Particle Characterization: Petroleum Applications

Mark Bumiller

Range of Applications

Exploration
Completion

Drilling fluid
Proppants

Processing

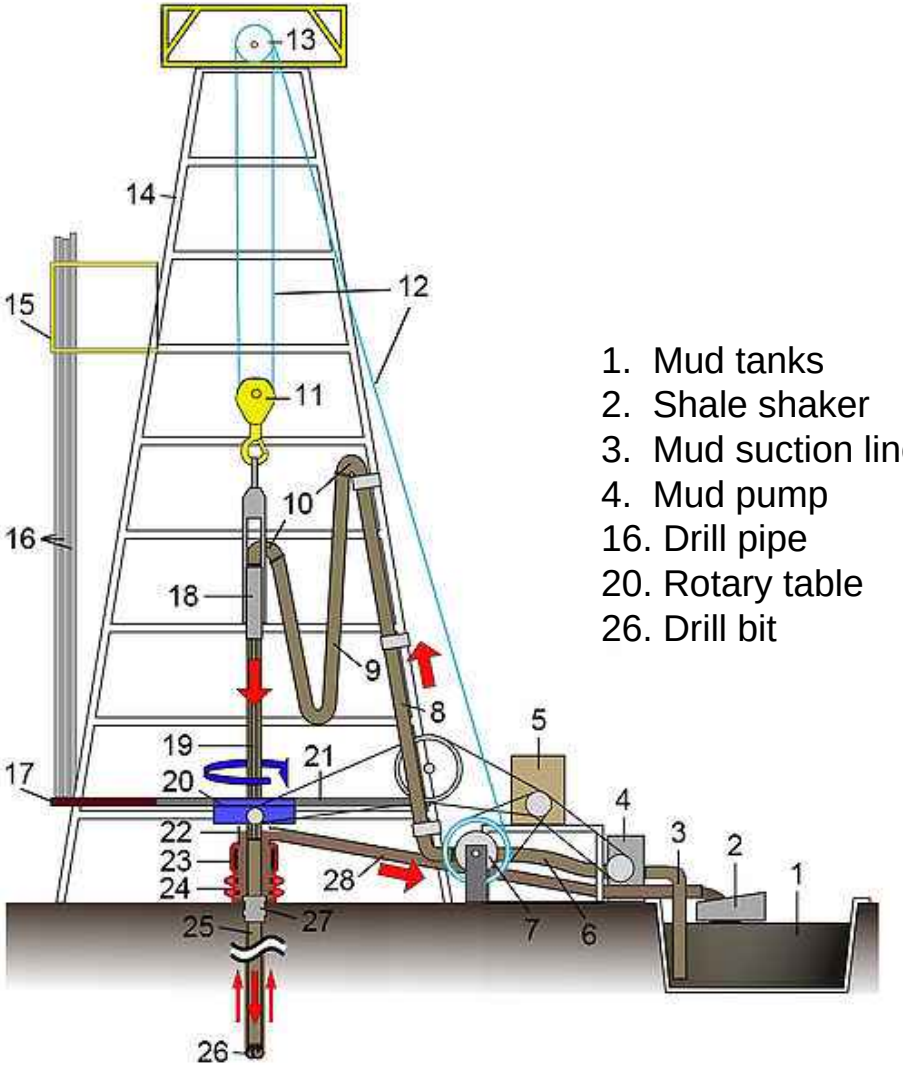
Oil in water emulsion
Solid particles

Refining

Catalysts

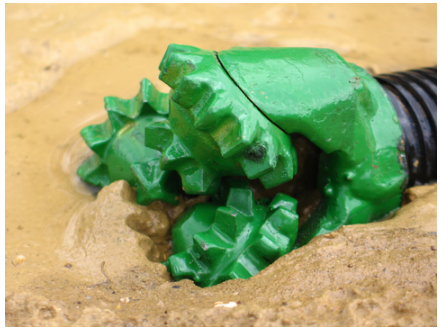
Techniques used: laser diffraction, dynamic image analysis, Acoustic spectroscopy, surface area analysis

Exploration: Drilling Rig



1. Mud tanks
2. Shale shaker
3. Mud suction line
4. Mud pump
16. Drill pipe
20. Rotary table
26. Drill bit

Drilling Fluid (Mud)



- Remove cuttings from well
- Suspend and release cuttings
- Control formation pressures
- Seal permeable formations
- Maintain wellbore stability
- Minimizing formation damage
- Cool, lubricate, and support the bit and drilling assembly
- Facilitate cementing and completion

Types of Fluids

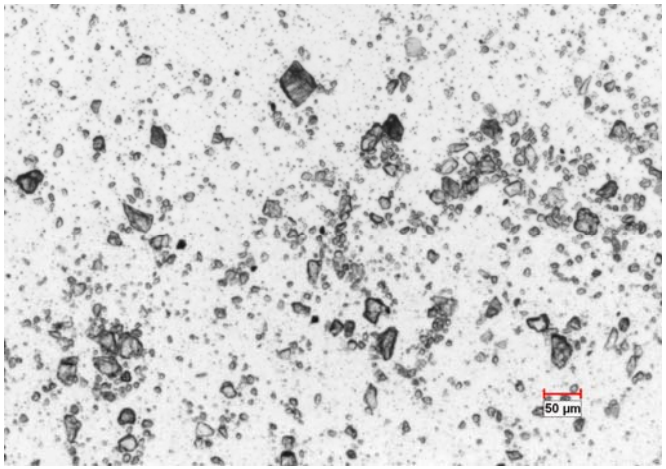
- Water-based mud (WBM)
  - Water, then clays and other chemicals
  - Most common is bentonite
  - Other chemicals for viscosity control, shale stability, enhance drilling rate, cooling, etc.
- Oil-based mud (OBM)
  - Base fluid is a petroleum product such as diesel fuel
- Synthetic-based fluid (SBM)
  - Base fluid is a synthetic oil

Particle Size of Drilling Fluids

- Standard methods include sieves & sedimentation
- Report residue > 75 µm & < 6 µm
- Diffraction can report this, + distribution information
  - D10, d50, d90
  - Quicker, more reproducible
- Recommended practices for barite:
  - RI: 1.64, 0.1 in 1.33 (water)
  - Dispersant: 1 g sodium pyrophosphate in 1000 cm^3 water
  - Mix paste of barite powder, dispersant
  - Add to system, %T not < 90-85%
  - Ultrasound for 60 sec
  - Measure

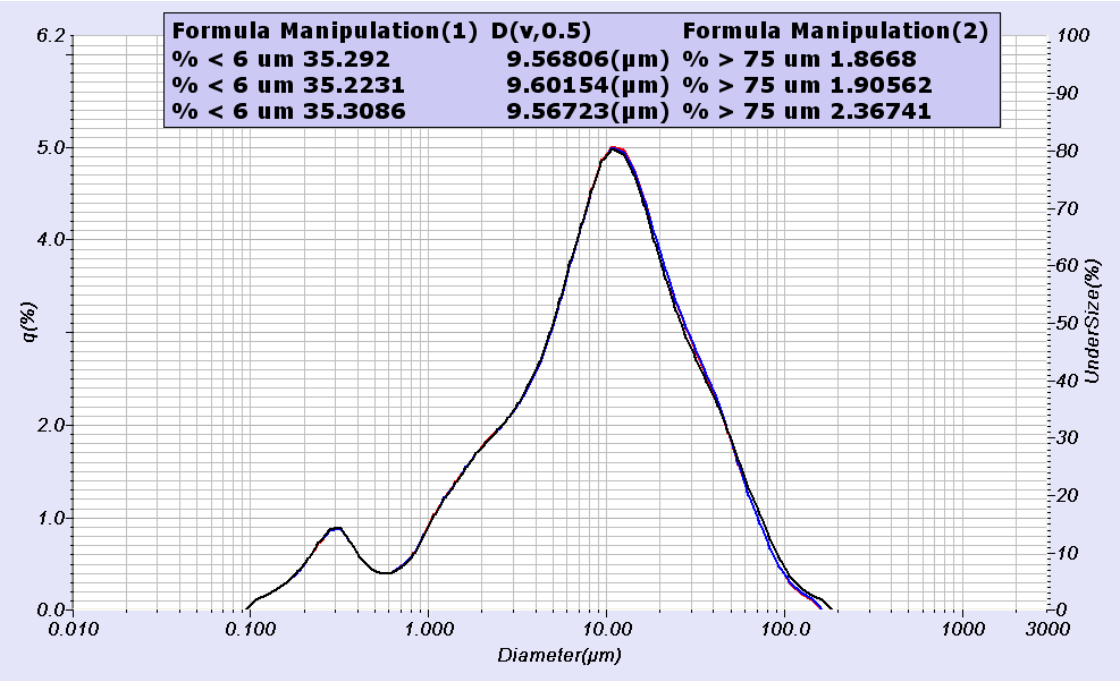
Particle Size of Drilling Fluid

Composition:
Bentonite 29 g/L
Xantham gum 2.9 g/L
P.A.C. (polymer additive) 2.9 g/L
Barite 15 g/L
NaOH 0.7 g/L



Procedure:
RI for barite = 1.64, 0.1
5 g of barite added to a beaker.
Dispersant* added to barite drop wise until smooth paste was created.
*Dispersant solution: 1 g of sodium pyrophosphate/1000 cm3 of solution.
Sampler was filled with DI water.
Barite sample added the sampler using clean spatula to desired concentration:
Transmission = 80-90%
Sampler circulation setting = 5
Ultrasound at level 7 applied to sample for 60 sec
Wait 30 sec after turning off the ultrasound
Perform particle size measurement
Repeat these steps for a total of 3 sub-samples from the original paste

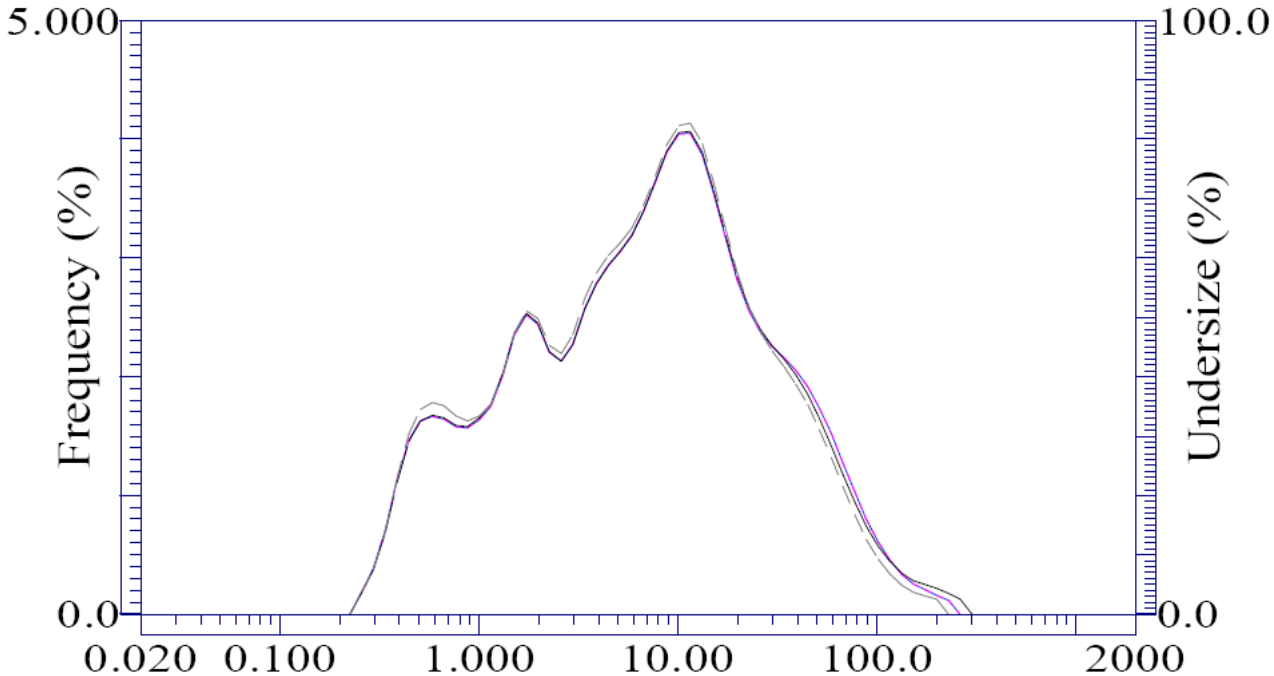
Particle Size of Drilling Fluid

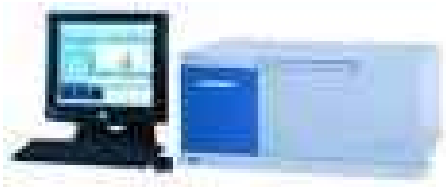


LA-950

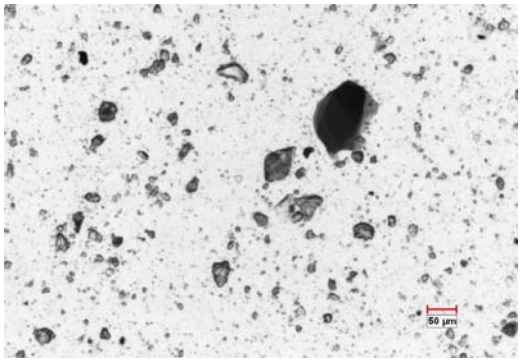
Sample Name	File Name	D(v,0.1)	D(v,0.5)	D(v,0.9)
Drilling Mud Sampling 2	200911301126035.NGB	1.211	9.568	38.269
Drilling Mud Sampling 2	200911301126036.NGB	1.212	9.602	38.490
Drilling Mud Sampling 2	200911301127037.NGB	1.211	9.567	39.830
Average		1.211	9.579	38.863
Std. Dev.		0.001	0.020	0.845
CV (%)		0.048	0.208	2.174
ISO 13320-1 (20.0, 15.0, 20.(		PASSED	PASSED	PASSED

Particle Size of Drilling Fluid

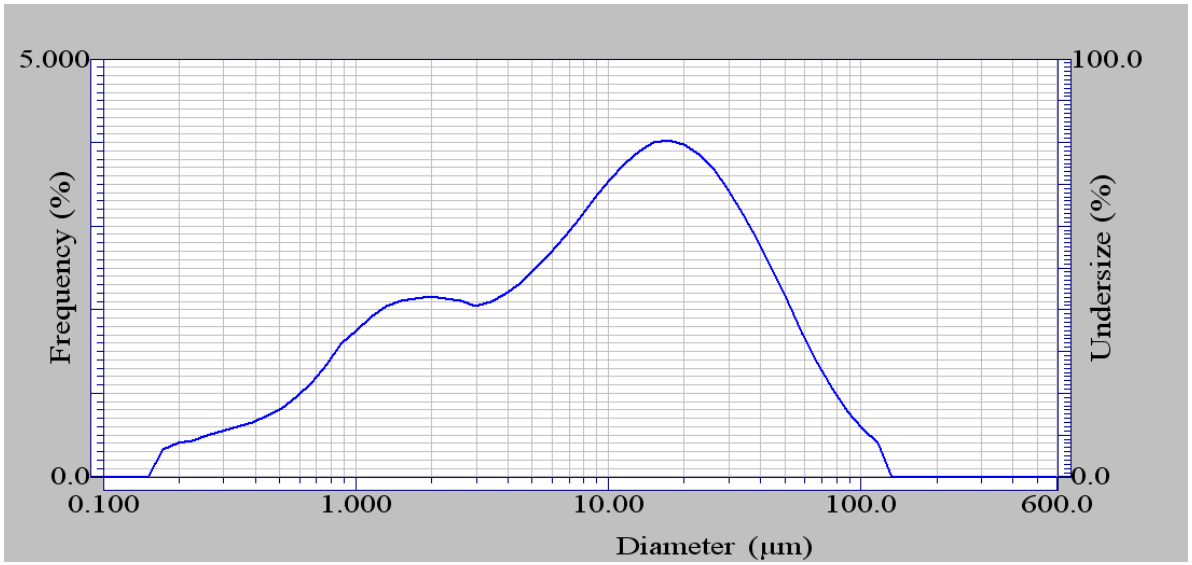




LA-930



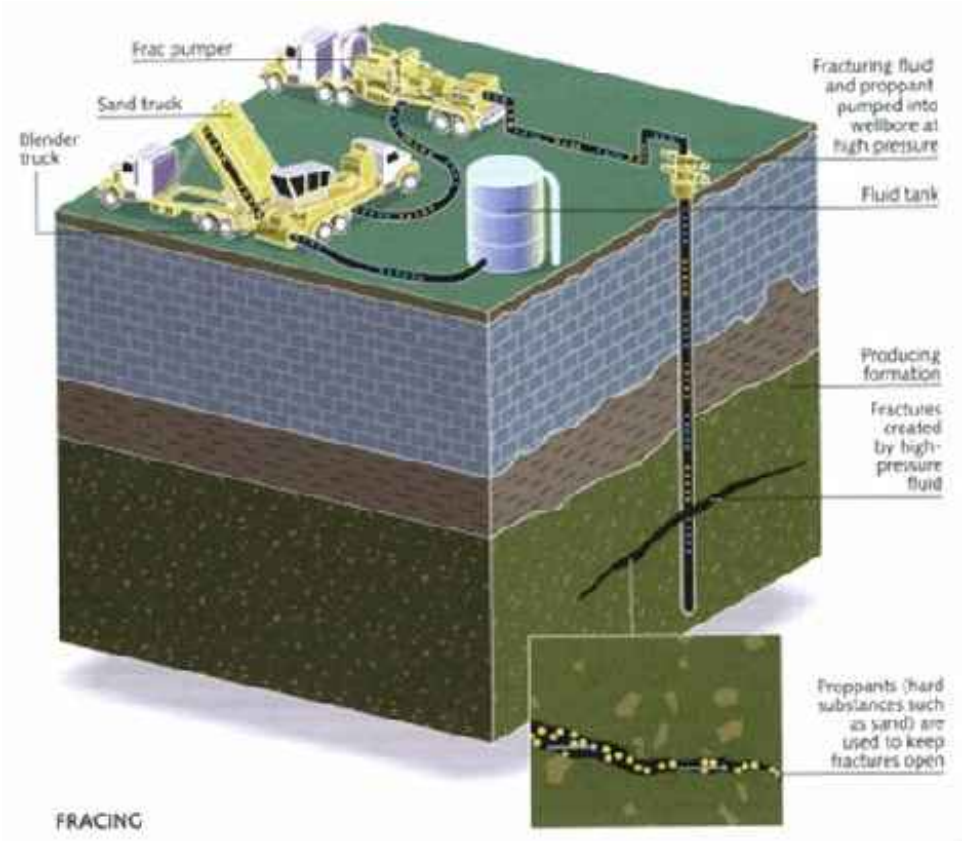
Particle Size of Drilling Fluid



LA-300
Smaller,
portable,
better for
field work
55lb/25 kg

Diameter on % :	(1)10.00 (%)-	0.875(µm)
	(2)50.00 (%)-	8.999(µm)
	(3)90.00 (%)-	40.147(µm)

Hydraulic Fracturing Proppant



20/40 sand

Proppants

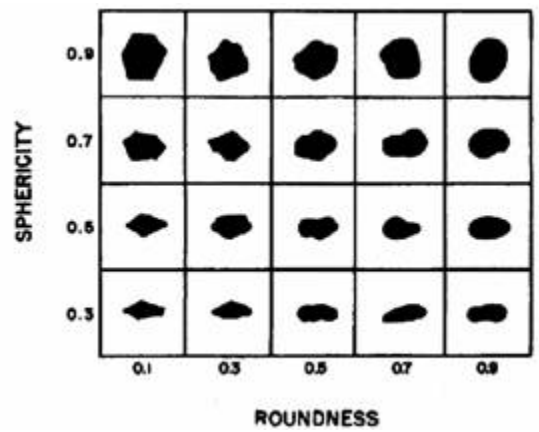
- Keep fractures open
- Improves permeability
- Often sand , specified by sieve sizes
  - 12/20, 16/30, 20/40, 40/70 (95% passes between these sieve sizes)
- Higher tech proppants, more expensive, higher performance

Ceramic Proppant Patent

"The resulting pellets have a sphericity of about 0.8-0.9, as determined using the Krumbein and Sloss chart."

TABLE 3

ASTM U.S. Standard Mesh Size	Equivalent Particle Diameter	Weight % Retained Typical 20/40 Intermediate Grade	Extended PSD-1
16	>1.18 mm	0.0	0.5
18	1-1.18 mm	0.0	6.0
20	0.85-1.0 mm	2.0	27.9
25	0.71-0.85 mm	38.0	24.7
30	0.6-0.71 mm	33.0	21.8
35	0.50-0.60 mm	24.0	12.8
40	0.425-0.5 mm	3.0	6.0
45	0.355-0.425	0.0	0.5
50	0.30-0.355	0.0	0.01



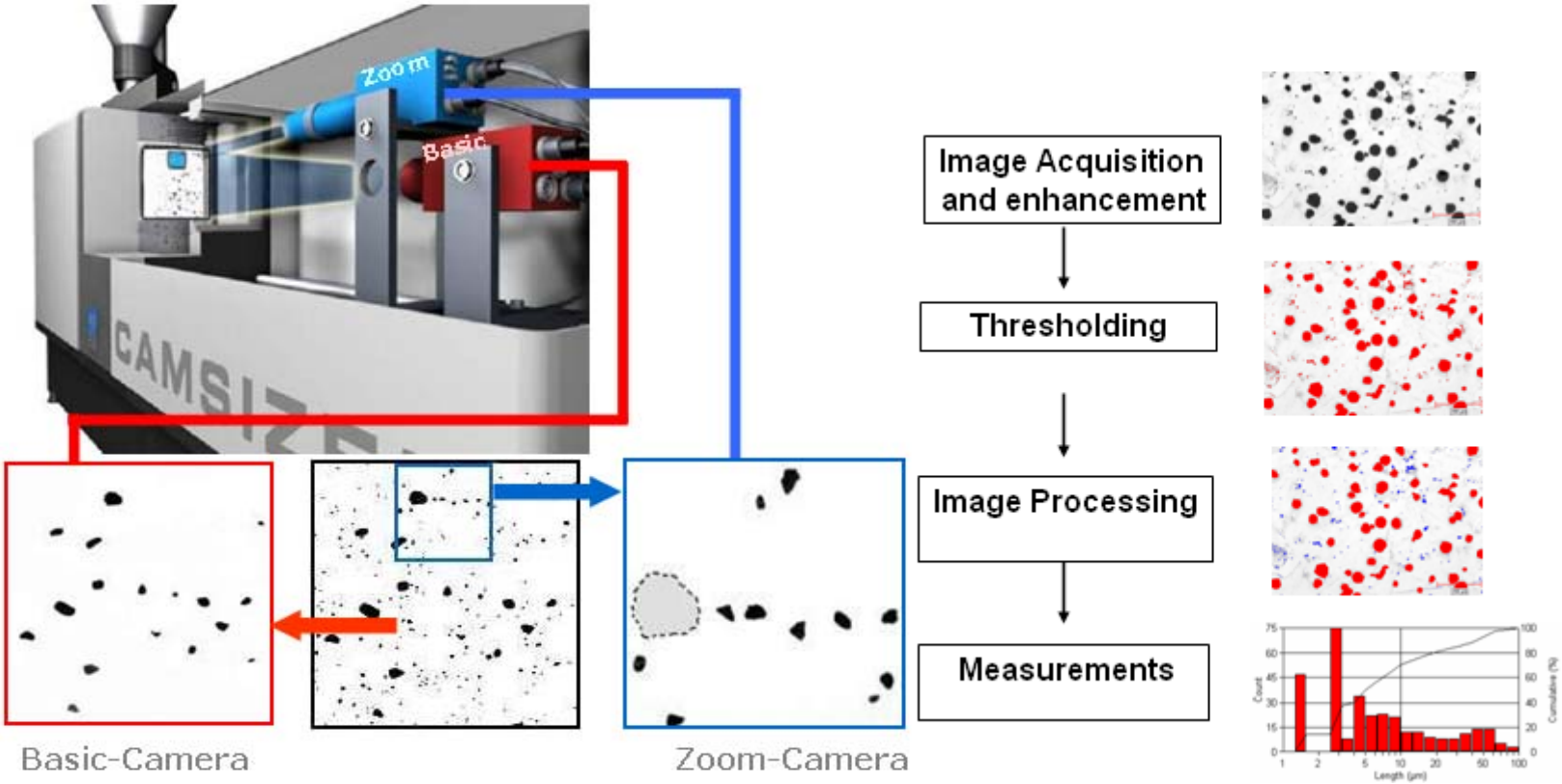
Proppant Spec Sheet*

Properties	Units/ Method (API)	Intermediate Density Bauxite						High Density Bauxite			
		Versaprop®	12/18 Interprop®	16/30 Interprop®	20/40 Interprop®	30/50 Interprop®	35/140 Interprop®-Hybrid (IP-H)	Ultraprop® Sintered Bauxite	16/30 Sintered Bauxite	20/40 Sintered Bauxite	30/50 Sintered Bauxite
		Sieve No. / % Retained	Sieve No. / % Retained	Sieve No. / % Retained	Sieve No. / % Retained	Sieve No. / % Retained	Sieve No. / % Retained	Sieve No. / % Retained	Sieve No. / % Retained	Sieve No. / % Retained	Sieve No. / % Retained
Typical Sieve Analysis	Min. 90% Retained Between Designated Sieves	18 / 8 25 / 56 40 / 36 <40 / TR	12 / 5 14 / 38 18 / 57 <18 / TR	16 / 4 20 / 84 30 / 12 <30 / TR	20 / 6 30 / 78 40 / 16 <40 / TR	30 / 5 40 / 80 50 / 15 <50 / TR	35 / 5 50 / 35 80 / 50 140 / 10 <140 / TR	18 / 8 25 / 56 40 / 36 <40 / TR	16 / 4 20 / 84 30 / 12 <30 / TR	20 / 5 30 / 75 40 / 20 <40 / TR	30 / 4 40 / 74 50 / 22 <50 / TR
Median Particle Diameter	mm inches	0.740 0.029	1.367 0.054	0.974 0.038	0.687 0.027	0.485 0.019	0.280 0.011	0.740 0.029	0.974 0.038	0.662 0.026	0.458 0.018
Specific Gravity	grams/cc	3.2	3.2	3.2	3.2	3.2	3.2	3.5	3.5	3.5	3.5
Acid Solubility	%	2.5	2.5	2.5	2.5	2.5	2.5	1.9	1.9	1.9	1.9
Shape/ Sphericity	Krumbein & Sloss	0.9	0.9	0.9	0.9	0.9	0.7	0.9	0.9	0.9	0.9
Absolute Volume	gal/lb	0.0374	0.0374	0.0374	0.0374	0.0374	0.0374	0.0347	0.0347	0.0347	0.0347
Bulk Density	grams/cc lbs/ft^3	1.88 117	1.88 117	1.88 117	1.88 117	1.88 117	1.88 117	2.04 127	2.04 127	2.04 127	2.04 127
Crush Resistance @ Stress %	5,000 (psi) 7,500 (psi) 10,000 (psi) 12,500 (psi) 15,000 (psi)	0.4 1.4 3.5 --- ---	1.4 8.0 13.0 --- ---	0.6 2.2 4.4 7.6 ---	0.3 0.8 2.5 4.0 ---	0.1 0.5 1.2 2.6 ---	--- 1.5 3.0 4.0 ---	--- 0.6 1.5 3.0 6.0	--- 1.0 2.5 4.5 9.0	--- 0.5 1.2 2.2 4.0	--- 0.2 0.6 1.0 1.5

*

Proppants: CAMSIZER

Size and shape from 30 µm – 30 mm



CAMSIZER Proppant Data*

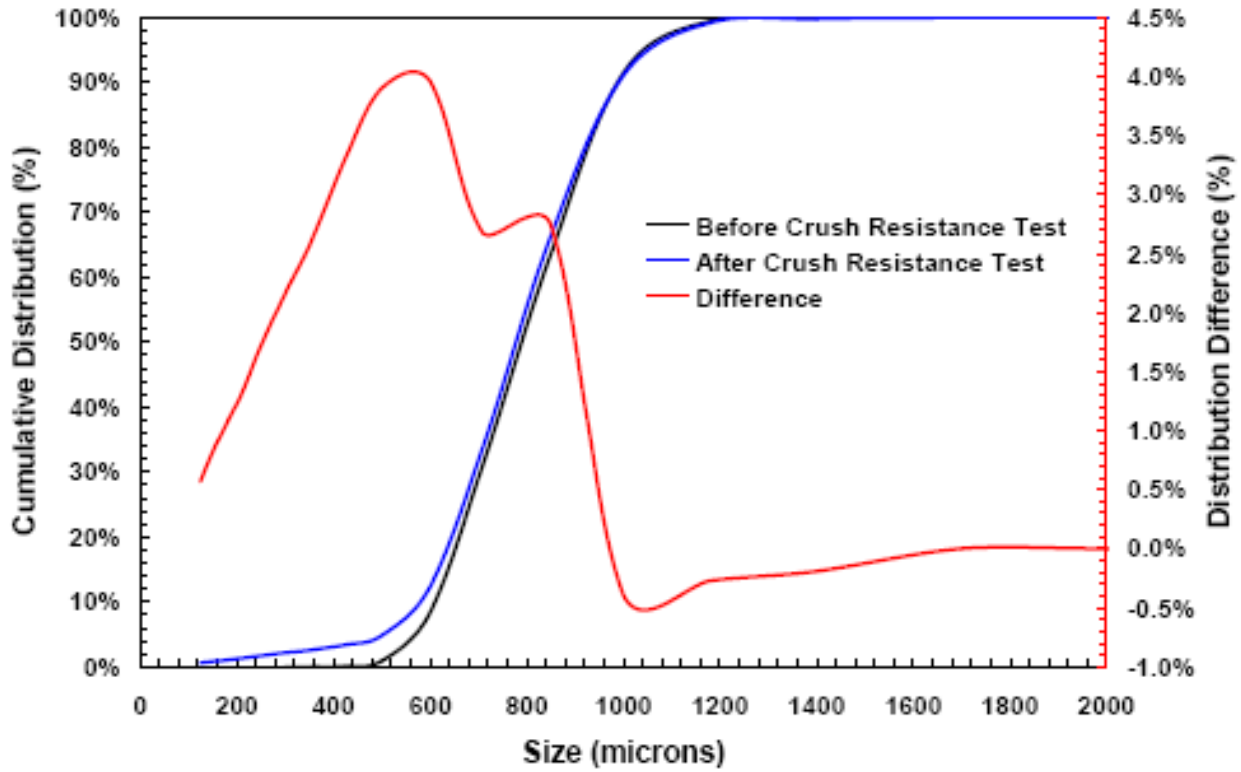
- API crush test of proppants:
- Mechanical forces compresses proppant in cell to desired P
- PSD is measured after release
- Crush resistance = wt% of proppant passing smallest sieve of originally specified PSD
- Only one sieve tray required, entire PSD changes during test
- Measuring entire PSD reveals more information

"An optical size analyzer can extract more information than physical sieve analysis about the behavior of proppants under stress while significantly reducing analysis time."

*Stephens et. al., Statistical Study of the Crush Resistance Measurement for Ceramic Proppants 2006 Annual SPE Meeting

CAMSIZER Proppant Data*

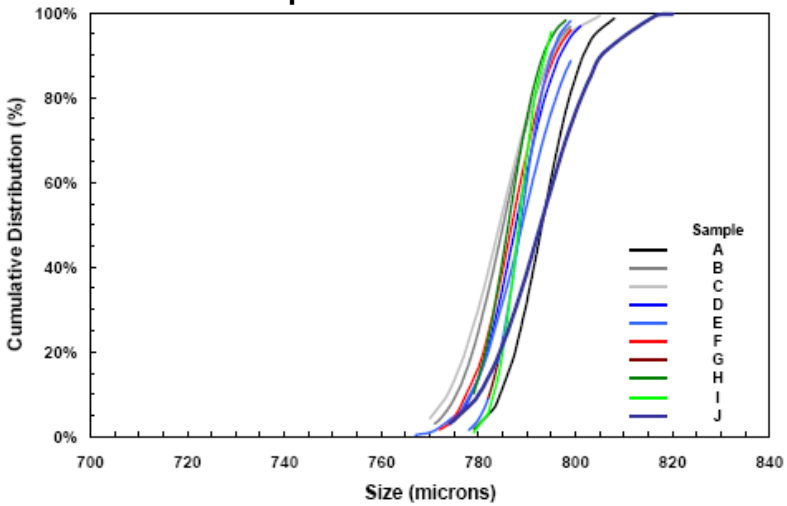
PSD before and after crush

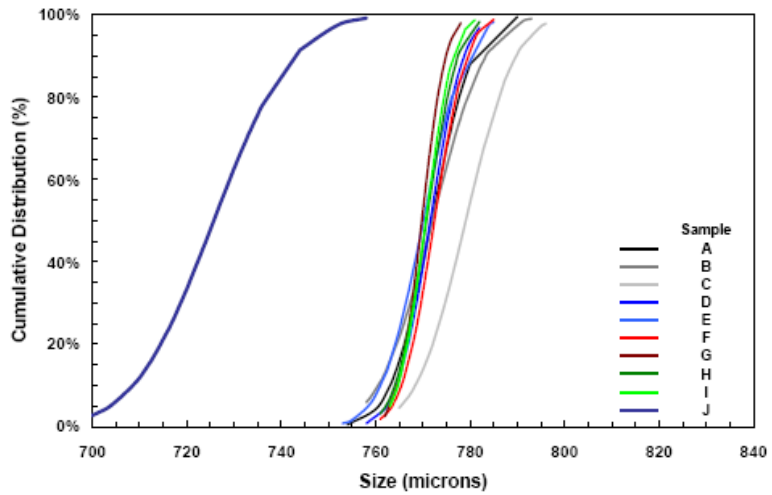


*Stephens et. al., Statistical Study of the Crush Resistance Measurement for Ceramic Proppants 2006 Annual SPE Meeting

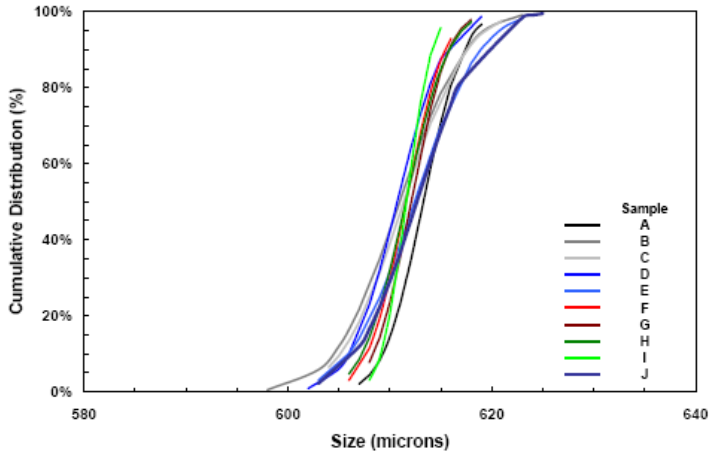
CAMSIZER Proppant Data*

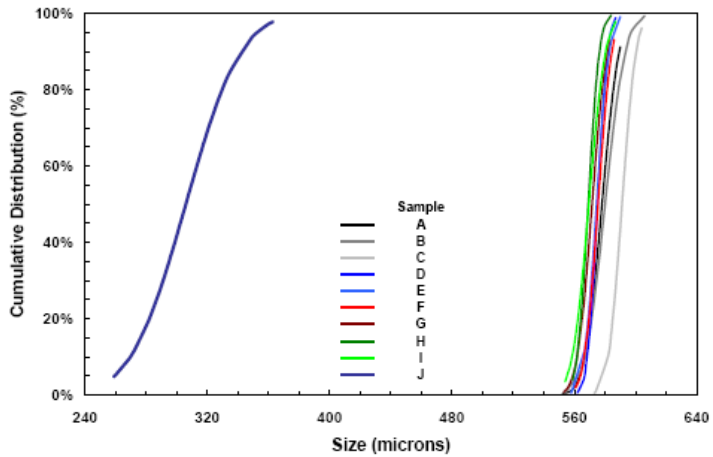
50th percentile before and after crush





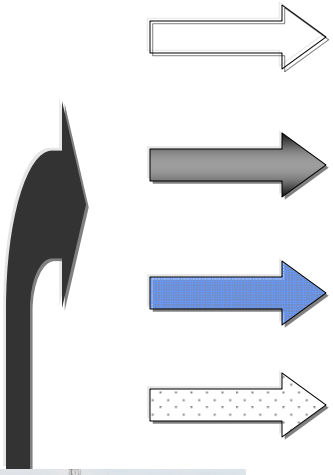
10th percentile before and after crush





*Stephens et. al., Statistical Study of the Crush Resistance Measurement for Ceramic Proppants 2006 Annual SPE Meeting

Production Fluid Separation

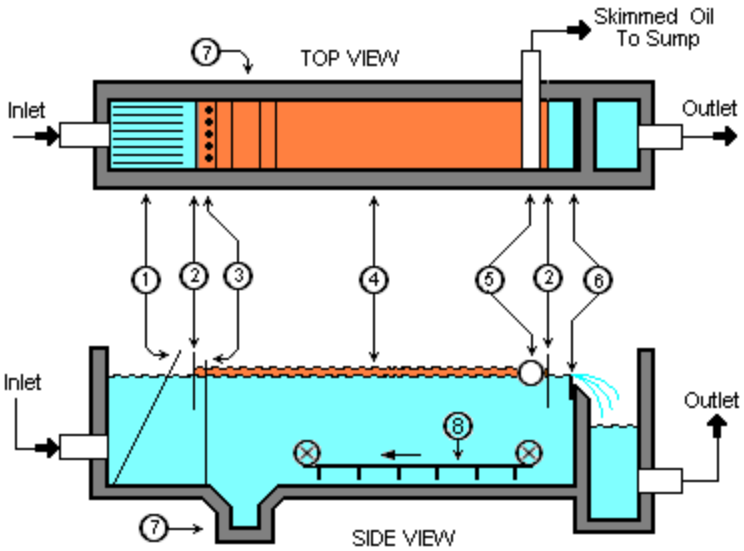


Gas

Oil

Water

Solids (particles)



1 Trash trap (inclined rods)
2 Oil retention baffles
3 Flow distributors (vertical rods)
4 Oil layer
5 Slotted pipe skimmer
6 Adjustable overflow weir
7 Sludge sump
8 Chain and flight scraper

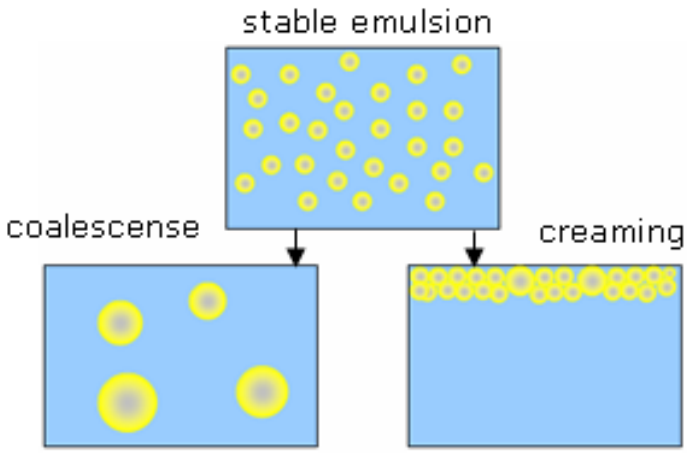
Oil/water separator

Production Fluid Separation

- Want to separate all phases
- Need to measure phase separation
  - Improve by adding surfactants
- Need to measure ppm oil in water
  - Required to measure if pumping water into ocean (off shore rigs)
  - Oil is valuable, separate as much as possible
- Possible to differentiate oil droplets from particles?

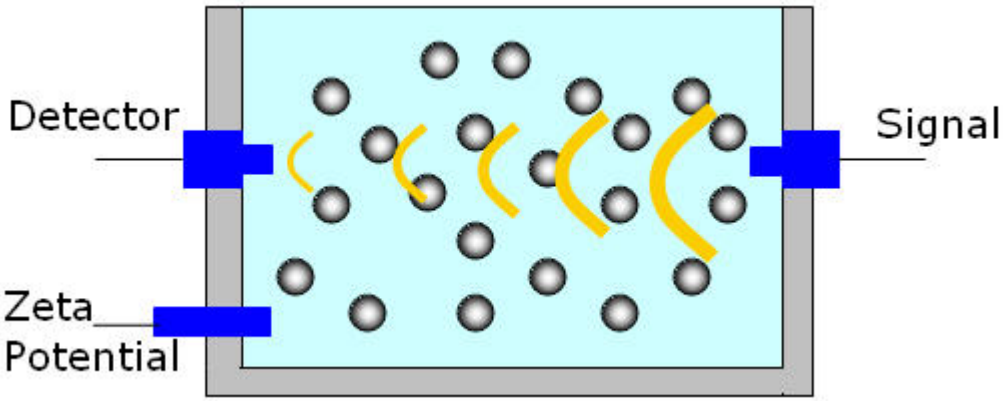
Emulsion Stability

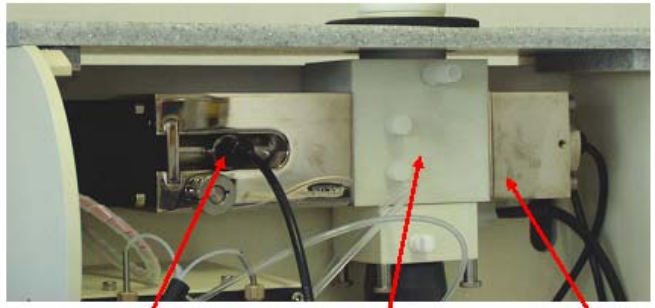
- Want to promote phase separation
- Recover oil, discharge water
- May want to add water to oil to reduce viscosity, easier to transport (orimulsion)

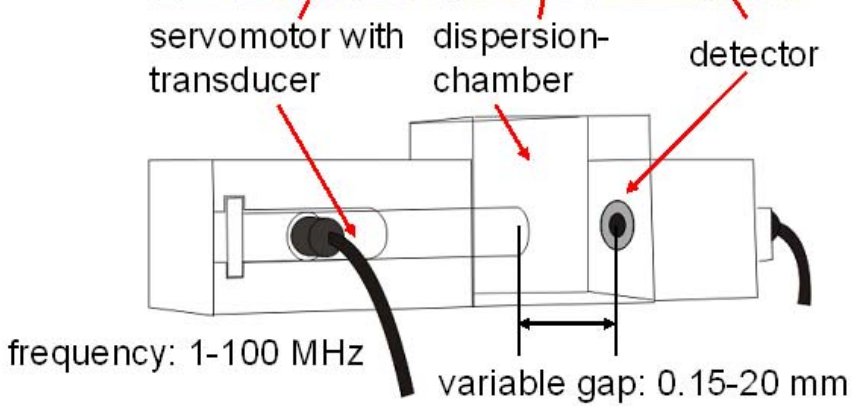


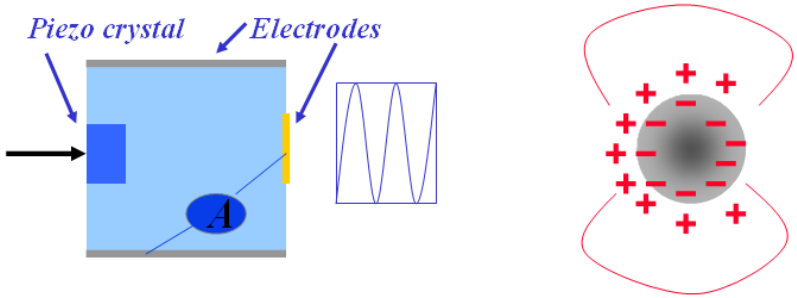
Emulsion Stability: DT1201

Measure particle size & zeta potential w/no dilution
Zeta potential (CVI) predicts dispersion stability





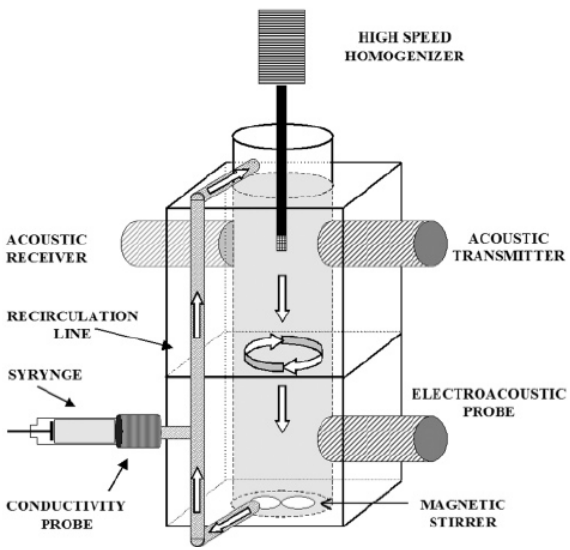




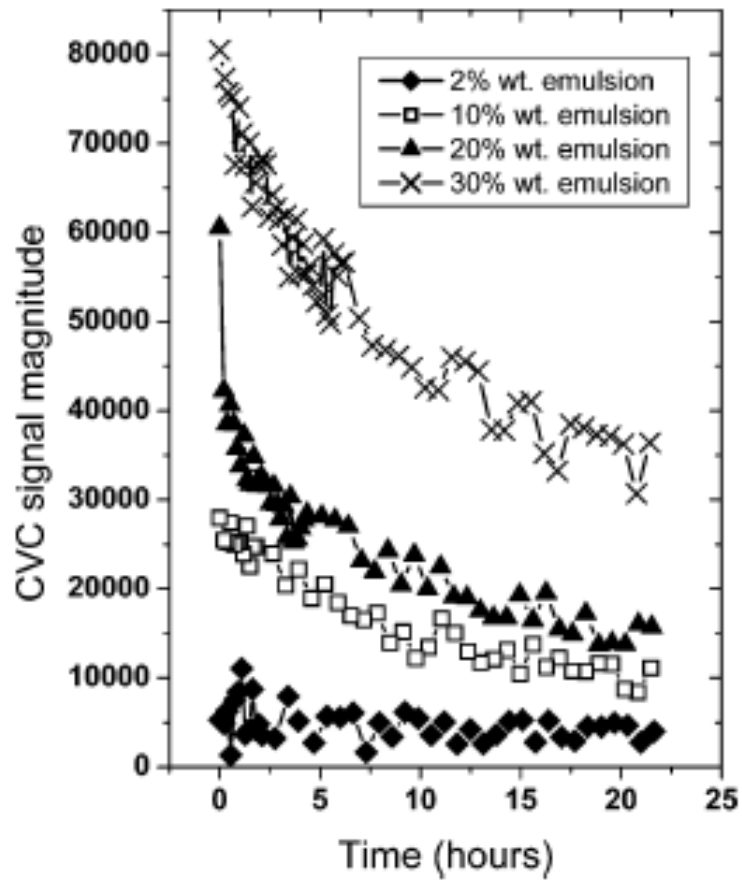
Colloid vibration current (CVC) ⇨ zeta potential

Bitumen Emulsion Stability*

- Bitumen diluted 1:1 w/toluene
- Diluted w/D_2O 2-30 wt %
- Homogenize 125 watt, 30,000 RPM

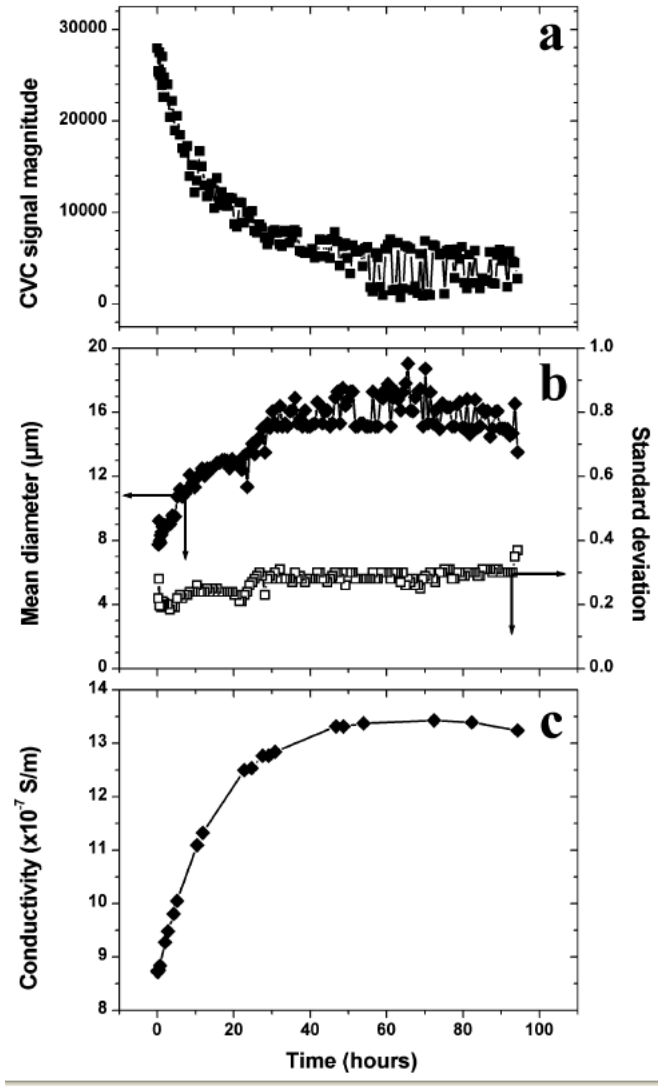


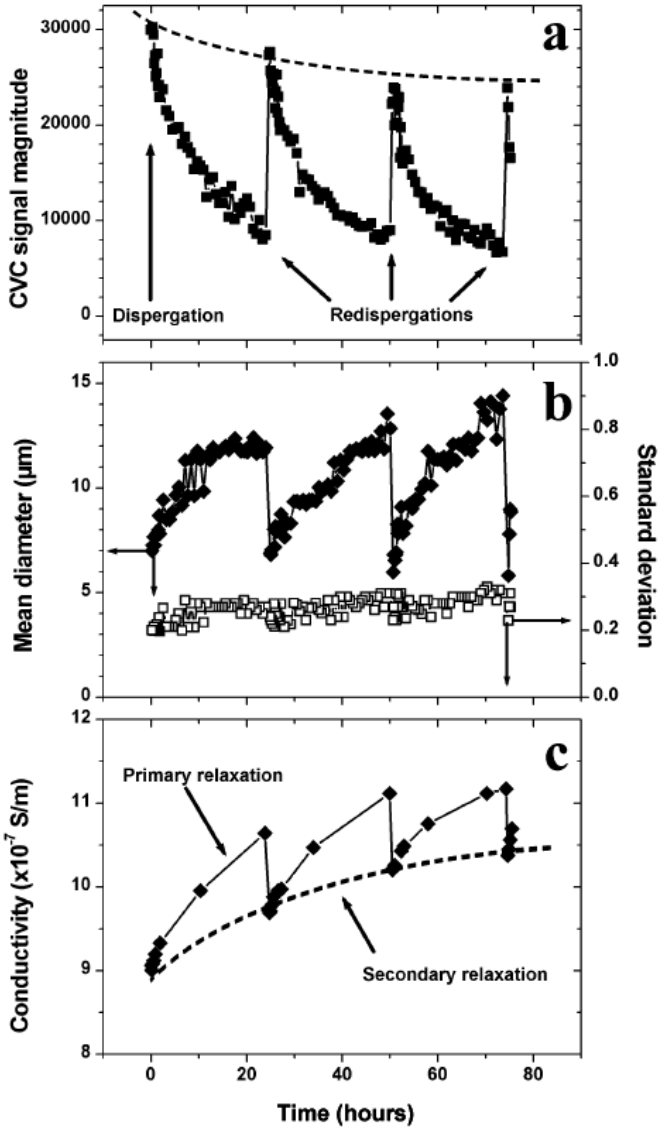
DT-1200 w/homogenizer



*Magual, A., Horvath-Szabo G., Masliyah, J.H. "Acoustic and electroacoustic spectroscopy of water-in-diluted bitumen emulsions", Langmuir, 21, 8649-8657 (2005)

Bitumen Emulsion Stability*





Crude Oil Demulsification

- Problem: Break the crude oil emulsion
  - In the shortest time
  - With less additive
  - Leave an unpolluted water phase

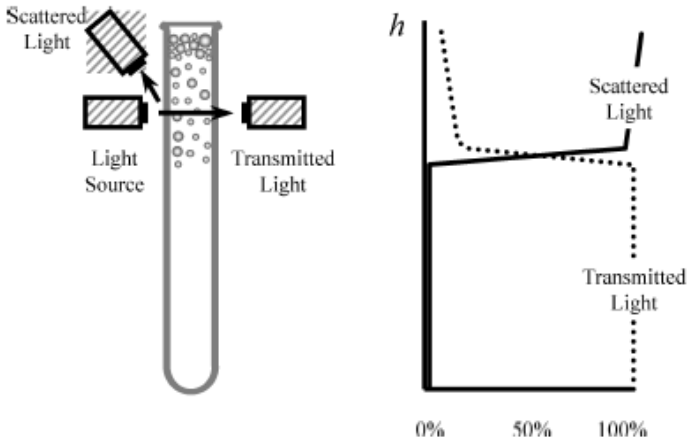
- Solution:
  - Find the optimal additive (cost, efficiency)
  - Need to be tested on each oil batch

➪ Identify the best surfactant for demulsification

Crude Oil Demulsification

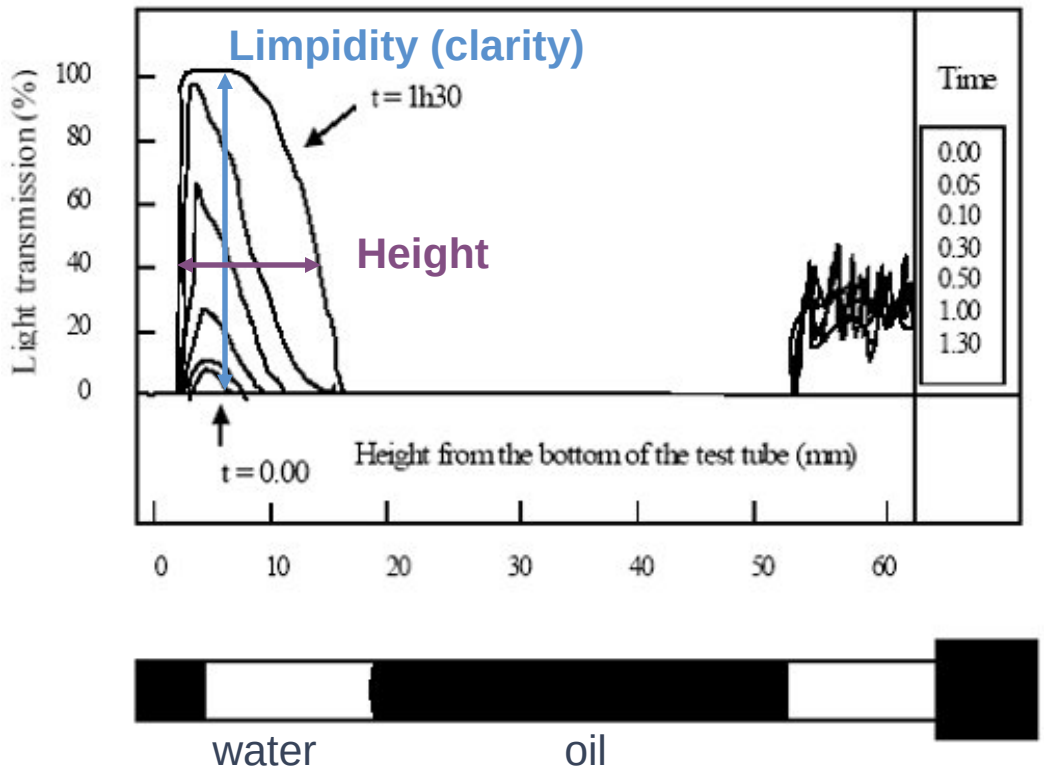
Study from French Institue of Petroleum (IFP)*

- Turbiscan test
  - Qualitative & Quantitative study
  - Selection of the best additive
  - Optimization of the additive concentration
  - Criteria:
    - width of T peak = height of separated water
    - height of T peak = Limpidity of separated water



*C. Dalmazzone, C. Noïk, «Development of New «green» Demulsifiers for Oil Production », SPE65041

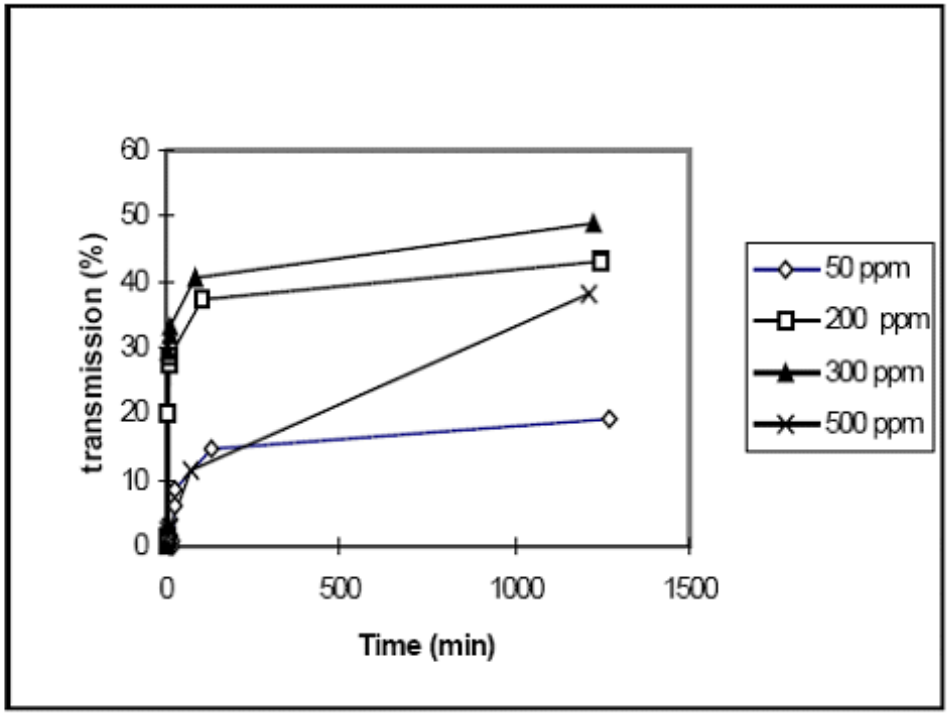
Crude Oil Demulsification



C. Dalmazzone, C. Noïk, «Development of New «green» Demulsifiers for Oil Production », SPE65041

Crude Oil Demulsification

« Separation rate » => %T = f(time)

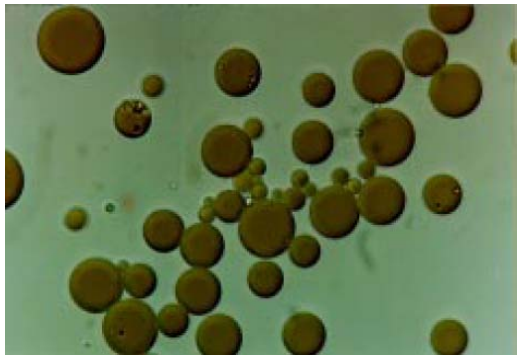


↳ 300 ppm is the best concentration

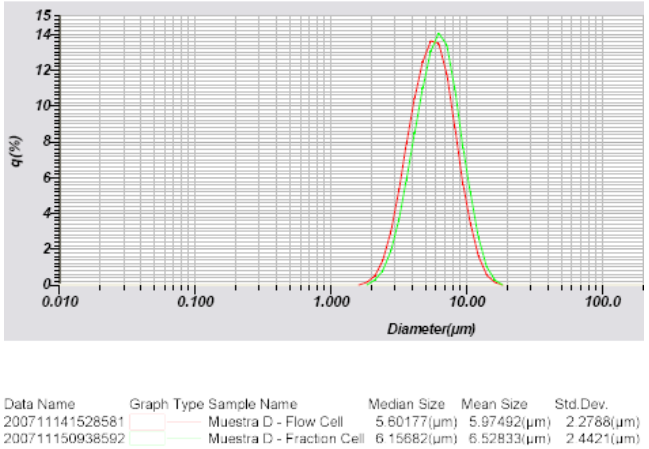
C. Dalmazzone, C. Noïk, «Development of New «green» Demulsifiers for Oil Production », SPE65041

Orimulsion: Emulsification

- Bitumen: sticky, tar-like form of petroleum so thick & heavy must be heated/diluted before it will flow
- World's largest deposit in Orinoco Belt, Venezuela
- Orimulsion made by mixing bitumen with ~ 30% fresh water + small amount of surfactant
- The result behaves similarly to fuel oil
- Used as commercial boiler fuel
- D50 ~ 14 µm

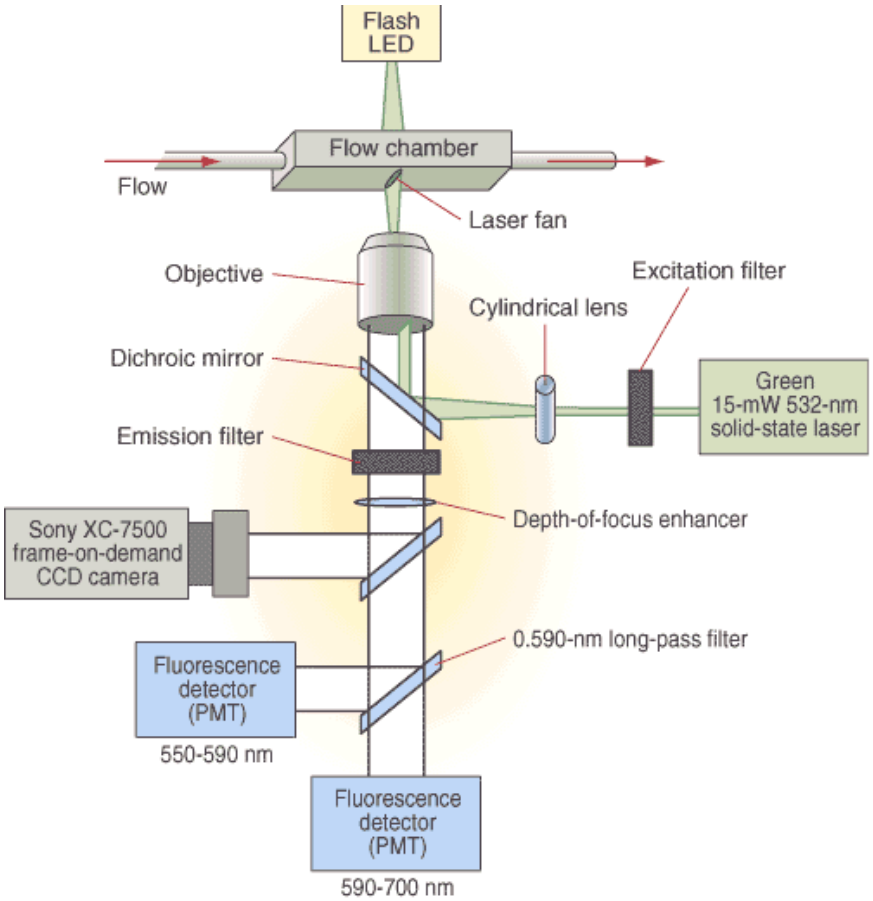


Orimulsion droplets



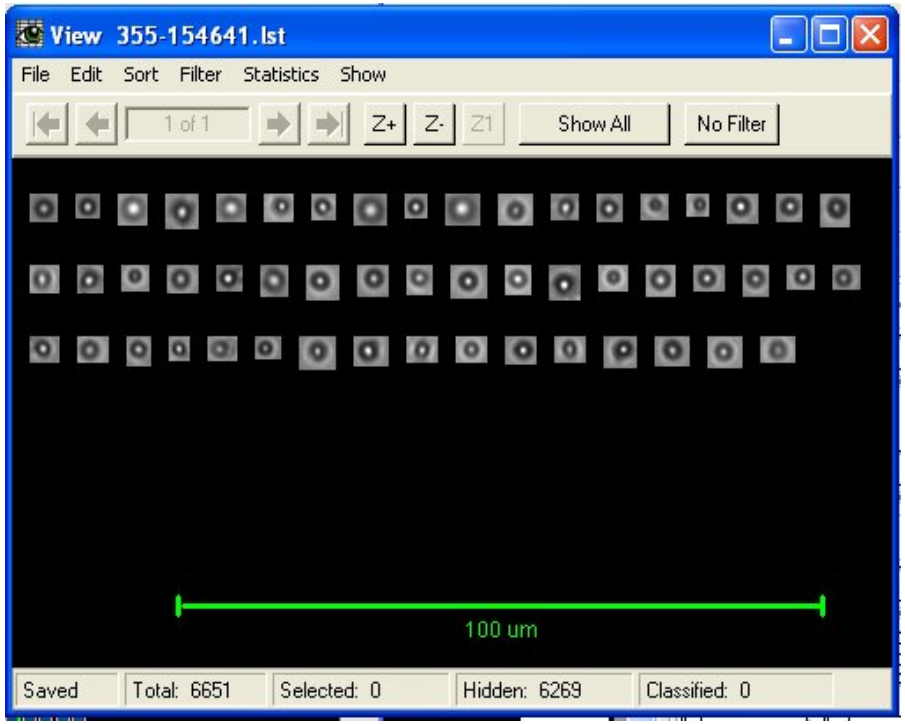
LA-950 result

Oil in Water after Separation

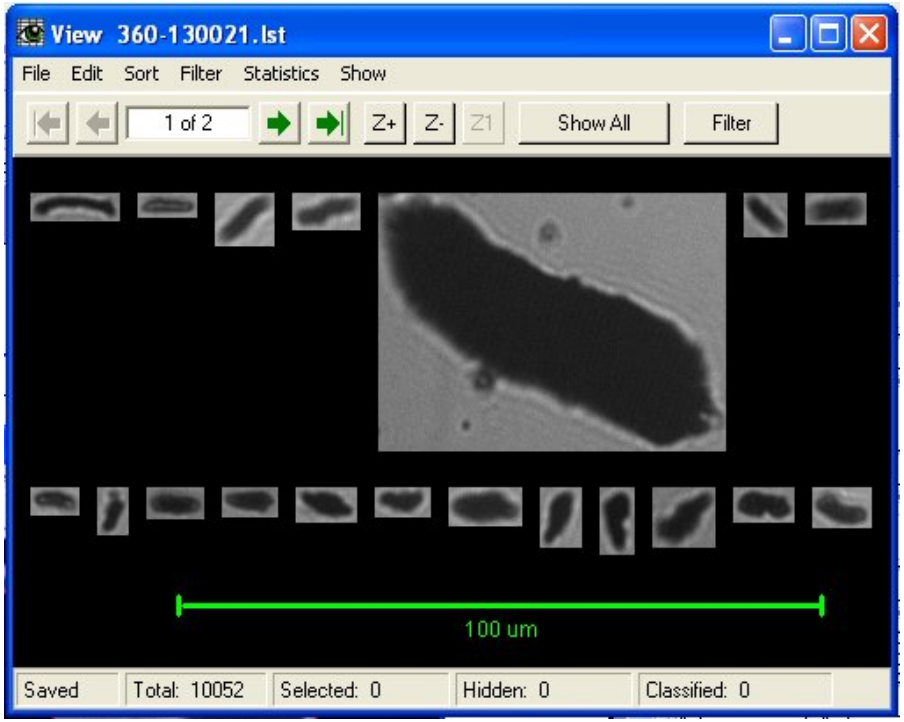


Flow chambers
50um – 1mm
2mm-6 mm

Droplets vs. Particles



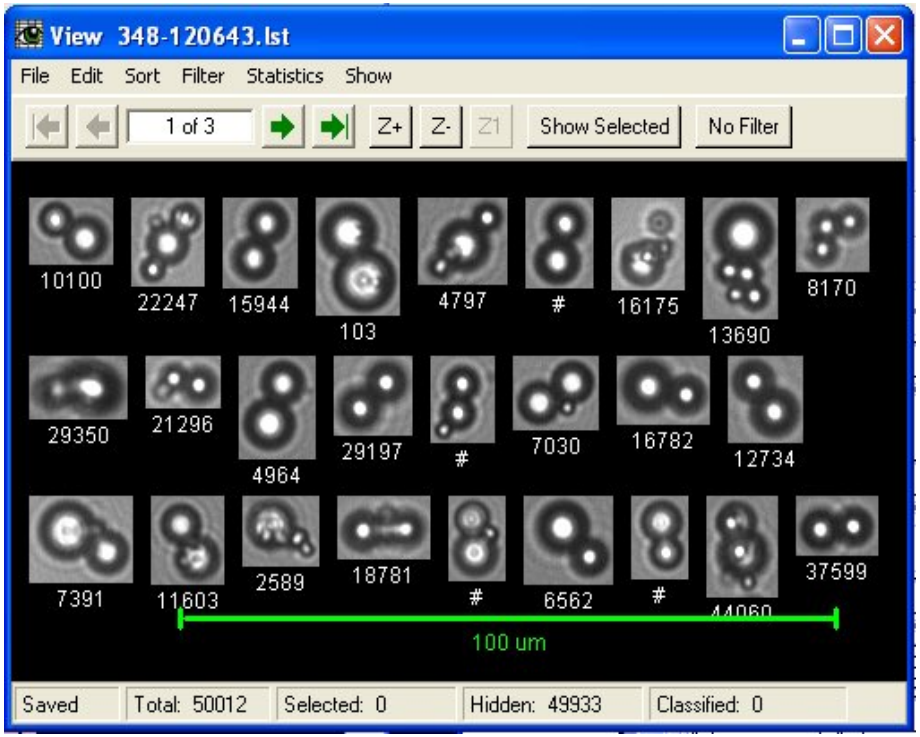
Oil droplets



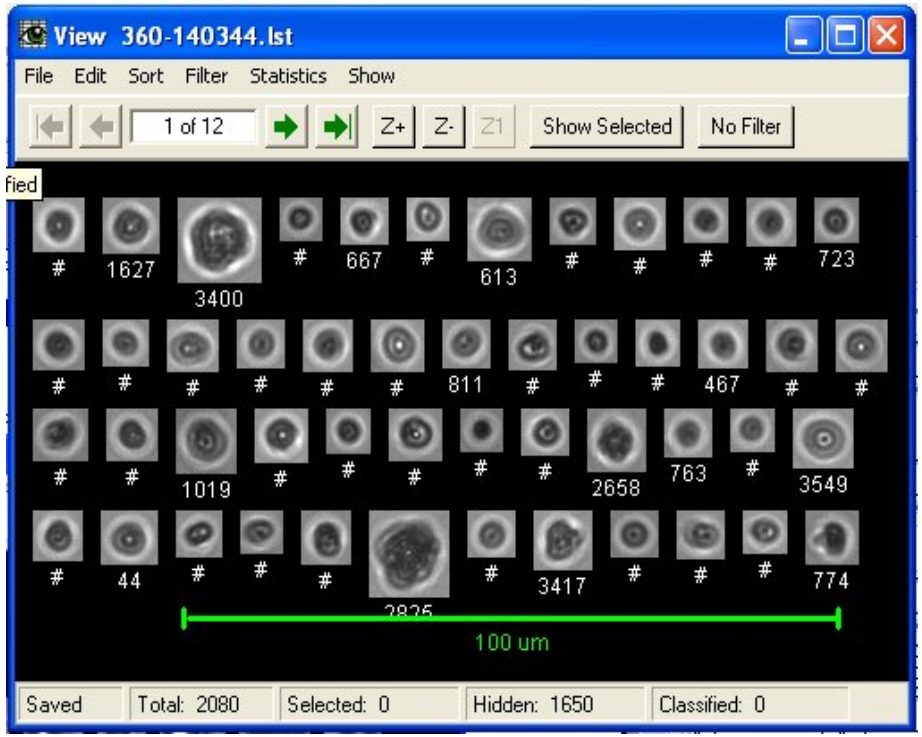
Iron sulfide particles

Distinguishing between two would be easy if they looked like this

Droplets vs. Particles



Agglomerated droplets

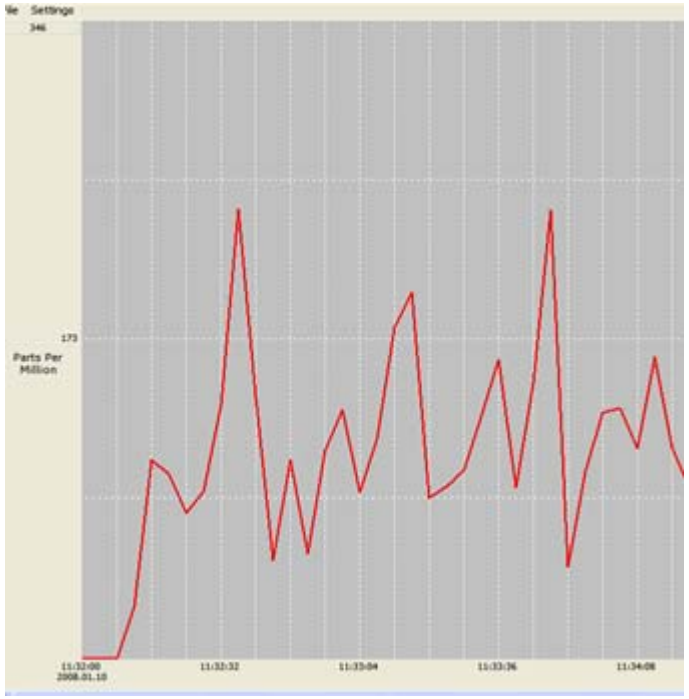


Sand

Pattern Matching Algorithm

- User selects examples of droplets, iron sulfide, sand particles
- Software looks at many size/shape values
- Chooses how to discriminate
- Bins each particle by type, counts
- Can calculate ppm oil

ppm



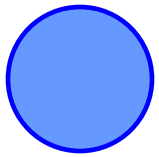
time

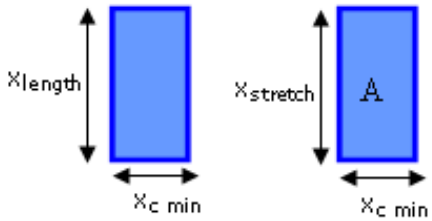
Refining: Catalysts

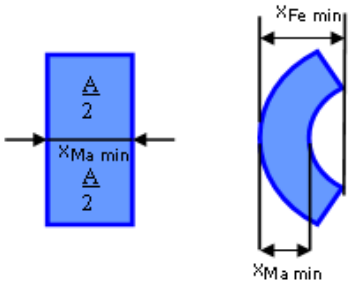
- Size, shape and specific surface area (SSA) affect catalyst performance
- Measure size using laser diffraction
- Measure size/shape by CAMSIZER
- Measure SSA using BET: SA-9600
  - Flowing gas BET method
  - Low price,operating costs, maintenance
  - Easy to use, fastest measurement time
  - No vacuum system required
  - Single or multi-point
  - Up to three samples simultaneously

Catalysts Size/Shape by CAMSIZER

- Spherical catalysts
  - Easy, no special effort
- Cylindrical catalysts
  - Length, width
- Bended extrudates
  - Use other parameters





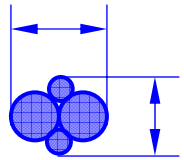


$$x_{length} = \sqrt{(x_{Fe\max})^2 - (x_{c\min})^2}$$

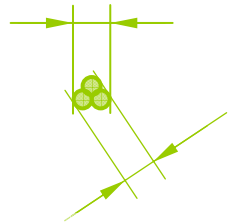
$$x_{stretch} = \frac{A}{x_{c\min}}$$

Catalysts Size/Shape by CAMSIZER

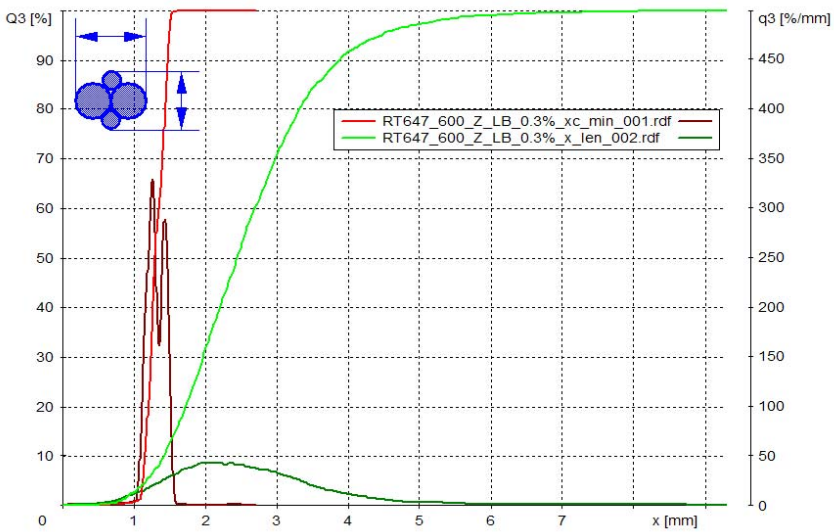
- Tri & quadralobe
  - Possible to distinguish between different diamteters
  - Shorter green distribution = length
  - Taller maroon distribution= width



Quadralobe catalysts



Trilobe catalysts



Summary

- Particle size, shape, surface area, other properties important from exploration to refining
- Key applications:
  - Drilling fluid
  - Proppants
  - Oil/water/solids separation
  - Catalysts
- Talk to HORIBA about all these requirements

Thank-you