Gas Retention and Release Experiments with Low Yield Stress Fluids - 11289

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ABSTRACT

The Waste Treatment and Immobilization Plant (WTP) facilities will generate process streams, which are characterized with relatively high concentrations of solids and non-Newtonian rheology (generally described with a Bingham plastic model). The current flow sheet of the WTP includes the use of pulse jet mixers (PJMs) throughout the plant to provide adequate mixing of the process fluids. The main objective of the experimental program conducted at Florida International University was to investigate gas retention and release properties of selected non-Newtonian fluids relevant to waste streams of the Hanford Tank Waste Treatment, and Immobilization Plant. Bench scale experiments were carried out using mixtures of kaolin-bentonite which define a range in particle size distribution, morphology, zeta potential and yield stress. The experimental program used in-situ generated oxygen generated by decomposing hydrogen peroxide catalyzed by a mixture of ferrous iron and EDTA to provide data about the volumes of retained gas and the release rates from the gelled simulant. A six foot column with diameter of 6 inches was connected to a mass spectrometer to provide high resolution time series of gas release. The retention potential of each simulant and the signature of the released gas (such as total volume, shape, and peak intensity) were determined. Studies conducted to date at SRS (Savannah River Site) and PNNL (Pacific Northwest National Laboratory) have addressed mostly steady state behavior of gas hold up in completely mixed fluids while transient behavior, which is significant for loss of power events, has not been investigated in a systematic manner. Moreover, data reported by Stewart et al [10] reported results from simulants with yield stress of 3, 13 and 30 Pa, however no systematic studies were conducted in the lower range of 0-10 Pa and only one point existed in this range (3 Pa) which does not provide sufficient basis for extrapolation in the lower range Therefore, this experimental program extends experimental studies previously conducted at SRS and PNNL by addressing the transient behavior of gas release, retention and release patterns for mixtures with yield stress lower than 5 Pa. The experimental data were analyzed to provide information about the equilibrium mass of oxygen contained in the gelled simulant and the initial rates of release of retained gas. Additional experiments are planned with mixtures that include antifoaming agents (AFA) and the data will be correlated with the physicochemical and colloidal properties of the simulants (particle size distribution, morphology, zeta potential, yield stress and Bingham consistency) to provide a better understanding of the governing factors for gas retention and release properties of fluids with yield stress parameters in the range of 0-10 Pa.

INTRODUCTION

Hydrogen gas generation by radiolysis and/or thermolysis has been recognized as a significant hazard within the WTP. The current control strategy is to maintain hydrogen concentrations below 25 percent of the lower flammability limit [1](LFL) [1]. The design approach involves providing sufficient dilution ventilation during all plant conditions (e.g., normal operating and upset conditions) and therefore requires

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[1] The LFL limit has been defined as 2.5% H_2

an accurate understanding of hydrogen generation rates within each WTP vessel. Dilution air is provided by the process vessel purge (PVP) system. The gas generation rate, together with the retention characteristics of mixed sludge waste was used to determine how much gas can accumulate in the waste in WTP vessels. Furthermore, the volume and rate of a gas released into a vessel headspace, not the generation rate, was used determine whether flammable concentrations result.

During normal operation, the mixing systems (PJM's, air spargers, recirculation) in the WTP vessels (including non-Newtonian waste slurries) must achieve safe, controllable release of flammable gases including hydrogen). Previous experimental studies using PJM mixing systems identified a formation of active mixing caverns at the bottom of the mixing vessels and a lack of mixing at the upper portion of the vessel, which may result in accumulation of gas produced by radiolysis in the stagnant fluid or gel. During loss-of-power events, PJMs may be operated intermittently on backup power for extended periods, which can result in gelling of the slurry and accumulation of hydrogen or other flammable gases in the gelled slurry. Upon restart of the PJMs, the mass of released hydrogen gas and rate of release must not create flammable conditions in the vessel headspace. Furthermore, the process flow sheet will consider the addition of an anti-foam agent (AFA) to the process streams to prevent surface foaming, which may modify the gas retention and release patterns of the waste fluids.

Several studies have been conducted by PNNL and SRS that quantify the steady state behavior of gas hold-up in completely mixed fluids. While these studies provide a fundamental understanding of the steady state gas hold up and the subsequent gas release behavior in normal operations, they fail to provide an adequate understanding of the gas dynamics in the loss of power events or transient conditions especially in a low yield stress range of 0-10 Pa. Therefore a test program was established at Florida International University to quantify the gas retention and release characteristics of low yield stress non-Newtonian fluids of varying rheology.

ASPECTS ALREADY INVESTIGATED

Radiolytic, thermal and chemical reactions in radioactive waste generate flammable gas mixtures. The rheological and physicochemical properties of the waste define the gas retention and release patterns which may present safety concerns for nuclear waste storage tanks, including Hanford's Single and Double Shell underground tanks and the waste processing vessels from the Waste Treatment and Immobilization Plant. Previous research work has investigated three areas: i) Underground waste storage tanks, ii) WTP process vessels and iii) The effect of addition of anti foaming agents in WTP process vessels.

Underground waste storage tanks: Earlier studies by Gauglitz et al [2, 3] and Rassat et al [4, 5] investigated the underground storage tanks and determined that the yield stress and particle size of the slurry are the two most important macroscopic parameters that govern the release and retention patterns of flammable gases. Gauglitz et al [6] provided theoretical and experimental analysis of the observed hysteresis of volume versus pressure in actual tanks and investigated a range of related phenomena (including solid and fluid mechanics processes) governing gas retention and release mechanism in waste slurries and porous media filled with both Newtonian and non-Newtonian fluids. The majority of earlier experimental work on retained volumes and rates of release of gas were calculated by measurements of the height of the slurry. The results from these studies have provided correlations for untreated waste and a fundamental understanding of the phenomena; however, they did not provide sufficient information to predict the gas-retention and release characteristics for non-Newtonian waste-slurries in WTP vessels mixed with PJMs and possibly with auxiliary spargers or recirculation pumps. Therefore, additional experiments were required to determine the gas retention and release parameters in WTP process vessels.

WTP process vessels: Guerrero [7] conducted studies of the quantities of gas accumulated in completely mobilized simulant (using PJM only or PJM plus sparging) and gas release patterns after simulation of loss-of-power events in the scaled Concentrate Receipt Vessel (CRV) Test Stand at SRNL. Russell et al [8] provided analysis for a series of gas hold up tests using simulants with yield stress between 7 and 44 Pa and Bingham consistency between 8 and 27 cP. The gas hold-up results were correlated with the rheology of the fluids, the geometry of the vessel, and the PJM characteristics. The gas release rates were correlated with three time constants and the tests provided correlations of the rheology, bubble size, and the gas retention and release properties of the fluids. Using hydrogen peroxide, Meyer et al [9] investigated the operation of PJMs for a range of simulants and provided analysis of the steady state parameters and the transient behavior of gas retention and release for a Half Scale Lag Storage vessel (HSLS) and for a Quarter Scale Lag Storage vessel (QSLS). Additional investigations were recommended for the effects of addition of antifoaming agents.

Addition of AFA: Stewart et al [10] and Guerrero et al [11] conducted bench scale tests and confirmed that mixtures with AFA (Dow Corning® Q2-3183A) increase gas retention. The experimental data showed unexpected increase of gas holdup for simulants with lower shear strength, which was attributed to reduced bubble size of the gases in the column and decreased mixing efficiency of the spargers for given flow rate. To resolve the observed discrepancies, Stewart at al [12] conducted additional studies at SRNL (using small scale column experiments) and PNNL (experiments in prototypic sparger-PJM systems) and provided a gas retention and release model which fits the experimental data. The studies at SRNL which analyzed the gas holdup data from small-scale and bench-scale impeller-type mixing systems confirmed the previously observed discrepancies [10 and 11] of greater gas holdup and retention for fluids with lower yield stress (clay was used for this comparison). The performance of an alternative AFA was analyzed using Dow Corning® 1520-US and provided results comparable to Dow Corning® Q2-3183A AFA; however, significantly higher concentrations were required (2500 ppm vs. 350 ppm respectively). Furthermore, the addition of noble metals to the AZ-101 waste simulant did not produce a catalytic gas retention effect with the AFA. Mass transfer coefficients for air bubbles generated by a prototypic sparger were obtained from the transient decay of dissolved oxygen concentration in initially saturated fluids. The addition of AFA reduced the mass transfer coefficient for AZ-101 simulant. The performance of an alternative AFA was analyzed using Dow Corning® 1520-US and provided results comparable to Dow Corning® Q2-3183A AFA; however, significantly higher concentrations were required (2500 ppm vs. 350 ppm respectively). Furthermore, the addition of noble metals to the AZ-101 waste simulant did not produce a catalytic gas retention effect with the AFA. Mass transfer coefficients for air bubbles generated by a prototypic sparger were obtained from the transient decay of dissolved oxygen concentration in initially saturated fluids. The addition of AFA reduced the mass transfer coefficient for AZ-101 simulant. The results obtained from the SRNL team confirmed previously observed greater gas holdup trends for fluids with lower yield strength. The prototypic sparger-PJM mixing system at the PNNL APEL facility (HSLS and QSLS vessels) investigated kaolin-bentonite clays and AZ-101 simulant with non-Newtonian rheological properties and provided analytical studies of the effect of gas composition, mass transfer during sparging and the addition of AFA on gas retention and release using simulants with 3, 13, and 30 Pa Bingham yield stress. The prototypic tests showed that gas retention due to in situ generation was higher at 30-Pa yield stress than at 13 or 3 Pa, though sparger holdup was higher at 3 Pa. The HSLS and QSLS tests concluded that the steady state gas hold up decreased with decreasing yield stress. The rate of gas release was slower at lower yield stress-3 Pa simulant took much longer (1 hr) to release the holdup compared to 13 and 30 Pa (less than 10 min). The reasons for the difference of holdup and retention behavior between the prototypic and non-prototypic have not been determined.

An analysis of the data from more than 650 experiments during the literature review revealed that no systematic studies had been done in correlating gas fraction as a function of simulant yield stress and other principal properties for low yield stress non-Newtonian fluids in the range of 0-10 Pa. Only a

handful of (<50) experiments investigated the gas retention and release patterns in 0-10 Pa range and that too primarily for completely mixed conditions as shown by the highlighted circles in Fig.1.
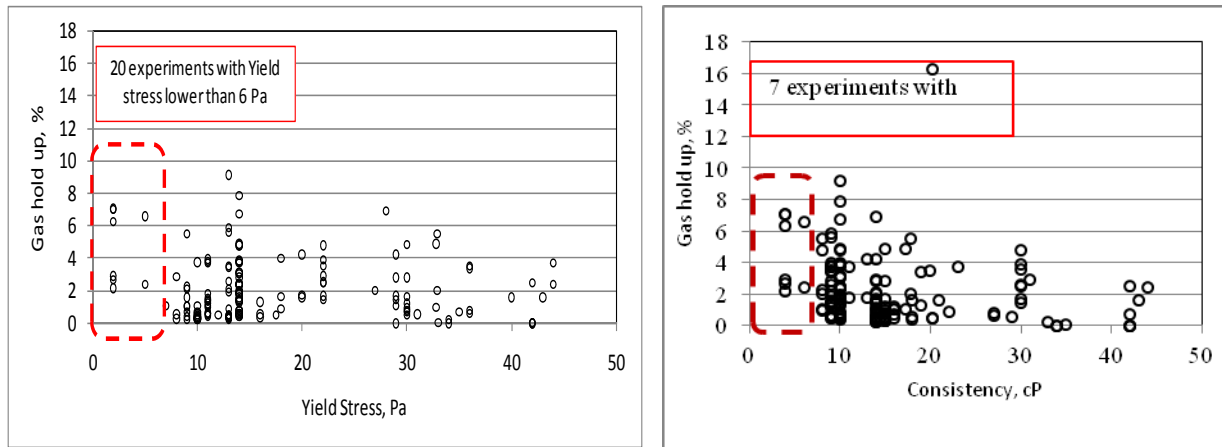

Fig.1. Experiments in the range of 0-10 Pa

Table I below shows the handful number of tests done to data in the 0-10 Pa range.

Table I. Literature Review Synopsis

Simulant Type	Yield Stress Range 0-2 Pa	Yield Stress Range 2-4 Pa	Yield Stress Range 4-6 Pa	Yield Stress Range 6-8 Pa	Yield Stress Range 8-10 Pa
AZ-101		3			2
AZ-101 + AFA	6	4	2		
Clay				4	16
Clay + AFA		4			
AZ-101		3			2

The experimental program initiated at FIU aims to conduct further research that will address the gap of knowledge in transient behavior and abnormal rates of gas release in waste fluids with yield stress lower than 5 Pa.

OBJECTIVE

The main objective of these tests was to study the gas retention and release behaviors of simulants during the release of gas from the "gelled" state with a yield stress range of 0-10 Pa. The tests would investigate:

Release rates of low yield stress simulants: Stewart et al [11] reported results from simulants with yield stress of 3, 13 and 30 Pa, however no systematic studies were conducted in the lower range of 0-10 Pa and only one point exists in this range (3 Pa) which does not provide sufficient basis for extrapolation in the lower. The retention potential of each simulant, the signature of the released gas (such as total volume, shape, and peak intensity) and release rates were determined.

Transient behavior: The uncertainty of the post DBE requires studies under a range of mixing and gas release conditions. Most of the previous studies focused on the gas holdup in completely mixed waste.

Additional work is needed to determine the gas release patterns after restart of the mixing system. The experimental program will model the transient behavior by releasing gas from gelled conditions.

EXPERIMENTAL SET-UP

The bench scale setup consisted of a 6 foot acrylic vessel filled with test liquid pumped from the bottom and a purged argon head space Fig.2 (left). The test vessel was connected to mass flow controllers and an inline mass spectrometer Fig.2 (top right). The simulant was mixed by a hybrid system consisting of impellers and spargers. The mixing system consisted of three impellers on a common shaft. A "rake" or sweep-impeller was added below the lower impeller to improve mixing in the volume below the lower impeller. This sweep impeller, design provided below, had an overall diameter of 4 inches. The impellers were located on a single 1/2-inch shaft supported at the top by the packed seal and at the bottom by a teflon bushing located at the top of the radial flow deflector. The shaft was driven through a flexible coupling to allow for ease of alignment of the motor drive and the shaft. The pitch orientation for both impellers was selected for upflow displacement of fluid during mixing, and the impellers were located on the shaft to avoid gas entrapment in the vortex under well-mixed conditions. The vessel contents were considered well mixed when movement of fluid at the walls of the vessel could be observed. In other words, the entire fluid volume was fully sheared. In this regards, visual observations of fluid movement at the walls and the shape of the surface vortex was used to guide setting the mixer speed. The mixer shaft speed (RPM) was monitored continuously with a laser tachometer and controlled manually. In addition to impellers, a set of spargers were used to aid in mixing. The spargers were fitted via a cross connector and nitrogen gas was injected from the bottom through a stainless steel tube to ensure mixing across the entire test vessel Fig.2 (top bottom). The injected bubbles were directed at the tip of the lower impeller to break up the larger bubbles into smaller bubbles. The sparger flow rate was controlled by a mass flowmeter.

Fig.2. Experimental Set-up (left), mass spectrometer (top right), and spargers (bottom right)

Once the simulant was mixed, oxygen was generated in-situ via hydrogen peroxide decomposition. Simulant mixing was stopped, and the waste was allowed to gel for 18 hours while hydrogen peroxide decomposed to form oxygen bubbles within the gelling fluid to represent intermittent mixing cycles during loss of power (non-steady mixing conditions). After completing the reaction of hydrogen peroxide decomposition, the vessel was mixed while purging the headspace of the vessel with argon and measuring the composition of released gas. The rates of release and the volume of gas retained and released were correlated with the hydrodynamic and rheological properties of the simulants. The experimental parameters were used to determine the retention potential of each simulant and the signature of the released gas (such as total volume, shape, and peak intensity).

SIMULANT

The test program required the simulant to be safe to use as loading, unloading and sampling of the simulant could expose the personnel to the simulant and working in protective equipment for several hours would add undesirable complications. A transparent simulant was desired to observe the bubbles visually. Yield stress was the primary control variable for the proposed tests (Table II); hence a simulant whose Non-Newtonian rheological properties varied by dilution with water was required. Because testing is going to take over several weeks the simulant needed to exhibit constant rheological properties and be stable. Due to a large numbers of tests proposed to be conducted, an inexpensive simulant was also desired to minimize the impact on project costs.

Table II. Significant Simulant Properties for Hybrid Mixing Systems and Goal Values

Property	Goal Values
Density	1.2g/ml
Bingham Yield Stress	0-10 Pa
Bingham consistency	10 cP

Based on the test requirements kaolin-bentonite mix was found to be the most suitable. Kaolin-bentonite (80/20 ratio) clay mixtures have been used extensively by PNNL and SRNL in their gas retention and release tests and hence makes it the ideal candidate to compare with the previous test results Moreover it shear thins with yield stress and is unaffected by shear, temperature, microorganisms, enzymes or UV light The clay mix is also inexpensive and easy to make considering the large volumes that need to be prepared and tested.

IN-SITU GAS GENERATION AND RELEASE TESTS

The in-situ generation and release tests used oxygen generated through decomposition of H_2O_2 as the surrogate gas for hydrogen. The level of oxygen hold-up in the gelled simulant was controlled by the amount of H_2O_2 added to the simulant before gelling.

The in-situ gas retention and release tests consisted of: (a) Addition of hydrogen peroxide (H_2O_2) to the simulant and mixing it over a period of about 2 minutes to assimilate the peroxide into the simulant. The mixing is then ceased and the oxygen generated in-situ from peroxide decomposition was allowed to accumulate in the simulant. The simulant was allowed to gel for 18 hours. (b) At the end of the 18-hour gel period, the simulant was mixed to measure the gas release rate. The physical characteristic of the in-situ generated gas through peroxide decomposition was presumed to resemble that of hydrogen accumulated from radiolysis in the gelled waste.

Test Matrix In-Situ Generation

A total of four experiments were conducted under different operating conditions. The initial conditions, including the targeted gas hold-up based on stoichiometric calculations assuming completion of hydrogen peroxide decomposition are given in (Table III)

Table III. Test Matrix for In-situ Generation of Oxygen

Test	Simulant	$V_{simulant}$	V_{oxygen}	Holdup, L	Holdup, %	T, °C	Mixer speed, RPM
C01	Clay	22.23	2.36	1.51	6.79	24-25	198-208
C02	Clay	22.27	2.00	1.42	6.36	23-25	196-207
C03	Clay	22.33	2.41	1.54	6.89	22-23	197-207
C04	Clay	22.17	2.07	1.32	5.95	23-24	194-200

Oxygen signatures during release for experiment C01 and the corresponding rates are shown in Fig. 3.
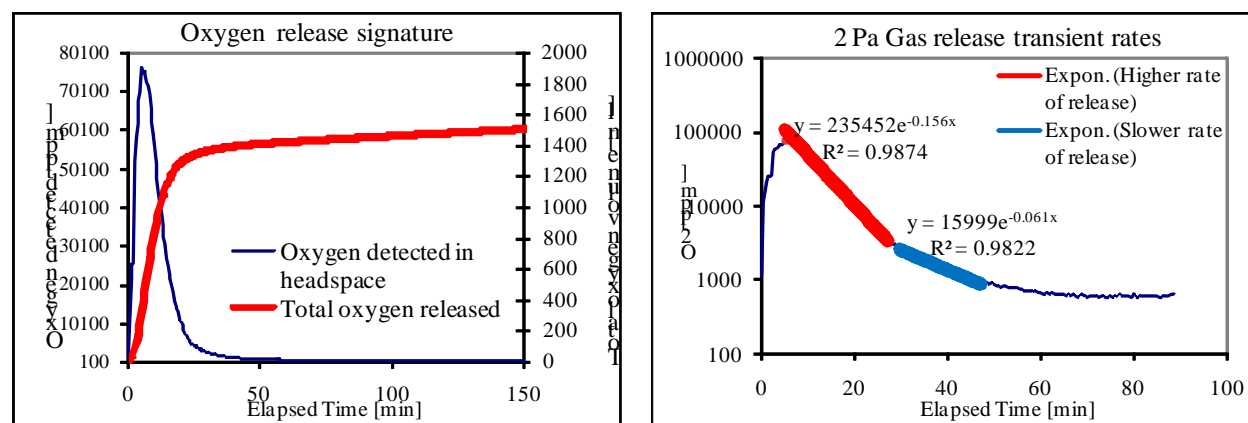

Fig. 3. Oxygen release signature-C01 (left) and Oxygen release kinetic parameters (right).

RESULTS AND DISCUSSION

The level of oxygen hold-up in the gelled simulant was controlled by the amount of hydrogen peroxide added to the simulant before gelling. The tests were planned to measure release rates from 2 levels of hold-up. The gel hold-up levels achieved during these tests and released volumes are summarized in Table IV. These were estimated from the effluent gas oxygen and nitrogen concentrations and the purge gas injection rates.

Table IV. Summary of In-situ Gas Retention and Release Test Results for Kaolin-Bentonite

Test	Test Mode	Simulant	Yield Stress [Pa]	V_{oxygen} [L]	Hold up [%]	O_2 released [%]
K01	Gas Retention Gel Release	Clay	2	2.36	7.45	99.34
K02	Gas Retention Gel Release	Clay	4	2.00	7.58	99.08
K03	Gas Retention	Clay	6	2.28		

	Gel Release				6.36	99.93
K04	Gas Retention	Clay	8	2.24		
	Gel Release				6.25	98.05

Analysis of the release signature curve shows that gas is released at two different rates representing two different regions; a fast release rate with a short time constant due to direct mobilization of the slurry wherein the majority of the gas gets released (as evident by a rapid linear drop) followed by a slower release rate with a longer time constant representing the erosion process wherein the gas is released slowly at a constant rate due to a less well mixed or unmixed slurry.

The initial rates of release for the kaolin-bentonite tests for the yield stress range of 2-10 Pa were calculated by fitting the release signature curve by an exponential trend. The release curve follows the first order kinetics. It was assumed that the gas release is the combination of two release rates each with a different peak rate and time constant.

For the estimation of gas release parameter in clay simulants, four clay simulants were tested for gel release behavior with varying rheology: (i) 14 wt % Clay simulant (2 Pa yield stress) (ii) 17 wt % Clay simulant (4 Pa yield stress) (iii) 19 wt % Clay simulant (6 Pa yield stress) and (iv) 22 wt % Clay simulant (8 Pa yield stress). The 17 wt% clay simulant has about 50% of the yield strength of the 22% Clay simulant. Therefore, it would be reasonable to expect higher release rate from the 17 wt% clay simulant than the 22 wt % Clay simulant under similar initial gas hold-up based on bubble rise velocities. This trend cannot be clearly noted in the release rates measured for the two simulants at an initial gel hold-up of about 6.3% vol. The estimated gel release rates for the clay simulant from the 1 hold-up levels are shown in
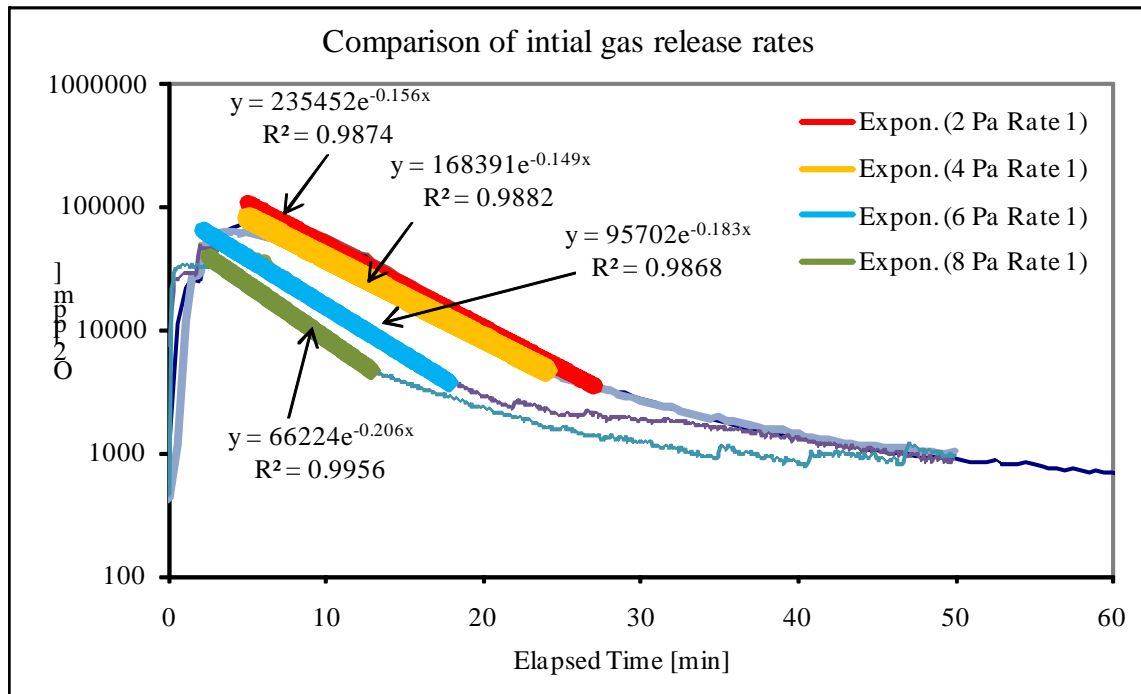

Fig. 4. The 2 Pa clay simulant takes about 15 minutes to release 60% of the total hold-up compared to the 10 minutes it takes for the 6 Pa and 5 minutes it takes for the 8 Pa to release about 80% of the total hold-up.
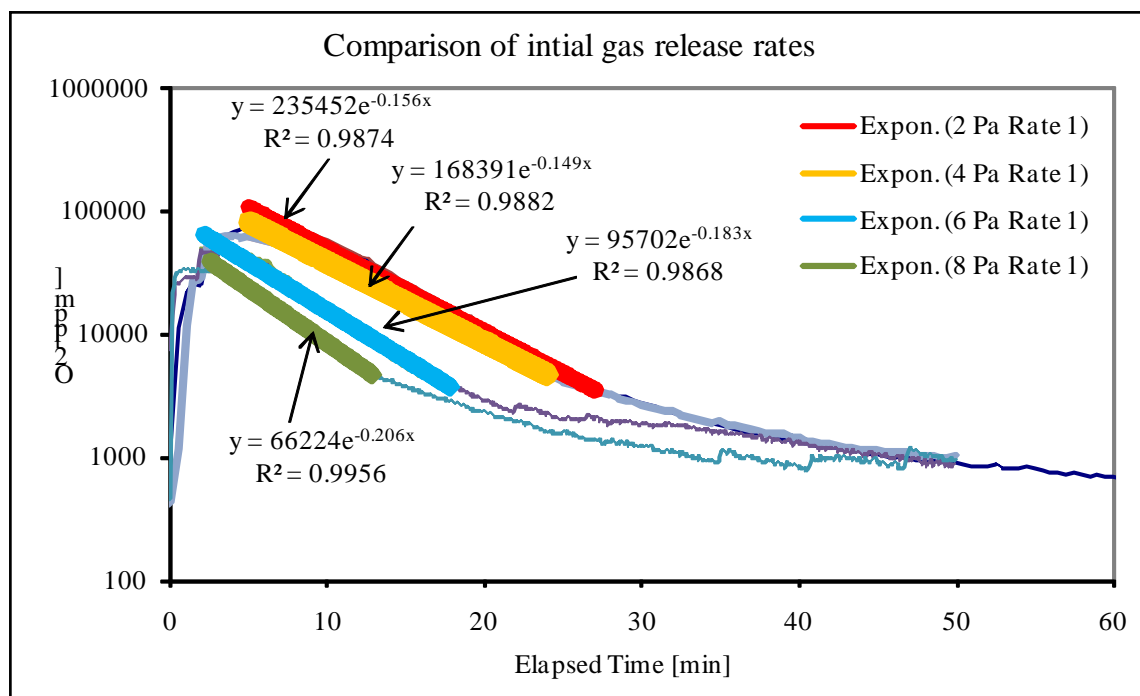
Fig. 4. Total volume of gas detected from release.

The gel release rate estimates clearly point to an initial period (on the order of 15 minutes) of relatively higher rates of release, upon commencement of mixing. Furthermore, the estimates indicate that this period of release was more prolonged at the lower yield stress with similar initial hold up. The higher initial release rate is attributed to greater coalescence of the smaller gelled bubbles into larger bubbles, and the higher spatial density of bubbles in the simulant at the commencement of mixing. The gradual drop in the release rate is attributed to depletion of the larger bubbles and to lowered spatial density of the bubbles.

Next, the volume of oxygen released was normalized relative to the total volume released over the entire release period. An assumption that 100% of the held-up oxygen is released over the duration of the release period is made. Under this assumption, a clear order seems to appear in the rate of release of oxygen. Clearly, in the initial surge of gas release upon commencement of mixing, a greater fraction of the hold-up is released with higher yield stress simulants. In the tests, within about 15 minutes approximately 60-85% of the hold-up was released. Table V summarizes the more important parameters that have been determined from the experimental program.

Table V. Summary of Release Parameters

Experiment	Simulant	$k_{1, 1/min}$	$k_{2, 1/min}$	O_2 Peak	Volume^2, mL	Volumetric Fraction released
C01	Clay 14 %	0.156	0.061	76%	860	3.87%
C02	Clay 17 %	0.143	0.075	63%	720	3.23%
C03	Clay 19 %	0.183	0.033	48%	509	2.28%
C04	Clay 22 %	0.205	0.089	32%	353	1.59%

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[2] Volume released in the first 15 minutes

CONCLUSIONS

The experimental program initiated at Florida International University extends experimental studies previously conducted at SRS (Savannah River Site) and PNNL (Pacific Northwest National Laboratory) by addressing the transient behavior of gas release, retention and release patterns for mixtures with yield stress lower than 5 Pa, and provides experimental correlations between release rates.

For the in-situ generated gas, a substantial fraction (65 to 85%) of the gas held up is released in a surge of gas release during the first 15 minutes after the commencement of mixing of the gelled simulant. The gel release rate estimates clearly point to an initial period (on the order of 15 minutes) of relatively higher rates of release, upon commencement of mixing. Furthermore, the estimates indicate that this period of release was more prolonged at the lower yield stress with similar initial hold up. The higher initial release rate is attributed to greater coalescence of the smaller gelled bubbles into larger bubbles, and the higher spatial density of bubbles in the simulant at the commencement of mixing. The gradual drop in the release rate is attributed to depletion of the larger bubbles and to lowered spatial density of the bubbles.

Approximately 95 to 98% of the total gas release occurs in the first 120 minutes from the start of gel mixing. Also, virtually all the gas held up was released within 180 minutes, the test time period used for monitoring the release.

Even though significantly different in rheology, the clay simulant with varying rheology tested exhibit similar gas release behavior within the range of initial gel hold-ups in these tests. Additional tests will be carried out with laponite, xanthan gum and AZ-101 with mixtures of anti-foam agents and the release profiles will be compared to that of clay. Moreover the effect of AFA on the gas release behavior for each of the selected simulant will be investigated.

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