



PNNL-19094

Prepared for the U.S. Department of Energy
under Contract DE-AC05-76RL01830

Shear Strength Correlations for Kaolin/Water Slurries: A Comparison of Recent Measurements with Historical Data

CA Burns
PA Gauglitz
RL Russell

January 2010



Pacific Northwest
NATIONAL LABORATORY

*Proudly Operated by **Battelle** Since 1965*

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency thereof, nor Battelle Memorial Institute, nor any of their employees, makes **any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights.** Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof, or Battelle Memorial Institute. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

PACIFIC NORTHWEST NATIONAL LABORATORY
operated by
BATTELLE
for the
UNITED STATES DEPARTMENT OF ENERGY
under Contract DE-ACO5-76RL01830

Printed in the United States of America

**Available to DOE and DOE contractors from the
Office of Scientific and Technical Information,
P.O. Box 62, Oak Ridge, TN 37831-0062;
ph: (865) 576-8401
fax: (865) 576 5728
email: reports@adonis.osti.gov**

**Available to the public from the National Technical Information Service,
U.S. Department of Commerce, 5285 Port Royal Rd., Springfield, VA 22161
ph: (800) 553-6847
fax: (703) 605-6900
email: orders@nits.fedworld.gov
online ordering: <http://www.ntis.gov/ordering.htm>**

Shear Strength Correlations for Kaolin/Water Slurries: A Comparison of Recent Measurements with Historical Data

CA Burns
PA Gauglitz
RL Russell

January 2010

Prepared for
the U.S. Department of Energy
under Contract DE-AC05-76RL01830

Pacific Northwest National Laboratory
Richland, Washington 99352

Executive Summary

Recent experiments were conducted at Fauske and Associates, LLC (FAI) with kaolin/water mixtures to determine the stability of vessel-spanning bubbles. This report documents shear strength measurements of kaolin clay samples that were prepared at FAI for these tests. These shear strength measurements were conducted at Pacific Northwest National Laboratory (PNNL) for CH2M Hill Plateau Remediation company and were performed in collaboration with FAI. Four kaolin/water mixtures were obtained by PNNL from FAI. The shear strengths of these mixtures were measured, and a correlation was developed relating shear strength and wt% clay. This correlation is needed to estimate the shear strengths of specific kaolin/water mixtures used in the vessel-spanning bubble tests. The correlation for the FAI clay samples is compared with correlations of previously reported data. The FAI clay samples have similar shear strengths to previous data, but the results and correlation are also sufficiently different that the new correlation is needed to estimate the shear strength of kaolin/water mixtures used at FAI in the vessel-spanning bubble tests.

Acronyms

APEL	Applied Process and Engineering Laboratory
LHS	left hand side
DI	de-ionzied (water)
FAI	Fauske and Associates, LLC
FY	fiscal year
NIST	National Institute of Standards and Technology
PNNL	Pacific Northwest National Laboratory
QA	quality assurance
RHS	right hand side

Contents

Executive Summary	iii
Acronyms.....	v
1.0 Introduction.....	1.1
2.0 Background.....	2.1
3.0 Instrumentation	3.1
4.0 Test Approach.....	4.1
4.1 Instrument Performance Check	4.1
4.2 Simulant Description	4.3
4.3 Shear Strength Testing.....	4.3
5.0 Test Results.....	5.1
5.1 FAI Clay Shear Strength Results.....	5.1
5.2 Previous Data.....	5.2
6.0 Discussion.....	6.1
7.0 Conclusions.....	7.1
8.0 References.....	8.1
Appendix A: Shear Strength Plots	A.1

Figures

2.1. Typical Shear Strength Experimental Setup	2.2
2.2. Example of a Shear Strength Torque Versus Time Curve.....	2.3
5.1. FAI Clay Average Measurements and Correlation Derived from the Data.....	5.2
5.2. Powell et al. (1995a) Clay Shear Strength Data with Correlation Derived from the Data	5.3
5.3. Powell et al. (1995b) Clay Shear Strength Data with Correlation Derived from the Data	5.4
5.4. Gauglitz et al. (2001) Clay Shear Strength Data with Correlation Derived from the Data	5.5
5.5. Rassat et al. (2003) Clay Shear Strength Data with Correlation Derived from the Data.....	5.7
6.1. Previous Shear Strength Correlations Compared	6.1
6.2. Shear Strength Correlations Compared with Current Data.....	6.2

Tables

2.1. Vane Immersion Depth and Container Geometry Constraints for Shear Strength Tests Using the Vane Technique.....	2.3
3.1. Summary of Haake RV20 System with M5 Measuring Head	3.1
3.2. Vane and Cup and Rotor Measuring System Dimensions.....	3.1
4.1. Properties of Brookfield Fluid 50	4.2
4.2. Performance Check of RV20-M5 and Temperature Control Instruments Using Brookfield Fluid 50 Viscosity Standard.....	4.3
5.1. FAI Kaolin Clay Shear Strength Measurements.....	5.1
5.2. Powell et al. (1995a) Clay Shear Strength Measurements.....	5.2
5.3. Powell et al. (1995b) Clay Shear Strength Measurements.....	5.3
5.4. Gauglitz et al. (2001) Clay Shear Strength Measurements.....	5.4
5.5. Rassat et al. (2003) Clay Shear Strength Measurements	5.6

1.0 Introduction

This report documents shear strength measurements of kaolin clay samples that were used at Fauske and Associates, LLC (FAI) in testing the stability of vessel-spanning bubbles (Epstein and Gauglitz, 2010). This work was conducted at Pacific Northwest National Laboratory (PNNL) for CH2M Hill Plateau Remediation company and was performed in collaboration with FAI. Four samples of kaolin/water mixtures were obtained by PNNL from FAI, and the shear strengths of these samples were measured and are reported here. To estimate the shear strengths of kaolin/water mixtures over a range of wt% solids for specific vessel-spanning bubble experiments, the measured shear strengths were correlated with an exponential equation relating shear strength and wt% kaolin. The correlation for the FAI clay samples is compared with correlations of previously reported kaolin/water mixture shear strength data.

The test setup, rheology background, and instrumentation are presented in Sections 2 and 3. The test approach and simulants are discussed in Section 4. The test measurements and data reduction are provided in Section 5 along with correlations of existing kaolin shear strength data. Section 6 provides an assessment of the FAI samples and compares them with previous data. Conclusions are provided in Section 7.

2.0 Background

Rheology is the science of material flow and deformation. For fluid systems, including pure liquids, mixtures of liquids, and suspensions of solids in liquids, the rheological properties of that system describe how it responds to an applied force or stress. When applied to solids, stress induces a strain or finite deformation in the material. When applied to pure liquids, stress causes a continuous deformation of the substance or, in simpler terms, fluid flow. Suspensions of solids in liquids or liquid mixtures with internal structure can show a combination of both solid- and liquid-like behavior. In addition, the response of materials to force and deformation may not be constant. Changes in the internal structure of materials that occur as a result of mechanical and chemical processes, such as breakage, precipitation of solids, and gelation, may alter the macroscopic flow and deformation properties. For the current study, the rheology of kaolin/water slurries is considered. A single region of slurry flow behavior is considered: incipient motion in a paste composed of kaolin solids in water.

For a kaolin slurry, a finite stress must be applied before the settled solids will begin to flow. The stress required to transition the settled solids from elastic deformation to viscous flow is referred to as the shear strength, and its origin can be attributed to static and kinetic friction between individual particles and/or aggregates, the strength of the matrix supporting the coarse fraction (i.e., the interstitial fluid), and settled solids cohesion arising from interparticle adhesive forces such as van der Waals forces. The resistance of settled solids to motion can be quantified through shear strength testing.

The vane method (Nguyen and Boger, 1985) was used to measure the shear strength of kaolin slurry. For the vane method, the stress required to begin motion is determined by slowly rotating a vane immersed in the test sample while continuously monitoring the resisting torque as a function of time. A material's static shear strength is then associated with the maximum torque measured during the transition from initial to steady-state vane rotation. A typical experimental setup for measuring shear strength with a vane is shown in Figure 2.1. For the current tests, the kaolin slurry did not settle, so a supernatant layer (water) in Figure 2.1 was not present. A sludge/slurry sample is placed in a container of radius R_{cont} and a vane tool attached to a viscometer (i.e., a torque sensor) is immersed into the settled solids portion of a sludge or slurry to a depth h (relative to the top of the vane blades). The vane blades have a radius, R , and a height, H . The vane is then slowly rotated at a constant rotational speed, Ω . The torque versus time profile is recorded and the maximum torque required to initiate rotation determined. The shear strength is then calculated from this maximum torque based on the assumption of a uniform stress distribution on the known vane tool geometry.

An example torque versus time curve is shown in Figure 2.2. The maximum torque corresponds to the onset of plastic deformation. Here, the stress applied by vane rotation is finally sufficient to overcome frictional, cohesive, and other structural forces stabilizing the settled solids. The maximum torque required for incipient plastic deformation is dependent on vane geometry. To account for vane geometry effects, the shear strength is expressed in terms of a uniform and isotropic stress acting over the surface area of the cylinder of rotation swept out by the vane. This uniform stress (i.e., the shear strength of the material) is related to the maximal torque during incipient motion by the equation:

$$\tau_{ss} = \frac{M_{max}}{4\pi R^3 \left(\frac{H}{2R} + \frac{1}{3} \right)} \quad (2.1)$$

Here, τ_{ss} is the shear strength [N/m²], M_{max} is the maximum torque [N·m], and R and H are the radius and height of the cylinder of rotation swept out by the vane [m]. Because the shear band observed upon slow rotation of the vane does not extend appreciably beyond the vane paddles, R and H are taken to be the dimensions of the vane itself.

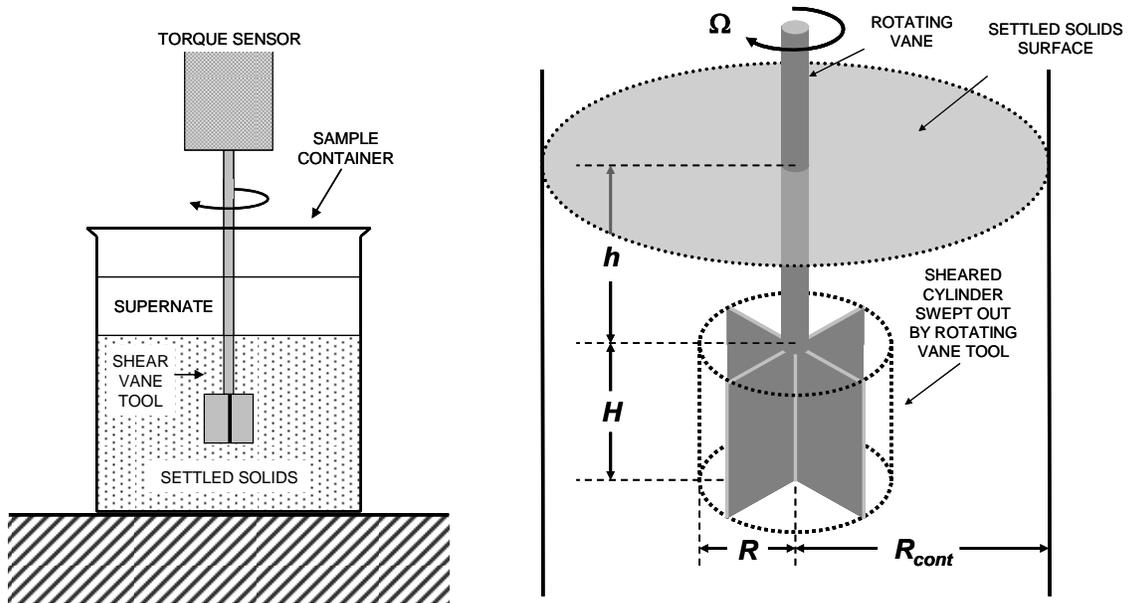


Figure 2.1. Typical Shear Strength Experimental Setup

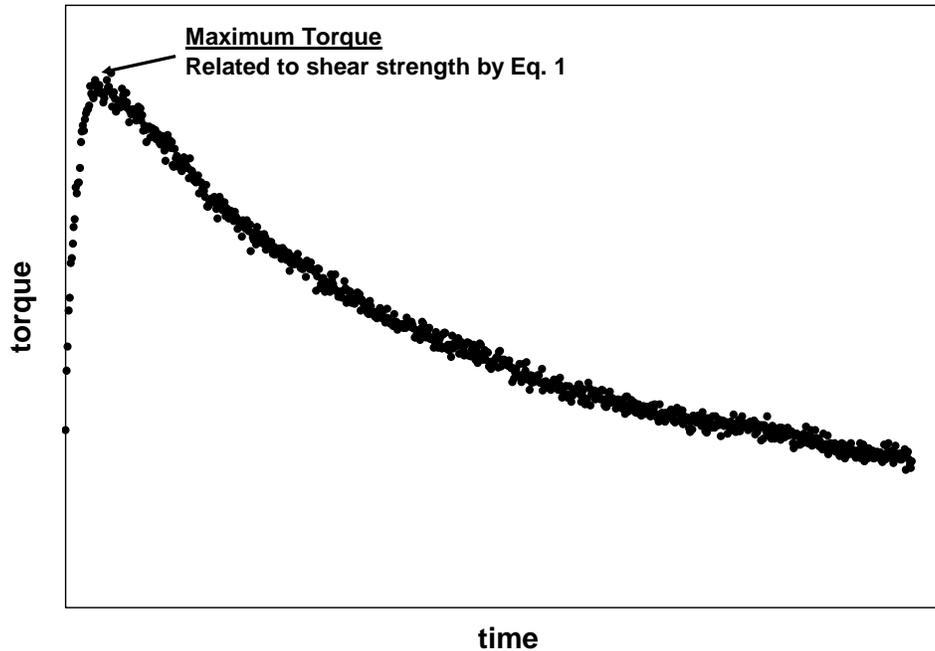


Figure 2.2. Example of a Shear Strength Torque Versus Time Curve

The proximity of the vane to the sample container inner surfaces as well as to the free surface of the settled solids can impact shear strength results. As such, certain geometric constraints must be satisfied for the test to be considered independent of container geometry. These constraints are outlined in Table 2.1 (along with example constraint dimensions for a 4×16 mm vane tool, which is the vane tool geometry employed in the current study).

Table 2.1. Vane Immersion Depth and Container Geometry Constraints for Shear Strength Tests Using the Vane Technique

Constraint	Criterion	For 4×16 mm (R×H) Vane
Vane height (H) to radius (R)	$H < 7R$	$H < 28 \text{ mm}$
Container radius (R_{cont}) to vane radius (R)	$R_{\text{cont}} > 2R$	$R_{\text{cont}} > 8 \text{ mm}$
Immersion depth (h) to vane height (H)	$h > H$	$h > 16 \text{ mm}$
Separation between bottom of vane and container floor (h_{floor})	$h_{\text{floor}} > 0.5H$	$h_{\text{floor}} > 8 \text{ mm}$

3.0 Instrumentation

The shear strength was measured with a Rotovisco® RV20 Measuring System equipped with an M5 measuring head and RC20 controller. These components were purchased from HAAKE Mess-Technik GmbH u. Co. (now the Thermo Electron Corporation, Madison, WI 53711). This system is located in the Applied Process and Engineering Laboratory (APEL), Room 112. The M5 measuring head (SN# 920099) is a “Searle” type viscometer capable of producing rotational speeds up to 500 RPM and measuring torques up to 0.049 N·m. The minimum rotational speed and torque resolution achievable by this measuring head are 0.05 RPM and 0.49 mN·m, respectively. Table 3.1 summarizes the M5 measuring system information.

Table 3.1. Summary of Haake RV20 System with M5 Measuring Head

Analyzer:	Rotovisco® RV20 Measuring System M with M5 Measuring Head.
Measurement principle:	Controlled Rate
Serial Number:	920099
Torque Sensor Range	0.49 to 49 mN·m
Rotational Rate Range	0.05 to 500 RPM

Specific measurement tools, such as cup and rotor assemblies and shear vanes, are attached to measure selected rheological properties. Shear strength measurements employed a 4-mm × 16-mm shear vane tool. The dimensions of the vane measuring system are listed in Table 3.2.

Table 3.2. Vane and Cup and Rotor Measuring System Dimensions.

Measuring System	Vane/Rotor Radius	Vane/Rotor Height	Cup Radius	Gap Width
Vane Tool	4 mm	16 mm	> 8 mm ^(a)	> 4 mm ^(a)

(a) Vane tests must satisfy the requirements outlined in Table 2.1.

A remote computer connection using the RheoWin Pro Job Manager Software, Version 2.96 (1996), was used to control the rheometer and acquire data. The RheoWin software serves as a central program for obtaining, processing, and recording to disk data from the RV20-M5 measuring system. During measurement, the software automatically converted rotor torque readings into shear stresses based on the appropriate A-factor conversion, such that

$$\tau = AM \tag{3.1}$$

For vane tools, the A-factor is defined as:

$$A = \frac{1}{4\pi R^3 \left(\frac{H}{2R} + \frac{1}{3} \right)} \tag{3.2}$$

The A-factor for the 4 mm × 16 mm vane tool sensor system is ~533,000 m⁻³. The RheoWin software also allows post-measurement processing and interpretation of data. Specifically, it can be used to determine maxima points in shear strength testing.

4.0 Test Approach

4.1 Instrument Performance Check

As required by procedure RPL-COLLOID-02, Rev. 1, *Measurement of Physical and Rheological Properties of Solutions, Slurries, and Sludges* (Daniel 2007), the performance of the Haake RV20-M5 rheometer in APEL/112 must be verified at the beginning of each series of analyses (with the period between performance checks not to exceed 30 days during use). Checks are performed using Newtonian viscosity standards certified by methods traceable to the U.S. National Institute of Standards and Technology (NIST). Checks verify that the Haake RV20-M5 rheometer can measure the standard's viscosity to within 10% for fluids of 10 cP or greater and to within 15% for fluids less than 10 cP at the temperature listed on the certificate of analysis (hereafter referred to as the list viscosity). Verification of the Haake RV20-M5 in APEL/112 involves

- 1) measuring Newtonian viscosities at 25°C and ambient temperatures with a second reference bench top rheometer (the Haake RS600 located in the APEL)
- 2) verifying that the RS600 measures the viscosity standard to within the limits defined by RPL-COLLOID-02, Rev. 1, at 25°C
- 3) measuring the viscosity of the standard at ambient temperature on the RV20-M5 rheometer system
- 4) comparing RS600 and RV20-M5 viscosities at ambient temperature to verify that they agree within acceptable limits of tolerance.

This three-point check allows verification of the RV20-M5 rheometer performance at ambient temperature. To verify the capability of the RV20-M5 rheometer to properly determine torque and stress, two conditions must be satisfied: measurements of viscosity at ambient temperature on the RV20-M5 must agree to measurements on the cold bench-top rheometer within 10%. The rheometer software (RheoWin 2.96, 1996) is also verified during this check, as it is used to calculate the Newtonian viscosity of the standard viscosity fluid tested.

For the measurements described in this report, the performance check employed a General Purpose Silicone Fluid purchased from Brookfield Engineering Laboratories, Inc. (Middleboro, Massachusetts, USA, 02346). Silicone oil viscosity standards are single phase liquids and have no suspended solids. Testing employed Brookfield Fluid 50. Table 4.1 provides a summary of this viscosity standard's properties.

Table 4.1. Properties of Brookfield Fluid 50

Fluid	50
List Viscosity	48.0 cP
Acceptable Range ^(a)	43.2 to 52.8 cP
Temperature	25°C
Lot Number	062408
Expires ^(b)	May 4, 2010

(a) As defined by RPL-COLLOID-02, Rev .1
(b) Expires 1-year after opening (standard opened May 4, 2009)

Performance checks consisted of temperature-controlled flow-curve measurements that employed the MV2P measuring cup and rotor. The measurements reported herein were covered by a single performance. Performance verifications were executed as follows:

- 1) The MV1 rotor (concentric cylinder geometry) was installed on the M5 measuring head.
- 2) Approximately 40 mL of viscosity fluid was added to the MV1 cup.
- 3) The measuring cup was installed into the measuring system by slowly raising it on a laboratory jack stand. During installation, the rotor volume displaces the viscosity standard fluid, forcing it to fill the gap between cup and rotor. While the cup was being raised, the liquid level relative to the top of the rotor was monitored through an opening in the top of the measuring system. The cup was raised until the test material was observed to spill over the top of the rotor. Before continuing, an attempt was made to remove the excess viscosity standard from the top of the rotor using a plastic transfer pipette. However, typically 1 to 3 mL of excess test liquid remains in the upper rotor recess during flow-curve measurement.^(a)
- 4) The flow curve (shear stress versus shear rate) data were measured. Rheological analysis was performed over an 11-min period, split into three intervals. Over the first 5 minutes, the shear rate was gradually increased from zero to 1000 s⁻¹. For next minute, the shear rate was held constant at 1000 s⁻¹. For the final 5 minutes, the shear rate was gradually reduced back to zero. During this time, the resisting torque and rotational rate were continuously monitored and recorded.

After the measurement, flow curve data were analyzed with the RheoWin 2.96 Pro software to determine the standard viscosity measured on both RV20-M5 and RS600 systems. The performance check is considered acceptable when the relative percent difference between measured and list viscosity was less than 10% for fluids with listed viscosities greater than or equal to 10 cP or was less than 15% for fluids with list viscosities less than 10 cP. For Brookfield Fluid 50 (see Table 4.2), the acceptable range of viscosity is 43.2 cP to 52.8 cP at 25°C. Viscosities at temperatures other than 25°C were not provided by the manufacturer.

Table 4.2 lists the results of the performance verification/check. The performance of the RV20-M5 measuring and temperature control systems was verified to be acceptable.

- (a) When the rotational rate of the rotor is sufficiently high, any excess material in the upper recess of the rotor can migrate from the top of the rotor to the gap (through inertia). This can lead to a change in the measured slope of the flow curve and flow and flow-curve hysteresis. The migration of excess material is often characterized by a slope discontinuity in the flow curve. Such discontinuities were excluded from analysis of flow curve data.

Table 4.2. Performance Check of RV20-M5 and Temperature Control Instruments Using Brookfield Fluid 50 Viscosity Standard

Period of Performance	Instrument	Temperature [°C]	Viscosity [cP]		Acceptable ^(a)
			List ^(b)	Measured	
Opening 8/3/2009–9/3/2009	RS600	25	48.0	51.43	Yes
	M5	Ambient (18.5)	n/a	56.95	Agrees with RS600 ^(b)
	RS600	Ambient (18.5)	n/a	57.89	Agrees with M5 ^(b)

(a) As per RPL-COLLOID-02 Rev. 1, the acceptable range for Brookfield Fluid 50 (calculated as $\pm 10\%$ of the list viscosity of 48.0 cP) is 43.2 to 52.8 cP at 25°C.

(b) List viscosities at temperatures other than 25°C are not provided by the manufacturer. Viscosity measurements at ambient temperature were conducted on two measurement systems (RV20-M5 and RS600); results were to agree within 10% to show acceptable performance.

4.2 Simulant Description

Three EPK kaolin samples (Edgar Minerals Inc., Edgar, FL) of different solids concentrations and one kaolin clay from Sigma Aldrich were provided by FAI for shear strength testing at PNNL. The samples were made up by FAI, labeled with solids content, and shipped to PNNL. No attempt was made to verify the solid content of the samples provided. The clay samples were prepared by FAI in de-ionized water from their laboratory and were mixed with a blade rotated by an electrical drill motor. Even though de-ionized water was used, the shear strength of clay/water mixtures can be affected by small differences in water chemistry, such as pH and ionic content. For this reason, it is important to characterize representative samples of the kaolin/water mixtures used in the vessel spanning bubble testing at FAI. Before performing shear strength measurements at PNNL, the samples from FAI were mixed for a period of 2 minutes and then allowed to age undisturbed for 1 hour before shear strength measurements were taken; the results obtained for kaolin/water mixtures provided by FAI are given in Table 5.1.

4.3 Shear Strength Testing

Shear strength testing was conducted as follows:

- 1) A 4×16 mm (radius by height) shear vane tool was installed on the measuring head.
- 2) The sample jar being tested was opened and positioned on a laboratory jack stand directly beneath the measuring head/vane.
- 3) The lab jack was slowly raised until the top of the vane blades were (typically) 1-vane height (16 mm) below the surface of the settled solids. It should be noted that for the 50-wt% solids sample, the vane was immersed 1/2-vane height (8 mm) because of insufficient sample volume.
- 4) The vane was slowly rotated at 0.3 RPM for 60 seconds. For the entire duration of rotation, the time, rotational rate, and vane torque were continuously monitored and recorded.
- 5) At the completion of testing, the vane was removed from the settled solids, rinsed clean of residual solids with de-ionized (DI) water, and dried before the next test. The sample jar was closed and set aside.

At the end of the measurement, the software parsed the shear stress versus time data and determined and reported the maximum measured shear stress (i.e., the material's shear strength). The curve of shear

stress versus time was visually inspected using the RheoWin software to verify that the appropriate stress maximum was selected. All information relevant to the measurement, including raw and calculated measurement results and sample information, were saved to disk using the RheoWin file format and a unique filename identifier. It should be noted that shear strength measurements were conducted at ambient room temperature, which was recorded using a thermocouple and temperature display. The torque versus times curves are given in Appendix A.

5.0 Test Results

5.1 FAI Clay Shear Strength Results

Table 5.1 gives the shear strength results for the FAI clay samples. The uncertainties in the average values given in Table 5.1 vary from 5% to 19% of the mean value, and the average of these uncertainties is about 10%. This is similar to the uncertainties reported by Powell et al. (1995b). Previous kaolin shear strength data have been successfully fit with an exponential equation (see Section 5.2 for examples), and this approach should be suitable for the FAI clay samples. For the EPK kaolin samples, the average value of the three independent measurements was used in developing a correlation. This exponential equation provides a good fit with an R^2 value of 0.997 and is shown in Figure 5.1 together with the data.

$$y = 0.0005\exp [0.2497 * (\text{wt\% kaolin})] \quad (5.1)$$

The results obtained for the 50 wt% solids for the Sigma Aldrich kaolin clay are significantly higher than those of the EPK clay. Although not measured, it is likely that the Sigma Aldrich kaolin had a smaller average particle size in comparison to the EPK kaolin. The vessel spanning bubble tests reported in Epstein and Gauglitz (2010) only used the EPK kaolin mixtures, and the shear strength results for the Sigma Aldrich sample are reported here to document the measurements made on the received sample.

Table 5.1. FAI Kaolin Clay Shear Strength Measurements

Wt %	Test	Aging Time [hrs]	Location	Immersion Depth [mm]	Shear Strength [Pa]
50 EPK	1	1	Center	8	139
	2	1	RHS	8	122
	3	1	LHS	8	121
	Avg				127 ± 24^(a)
54 EPK	1	1	Center	16	373
	2	1	RHS	16	383
	3	1	LHS	16	369
	Avg				375 ± 18^(a)
57 EPK	1	1	Center	16	743
	2	1	RHS	16	720
	3	1	LHS	16	706
	Avg				723 ± 47^(a)
50 (Sigma Aldrich)	1	1	Center	16	540
	2	1	RHS	16	551
	3	1	LHS	16	515
	Avg				535 ± 46^(a)

(a) Reported uncertainty is the 95% confidence limit (calculated as 4.3 times the standard error of the mean).

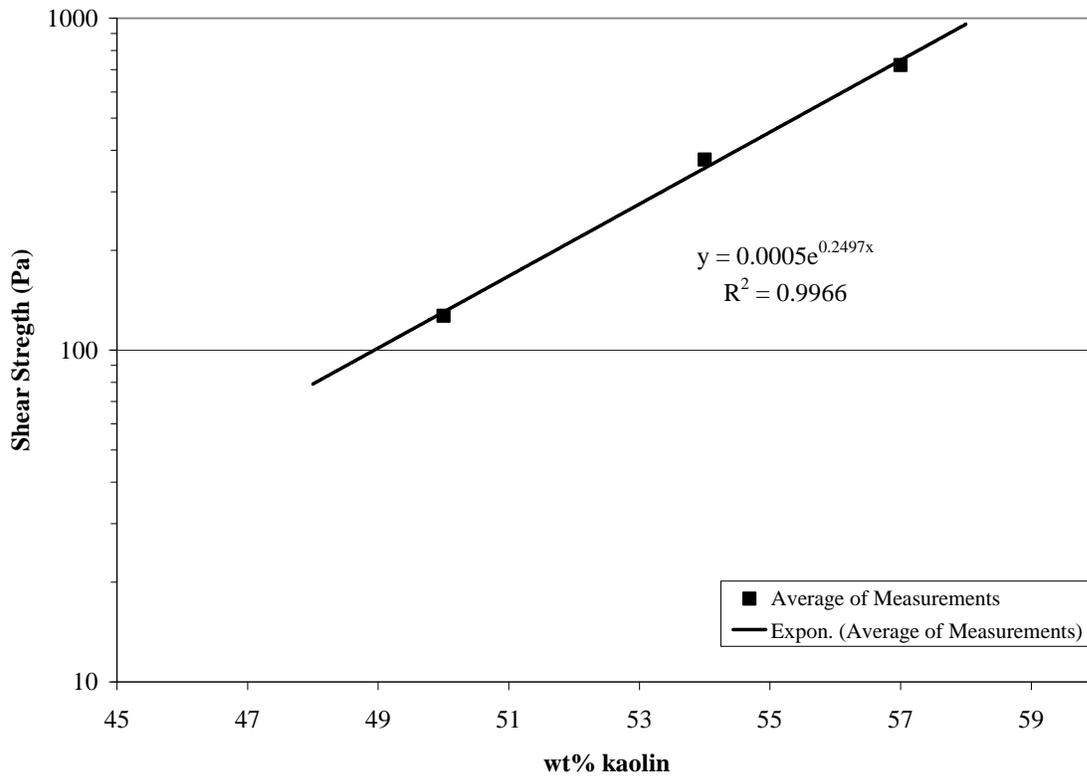


Figure 5.1. FAI Clay Average Measurements and Correlation Derived from the Data

5.2 Previous Data

Four sets of shear strength data were collected previously on various EPK kaolin and water clay slurries. These tests followed similar, though not identical, methods using a shear vane and an aging period of about an hour. These data are reported here in chronological order and will be compared to the current data obtained. The first set of data is from Powell et al. (1995a). It was collected in fiscal year (FY) 1993 and is shown in Table 5.2 and Figure 5.2. These shear strength data correspond to a concentration range of 58-wt% to 67-wt% kaolin and were fit to an exponential curve given by Equation 5.2 with an R^2 of 0.962:

$$y = 0.0243\exp [0.1786 * (\text{wt\% kaolin})] \quad (5.2)$$

Table 5.2. Powell et al. (1995a) Clay Shear Strength Measurements

Wt% Kaolin	Shear Strength (Pa)
58.3	874
60.1	1050
63.9	1810
65.0	3070
67.2	4050

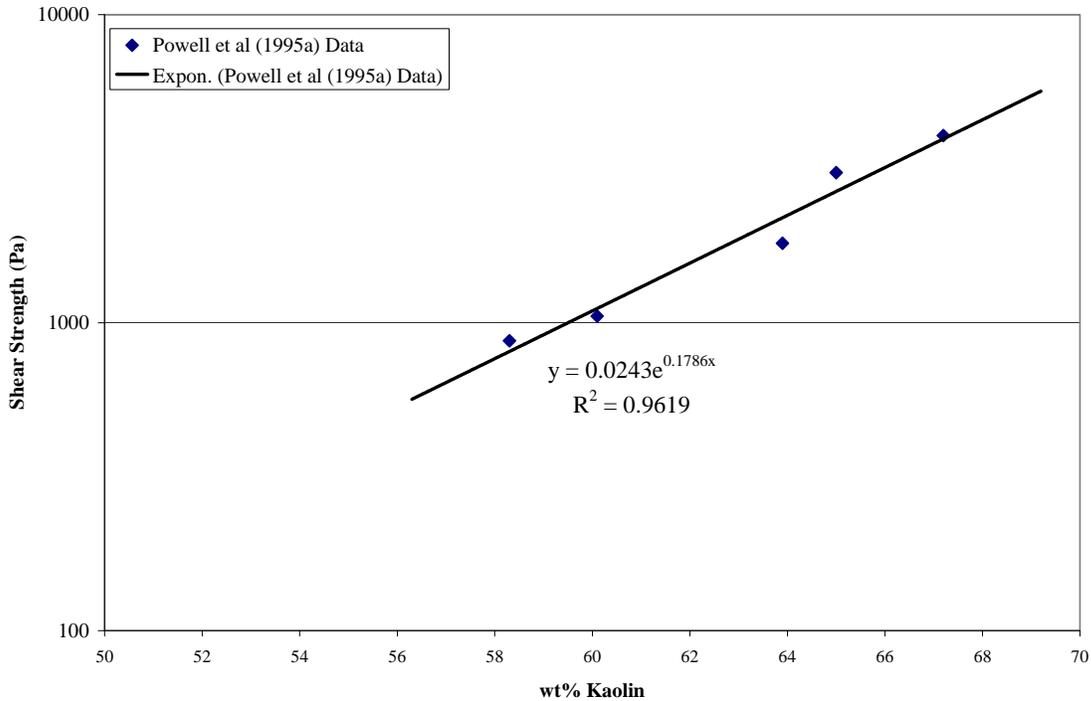


Figure 5.2. Powell et al. (1995a) Clay Shear Strength Data with Correlation Derived from the Data

The next set of data was also measured by Powell et al. (1995b) in FY 1994 and is shown in Table 5.3 and Figure 5.3. These shear strength data correspond to a concentration range of 54 wt% to 66 wt% kaolin. It appears that the data of Powell et al. (1995a) presented in Table 5.2 had slightly higher values than the Powell et al. (1995b) data in Table 5.3. These data were fit to an exponential curve given by Equation 5.3 with an R^2 of 0.996:

$$y = 0.0196 \exp [0.18 * (\text{wt\% kaolin})] \tag{5.3}$$

Table 5.3. Powell et al. (1995b) Clay Shear Strength Measurements

Wt% Kaolin	Shear Strength (Pa)
54.2	350
58.1	640
64.2	2070
65.3	2600
65.4	2670
66.2	2710
66.4	3050

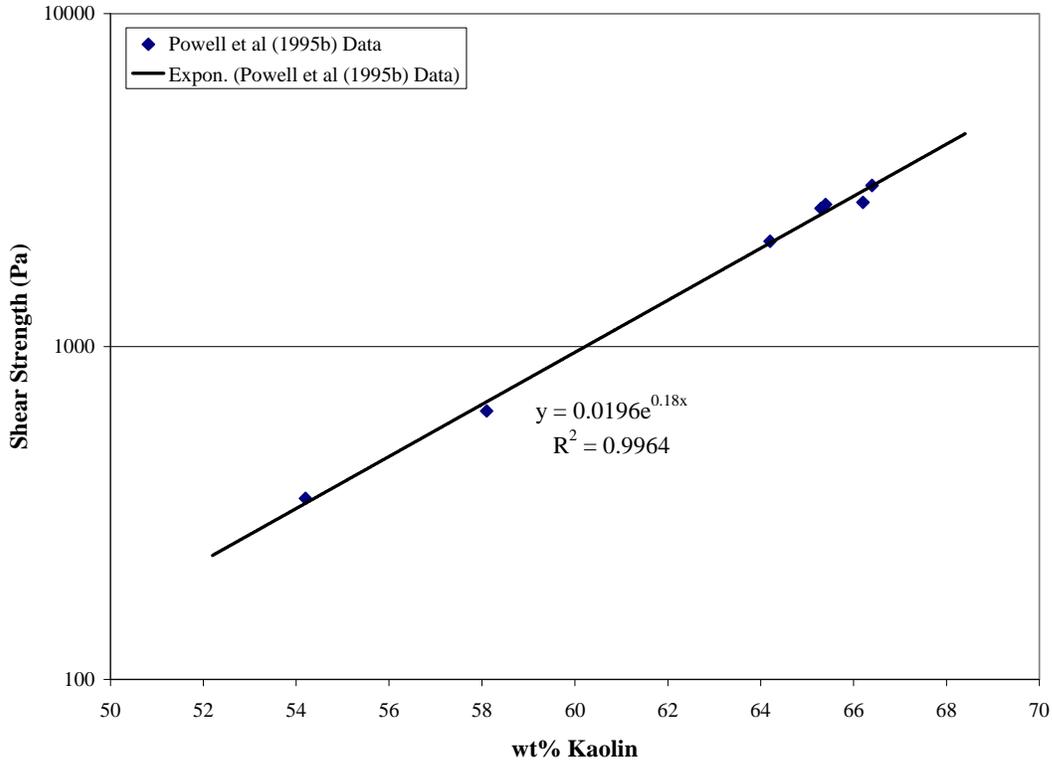


Figure 5.3. Powell et al. (1995b) Clay Shear Strength Data with Correlation Derived from the Data

The next set of data was obtained by Gauglitz et al. (2001) and is shown in Table 5.4 and Figure 5.4. These shear strength data correspond to a concentration range of 30 wt% to 60 wt% kaolin. These measurements were taken over a much broader range of solids with the upper values spanning nearly the same range as the Powell data in both Table 5.2 and Table 5.3. These data were fit to an exponential curve given by Equation 5.4 with an R^2 of 0.998:

$$y = 0.0119\exp [0.1921 * (\text{wt\% kaolin})] \quad (5.4)$$

Considering that the R^2 value is close to 1, it is clear that these data are fit closely by the exponential curve

Table 5.4. Gauglitz et al. (2001) Clay Shear Strength Measurements

Wt% Kaolin	Shear Strength (Pa)
30	3.71
35	8.89
40	28.7
45	70.1
50	198
55	444
60	1125

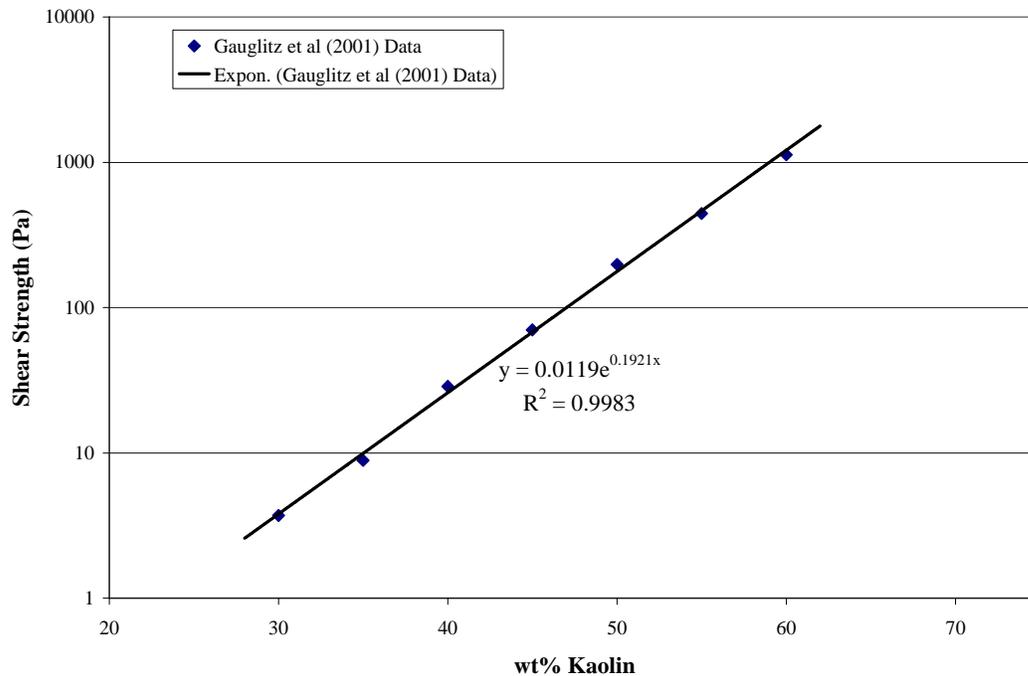


Figure 5.4. Gauglitz et al. (2001) Clay Shear Strength Data with Correlation Derived from the Data

The last set of data was collected by Rassat et al. (2003) and is shown in Table 5.5 and Figure 5.5. These shear strength data correspond to a concentration range of 40 wt% to 62 wt% kaolin. These values tended to be slightly lower than the other data measured. These data were fit to an exponential curve given by Equation 5.5 with an R^2 of 0.985:

$$y = 0.0297\exp [0.1715 * (\text{wt\% kaolin})] \quad (5.5)$$

The shear strength results for the samples with the highest and lowest kaolin concentration are seen to deviate from the exponential correlation, but the majority of the data support using an exponential correlation.

Table 5.5. Rassat et al. (2003) Clay Shear Strength Measurements

Wt% Kaolin	Shear Strength (Pa)
35	20
37.5	23
40	31
42.5	39
45	52
45	69
45	50
47.5 ^(a)	89
50	122
52.5	210
55	321
57.5	516
60	810
60	790
60	840
62.5	1270
65	2010
65	2300
65	2250
70	8000

(a) This data point was not included in the correlation given by Rassat et al. (2003).

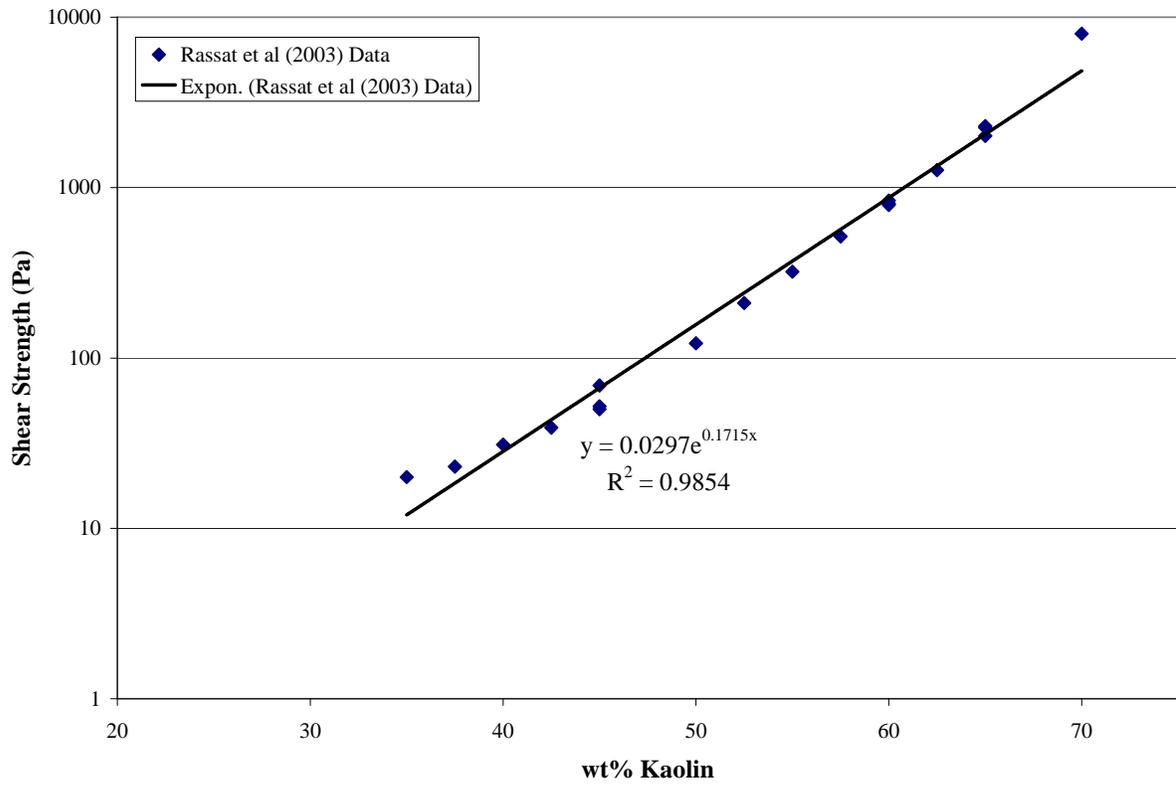


Figure 5.5. Rassat et al. (2003) Clay Shear Strength Data with Correlation Derived from the Data

6.0 Discussion

Similar shear strength correlation studies have been reported previously for EPK kaolin clay, and the data fit well to exponential functionality as expected. The correlations of these previous data presented in Section 5.2 are shown together in Figure 6.1 for comparison. As the wt% solids increases, so does the variation observed in the previously reported data. This may in part be due to the difficulties of making and handling high shear strength materials. The range of the FAI EPK kaolin samples was 50 wt% to 57 wt% solids, which is in the range of both the Gauglitz et al. (2001) and Rassat et al. (2003) data. The Powell et al. (1995a and 1995b) data only had three shear strength measurements within the kaolin concentration range of this current study. The rest correspond to higher kaolin concentrations. Therefore, the Powell et al. (1995a and 1995b) data were not used for comparison against the FAI EPK clay results. The correlation derived from the Rassat et al. (2003) data is considered the best correlation due to the number of data points measured along with this work being carefully documented, including repeatability tests.

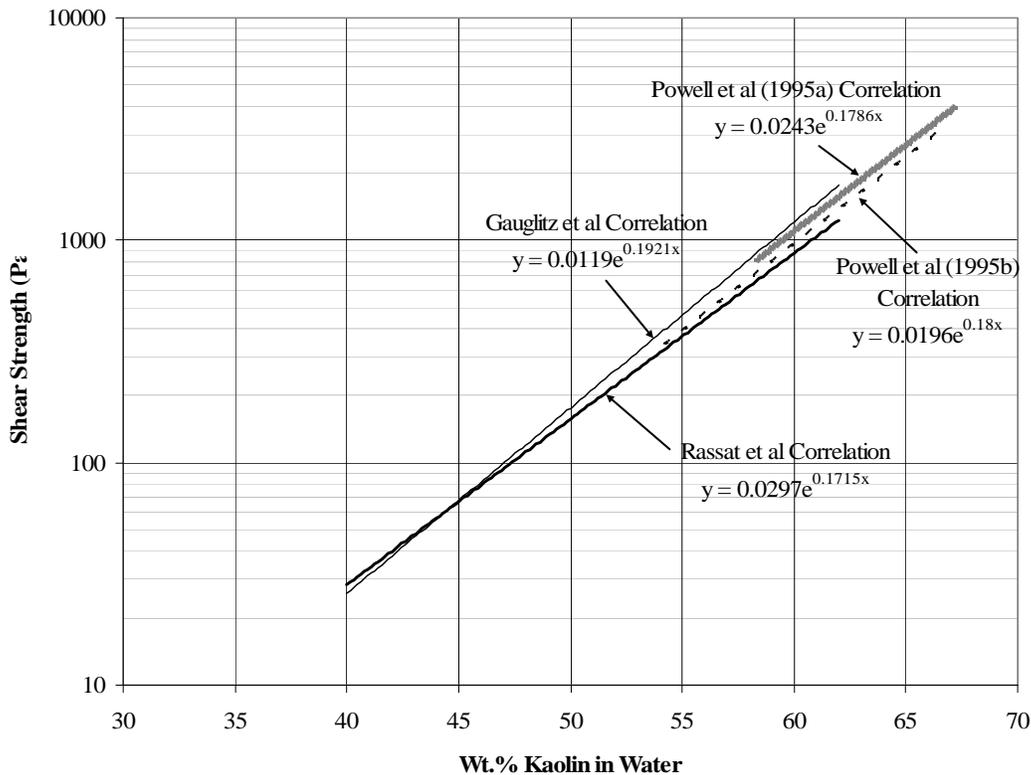


Figure 6.1. Previous Shear Strength Correlations Compared

The three FAI EPK clay data points, measured in triplicate and averaged and fit to an exponential curve in Section 5.1, are compared in Figure 6.2 to the previous correlations discussed above. The specific comparison is with the correlations from the data in Gauglitz et al. (2001) and in Rassat et al. (2003). As mentioned above, the Rassat et al. (2003) correlation represents the best correlation from the previous work. Although similar, the FAI clay shear strength data and correlation differ from the Rassat et al. (2003) correlation, At low solids concentrations, the observed shear strength of the FAI clay was lower than the Rassat et al. (2003) correlation predicted, and at high solids, it was higher than the Rassat

et al. (2003) correlation prediction. Because the FAI clay samples have shear strengths different from previous data, the new correlation is needed to estimate the shear strength of kaolin/water mixtures used at FAI in the vessel-spanning bubble tests.

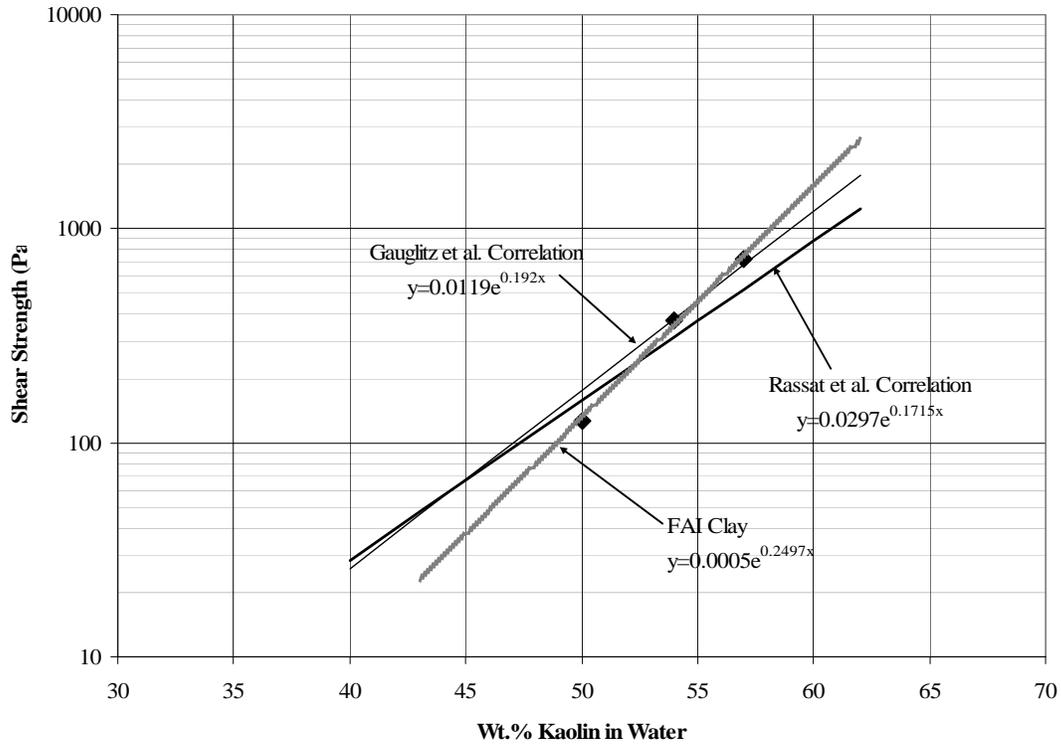


Figure 6.2. Shear Strength Correlations Compared with Current Data

7.0 Conclusions

The FAI EPK clay samples measured in this study could not be predicted using the standard Rassat correlation (2003) currently employed. A new correlation equation was produced for the FAI EPK kaolin data and is given in equation (7.1) below.

$$y = 0.0005 \exp [0.2497 * (\text{wt\% kaolin})] \quad (7.1)$$

Shear strength measurements can be affected by variables such as water quality, sample handling, mixing, gel time, and to some extent, the geometry of the measuring device used, which determines both the sensitivity and range of the measurement. Given the deviations observed from the Rassat correlation (2003) for the FAI kaolin clays over the concentrations used in this current study and the fact that the FAI clay was well fit by an exponential equation, the new correlation should be used to estimate the shear strength of kaolin/water mixtures used at FAI in the vessel-spanning bubble tests. The average uncertainty in the data used to develop the correlation is about 10%.

The new correlation is based on samples spanning a small range in wt% kaolin. Using the correlation to estimate strengths outside the range of the data (extrapolate) is reasonable because the exponential relationship is well established. However, the uncertainty in the estimated shear strength increases with extrapolation.

8.0 References

Daniel RC. 2007. PNNL Technical Procedure, “Measurement of Physical and Rheological Properties of Solutions, Slurries and Sludges.” RPL-Colloid-02 Rev. 1, Pacific Northwest National Laboratory, Richland, Washington.

Epstein M and PA Gauglitz. 2010. *An Experimental Study of the Stability of Vessel-Spanning Bubbles in Cylindrical, Annular, Obround and Conical Containers*. Fauske & Associates Report FAI/09-272, Rev. 1 (January), Burr Ridge, Illinois.

Gauglitz PA, G Terrones, SJ Muller, MM Denn, and WR Rossen. 2001. *Mechanics of Bubbles in Sludges and Slurries*. PNNL-13748, Pacific Northwest National Laboratory, Richland, Washington.

Nguyen QD and DV Boger. 1985. “Direct Yield Stress Measurement with the Vane Method.” *Journal of Rheology* 29(3):335–347.

Powell MR, GR Golcar, CR Hymas, and RL McKay. 1995a. *Fiscal Year 1993 1/25-Scale Sludge Mobilization Testing*. PNL-10464, Pacific Northwest National Laboratory, Richland, Washington.

Powell MR, CM Gates, CR Hymas, MA Sprecher, and NJ Morter. 1995b. *Fiscal Year 1994 1/25-Scale Sludge Mobilization Testing*. PNL-10582, Pacific Northwest National Laboratory, Richland, Washington.

Rassat SD, LM Bagaasen, LA Mahoney, RL Russell, DD Caldwell, and DP Mendoza. 2003. *Physical and Liquid Chemical Simulant Formulations for Transuranic Wastes in Hanford Single-Shell Tanks*. PNNL-14333, Pacific Northwest National Laboratory, Richland, Washington.

Appendix A: Shear Strength Plots

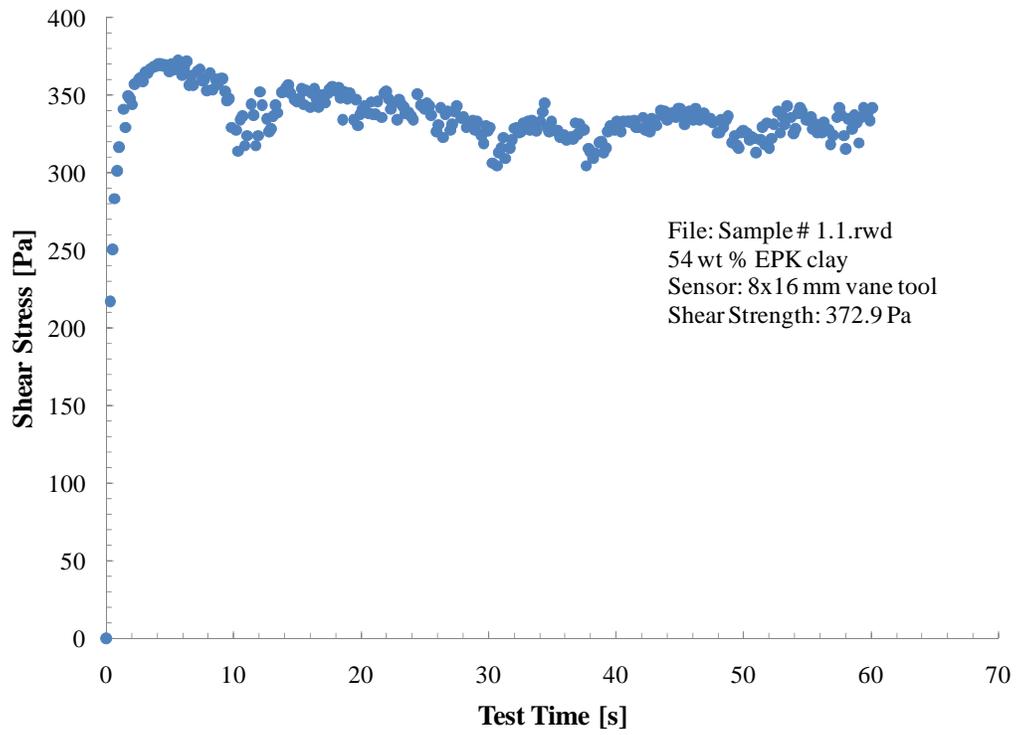


Figure A.1. 54 wt% EPK Clay, Measurement 1

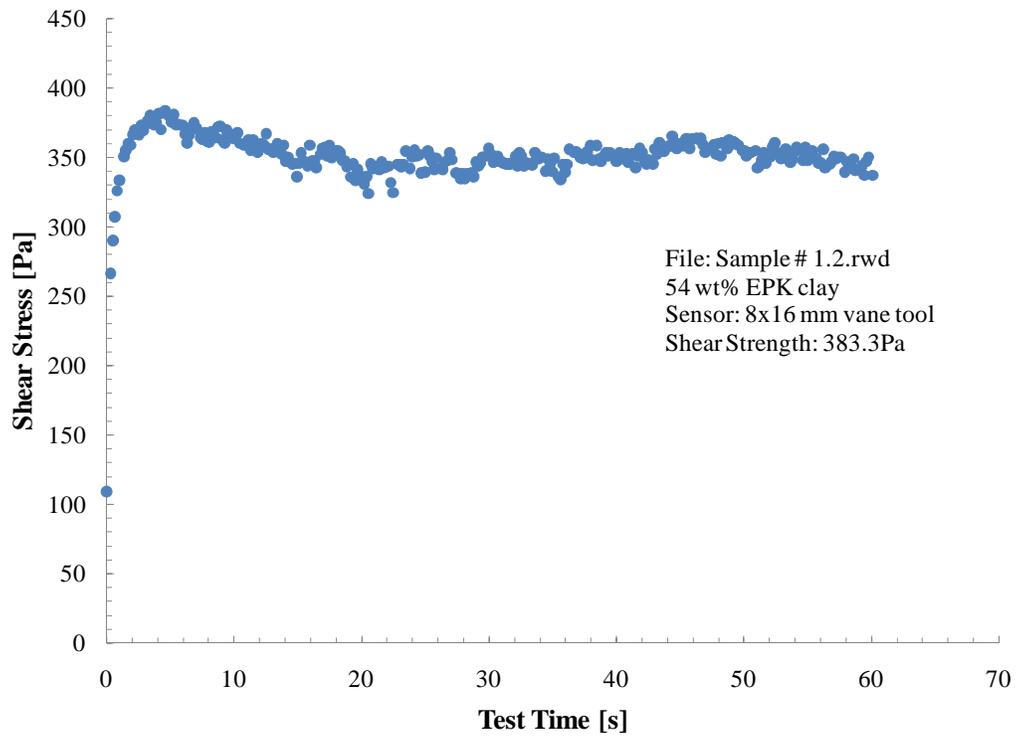


Figure A.2. 54 wt% EPK Clay, Measurement 2

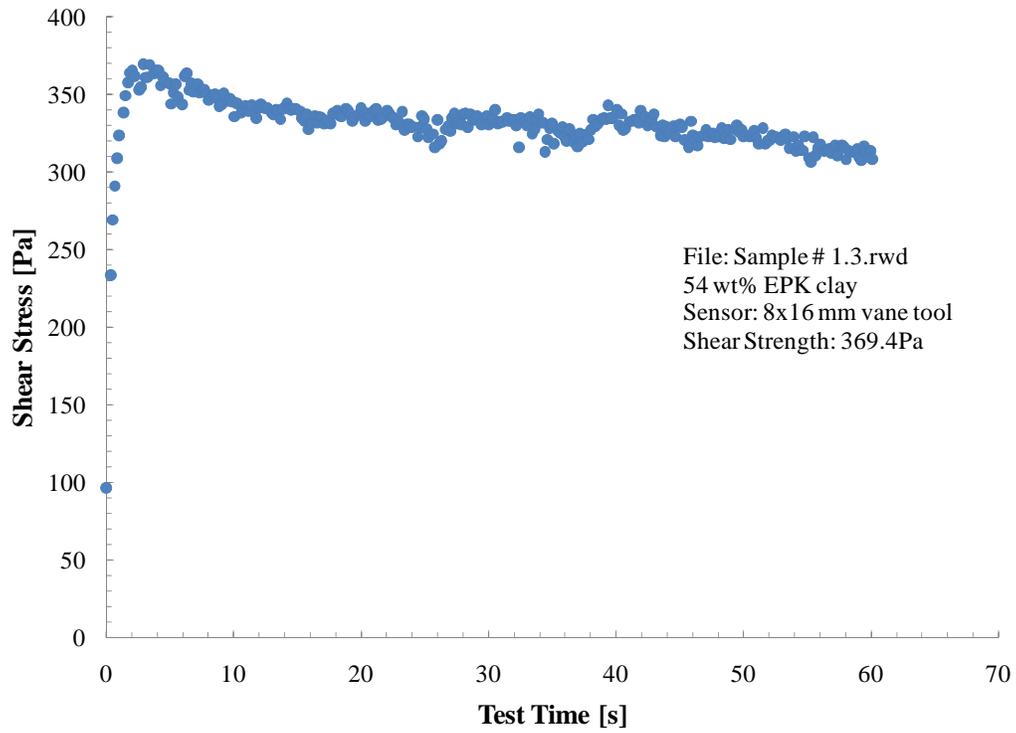


Figure A.3. 54 wt% EPK Clay, Measurement 3

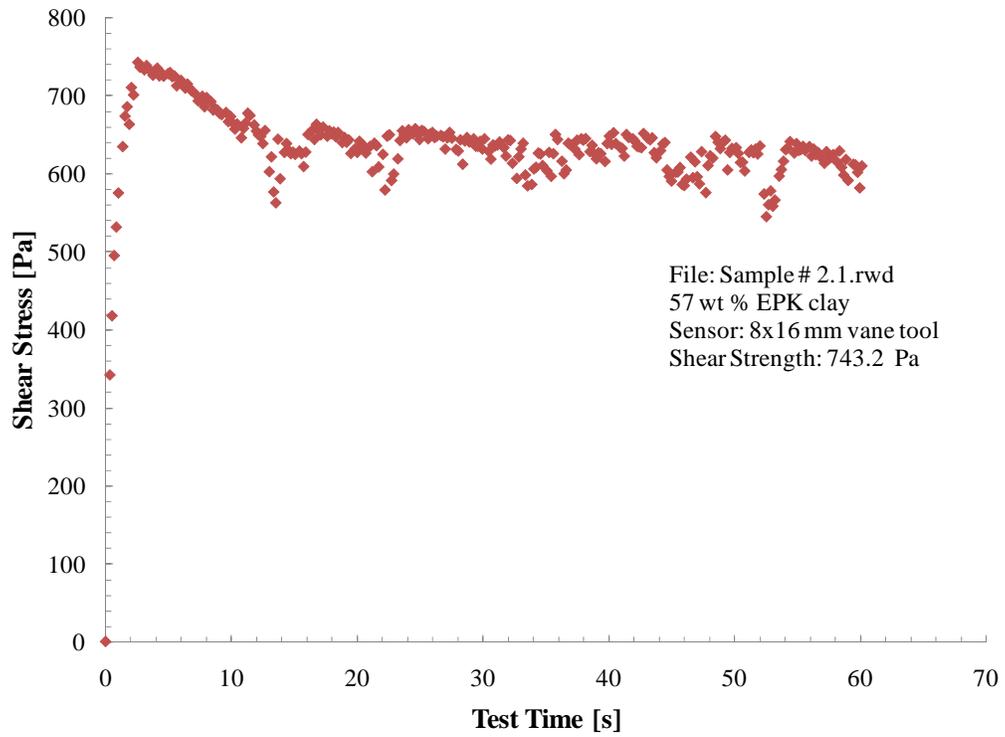


Figure A.4. 57 wt% EPK Clay, Measurement 1

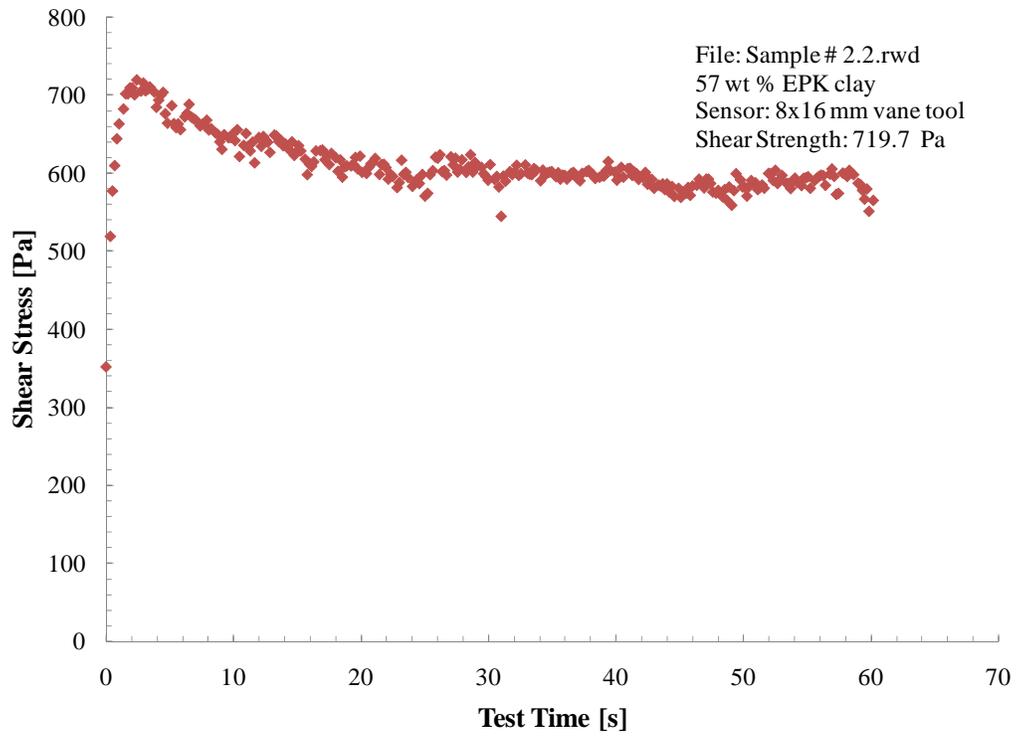


Figure A.5. 57 wt% EPK Clay, Measurement 2

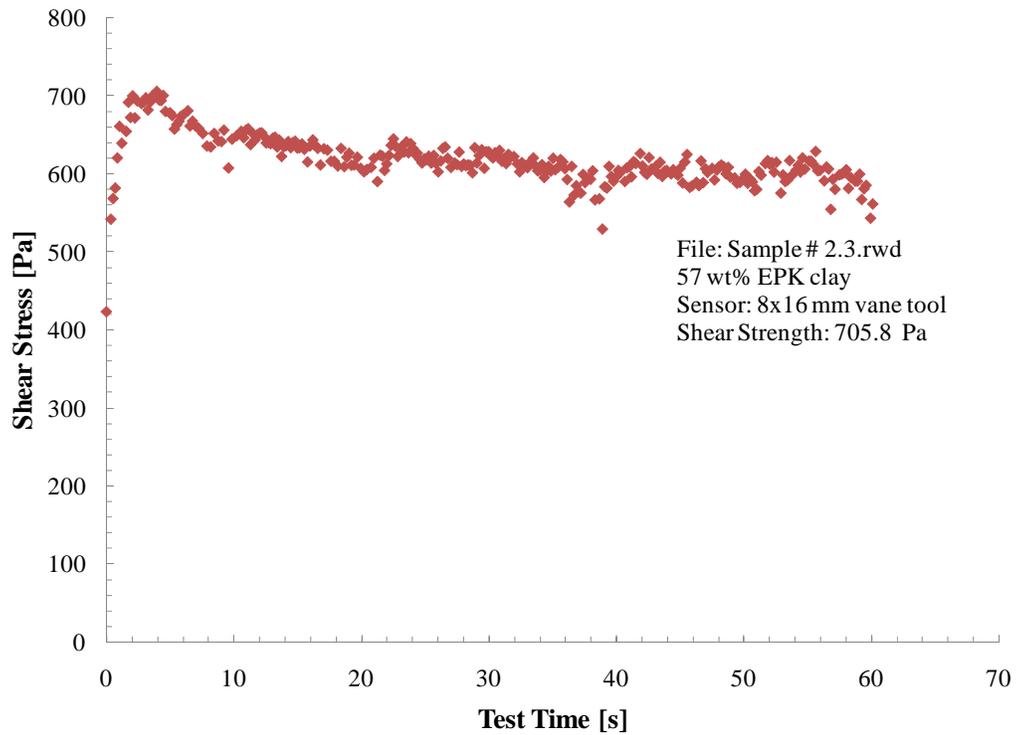


Figure A.6. 57 wt% EPK Clay, Measurement 3

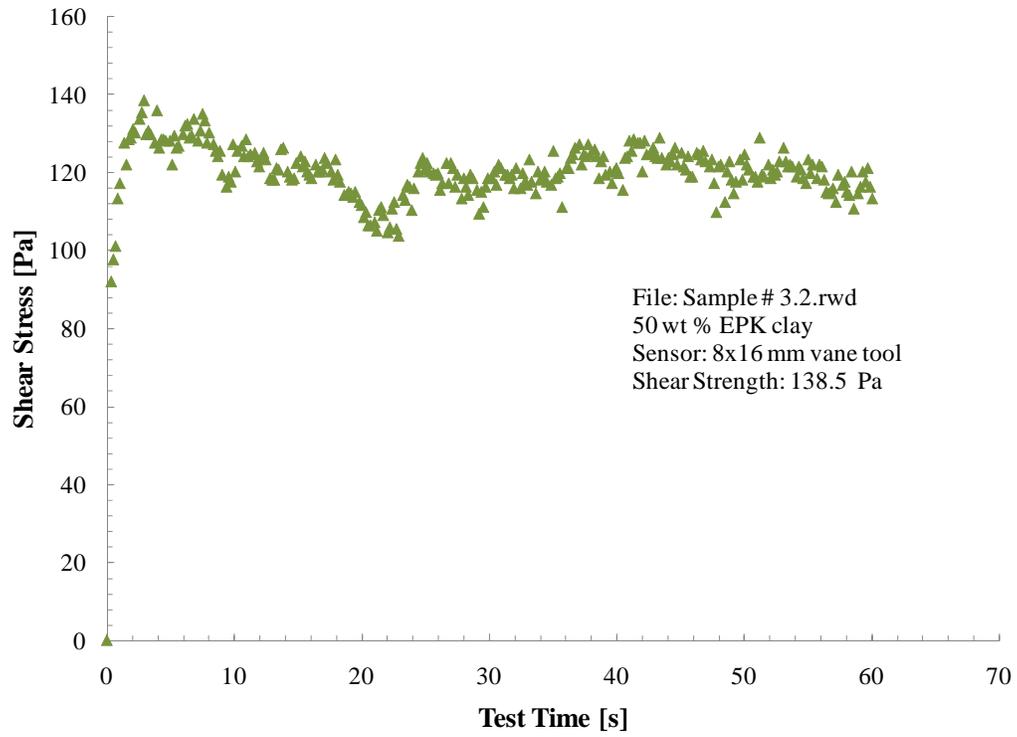


Figure A.7. 50 wt% EPK Clay, Measurement 2

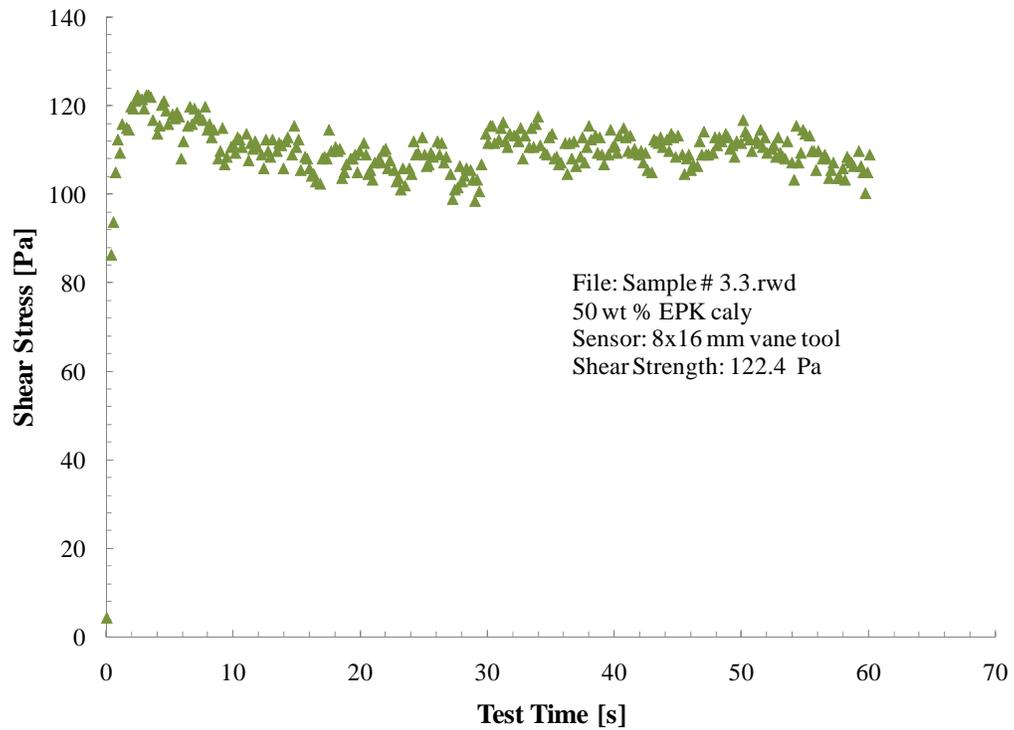


Figure A.8. 50 wt% EPK Clay, Measurement 3

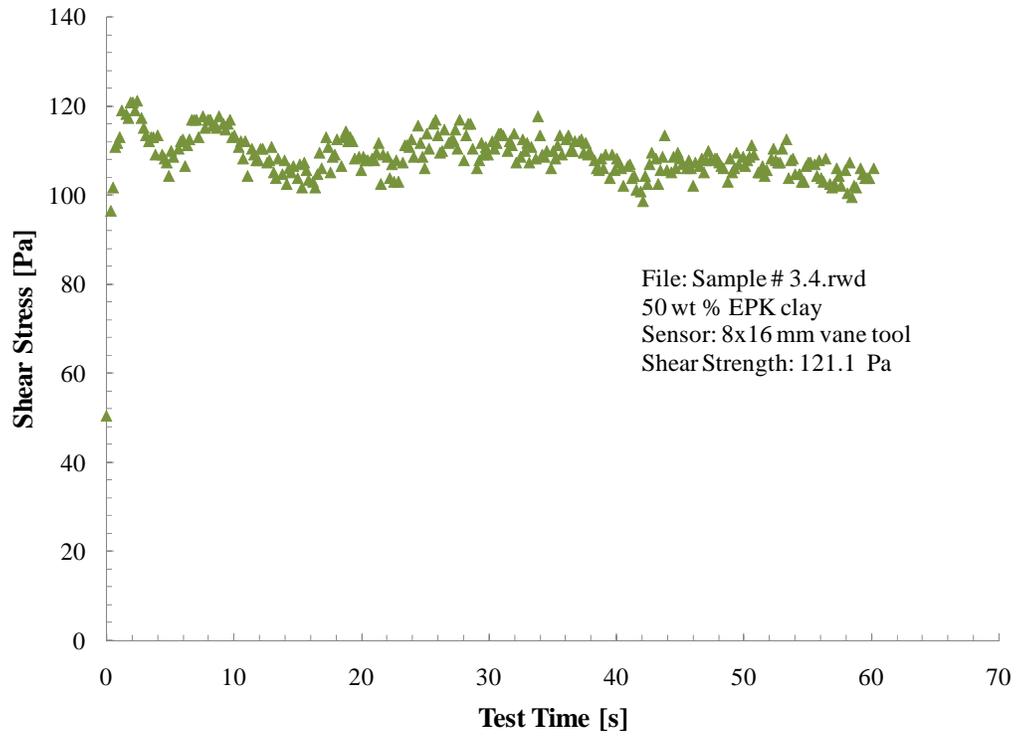


Figure A.9. 50 wt% EPK Clay, Measurement 4

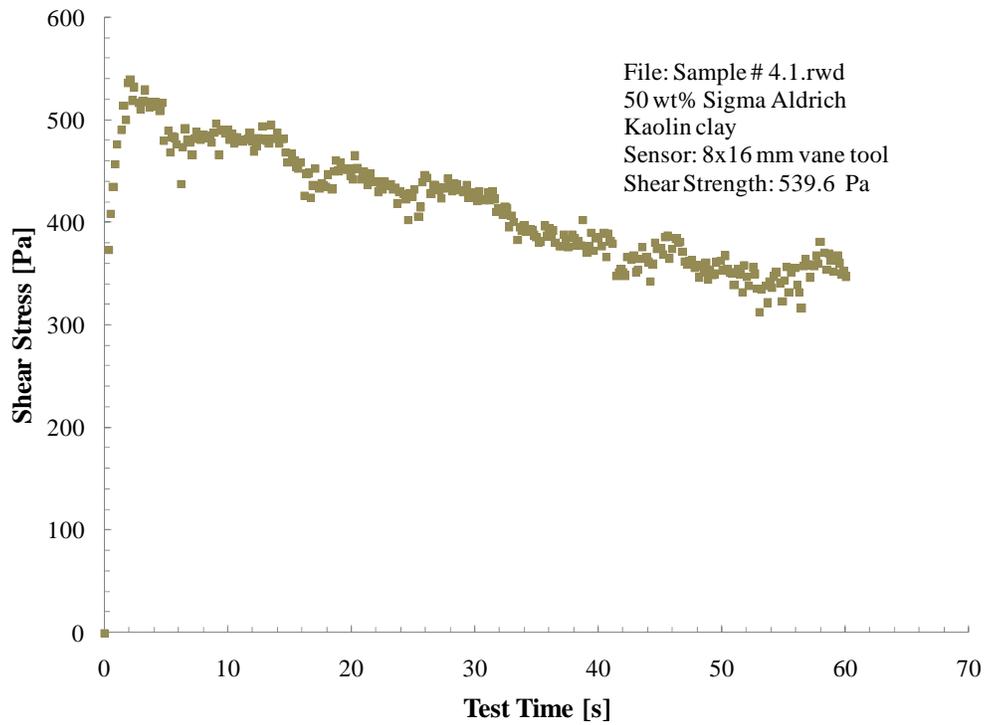


Figure A.10. 50 wt% Sigma Aldrich Clay, Measurement 1

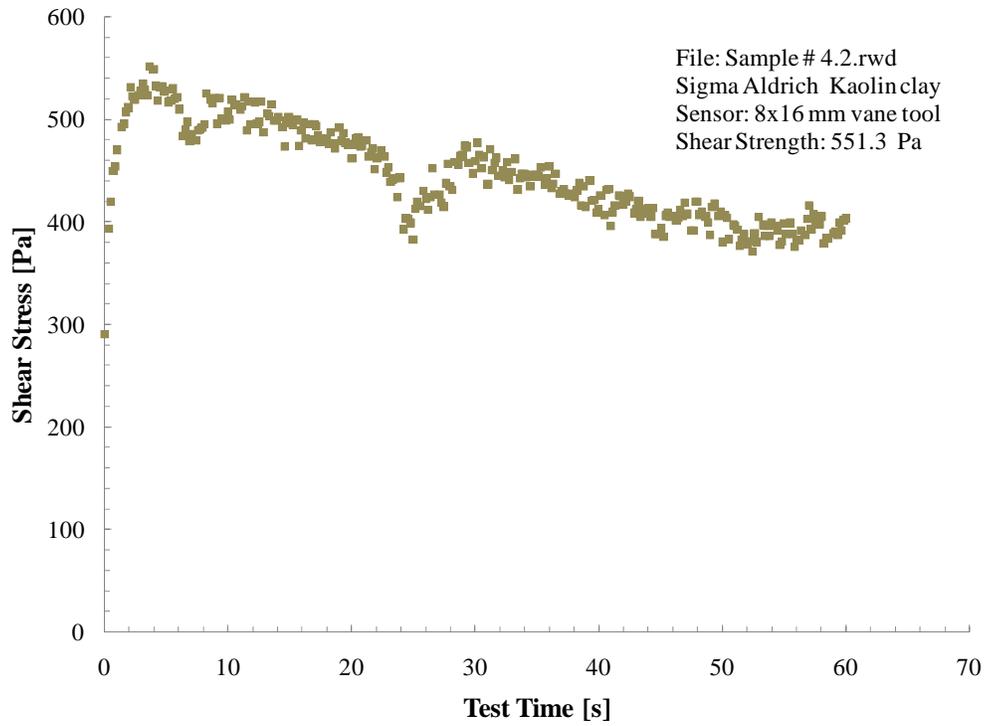


Figure A.11. 50 wt% Sigma Aldrich Clay, Measurement 2

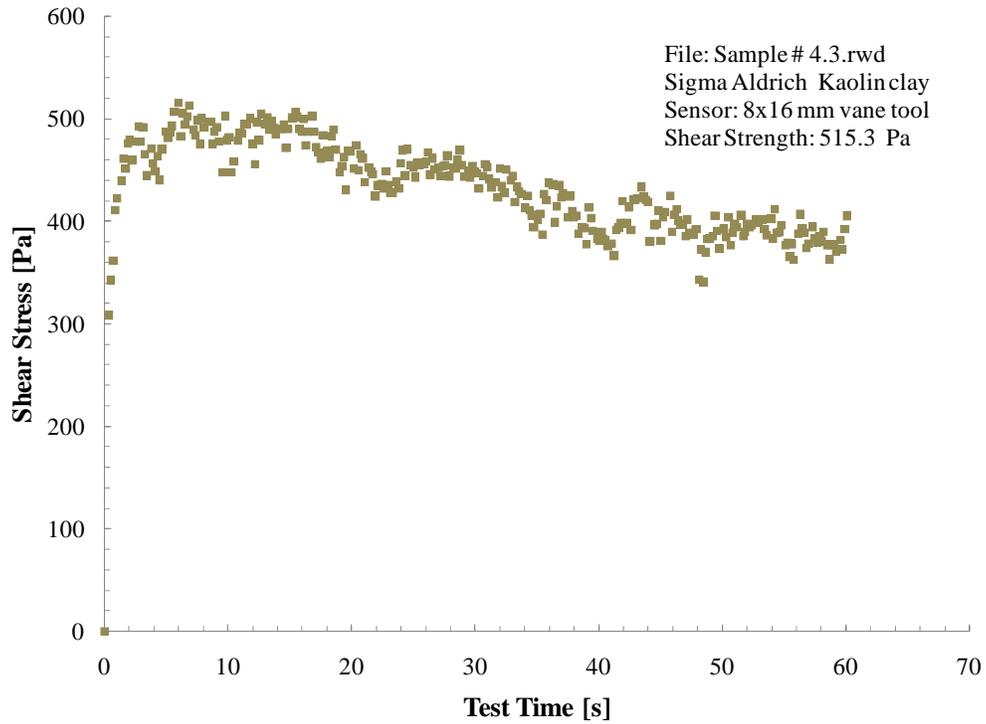


Figure A.12. 50 wt% Sigma Aldrich Clay, Measurement 3

Distribution

**No. of
Copies**

**No. of
Copies**

OFFSITE

ONSITE

1 M Epstein
Fauske and Associates, LLC
16W070 83rd Street
Burr Ridge, IL 60527

17 Pacific Northwest National Laboratory
C. A. Burns P7-25
R. C. Daniel P7-22
P. A. Gauglitz (10) K7-15
A. J. Schmidt P8-60
S. D. Rassat K6-28
R. L. Russell K6-24
B.M. Thornton K7-07
B. E. Wells K7-15
Information Release (pdf)

3 CH2M HILL Plateau Remediation Company
J. P. Sloughter A3-06
M. E. Johnson A0-26
STP Project File (N. Fouad) H6-08



Pacific Northwest
NATIONAL LABORATORY

902 Battelle Boulevard
P.O. Box 999
Richland, WA 99352
1-888-375-PNNL (7665)

www.pnl.gov



U.S. DEPARTMENT OF
ENERGY