AIP | Review of Scientific Instruments

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Citation: Review of Scientific Instruments **73**, 2479 (2002); doi: 10.1063/1.1480451
View online: http://dx.doi.org/10.1063/1.1480451
View Table of Contents: http://scitation.aip.org/content/aip/journal/rsi/73/6?ver=pdfcov
Published by the AIP Publishing

REVIEW OF SCIENTIFIC INSTRUMENTS VOLUME 73, NUMBER 6 JUNE 2002

Design of a *z*-axis translating laser light scattering device for particulate settling measurement in dispersed fluids

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(Received 31 August 2001; accepted for publication 30 March 2002)

A user friendly, Labview™ controlled, prototype settling device has been designed and built that incorporates a laser light source and detector fixtured to a *z*-axis translating stage. The Labview™ data interface drives the unit and captures data in the form of scattered intensity as a function of *z*-axis location. We present some examples of sample output from low- and high-density particles settling in epoxy fluids of various viscosities. This device maps all of the expected settling regimes and, more importantly, valuable scattering information about partial settling is found in instances without a discrete mud line. The effect of resin viscosity on settling has been measured and corresponds well to model predictions. Measurement of settling data in the compression regime was also achievable, validating descriptive models. © *2002 American Institute of Physics.*

[DOI: 10.1063/1.1480451]

I. INTRODUCTION

Sedimentation is commonly observed in both industrial and natural situations. Settling occurs with waxes in crude oils,[1] mining separations involving flocculation and/or froth floatation,[2,3] paints, adhesives,[4,5] and other slurry mixtures where particles are all suspended in liquid mixtures. The most common settling processes are seen in water treatment facilities where settling tanks separate solid particulates from the millions of gallons of water processed through a plant each day.[4–6] Natural settling also occurs with dispersed slit flowing into lakes and ponds[7] following rainfall. Closely related is the coagulation of oceanic plankton, which is linked to the presence of suspended minerals.[8,9]

The settling of particles through a fluid due to a driving force makes accurate characterization daunting. Settling suspensions can be grouped into four categories.[4] Class-1 clarification occurs when dispersed particles settle as single particles in a fluid, while Class-2 clarification results from flocculent particles settling. With more concentrated solutions, hindered hydrodynamic forces can hold particle clusters closer together. In ''zone settling,'' the particles fall out of suspension as a front, with a discrete boundary called a mud line. Highly concentrated slurries act more like porous media through which liquid matrix is further compressed by the particles mass. This type of settling is called compression.

For a single dense particle falling out of a less dense solution, its terminal velocity U in the low Reynolds number regime, can be generally expressed as[10]

$$U=\frac{(\rho_p-\rho_l)Vg}{3\pi\mu d}, \tag{1}$$

where ρ_p and ρ_l are the densities of the particle and liquid phase, respectively, V and d are the particle volume and diameter, respectively, μ is the velocity of the fluid, and g is the gravitational constant. After initially accelerating, the particle's vertical position moves linearly with time. The same constant velocity is observed for nonaggregating rigid particles falling ''en masse'' in the zone regime.

Settling has been assessed experimentally by several techniques such as batch settling tests, aliquot sampling at various depths, and sedimentometers. Batch settling columns require a strong boundary between the settled and dispersed zones. With aliquot sampling,[4] measurements of particulate concentration with depth are direct and do not require a strongly identifiable mud line, but the fluid is disturbed during extraction.

Sedimentation analyzers[11] consist of reeds and plates submerged in the settling mixture column. As particles settle, some collide with the reed and the plate. A connected detector analyzes the minute movements of the plate. The sensitivity of the unit is based on the number of plates. While sensitive, the presence of the reeds may also upset the normal settling flow pattern. Other devices[12] identify the maximum gradients in light transmission that would occur at the mud line. The unit is connected to a transducer translating with the largest gradient in transmission with depth, yielding a mud line settling velocity. Thus one location is identifiable with this technique neglecting other settling processes away from the largest gradient in sediment.

The identification of a mud line and a corresponding mud line velocity are key to defining the rate of overall settling to which models have been ascribed. Complicating factors arise when there is no discernable mud line. Similarly, there may also be settling gradients within both the clarified and settled zones. No current unit allows for a more detailed interpretation. It was with these limitations in mind that a new type of measurement tool was designed and built.

Like the latter technique, our device is based on light

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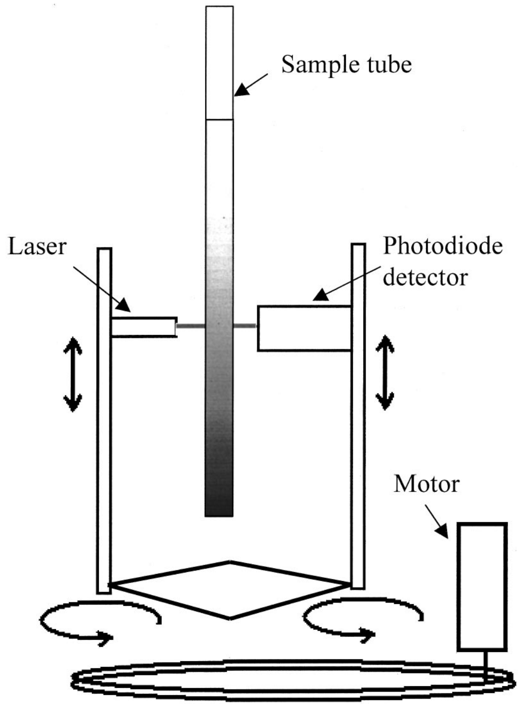

FIG. 1. Diagram of the settling characterization device.

scattering: a large enough difference in the index of refraction between fillers and matrix yields a scattering profile in which larger concentrations of particles lead to more scattering. Calibration curves are required to associate a particle concentration value to the measured scattering data. Once that is established, maps of the particle concentration can be created by measuring light transmittance across an unknown sample's depth. Particle front movement can be tracked by continually remapping the sample. Results validating our prototype machine's capabilities are presented. The tests include epoxy resin formulations mixed with either encapsulated imidazole particles or carborundum particles.

II. EXPERIMENT

A. Basic device layout

A schematic of the unit is drawn in Fig. 1. The machine is comprised of a measurement platform containing a 5 mW red laser operating at 635 nm (NVG Inc.), insulated from the chassis, and a silicon photodiode (Melles Griot Corp.). The entire platform is mounted on a threaded machine nut on a pair of threaded machine rods. A motor underneath the platform turns these threaded machine rods to raise or lower the measurement platform along the height of the sample column. The power from the device is supplied via a 24 V power supply, controlled by four solid state relays.

Labview™ provides the 5 V power required to feed the laser. The silicon photodiode is mounted on an adjustable protractor on the measurement platform. The photodiode has angular control to record scattered and transmitted light, sending intensity to the controlling computer as an analog voltage signal. Labview™ also receives user inputs that define and control the sampling rate and the stage amplitude.

Stage height is tracked using a magnetic counter relating the number of rotations the threaded machine rods make. The system can record data at 1 mm intervals. The entire apparatus is covered to eliminate spurious ambient light.

B. Materials

All tested resins contained a mixture of EPON® Resin 828 epoxy and HELOXY® Modifier 107 (Shell Inc.). The epoxy is a bisphenol A/epichlorohydrin based resin, with an ambient viscosity of about 20 000 cP and a density of 1.304 g/cm^3. The viscosity modifier is a diglycidylether of cyclohexane dimethanol, with a viscosity of about 60 cP and a density of 1.092 g/cm^3. Both fluids are slightly beige colored but clear.

Two types of particles have been used. First, wax encapsulated imidazole catalyst particles (Intelimer® 7004) were provided by Landec. These beige particles have a mean diameter of 10 μm and a density ~0.308 g/cm^3. Thus, they settle upward in the fluid. Denser black SiC particles (2.297 g/cm^3) with mean size of 2 μm were also used.

For each particle/resin-mixture-composition combination, several samples with known particle concentrations were measured, providing calibrations curves to convert the voltage signals measured by the photodiode into concentration values.

III. RESULTS AND DISCUSSIONS

Four tests have been run to demonstrate machine capabilities. The first test compared the machine's measurements with visible observed trends in an epoxy–SiC mixture. The second test proved the device's ability to detect concentration changes where none are visually observable using an epoxy–catalyst particle mixture. Epoxy–SiC dispersions with various resin viscosities were then used to demonstrate the ability to measure a settling velocity. A final test where an epoxy–catalyst mixture was allowed to settle completely demonstrated the machine's ability to measure all the settling regimes without an observable mud line.

A. Proof of concept

For the first test, two settling samples were constructed and recorded simultaneously, one photographically and the other with the settling device, at 10 min intervals. The samples were a 50/50 mixture of EPON 828 and HELOXY 107 with 6 wt. % SiC particles. These particles are denser than the resin, settling towards the bottom of the vessel, the results of which are shown in Fig. 2. The settling scans were performed between the horizontal line drawn on the pictures (mark 0 in height) and the black tape on the top of the settling vessel (top of the pictures). On the recorded scans, the feature at the extreme right corresponds to the black tape. Then, moving left, the light was transferred without absorption through the air gap above the resin. The protuberance in the recorded signal corresponds to the fluid meniscus at the top of the sampling column. This first part of the concentration/height curve is identical for the scans at the different times. The left part of the scans shows lines of decreasing slope for increasing time. This corresponds to the

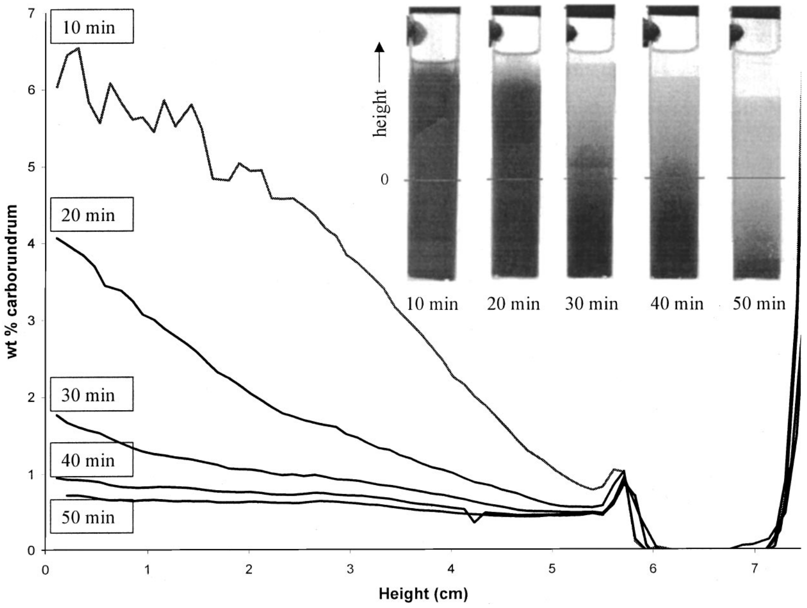

FIG. 2. Comparison of device recorded data and visual observation of carborundum particles settling in the resin.

gradual decreasing concentration of the top part of the settling vessel as particles settle down. This change in particle concentration can be observed visually on the pictures. What is noticeable both on the pictures and the scans is a mud zone rather than a discrete mud line. This first test with color contrasting particles shows the reality of the settling scan features. It also shows the difficulty in defining an absolute mud line, one reason why traditional batch settling lacks precision.

B. Pushing down the sensitivity limit

The second test used the same idea of recorded scan/picture comparison, and was recorded with the settling of 1 wt. % catalyst particles in a mixture of 10% EPON 828 and 90% HELOXY 107. These particles settled upward in the mixture. The settling scan and corresponding picture are displayed in Fig. 3. Features at the top of the scan are associated with the black tape and air gap on the settling vessel picture. Foam at the top of the fluid was created by air bubbles trapped in the viscous resin and is shown as a sharp peak on the settling scan. Below that surface foam, the resin/particle mix appears homogeneous on the picture and visually. On the other hand, a concentration gradient is observed on the settling scan, with the particle concentration increasing from the bottom to the top of the vessel. This test shows that the settling device can observe sedimentation even when no mud line exists.

C. Effect of viscosity on measured settling

Three different viscosities were obtained by changing the amount of modifier in the resin mix: 60, 108, and 384 cP. The settling behavior of 5 wt. % SiC particles in these three resin mixtures was recorded with the settling device using short, 1.5 min time intervals between the scans. From the concentration/height curves, isoconcentration heights at each

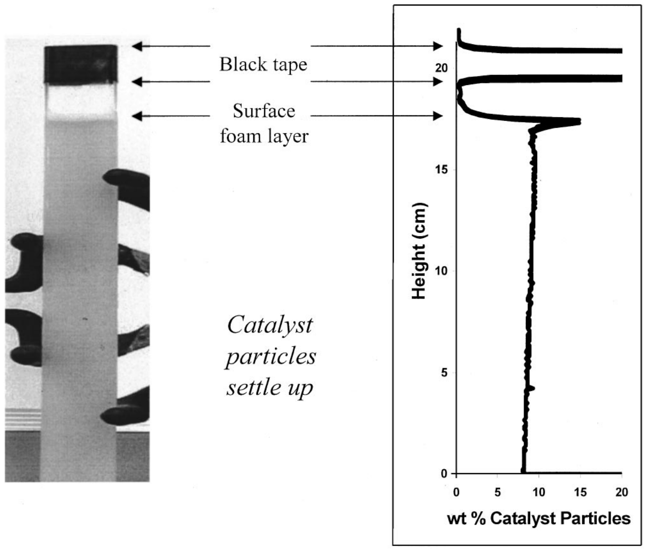

FIG. 3. Comparison of device recorded data and visual observation in case of no visible mud line.

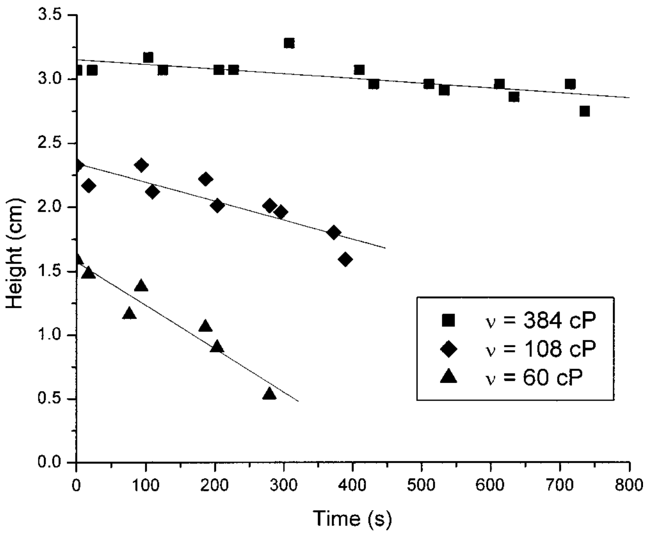

FIG. 4. Settling curves for three resin viscosities.

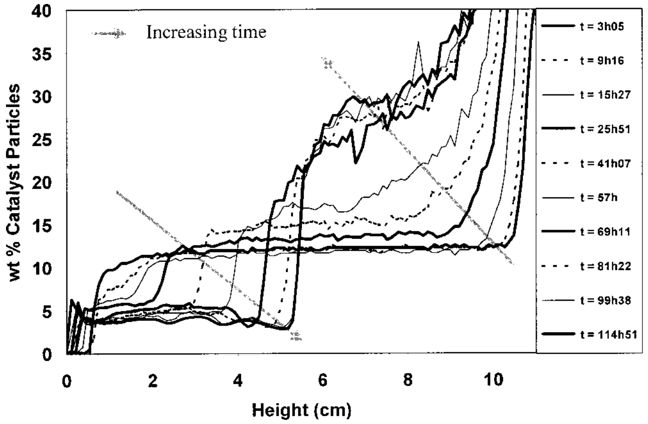

FIG. 5. Successive settling scans of catalyst particles settling in the resin (only selected scans are shown for clarity).

recorded time have been extracted. Figure 4 shows the height vs time data corresponding to a concentration of 1.5 wt.% for the three resin viscosities. First, a linear variation of the height as a function of time is obtained for the three viscosities, corresponding to a constant settling rate, which is expected with zone settling. Second, these constant settling rate values decrease with increasing resin viscosity, fitted by a power law with an exponent of -0.92. Comparing this result with the expression for the terminal velocity for low Re number particle transport [Eq. (1), $U \propto \mu^{-1}$], the SiC/modified epoxy resin mixture is described successfully by this model. This result also shows that the device yields a precise, quantitative measurement of settling.

D. Full picture of the settling process

For the final test, the concentration in a mix of 98% modifier and 12% catalyst particles was recorded every 3 h during a week using the settling device, allowing ‘‘complete’’ settling to occur. Only a select series of scans are shown in Fig. 5. Since the resin is denser than the particles, the concentration of the half-top of the settling vessel (right-hand side of the graph) increases with time, while that of the half-down (left-hand side) is decreasing. These settling curves include several interesting features. First, the clarified fluid still contains ~4% of particles. Second, a sharp drop in concentration marks the transition between the supernatant fluid and the settled particles, but is still not visible to the naked eye because of the resin opacity and the color similarity between particles and resin. Finally, the compaction zone is marked by a noisier signal, which may correspond to the ‘‘volcano’’ effect described by Pearse,[13] by which the fluid still trapped within the particles is evacuated through channels. Height/time values corresponding to a concentration of 8% of particles have been extracted from the recorded settling scans and are plotted in Fig. 6. The first part of that height vs time curve is linear, clearly associated with zone settling. After 2 days (5000 min), the settling rate transitioned towards a constant height, characteristic of the compaction regime. A closer look to that part of the curve should allow the extraction of the slow variation of the isoconcentration heights vs time.

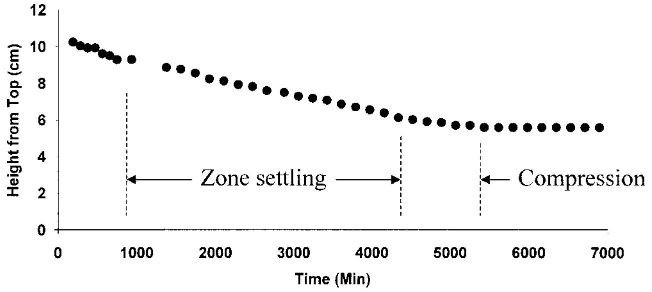

FIG. 6. Settling curve for the catalyst particles in the resin.

A new settling characterization device that can accurately and precisely measure the more comprehensive settling trends of a fluid dispersion has been successfully demonstrated. Measurements made with it correspond very well to visually observable trends. Even where no mud line was visible, the settling device extracted particle concentration gradients in the fluid. As a way to validate the quantitative results obtained from the settling scans, the effect of the resin viscosity on the particle settling rate has been measured and corresponds to what sedimentation theories in the low Re number regime predict. In addition, a quantitatively precise picture of the particle concentration in the fluid over the whole settling vessel can be obtained. This device may lead to a more precise way to measure settling in the compression regime, where the particles are moving much more slowly in the fluid. The settling device described here should find use in numerous applications and allow for a more accurate observation of settling.

ACKNOWLEDGMENTS

The authors thank Dr. Gray Pickrell and Dr. Russ May of the Virginia Tech Photonics Group for their help and useful discussions. We also acknowledge the National Science Foundation Summer Undergraduate Research Program (SURP) for the support of D.L.H. We would also like to acknowledge the Office of Naval Research (ONR No. N000014/99/1/0946) for support of other components of this research.

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