Advanced Particle Sensors LLC

particle characterization for the real world ™

Review: In-Process Characterization of Suspensions and Slurries

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Abstract

This presentation provides highlights of a comprehensive literature review of new and emerging methods for in-process characterization of suspensions and slurries. Most of the 165+ reviewed publications (selected from a field of over 3200 search hits) describe variations of known methods or ideas but nonetheless represent current applications. Innovative or otherwise noteworthy developments are identified in this presentation and will be described more fully in the report.

Outline of Presentation

(Numbering follows the outline presented at the Winter meeting in Philadelphia.)

Scope of Review

New and emerging methodologies for in-process characterization of suspensions and slurries, encompassing

- Particle size distribution: from nm to mm, including detection of very small particles in the presence of large particles
- Particle structure: shape, porosity, fractal dimension, density, etc.
- Suspension/slurry density, gas volume fraction, phase distribution
- Rheological properties

Automated methods for taking and preparing samples for analysis are also in scope.

4.1 PSD via Acoustic Methods

High level of activity in this area, with few recent innovations

Acoustic Emission (from particle impacts, larger particles create louder sound)

- AE is well-established in minerals processing, with intrusive and non-intrusive clamp-on sensors. A recent patent to CiDRA (Newton and Fernald 2019) measures particle size in a grinding circuit by comparing the power spectrum of the sound to an empirical model. Percent oversize is estimated.

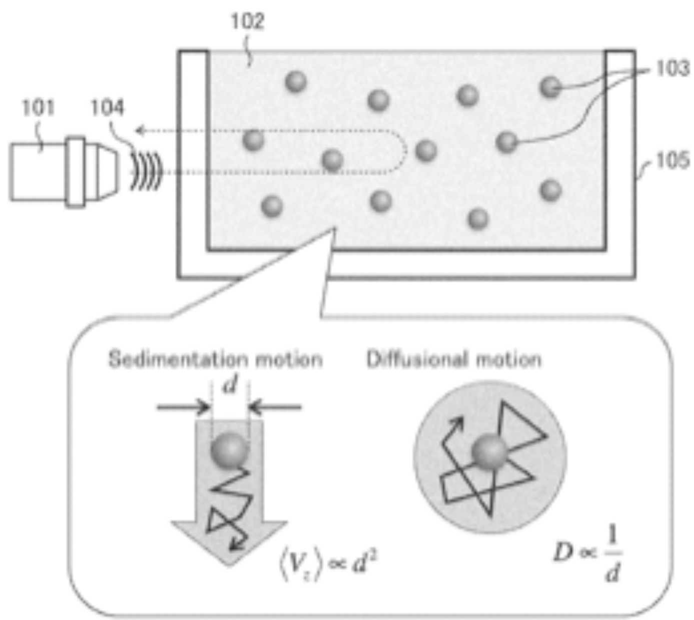
CiDRA PST mounted on hydrocyclones (cidra.com)

- A more rigorous approach was investigated at Strathclyde using a finite element code and Hertz contact theory to analyze particle collisions with the vessel wall (Tramontana et al. 2015). Experimental validation was provided using 3 broad size classes covering 0-850 μm. There have been no follow-up studies citing this work.

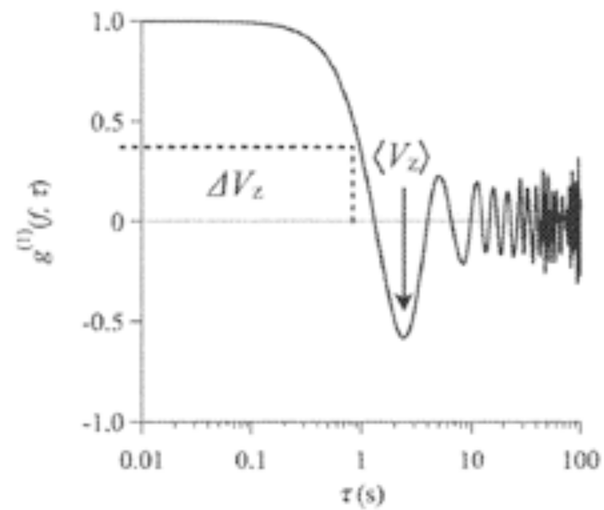
PSD via Acoustic Methods, cont'd

Dynamic Sound Scattering (ultrasonic analog of Dynamic Light Scattering)

- Proposed conceptually by Bodner and Inozemtsev (1977)
- Various authors have discussed measurement of particle velocity
- Norisuye (2018) patented an implementation that can monitor particle size over 3-30 μm in opaque, concentrated, sedimenting systems.



Norisuye, Fig. 16



Norisuye, Fig. 3c

4.4 PSD via Optical Methods

4.4.1 Absorption and spectroscopy

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- Statistical Extinction Method (Dannigkeit et al. 2016; Schwarz et al. 2018) measures mean size (1-1000 μm) up to 30 vol%, by analyzing transmission fluctuations in a collimated light beam with a spatial filter or pinhole detector.
- Raman spectroscopy has been used to measure size in suspensions and emulsions (Owen 2018; Houben et al. 2015).

4.4.3 FBRM & 1-D Optical Scanners (chord length distributions)

- Deconvolving the PSD from CLD data requires an instrument response model that is typically empirical (e.g. Kernick 2018). Irizarry et al. (2017, 2020) create such models based on the response for 5 different size fractions.
- Szilaǵyi and Nagy (2018) provide a fast simulation of CLD for a given PSD (the "forward problem") for control of crystallization.
- Other applications include contamination detection in drilling mud and monitoring flocculation in a minerals grinding circuit.

PSD via Optical Methods, cont'd

Interferometry

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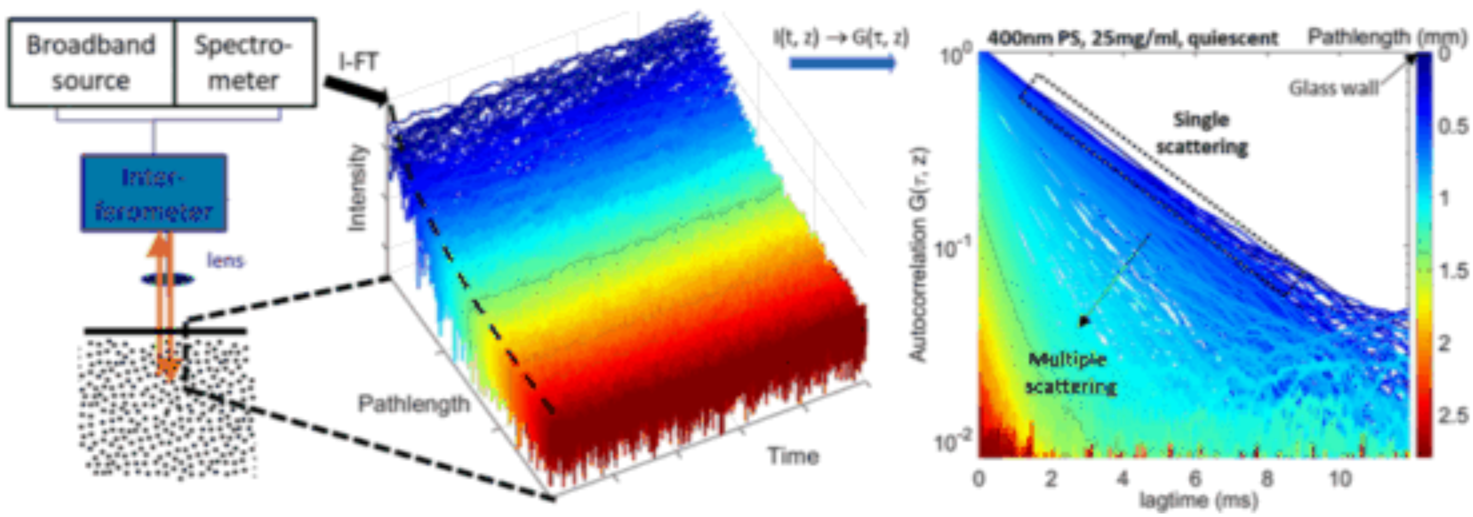
- InProcess-LSP (www.inprocess-lsp.com) introduced "NanoFlowSizer" based on Spatially Resolved Dynamic Light Scattering (Besseling et al. 2019) to measure size in turbid flowing nanosuspensions.



Besseling et al., Fig. 1

- A similar approach is described by Guzman-Sepulveda et al. (2015), who used a fiber coupler rather than an interferometer (fixing the path length).

PSD via Optical Methods, cont'd

Scattering / Diffraction

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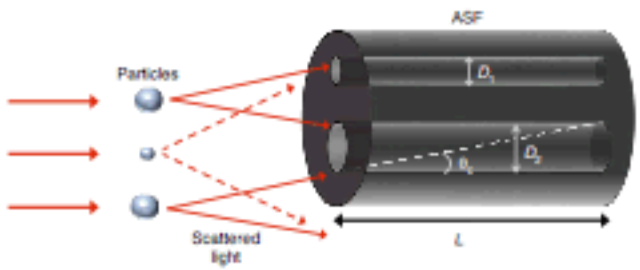
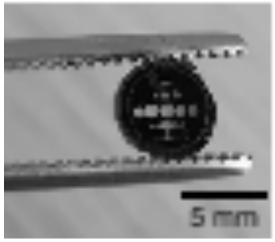
- Hussain et al. (2020) propose a cheap and compact size analyzer based on a custom Angular Spatial Filter (Pruneri et al. 2018) and a CMOS camera. Sizes 13μm to 150μm at concentrations up to 4 wt% were measured.





Hussain et al., Figs. 1 & 2

- Wyatt (2018) describes how to extend the Rayleigh-Gans approximation (usually limited to sizes <50 nm) to 2000 nm by extrapolating the forward ($\theta=0°$) intensity from measurements at multiple scattering angles.
- Several patents were filed or issued on "next generation" improvements to existing diffraction instruments (to be listed in the report).

5.2 Particle Density

- Guzman-Sepulveda et al. (2015) use low-coherence DLS to determine the Stokes' settling velocity in colloids, from which mass density is calculated. See also Ishii et al. (2010). Time scale is hours!
- Al-Lashi and Challis (2015) discuss the application of ultrasonic spectroscopy to suspensions of solid particles in order to estimate density, if the other properties are known.
- For oceanographic applications, Hurley et al. (2016) use a Sequoia Scientific (sequoiasci.com) submersible laser diffraction system to estimate the mass density of particles by assuming beam attenuation is proportional to density.

5.3 Fractal Dimension

Imaging Techniques

- Fractal dimension is commonly determined by image analysis, e.g. of flocced sediment in vessels (MacIver & Pawlik 2017); capillary suspensions (Bossler & Koos 2016, Bossle et al. 2018); and coagulation of wastewater (Ratnaweera & Fettig 2015, Ren et al. 2017).
- Yu (2014) demonstrated the use of a 3-layer artificial neural network to analyze output from an imaging system and predict suspended solids removal efficiency for chemical coagulation in wastewater treatment plants.
- Cao et al. (2014) have extracted fractal dimension with digital holography to monitor flocculation of algal blooms with clay.

Fractal Dimension, cont'd

Optical Methods

- Small-angle light scattering (Jung et al. 1995) is an established technique for estimating fractal dimension. Kuśnierz & Wiercik (2016) applied it to diluted sludge, and Bowers et al. (2017) used it to study marine flocs.
- Expósito et al. (2019) have proposed a new method to estimate fractal dimension of microalgal cultures by correlating the suspension chord length distribution with the flocs average geometry through a machine learning model.

5.5 Shape

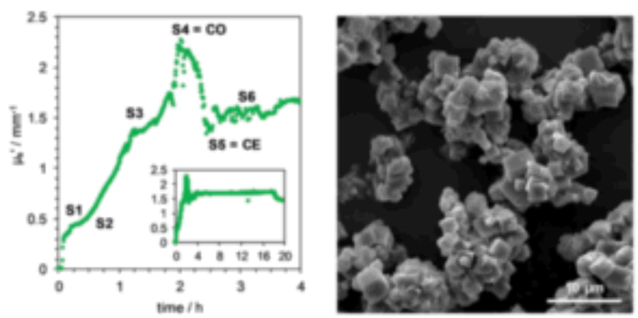
Imaging Methods

- Huo et al. (2016) propose a strategy for in-situ crystal shape and size measurement for monitoring crystallization, including a new shape feature index based on the COV of distance from perimeter points to the centroid.
- Cardona et al. (2018) tackle the issue of removing out-of-focus objects from the particle statistics, using the Gaussian derivative method of Geusebroek et al. (2000) to assess the focus.
- Lu et al. (2018) address the challenge of image segmentation in the presence of varying backgrounds and bubbles in a 4 L crystallizer.
- ★ Heisel et al. (2019) built a neural network to discriminate between single crystals and agglomerates independent of shape.
- Several groups have used stereo imaging to obtain 3D crystal shape (Rajagopalan et al. 2017), measure agglomeration (Huo et al. 2017), or estimate growth kinetics (Zhang et al. 2017).

Shape, cont'd

<u>Optical Methods</u>

- Mostafavi et al. (2014) noted that particle shape can influence the results of many on-line characterization techniques. Turning this to advantage, <u>aspect ratio</u> has been measured from laser diffraction data (Scott & Matsuyama 2014, Villa et al. 2016) and – in theory – angularly-resolved Vis-NIR spectroscopy (Stoliarskaia et al. 2019).
- Aspect ratio of gold nanorods was estimated via DLS by taking the ratio of the rotational and translational decay rates (Muschol 2014).
- ★ Häne et al. (2019) used Photon Density Wave spectroscopy (Fishkin et al. 1996, Bressel et al. 2013) to monitor particle formation of zeolites in a highly turbid suspension, noting that the reduced scattering coefficient becomes constant when no changes occur in particle size, number, <u>or shape</u>.

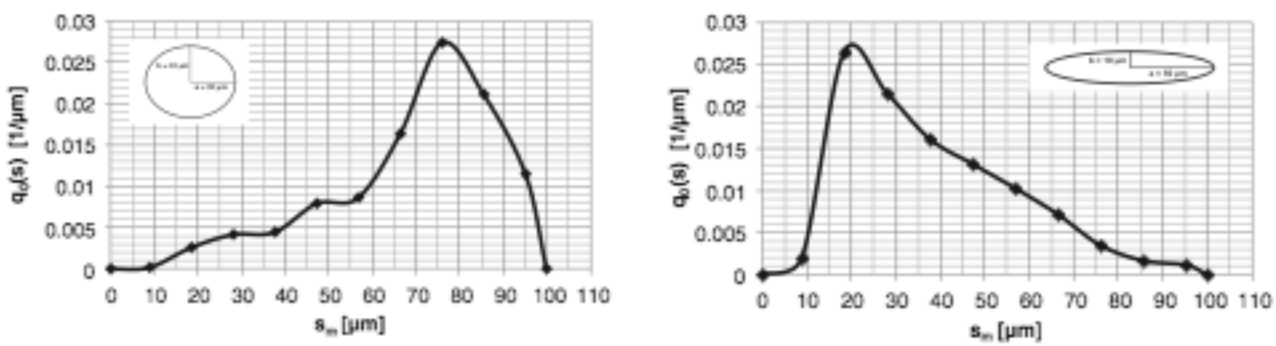


Häne et al. Fig. 2

Shape, cont'd

Optical Methods, cont'd

- Petrak et al. (2015) applied geometric reasoning to extract particle shape and aspect ratio from hypothetical chord length distributions generated by the Parsum probe (not taking instrumental bias into account). Parsum has a patent on this approach (Dietrich et al. 2018).



Petrak et al.
Figs. 6 & 7

Ultrasonic Method

- Ivanov et al. (2017) measured the diameter and aspect ratio of carbon nanotubes by comparing attenuation spectra of the colloid at rest (isotropic orientation) and in laminar flow (aligned particles).

6.1 Density of Suspension/Slurry

- Mechanical methods include measuring the weight of the slurry (Moth 2019) and monitoring the resonance frequency of a piezoelectric tuning fork in contact with the process slurry (Gonzalez et al. 2017).
- Recently, Lin et al. (2020) used a Neural Network to extract slurry density from Raman spectra in a 25 wt% paracetamol / ethanol suspension. [This approach requires an enormous effort to set up new applications.]
- Density can be measured from the ultrasonic reflection coefficient, which depends on the acoustic impedance (density x sound speed) of the slurry. Recent advances include a spool piece deign for large (<70 cm) steel pipe (Greenwood 2015) and application to cement slurry in undersea drilling (Cordeiro et a. 2016).

6.2 Gas Volume Fraction in Slurry/Suspension

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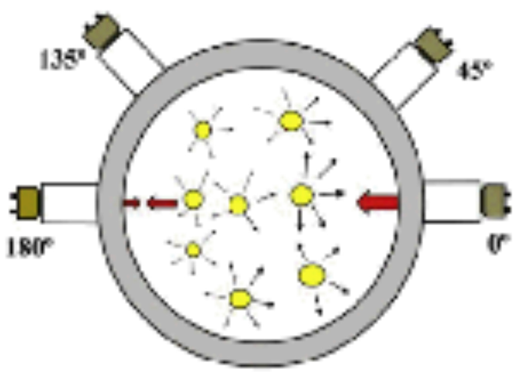
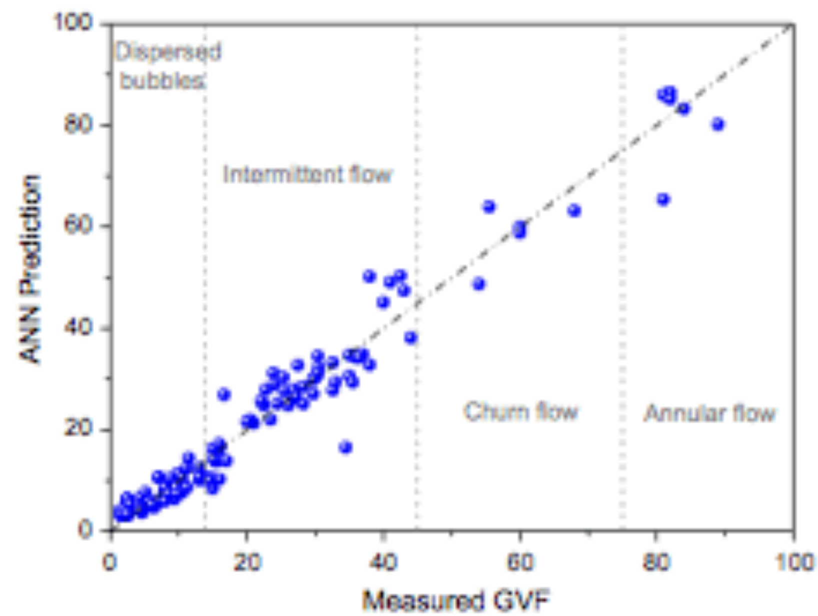
- An interesting approach (Figueiredo et al. 2016) recorded ultrasound scattered at 45, 135, and 180 degrees in a mutiphase flow experiment, then used a 4-layer neural network to extract gas volume, with surprisingly good results. [Note: the Atkinson-Kytömaa attenuation model they use is invalid over their range of frequency and particle size! Since they mention “the solids concentration is very small”, one assumes the particles contributed little to the total ultrasonic attenuation.]

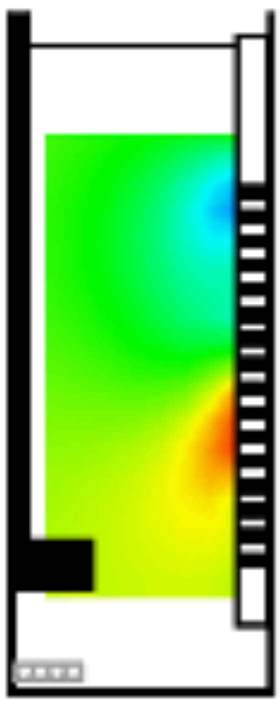




Figueiredo et al., Figs. 8 & 22

Gas Volume Fraction, cont'd

- Forte et al. (2019) demonstrated that a linear-probe ERT system (Industrial Tomography Systems, itoms.com) can measure axial profile of gas fraction and the global gas hold-up in a stirred tank, with minimal influence from the presence of particles in a gas/solids/liquid system.



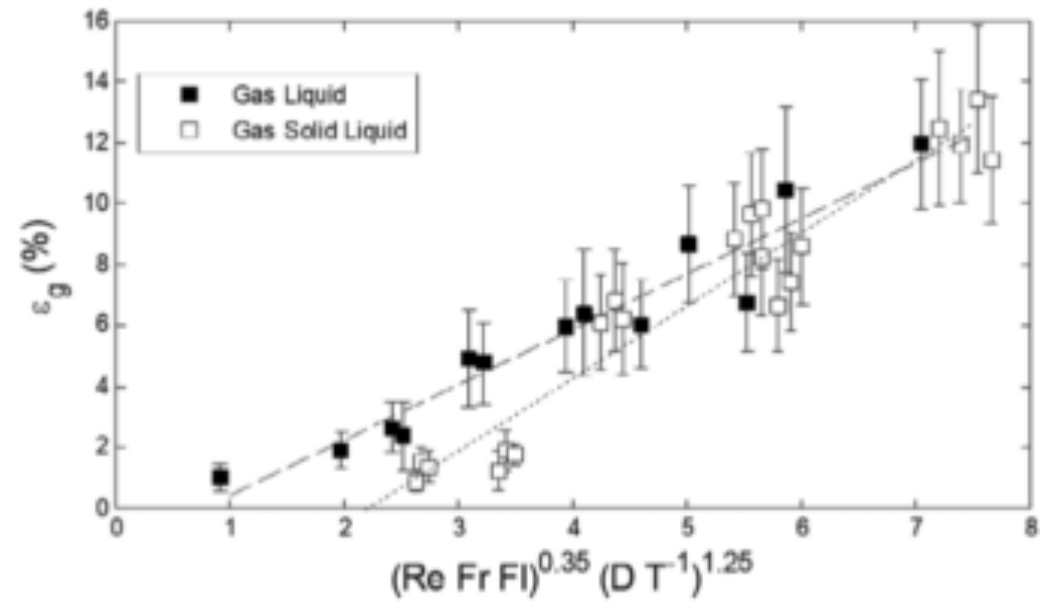


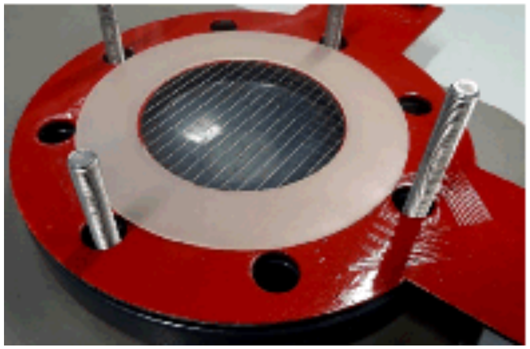
FIGURE 11 Global gas hold-up trend for two-phase and three-phase systems Solids concentration is 5 wt%

Forte et al., Figs. 6e & 11

6.3 Phase Distribution in Slurry/Suspension

Electrical Sensing

- Dos Santos et al. (2015) used invasive electrical mesh sensors to measure solid concentration distribution in slurries at 4000 frame/s with a spatial resolution of 6.25 mm. Tests used 250 micron sand at 1-35 vol%.



Dos Santos et al., Fig. 4

- Electrical Resistive Tomography (ERT) has long been used to measure concentration profile and homogeneity of mixing (e.g. Mann et al. 2001). A recent example is the measurement of slurry concentration profiles in a reactor to evaluate mixing performance of a particular impeller design (Mishra & Ein-Mozaffari 2016).

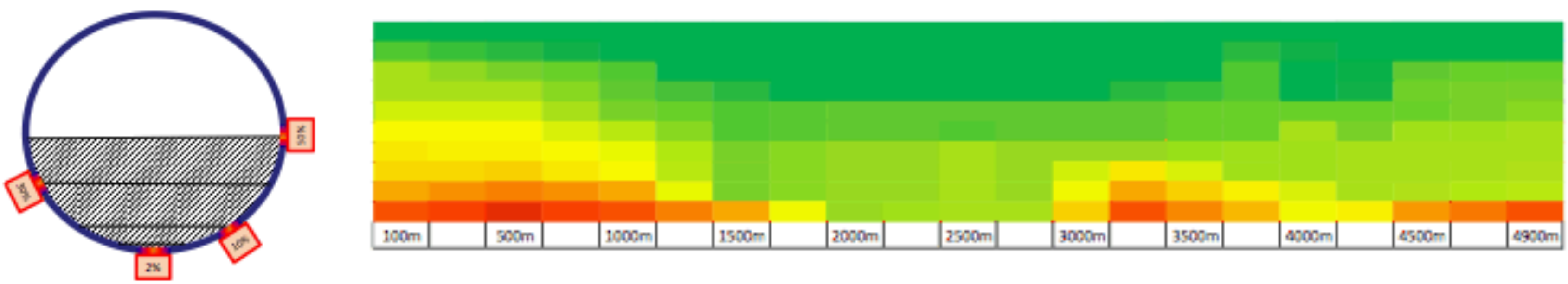
Phase Distribution in Slurry/Suspension , cont'd

Optical Method

- Fiber optic backscatter probes calibrated to give local solids (Mokhtari & Chaouki 2019)

Heat Transfer Method

- Ilgner (2017, 2018) noninvasively monitors sedimentation in slurry pipelines by measuring rate of heat removal from heated spots strategically placed along the line. Additional work (Ilgner & Kruger 2018) is aimed at monitoring dune height along the pipeline (simulation shown below).

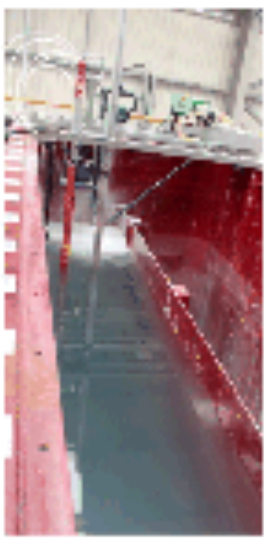


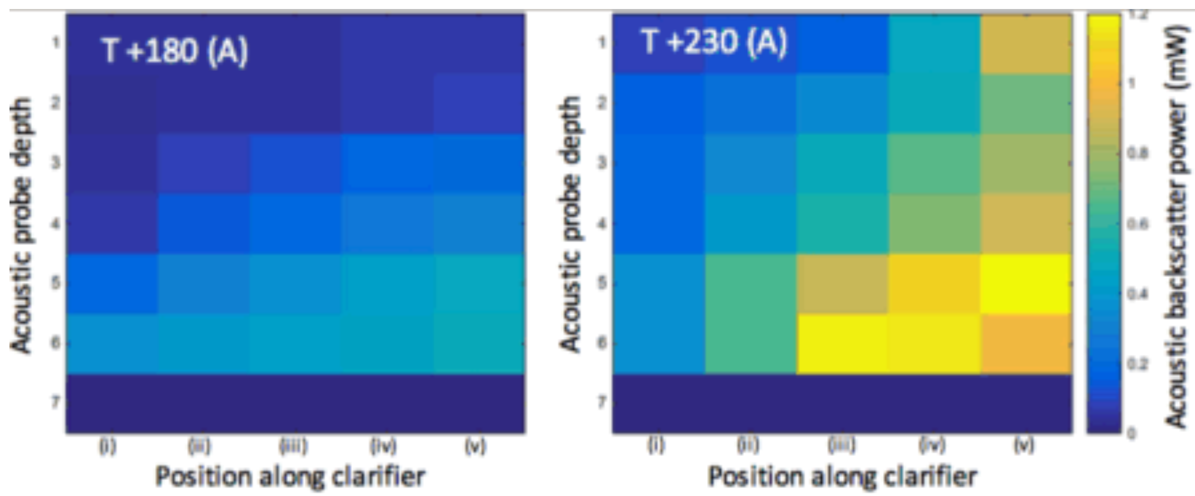
Ilgner & Kruger, Figs. 8 &13

Phase Distribution in Slurry/Suspension , cont'd

Ultrasonic Method

- Hunter et al. (2020) recently demonstrated the use of ultrasonic backscatter from a 5x7 transducer array to monitor the settled layer in a pilot scale sedimentation tank.

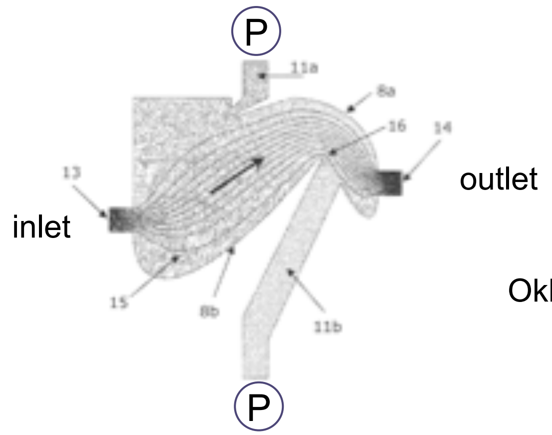




Hunter et al., Figs. 3 & 11 (Clarifier dimensions: 4.86 m long, 2.46 m high, 0.75 m wide)

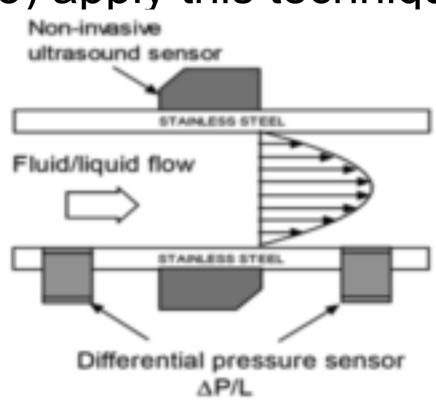
6.4 Rheology

- ★ Pressure drop across an innovative flow cell design (Okkels 2017a,b) is the heart of the on-line rheometer sold by Fluidan (fluidan.com).



Okkels (2017b) Fig. 4

- Kotzé et al. (2008) combined ultrasonic velocity profiling (to get shear rate), measure the pressure differential (to get stress), and take the ratio to find viscosity. Kotzé et al. (2015) apply this technique to characterize wastewater sludge.



Kotzé et al. (2015) , Fig. 2

Conclusions

- A comprehensive search of the scientific and patent literature revealed a few innovative characterization methods (and several instances of “rediscovering the wheel”). Some of these recent innovations have been commercialized.
- The bulk of the papers reviewed have demonstrated useful, and often new, applications of known methods (or variations of known methods).
- Many particle characteristics of interest can be measured by multiple modalities, including electrical, mechanical, optical , imaging and ultrasonic sensors.
- Characterization gaps still exist; for instance detection or measurement of small particles in the midst of large ones.