previously recorded for Hanford slurry wastes. The recent measurements, for sludges from three slurry tanks, had been obtained with a modern laser light-scattering instrument, the Horiba[b] Model LA-910.

Table I compares the Horiba results with results obtained previously with other instruments. (No previous measurements were recorded for tank AW-103.)

It could be argued that large particle sizes were not recorded previously simply because the instruments used were incapable of detecting them. The Horiba instrument has an upper measuring limit of 1020 μm, which exceeds those of all other instruments that had been used previously. Therefore, the particle size data review concluded that, to be conservative, the slurry modeling should be based on the measurements of the Horiba LA-910. The following statistical statements were made, based on the assumption that

the measured sludges from the three tanks constituted a representative sampling of all
Hanford tank sludges:

Table I. Comparison of Horiba LA-910 results with those of previous measurements *

Instrument	Upper Measuring Limit (µm)	Tank AW-103	Tank AZ-101	Tank C-104
Horiba Model LA-910	1020	84 ± 27 (15)	120 ± 3 (2)	116 ± 47 (4)
Brinkmann[c] Model 2010	150		10 ± 6 (5)	
Microtrac[d] Model UPA	6.5			1.8 ± 0.8 (4)
Microtrac Model X-100	1000			3.5 ± 0.3 (8)

* Results for all instruments except the Brinkmann 2010 are given as the means of the medians in µm for the measurements obtained, followed by ± the standard deviation in µm, followed by the number of measurements in parentheses. Results reported for the Brinkmann 2010 did not include medians; the mean of the means is reported for those measurements.

- The median particle diameter (particle size at the 50th percentile of the distribution) in Hanford tank sludges is estimated to be 110 µm.

- It may be stated with 95% confidence that the median particle size in Hanford tank sludges is less than 140 µm.

- It may be stated with 95% confidence that, in at least 95% of the sludge tanks, the median particle size is less than 274 µm.

The first statistic provides a best estimate; the second provides an upper bound for all the wastes taken as a whole; and the third provides a tank-by-tank upper bound. All the particle size statistics in this paper are for <u>volume</u>-based distributions, i.e., the 50th percentile is the point in the distribution that corresponds to half of the <u>volume</u> of the solids.[e]

Agglomeration

During the review of the particle size measurements, it became evident that measurement of particle properties had to account for the phenomenon of agglomeration. Studies in the mid-1990s (7) had pointed out that the particles in Hanford waste slurries, which may be on the order of micrometers in size, consisted of accretions, or agglomerates, of many very tiny fundamental particles that may be only a few nanometers in diameter. Since the fundamental particles are held into agglomerates only by very weak van der Waals forces, they are likely to be very fragile. Shear forces due to convective mixing, pumping, or turbulent flow would tear these particles apart. Conversely, when these forces ceased, the particle could coalesce once again. In fact, the concept of agglomerate particles in a flowing slurry has to be considered as a dynamic process, with particles breaking and re-forming continually. Figure 1 illustrates the response of large

agglomerate particles to mechanical force (in this case the force of the glass cover applied to the microscope slide).

Clearly, to be useful in the Hanford application, particle size measurements must account for the phenomena of agglomeration and attrition of agglomerates. Likewise, measurement of particle density must also accommodate agglomeration. Agglomerate particles are not perfectly packed solid objects, but contain considerable amount of liquid. Accordingly, the density of an agglomerate is a weighted average of the densities of the fundamental solid particles and of the interstitial liquid.
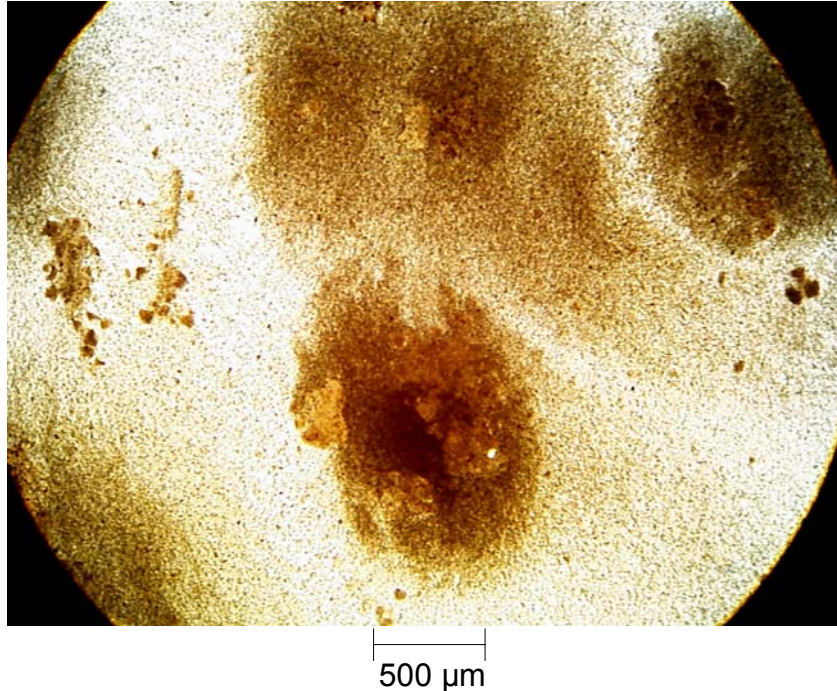

500 µm

Figure 1. Agglomerated waste from Tank SY-102. (8)

Simple pycnometric methods are often used to measure the density of particulate solids, but these techniques will not provide true particle densities of agglomerates, because the particulates must be separated from the liquid. In the case of agglomerates, removal of the liquid will change the structure, and therefore the volume, of the particles. In fact, agglomeration theory tells us that the density of agglomerates depends on agglomerate size (9). Because, as indicated above, agglomerate size depends on the hydrodynamic environment of the particle, measurement of agglomerate particles should be measured under relevant conditions, e.g., under conditions existing during pipeline flow.

Indeed, it would be more convenient to adopt slurry flow models that do not require particle size and particle density as inputs, e.g., a slurry flow model devised explicitly for agglomerates. Unfortunately, research in this area is not ready for practical application (10, 11).

DESCRIPTION OF TESTS

Slurry flow modeling based on the unexpectedly high measurements of the Horiba LA-910 led to pipe pressure requirements that greatly exceed the design bases for existing equipment and pipelines being constructed. This issue provided the incentive for further studies of the physical properties of waste particles. The primary goals for these studies, conducted in the year 2001, were to provide more certainty and confidence in the particle size measurement methods and to obtain particle size data for more tank wastes. Particle density values and information about the durability of the agglomerates were also to be obtained. The tests to attain these goals included particle size measurements, sieving tests on waste samples, settling tests, and microscopic observations. All these tests were performed on archived tank waste samples; in addition, certified particle size standards were used in the particle size testing and microscopic examinations.

Particle Size Testing

The objective of the particle size testing was to validate the measurements of the Horiba LA-910 analyzer. Measurement results of the Horiba instrument were compared with results obtained on identical samples with another analyzer, the Microtrac X-100 particle size analyzer. Particle size testing was conducted on particle size standards as well as on actual tank waste samples.

Particle size standards were selected to challenge the measuring capabilities of the instruments in the same manner as waste samples would. Therefore, the selected standards:

- Contained a wide range of particle sizes to emulate the expected range to be found in the wastes and to challenge the mathematical routines that convert the light-scattering pattern to particle distribution[f] and

- Were of relatively dense materials to challenge the ability of the analyzers to suspend the particles and present them to the interrogating laser light beam.

Particle size standards BCR 67 and BCR 130, quartz particle specimens certified by the Community Bureau of Reference, Commission of the European Communities, were found to satisfy these requirements.

Parametric tests were conducted with the two instruments to establish operating conditions (flow rate, stir rate, and sonication[g]) that would reliably suspend and disperse the particulates.

Composite slurry waste samples were prepared from laboratory archives of actual tank waste to represent each of seven sludge feed tanks. Since most of the samples had dried significantly during several years of storage in the laboratory hot cell, water was gently mixed in to yield a cake-batter consistency. Three of the tank composites were split into fractions and submitted for analysis on the two instruments. Composites for the other four tanks were analyzed on the Horiba instrument only.

Both of the particle size analyzers are laser light-scattering instruments. Measurements were made under a variety of instrumental conditions, including various flow rates through the sample loop, various stirring rates in the sample reservoir, and before and after subjecting the particles to ultrasonic energy (sonication). Both analyzers require that the particles be suspended as a very dilute slurry. A previously filtered solution of 1 molar (M) NaOH and 1M NaNO$_3$ was employed as the diluent for this purpose, which is similar to the tank waste liquid.

Sieving Studies

Additional verification of the particle size measurement methods was obtained from sieving studies. Weighed amounts of the waste sludge composites for four tanks were slurried with filtered 1M NaOH / 1M NaNO$_3$ solution. Each slurry was then poured through a vibrating stack of standard sieves, having mesh sizes of 500, 212, 106, and 53 μm. The sieve bodies were made of clear plastic, for chemical durability and to facilitate observation of the solids as they progressed through the sieves. Additional liquid was added until the progress of solids through the sieves ceased. After sieving, the sludge remaining on the sieves was allowed to drain and then weighed. The weight of the material that passed through all the sieves was obtained by difference.

Due to the large amount of sludge (20 to 40 g) used, these tests were performed in a hot cell. Because of the limited time and funding available, these tests were performed only once for each of the four tanks.

Settling Tests

As discussed above, pycnometry is not a suitable methodology for determining the density of agglomerates. As an alternative, an attempt was made to determine densities of agglomerate particles ρ_p indirectly *via* the relation for the rate of free settling u$_t$ of particles of known size D_p through a liquid of known viscosity and density ρ_L:

$$\rho_p = \rho_L \left(1 + \frac{3u_t^2 C_d}{4g D_p} \right)$$

(Eq. 1)

where g is the acceleration due to gravity, and C_d is the drag coefficient for an idealized spherical particle which is a function of the Reynolds Number (12). The first settling test concept was to microscopically observe individual particles as they descended under the influence of gravity through a quiescent volume of liquid. This concept was not implemented due to the extremely tight schedule of the project.

Instead, an alternative scheme, adapted from the traditional sedimentation-fractionation method of determining particle size (13) was used. In this method, a dilute, uniform suspension of the particles was prepared using the same filtered 1M NaOH / 1M NaNO$_3$ solution used for the other tests. The particles were allowed to settle for a pre-determined time period, and then the top 90% (approximate measure) of the material (liquid and still settling slurry) was quickly removed by vacuum and discarded. Fresh diluent was then added to the solids remaining in the bottom, settling was again allowed to proceed for the

same time period as before, and the top 90% of the liquid was removed. This procedure was performed a total of eight times for each sample.

According to the theory for this methodology, nearly all the particles remaining after the series of settlings should settle faster than a rate given by the maximum settling distance divided by the time allowed. For these tests, the settling rate criterion was 0.0865 cm/s. This limiting settling rate was back-calculated from rough scoping calculations using the slurry flow models discussed above such that it would be possible to transport slurries of more slowly settling particles virtually anywhere in the feed delivery system without exceeding existing or planned design pressure limits.

These tests were performed twice on samples from each of four slurry tanks.

Microscopic Observations

Photomicrographs were obtained for the BCR 67 and BCR 130 particle size standards and for composite samples for four slurry waste tanks. Automated image analysis was performed on the photomicrographs to obtain particle size distributions.

RESULTS AND DISCUSSION

The results of the laboratory testing were reported in detail in Bechtold et al. (14) and are summarized and discussed below.

Particle Size Testing

The results of the particle size distribution measurements for the BCR 67 and BCR 130 certified standards are given in Figure 2. This figure shows the average distributions obtained for each analyzer for each of the certified standards. In the case of the Horiba instrument, the average for each of the standard materials was obtained from two specimens, making four measurements on each. In the case of the Microtrac analyzer, the average for the BCR 67 standard was obtained from a total of 25 measurements made on three specimens. The average for Microtrac measurements on the BCR 130 standard was taken on a subset of 12 measurements from a total of 28 measurements obtained during parametric testing. The other 16 measurements in this set were found to be invalid, because the circulation rate of the sample through the instrument and/or lack of sonication were not sufficient to keep these relatively large and dense particles in suspension.

Figure 3 shows the particle size distributions obtained for a sludge from Tank AY-101 both before and after treatment with ultrasonic energy. Before sonication there is a peak representing particles >100 μm. After sonication this peak disappears completely. This behavior, typical of all the tank sludges subjected to particle size analysis, demonstrates the fragility of the agglomerates. In fact, simply circulating the sample through the particle size analyzer for several minutes was sufficient to decompose the larger agglomerates, even without sonication. The circulation in the Microtrac analyzer was so vigorous that the larger agglomerates were usually not observed.
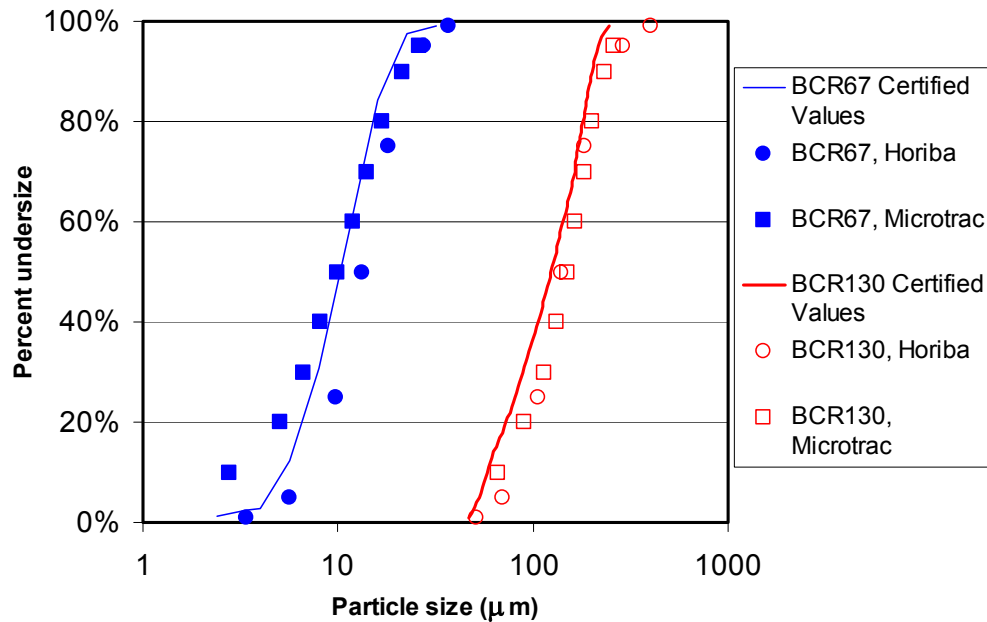
Figure 2. Results of particle size distribution measurements on certified standards.

A summary of the results of the particle size distribution measurements for the waste slurry samples is given in Figure 4. This plot shows the distributions obtained on the two instruments before and after sonication. Each curve is averaged over all the tank samples measured. For the Horiba instrument, samples from seven tanks were measured; for the Microtrac, only three tank samples were measured. The number of measurements averaged to obtain each curve ranged from a minimum of 18 to a maximum of 87. Of all the measurements obtained in this part of the test, only two from each instrument were rejected as outliers.

Sieving Studies

Table II shows the drained weight fractions of the sieved sludge samples. The table shows that 70% to 90% of the material passed through all the sieves. This result is consistent with the particle size distributions just discussed.

The next largest fractions are the materials that failed to pass through any of the sieves. These fractions are not consistent with the measured particle size distributions. There are several explanations for this occurrence, but the most likely are:

- The material contained large dry crumbs that had formed during sample storage and that had not sufficiently remoistened and softened before the sieving test.

- The material was not adequately suspended in the slurry before sieving and/or it was not vibrated and washed on the sieve enough to break up "clods."
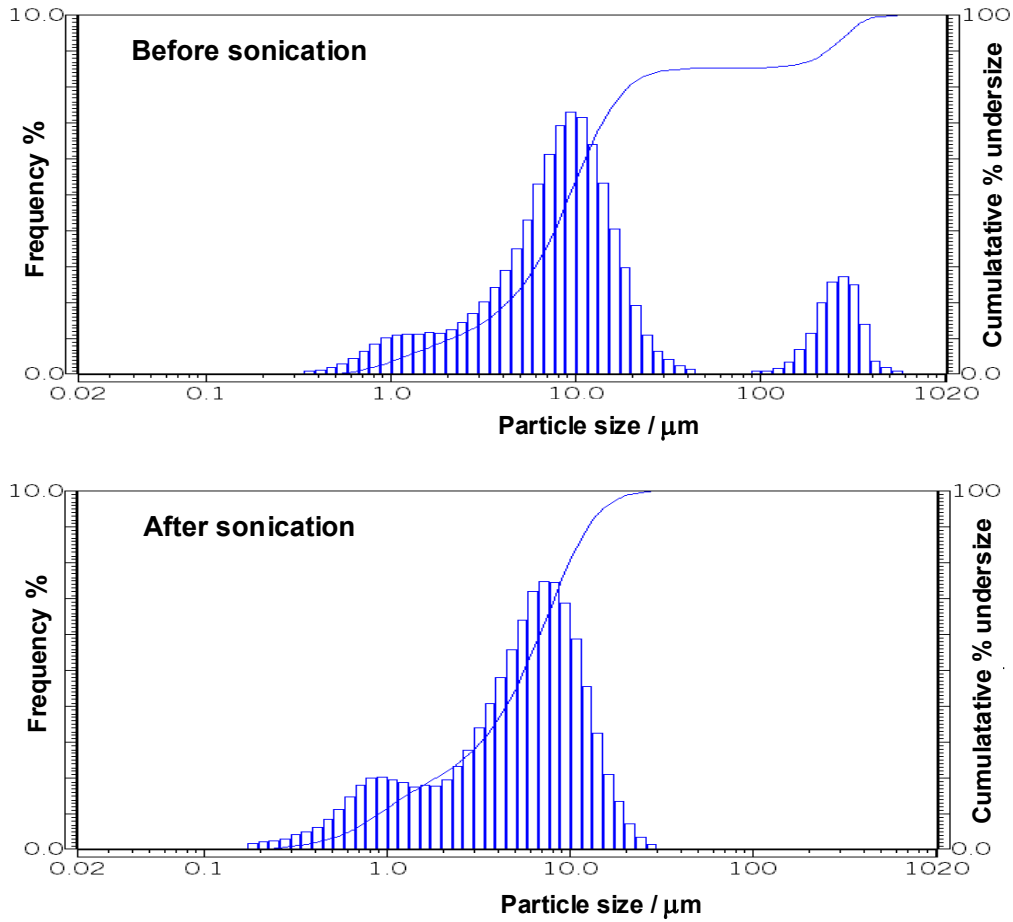
Figure 3. Particle size distributions obtained for Tank AY-101 sludge before and after sonication.
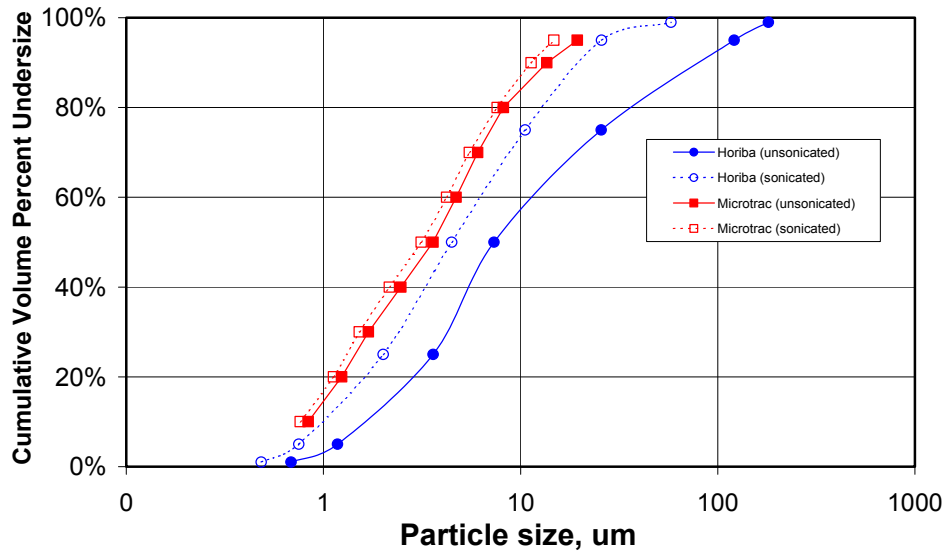

Figure 4. Particle size distributions averaged over all tanks measured with each instrument, both before and after sonication.

Table II. Drained weight percents of sieved sludge fractions

Tank	Size Range (µm) of Screen Fraction				
	<53	53-106	106 -212	212-500	>500
	Drained Weight Percent of Sieved Sludge Fraction				
AW-103	70.9	4.3	4.8	3.4	16.7
AY-101	85.4	2.3	1.5	2.1	8.7
C-104	87.3	6.6	1.1	1.8	3.1
SY-102	83.3	1.5	2.5	3.8	8.8

Particle size distributions were obtained in replicate (usually four measurements) for the sieve fractions that exceeded five weight percent. Table III compares the distributions obtained for the <53-µm fractions and the >500-µm fractions. The 50th, 95th, and 99th "percentiles" are the percentages of the particulate volume that is present in particles having diameters less than the indicated values.

Table III. Summary of particle size distribution results for the sieved fractions. Mean values are given (in µm) for the 50th, 95th, and 99th percentiles of the distributions. All measurements were obtained with the Horiba analyzer, without sonication

Tank	<53 µm fractions			>500 µm fractions		
	50th	95th	99th	50th	95th	99th
AW-103	153	293	391	63	235	337
AY-101	153	280	338	92	284	380
SY-102	126	392	476	181	551	714
C-104	319	454	549	Not measured		

This table shows that the particle size distributions of the <53 µm and the >500 µm fractions are quite similar. Most of the material in the <53 µm fractions was greater than 53 µm. This is evidence that the material re-agglomerated after sieving. The particle size distributions of the <53 µm fractions were also considerably higher than found for the unsieved tank composite samples. A possible explanation for this surprising result is that the lack of larger particles in the <53 µm fractions yield greater specific surface area of the primary particles. The greater area would provide more van der Waals interaction, which in turn would lead to stronger and therefore larger agglomerates.

The particle sizes found for the >500 µm fractions are also surprising, since most of the particles are considerably <500 µm. It is believed that the dry crumbs and unbroken clods mentioned above softened during storage under liquid after sieving and were readily broken up upon suspension prior to particle size measurement. Visual observations of the sieve fractions before and after storage support this explanation.

Settling Tests

The settled volumes of sludge remaining after the settling tests (the fast-settling fraction of the sludge) were compared with the initial settled volume of sludge going into each test. This provided an approximate value for the percentage of sludge that settled faster than the settling rate criterion (0.0865 cm/s) established for the tests. These volume fractions of remaining sludge are listed in Table IV.

Table IV. Volume percents of sludge remaining after eight settling steps

Tank	Vol% Settled	
	Test 1	Test 2
AY-101	7	9
C-104	4	9
AW-103	4	4
SY-102	6	4

The particle size distributions for the solids remaining in the cylinders after the settling tests did not exhibit the sharp cut-off predicted by the theory. This result most likely occurred because of additional break-up of the agglomerate particles each time the solids were re-suspended.

Microscopic Observations

The microscopic tests and image analyses yielded the following qualitative observations:

- For the quartz certified particle size standards, the observed particle sizes generally corresponded to the certification values.

- For the tank sludge composite samples, except for a minor amount of granular material at the bottom of one sample vial, the largest particle seen was 49 μm. The particles observed were generally too small and/or so ill formed that the chemical species could not be identified. Whereas many particles of a few micrometers or more appeared to have some transparency, indicating that they were individual crystalline fragments, other particles appeared only as dark smudges. The latter are apparently agglomerates of very small particles (<1 μm).

- In the sieved sludge fractions, the observed particle sizes rarely approached the mesh size of the relevant sieve:

    o In the 33 microscope images obtained for the four sludge fractions that had passed through a 53-μm sieve, only one particle was found to be greater than 20 μm.

- o In the 23 images obtained for the three largest >500-μm fractions, the largest particle observed was an unbreakable pea-sized chunk. The next largest particle observed was only 100 μm in diameter. Several other particles were about 50 μm, but all the rest were considerably smaller.

- The sludge fractions remaining from the settling tests contained the coarsest material observed, with particles ranging up to 400 μm in size.

It was generally concluded that neither the sampling nor the mounting methods used in the microscopic examinations were conducive to getting reproducible and defensible particle size distributions. The samples were not thoroughly dispersed before subsampling, and loose agglomerates would have been smashed when the cover slip was pressed on.

CONCLUSIONS

Particle Size

The particle size distributions measured for the quartz particle size standards gave good evidence that the two analyzers respond reliably to suspensions having a wide distribution of dense particles. This test demonstrated that the instruments suspend particles adequately under the test conditions and that the mathematical routines for converting light scattering patterns to particle size distributions are sufficiently reliable and accurate.

In the particle size measurements of the slurry wastes summarized in Figure 4, the most notable feature is that the Horiba instrument consistently yields larger particle sizes. Sonication causes the particle sizes measured on the Horiba to decrease significantly, but, in the case of the Microtrac, the decrease due to sonication was hardly noticeable. These results, coupled with the much closer agreement on the certified standards leads us to believe that the turbulence in the Microtrac is greater, causing the particles to break up to a greater extent.

These measurement results, coupled with theoretical considerations obtained from the scientific literature, gave good evidence that the solids contain sub-micrometer-sized particles that readily form accretions, i.e., agglomerate particles, ranging in size from a few micrometers to nearly a millimeter. The sludges also contain individual particles that measure more than a micrometer. These particles are probably also incorporated into the agglomerates, held by the van der Waals forces associated with the large surface areas of the much smaller particles. Moderate hydrodynamic shear forces readily disrupt the largest of these agglomerate particles, resulting in mixtures where most of the solids volume is present in particles of just a few micrometers.

Because methods are not available for scaling the laboratory data to the hydrodynamic conditions that are expected to exist during the actual process transfers of waste, laboratory data obtained under flowing conditions with minimal turbulence/disturbance were selected to be used for the slurry flow calculations. Particles present during

conditions of minimal turbulence/disturbance represent the largest particles that will be present in a moving stream and, therefore, will provide conservative (upper bound) estimates of pressure drop during slurry flow.

This conservative upper bound is represented by the right-most curve in Figure 4. This curve is shown more precisely by the row labeled "Mean" in Table V. Each value in this row gives the best estimate (i.e., the mean) over the seven tanks measured, each for a particular percentile. At the 50th percentile, for example, the mean value is calculated to be 7.3 μm. This indicates that 50% of the solids volume exists in particles with diameters less than 7.3 μm. The "95 UL" row gives 95%-confidence upper limits for the mean values.

The mean and the 95 UL values apply to the Hanford high-level wastes taken as a whole. However, since the wastes in the various tanks must be transferred individually, it is useful to provide statistics for individual tanks. The "tolerance limit" statistics given in the row labeled "95/95 TL" in Table V address this issue. The tolerance limits are upper 95% confidence limits for each percentile that are expected to be met in at least 95% of the Hanford high-level waste tanks. The tolerance limit values apply even to those tanks that have not yet been measured.[h]

Table V. Particle size results for the minimally agitated condition, averaged over the seven tank samples measured

	Percentile						
	1st	5th	25th	50th	75th	95th	99th
	Particle Size (μm)						
Mean	0.7	1.2	3.6	7.3	25.6	121.1	180.7
95 UL	1.0	1.6	5.0	10.4	47.3	184.4	276.5
95/95 TL	2.0	3.2	9.9	21.5	125.1	411.6	619.9

Particle Density

An attempt was made to determine the densities of the particles by means of settling rate studies. In these studies, the plan was to calculate the density from measured values of particle size and particle settling rate. However, this attempt was generally unsuccessful because of the extreme fragility of the agglomerate particles. Stirring to establish a uniform suspension of particles caused the particles to be broken up uncontrollably.

Because it has not been possible to provide laboratory measurements for the particle densities, the slurry flow calculations have proceeded with estimated particle densities. These estimated particle densities are weighted averages of the densities of the component minerals of the solids in the waste. This is a conservative over-estimate, since the mineral densities do not account for the interstitial liquid in the agglomerated particles.

Implication for Waste Delivery System Design

The work discussed in this paper has shown that the particles in Hanford tank waste sludges are soft agglomerates that attain size distributions dependent on the hydrodynamic environment and history. Recent particle size distributions obtained in flowing streams are generally consistent with historical particle size measurements of Hanford wastes. Even though a conservative upper limit has had to be invoked for particle density, slurry flow calculations based on the particle properties measured for tank composite samples have demonstrated that slurry wastes can be delivered to the Waste Treatment Plant at planned solids concentrations without exceeding the design pressure limits of the delivery system (1).

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FOOTNOTES

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[b] Horiba is a trademark of Horiba, Ltd., Kyoto, Japan.

[c] Brinkmann is a trademark of Brinkmann Instruments, Inc., Westbury, New York.

[d] Microtrac is a trademark of Leeds & Northrup Company, North Wales, Pennsylvania.

[e] Other workers may calculate particle size distributions in different ways, for instance, in terms of the numbers or surface area of particles, depending on the intended application of the data. For mass transport concerns, however, volume-based distributions are appropriate. If all the particles have the same density, volume-based and mass-based distributions are identical.

[f] In routine use, the analyzers are tested only with monodisperse standards, i.e., standards containing particles all of essentially the same size.

[g] The term "sonication" in this paper refers to the application of mild ultrasonic energy. Typically, the ultrasonic treatment is conducted with the 40 W, 39 kHz devices built into the particle size analyzers. However, a 40 W jewelry-cleaning bath was used in some cases.

[h] The latter statement assumes that particle size values at each percentile are normally distributed among the tanks, but does not require that the particle size distributions themselves obey the normal probability function.