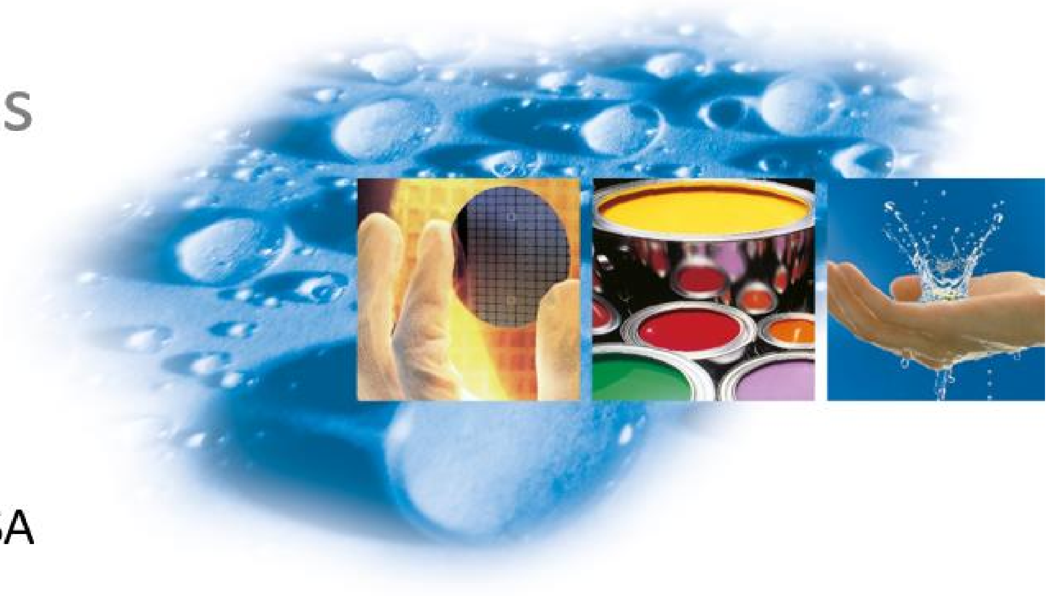
Coatings Trends & Technologies SUMMIT

Modern techniques in studies of interfacial behavior



Hieu Ngo, DataPhysics Instruments USA

dataphysics

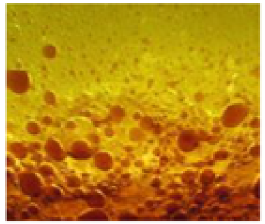
Emulsion and Dispersion Stability Device

- Instrument for analysing stability of emulsion and dispersion
- Temperature could be controlled to accelerate aging and simulate real-world conditions
- Uses a light-scattering technique to detect movement of particles over time, position, and temperature.

Classification of multi-component systems

- homogeneous mixtures
  mixed at the molecular level ("molecularly disperse") → one-phase
  - gas mixtures, alloys (metal + metal), solutions (solid, liquid or gas in liquid)

- heterogeneous mixtures (= dispersions)
  different compounds in distinct areas ("coarsely disperse") → multi-phase
  - solid in solid: blend (e.g. granite), muck (e.g. flint)
  - gas in solid: rigid foam (e.g. sponge)
  - solid in liquid: suspension (e.g. slurry)
  - liquid in liquid: emulsion (e.g. milk)
  - gas in liquid: foam (e.g. beer foam)
  - solid in gas: smoke, dust (e.g. cigarette smoke) } aerosols
  - liquid in gas: steam, vapour, fog } aerosols



- *the Device is designed to investigate liquid heterogeneous mixtures (i.e. suspensions, emulsions, foams) regarding their stability – or rather their de-mixing/destabilization is monitored…*

Scan Tower ST

→ Up to 6 scan towers ST can be connected simultaneously to the Main unit

Comprises:

→ Thermostatable **measuring chamber** into which the sample container is inserted from the top

→ Measuring chamber lid with electric motor (controlled via touch display or software)

→ Temperature sensor in the sample chamber

→ **Scanning plate** and associated electronics (stepper motor...)

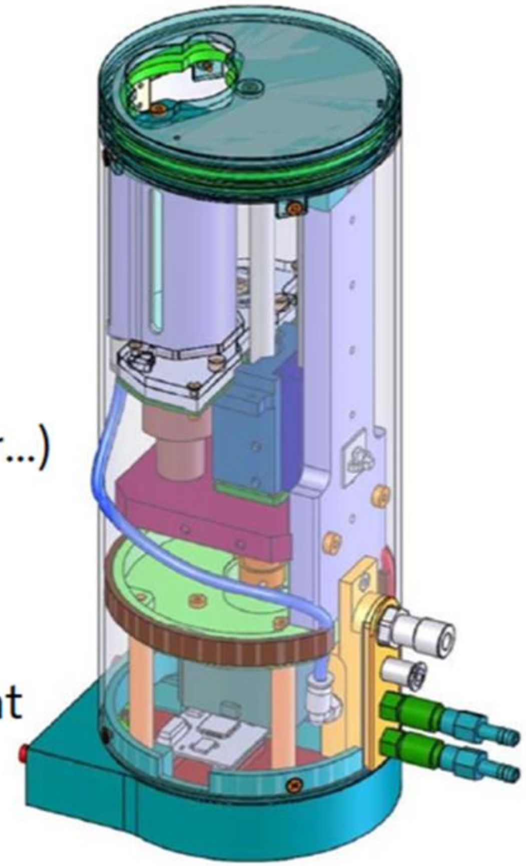
→ Protective liquid collection tray above electronics

Ports: (from bottom to top)

→ Liquid connections for cooling fluid/to thermo- or cryostat

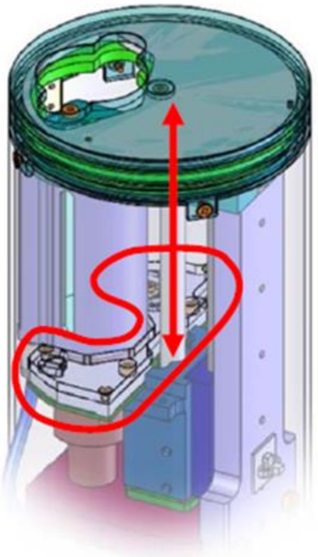
→ Gas connection for purging gas against condensation

→ Drain for liquid in the collection tray



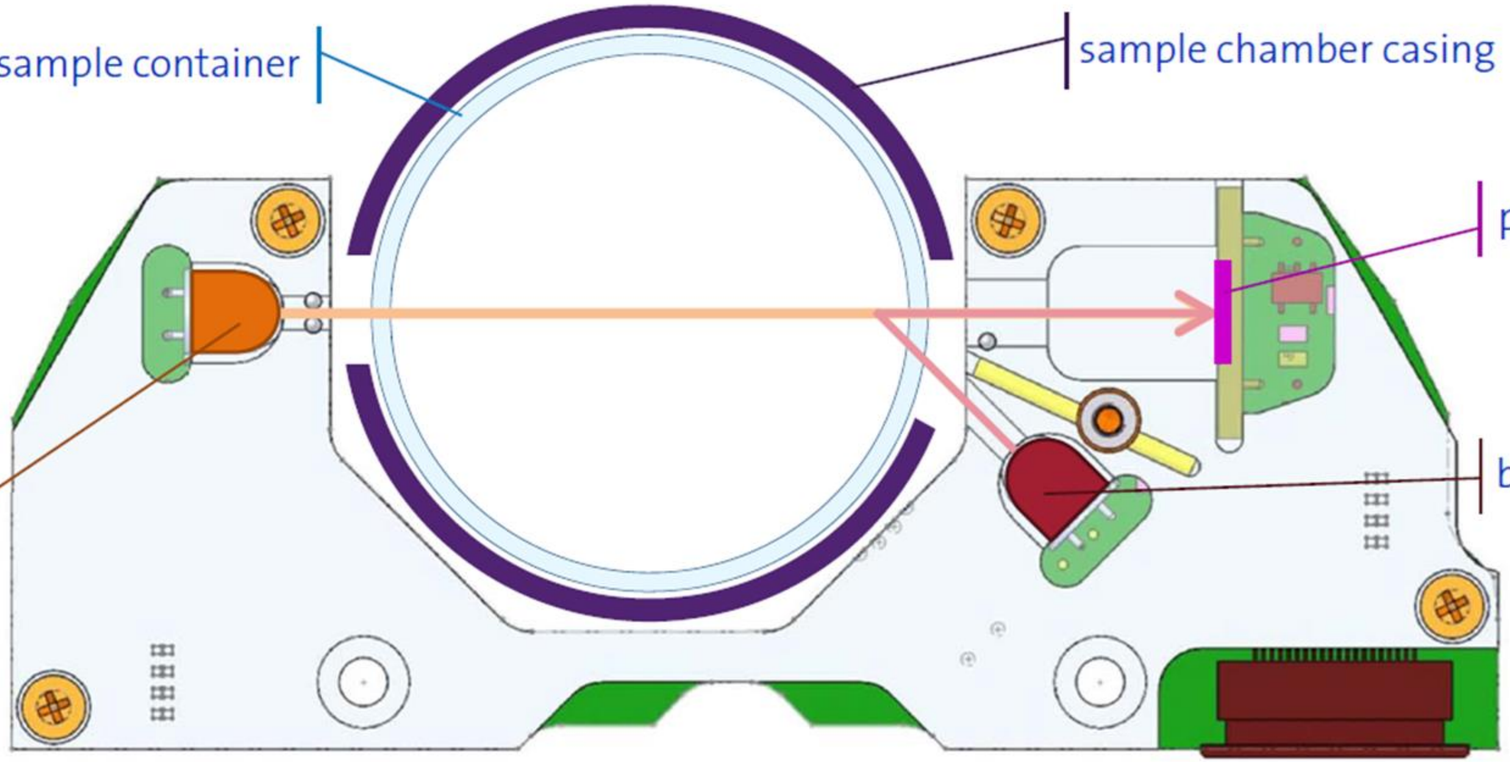
Scanning plate: Measuring principle

→ Centerpiece of the scan tower!



→ "Scanning" → Plate is moved along the sample height & collects data as a function of position

→ "Optical analysis" → Data: transmission & backscattering (in 45° angle)

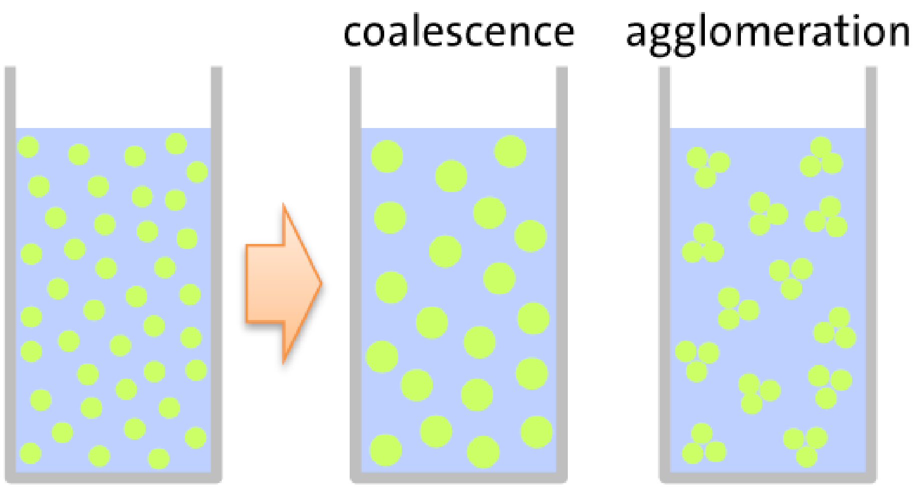


Destabilisation mechanisms of fluid mixtures

→ particle size increase (often irreversible) — global

- → coalescence (merging of particles)
  e.g. breakup of an emulsion or foam
- → agglomeration, aggregation
  e.g. precipitation of salt from brine solution

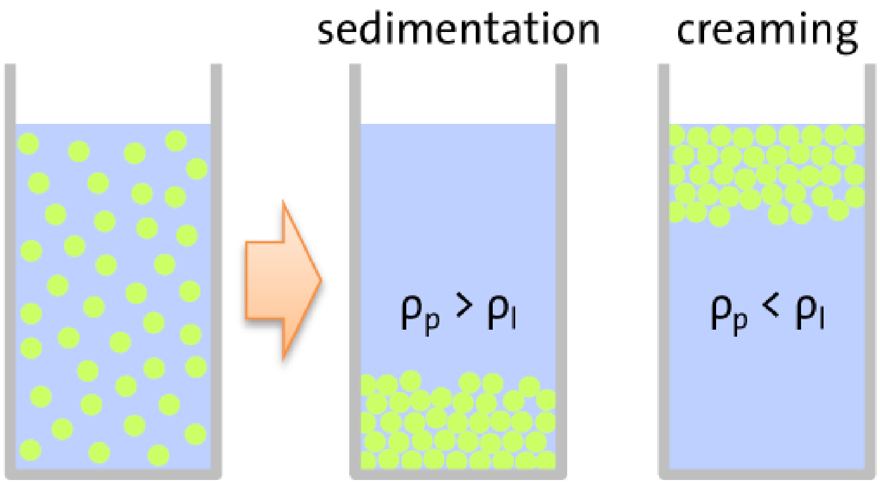
driving force: *reduction the of interfacial area between different phases (which reduces the energy of the system due to the interfacial tension)*



→ particle migration (usually reversible) — local: concentration changes

- → sedimentation
  e.g. solid particles in slurry
- → creaming
  e.g. fat droplets in milk

driving force: *gravity acting on phases with different density*

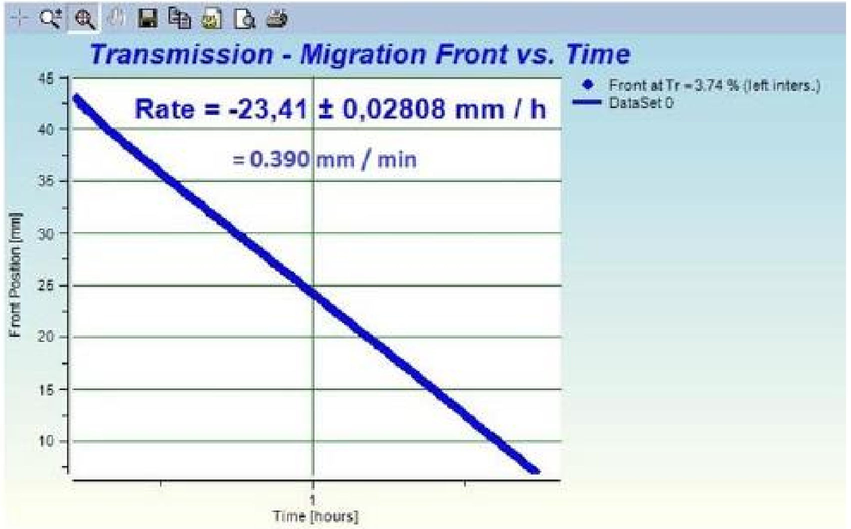


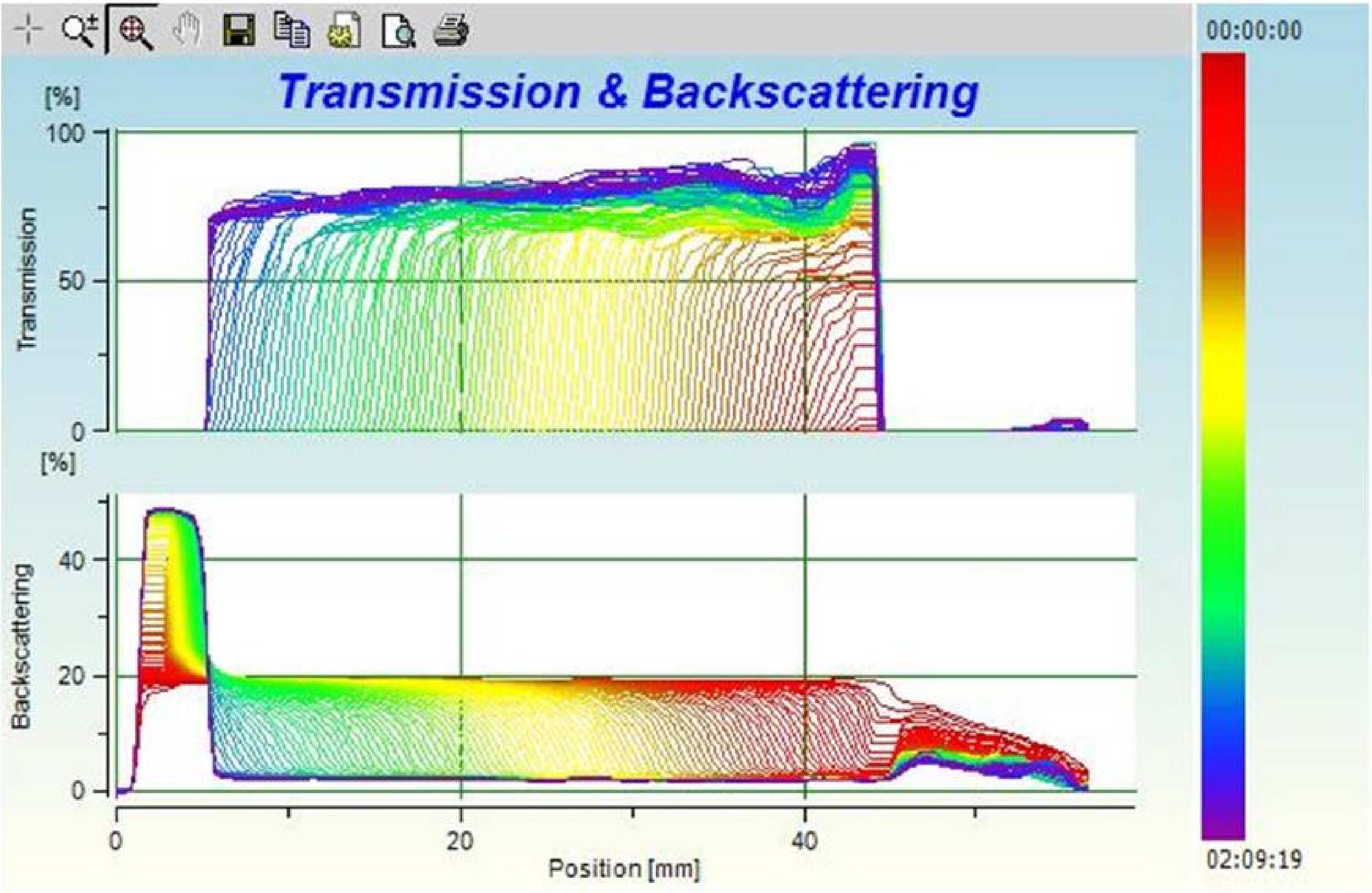
→ both mechanisms are recognized by optical analysis!

Sedimentation of polystyrene

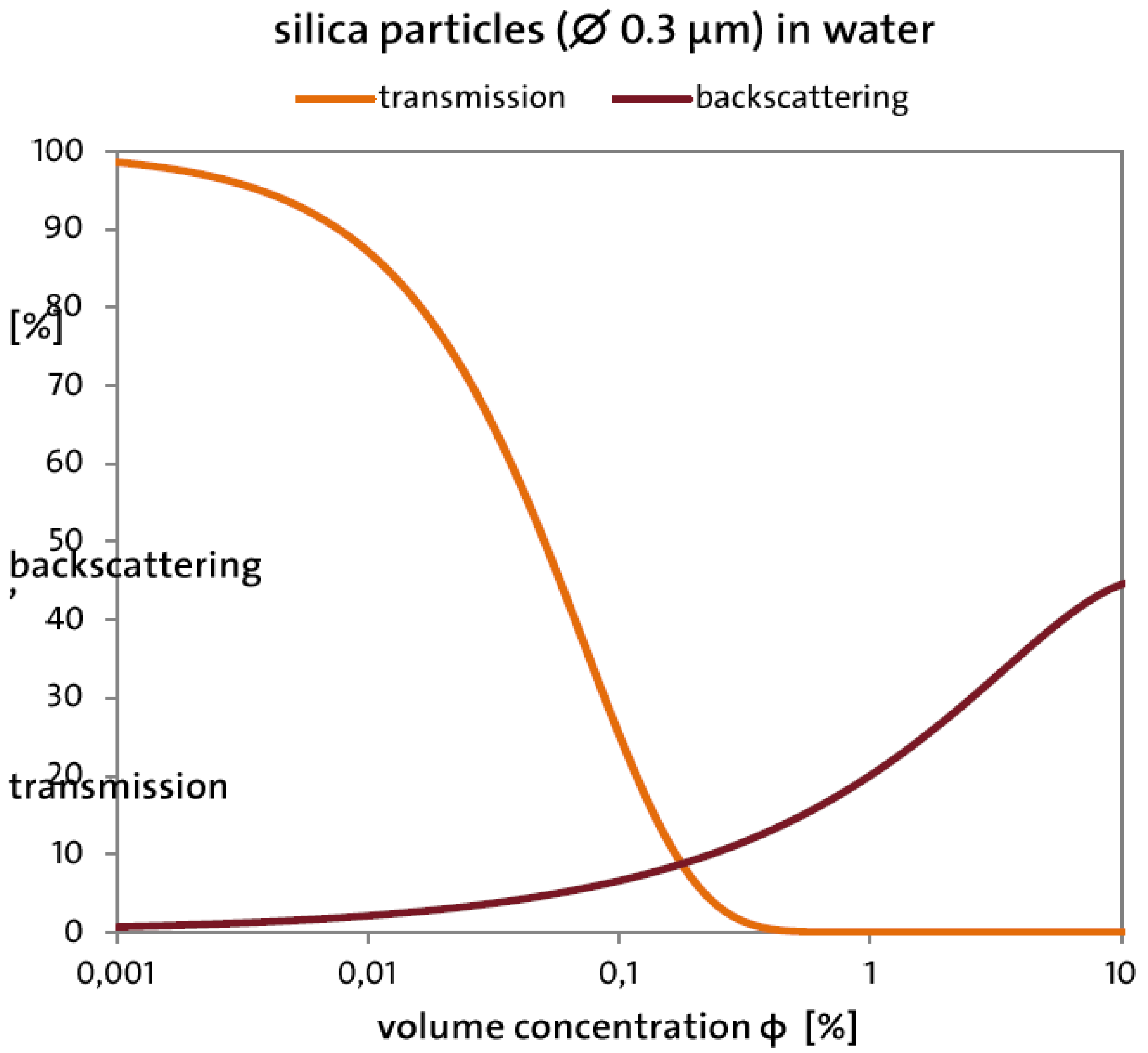
→ d = 15.3 µm

T = 30 °C





Optical analysis: Concentration dependence



increasing volume concentration ϕ:

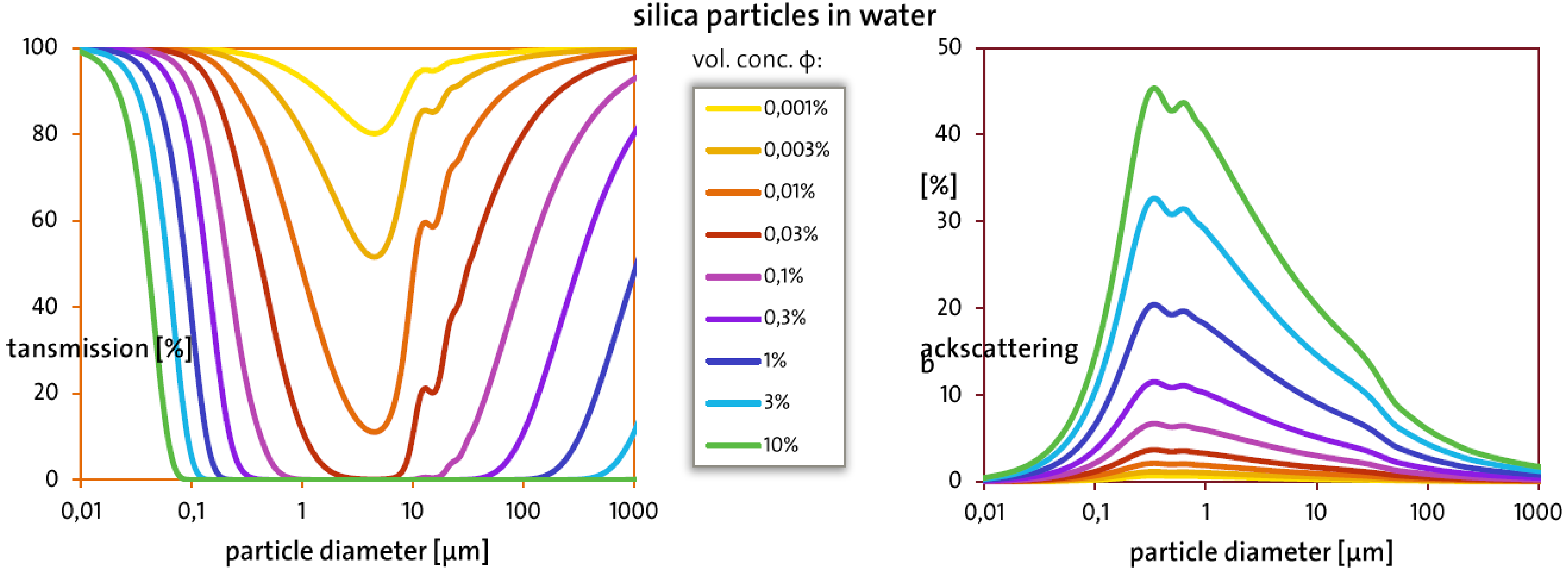
→ transmission decreases

→ more and more particles obstruct light passage

→ backscattering increases

→ more particles → higher backscattering potential

Optical analysis: Particle size dependence



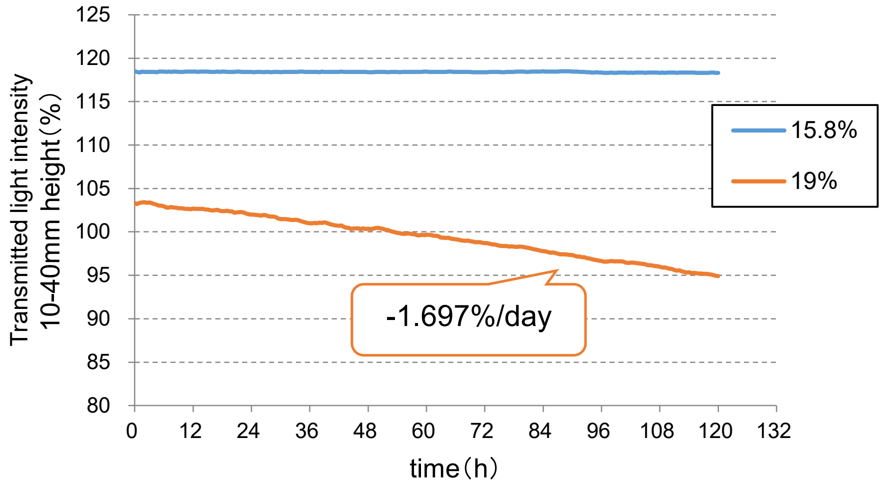
- → at constant volume concentration ϕ the number of particles decreases with increasing particle diameter!
  - → first particles become big enough to be encountered by the incident light
    → decreasing transmission, increasing backscattering
  - → then there are big but merely few particles and light is likely to just pass by
    → increasing transmission, decreasing backscattering

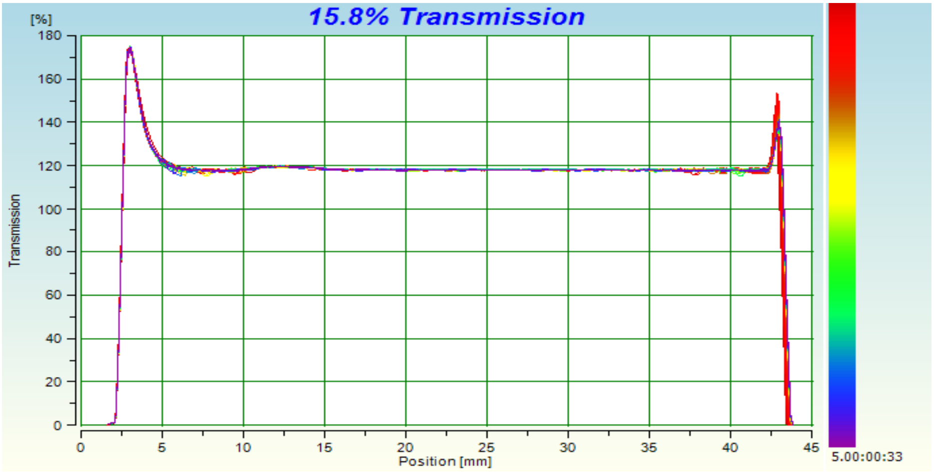
Example 1: Polymer Solutions

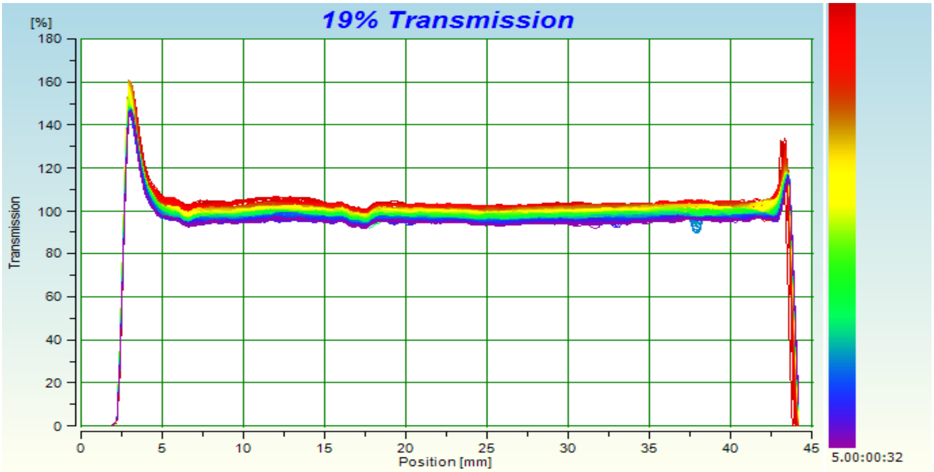
- Customer Information: Developer of polymers that are applied and processed as an aquatic solution to coat some surface by treatign it with the solution and setting it for drying
- Goal: Determination of the maximum polymer concentration that is table over a certain time.
- Samples: Customer sent us 2 samples with different concentrations of the polymer (15,8% polymer content and 19% polymer content

Experiment: Transmission measured over 5 days at room temperature for both samples

Evaluation with peak height method.



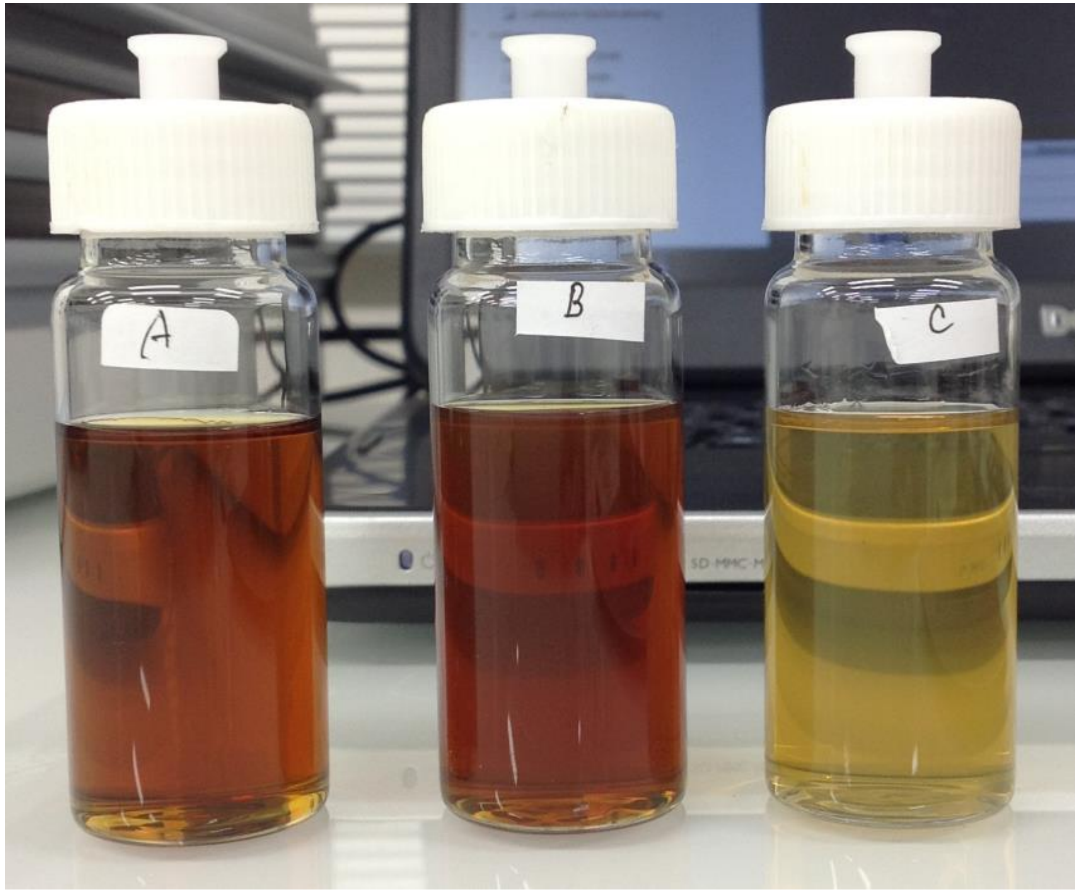




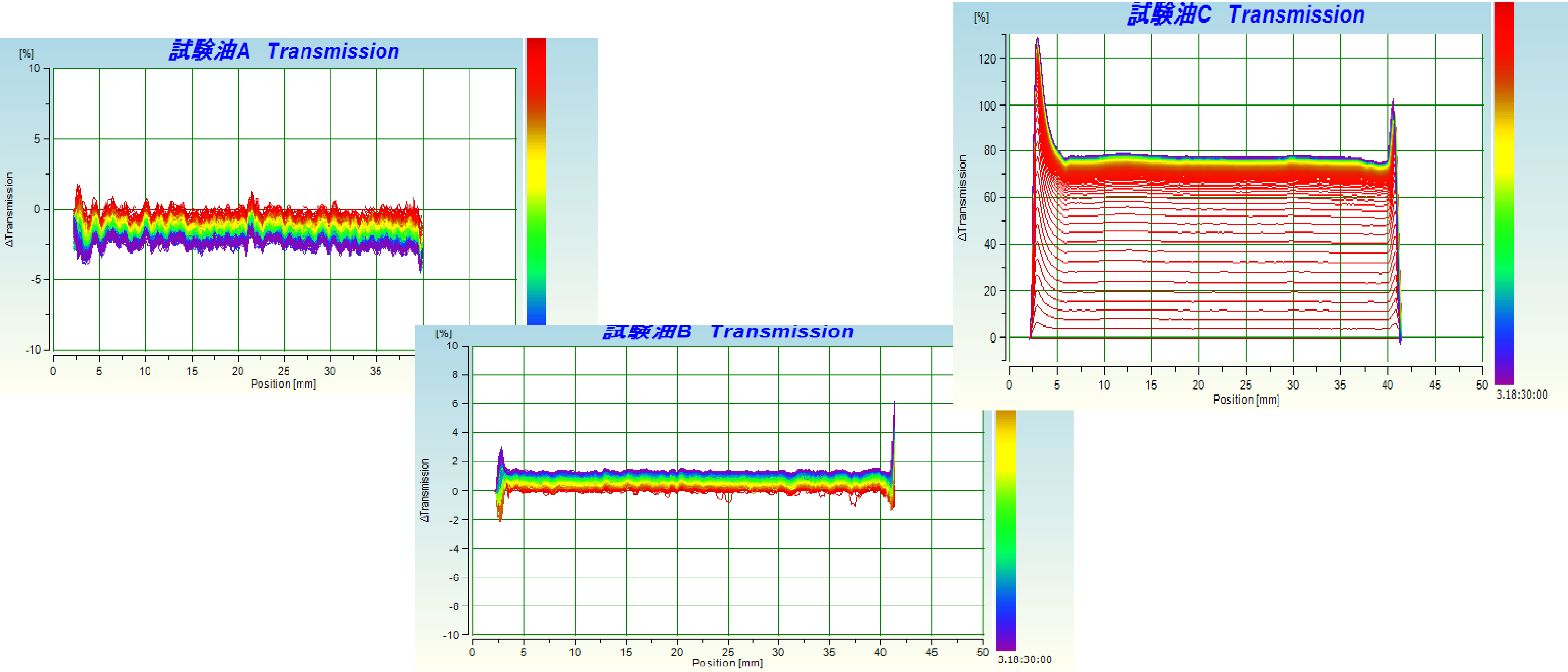
In the sample with 19% concentration, increasing cloudiness was observed over time.

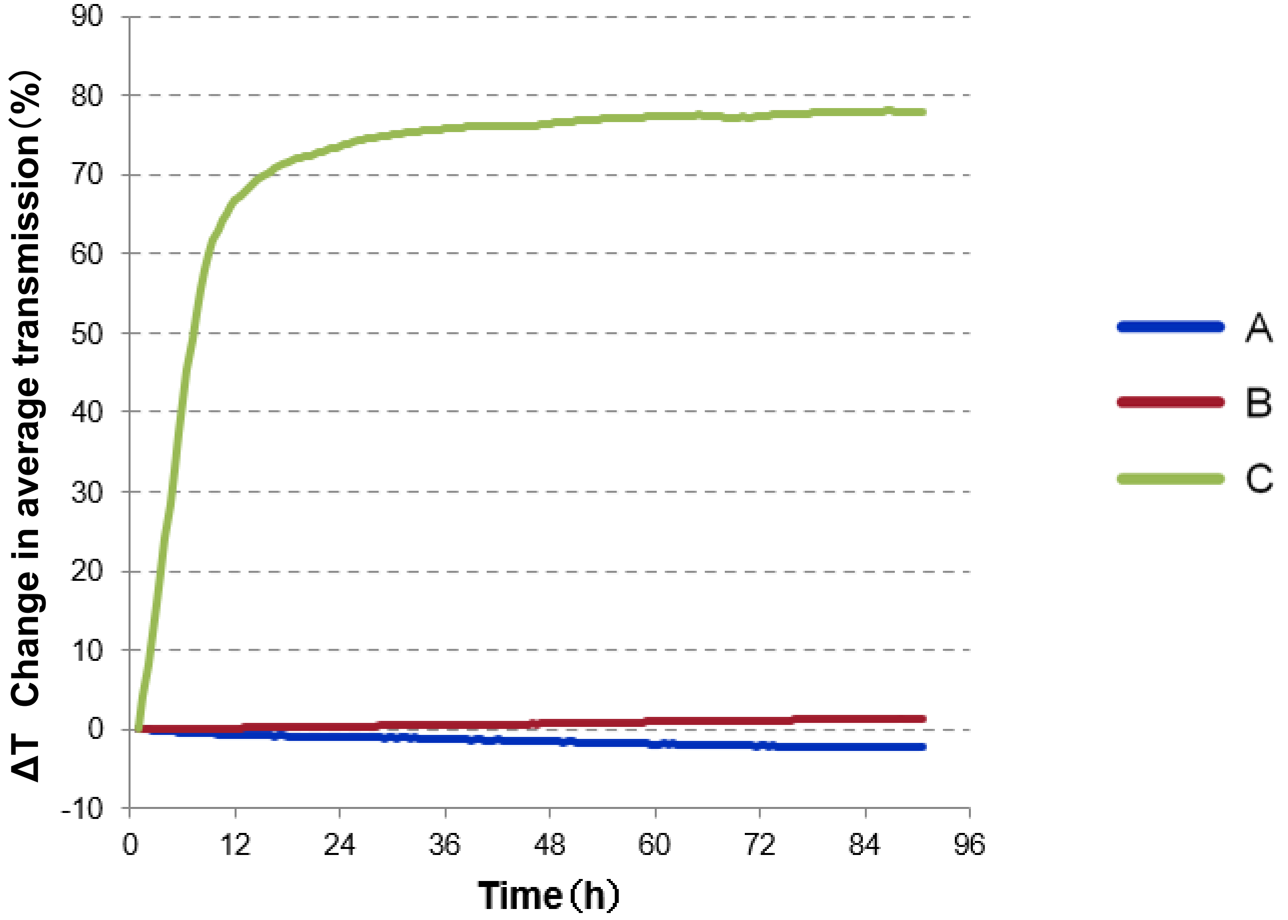
Example 2: Motor Oil

- Customer information: Developer of motor oils
- Process goal: Development of special oil with increased stability up to 60°C temp
- Samples for testing: 3 samples with different additives.
- Testing goal: Identification of the most stable product with the DataPhysics MS20



Experiment: Transmission measured for 3 days at 60 °C





	Change rate (%/day)	
	1-12 h	12-90 h
A	-0.58	
B	0.36	
C	164	2.24

Formulation C shows the lowest stability fast change especially in the first 12 h.

Stability Trend: B>A>C

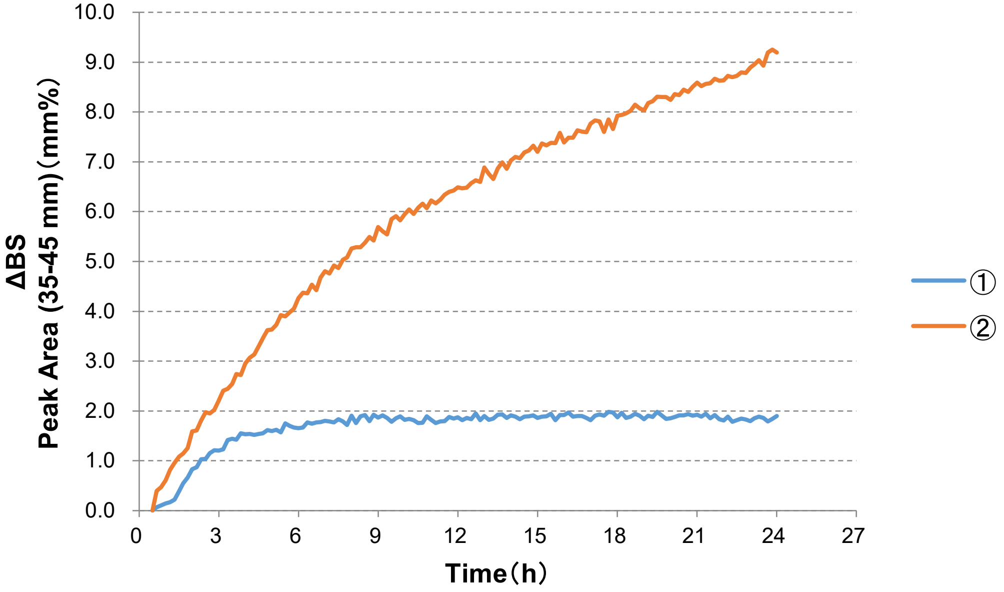
Example 3: Seasonings for processed meat

- Customer Information: Developer of food additives
- Goal: Increase shelf stability of a seasoning by addition of different emulsifiers
- Samples: Customer provided 2 samples in with different emulsifiers

Experiment: Backscattering measured for 1 day

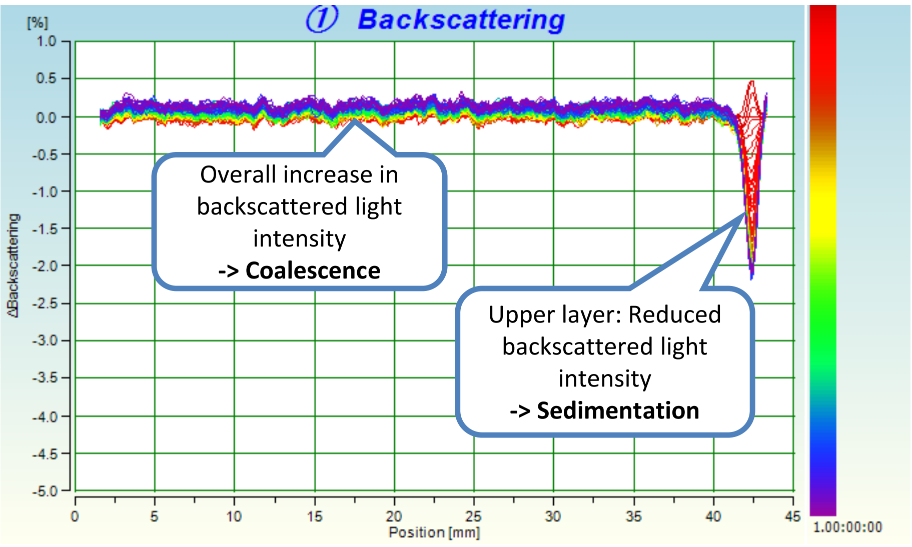
at room temperature for samples 1-2

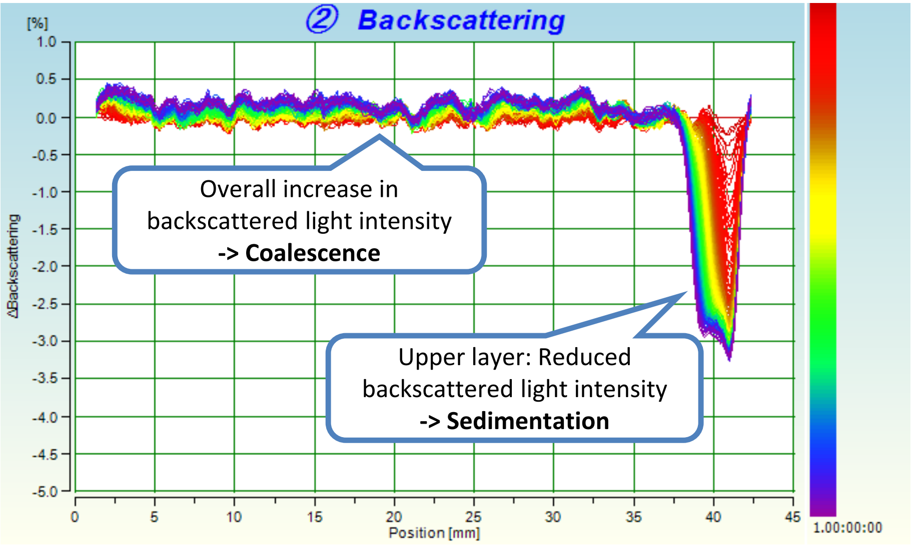
Samples show some sedimentation on top and a possible coagulation



	Peak Area(mm%/h)	
	0-6 h	6-24 h
①	0.29	-
②	0.72	0.25

Stability trend: ①>②



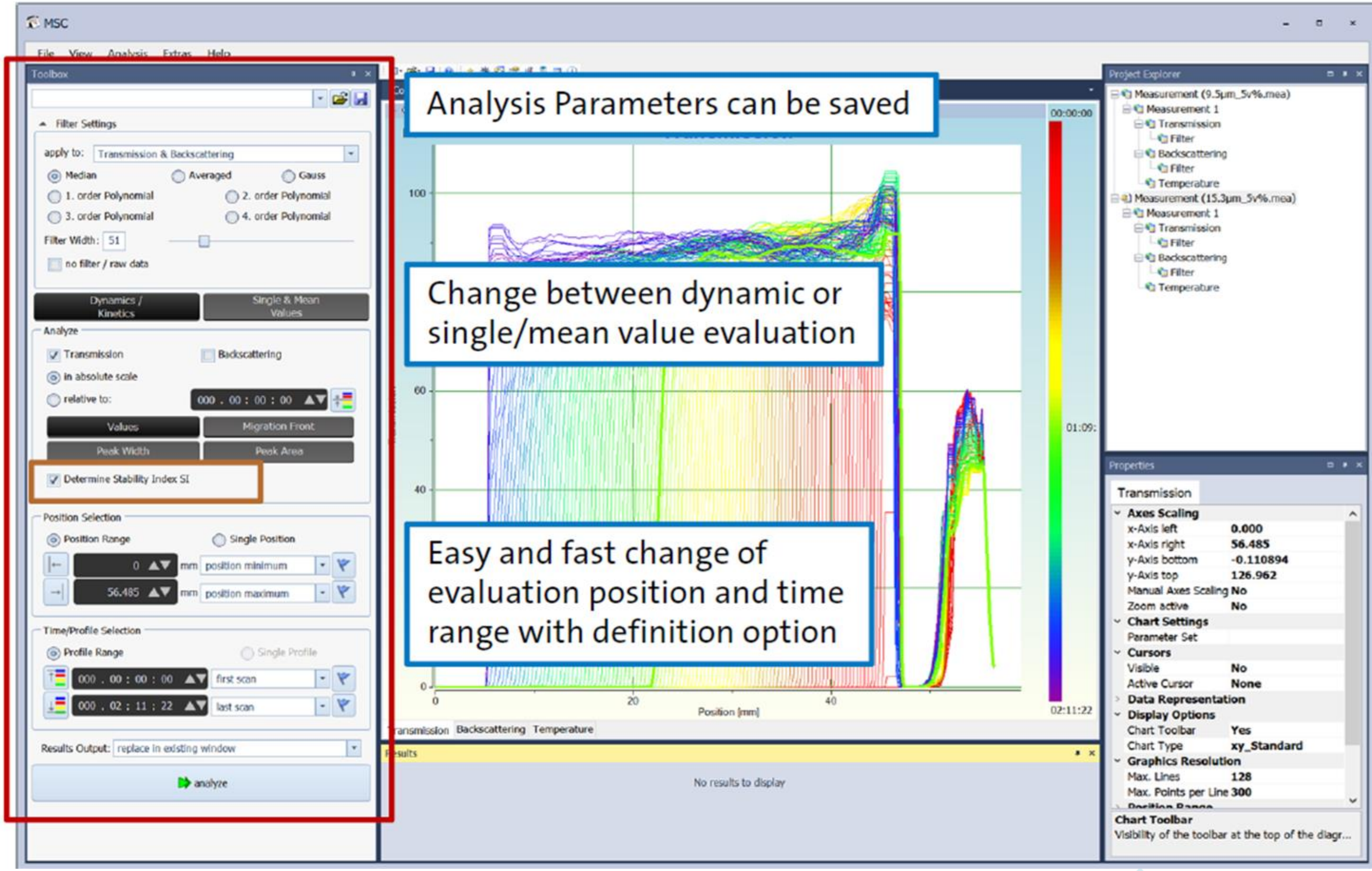


Analysis Software : New look of Analysis Methods

Various filtering methods live change of filter width

Simultaneous evaluation of transmission and backscattering possible

Choice how new analysis should be displayed
→ Possibility for direct comparison of evaluated results

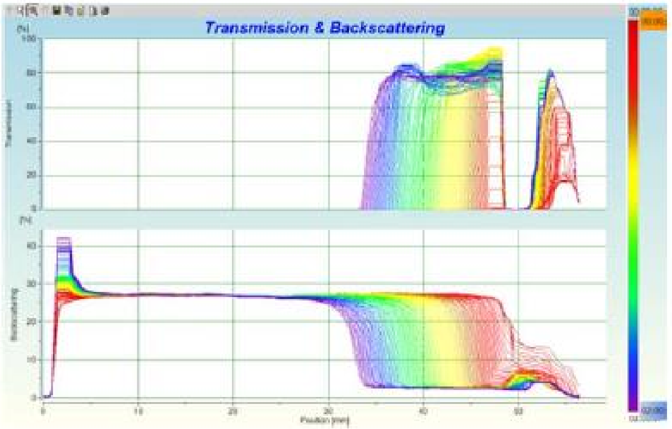


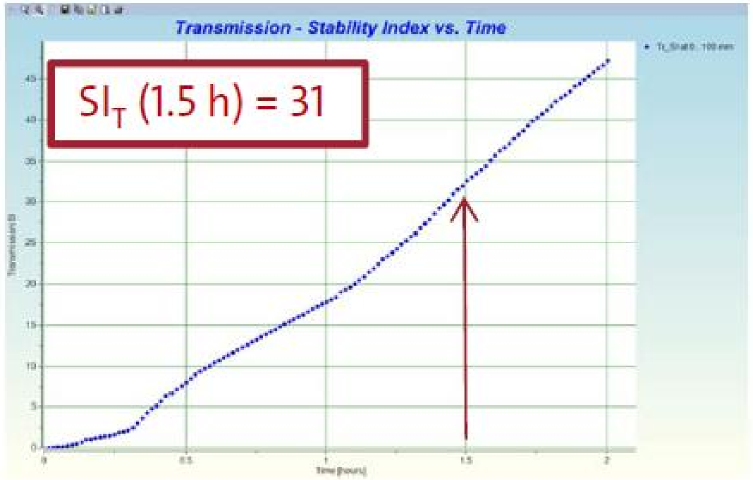
Determination of Stability Index (SI) within the Values method

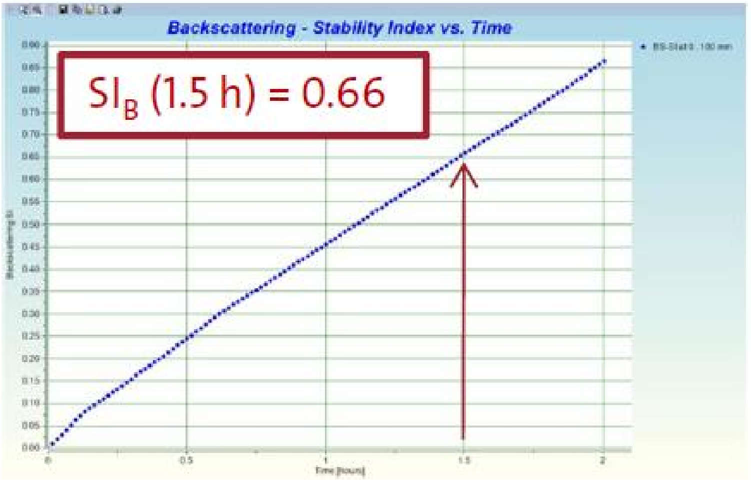
→ Stability of the system independent of the destabilization mechanism

→ *Which system is more stable?*

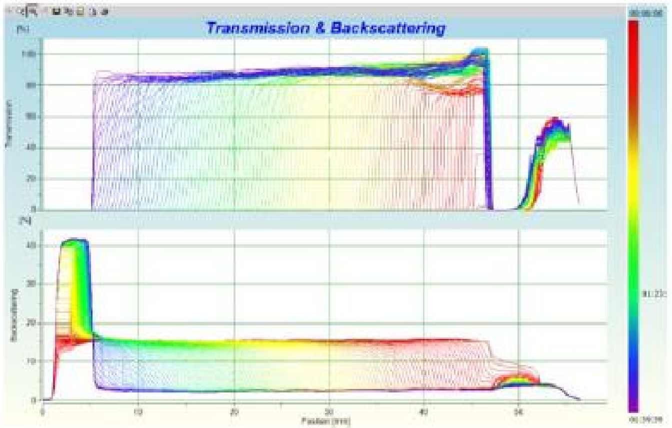
Sample 1:
Small Particles

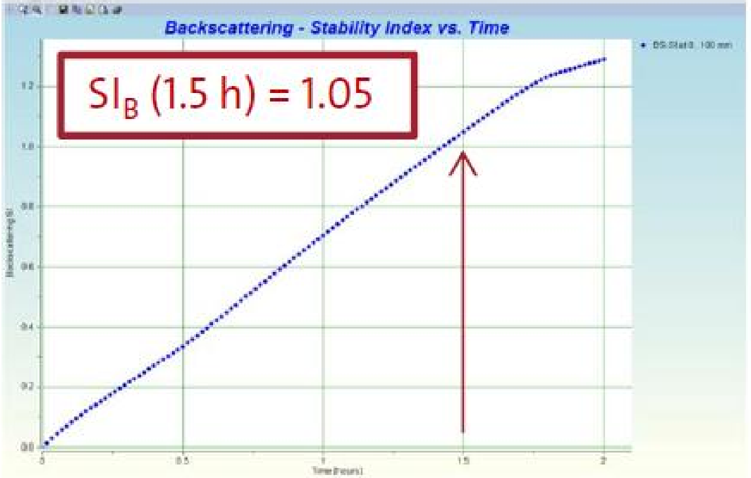






Sample 2:
Big Particles

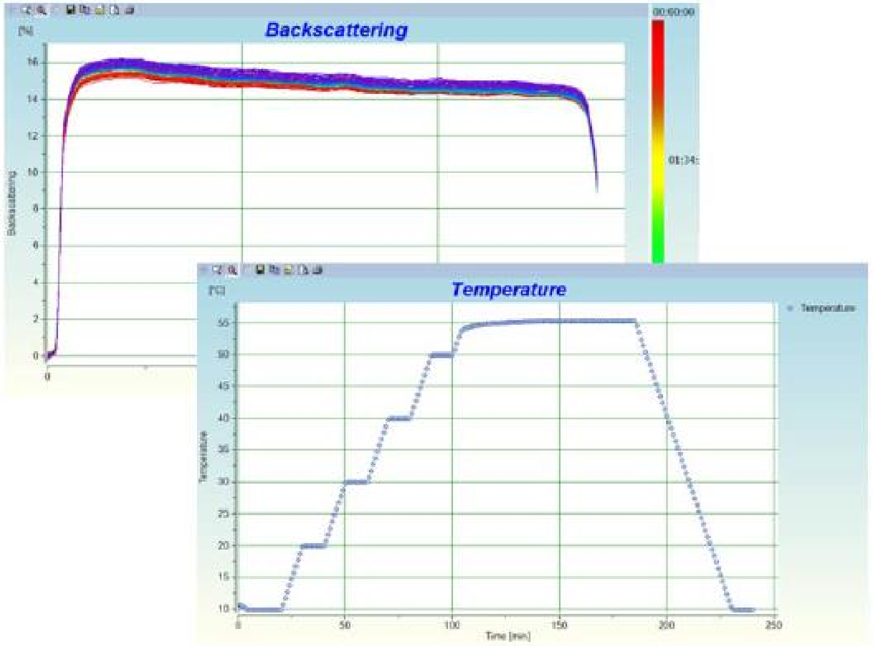


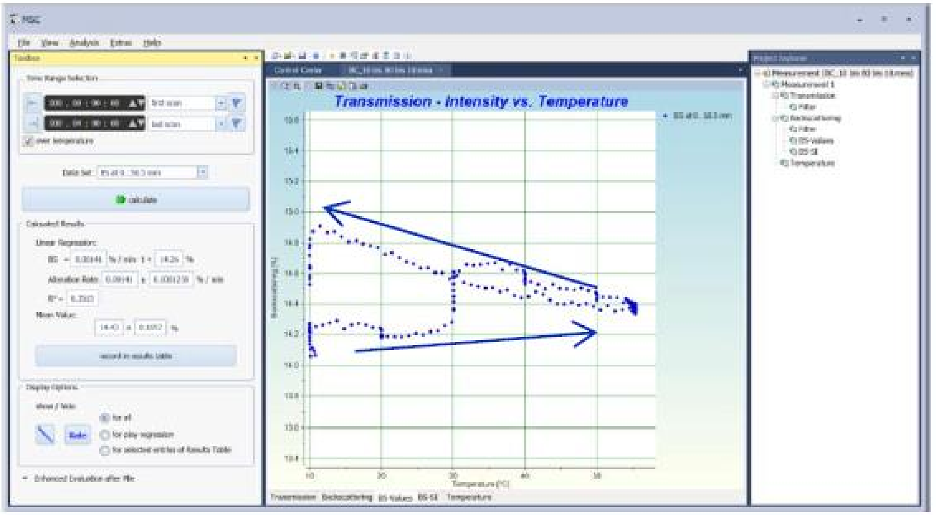


→ Sample 2 is less stable than sample 1

Graphical representation of 'Intensity vs. temperature'

→ Influence of temperature on backscattering and transmission intensity can directly be displayed

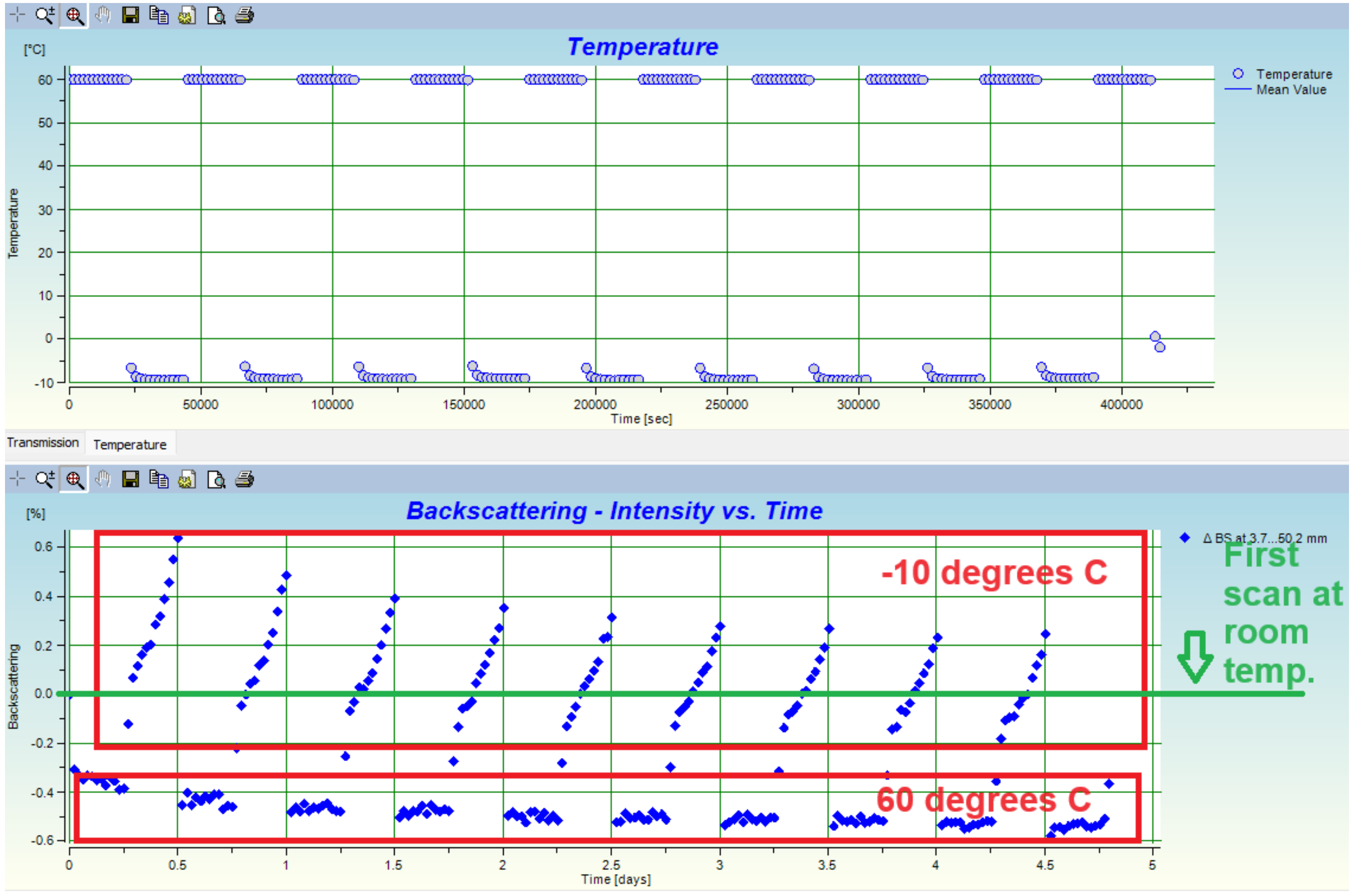


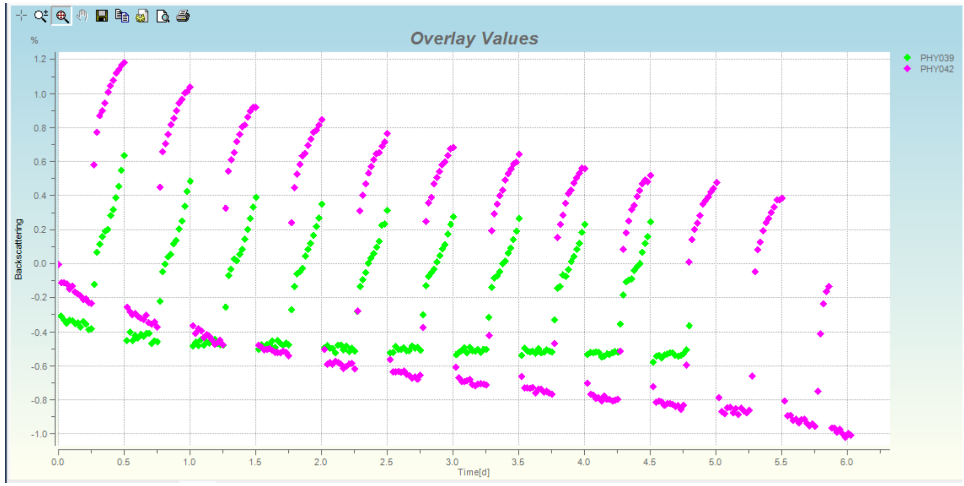


→ Sample destabilization with temperature is irreversible!

- Simple insulating sleeve of the tower allows temperature range for the measurement starting from -10 °C and up to 80 °C.
- Insulation of the tower makes it more heat efficient in the full range of temperatures.
- Measurements below 30 °C require external cooling circulator and dry gas purging to prevent condensation inside the tower.

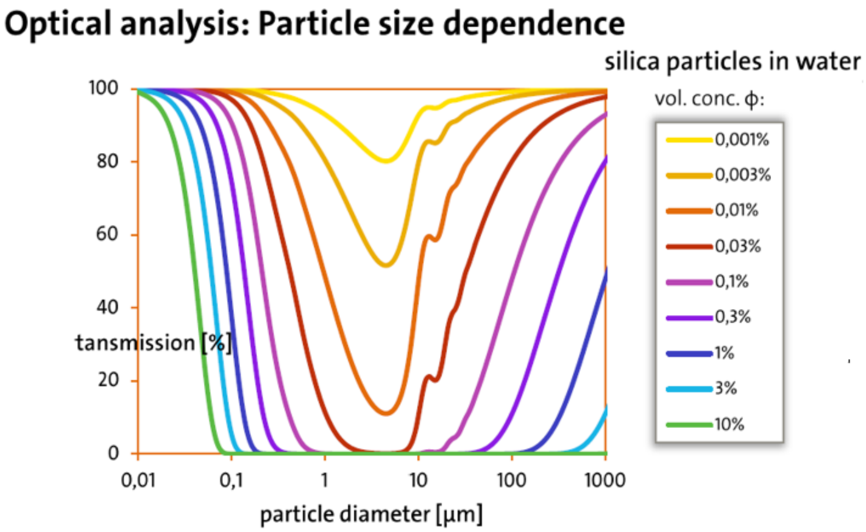
Backscattering of two samples cycling between -10 and +60°C

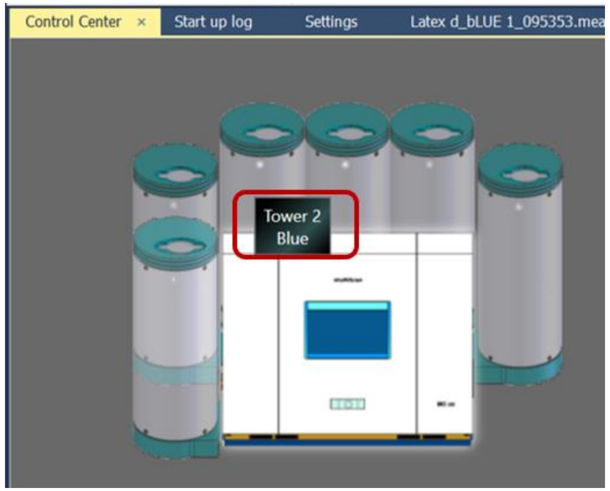


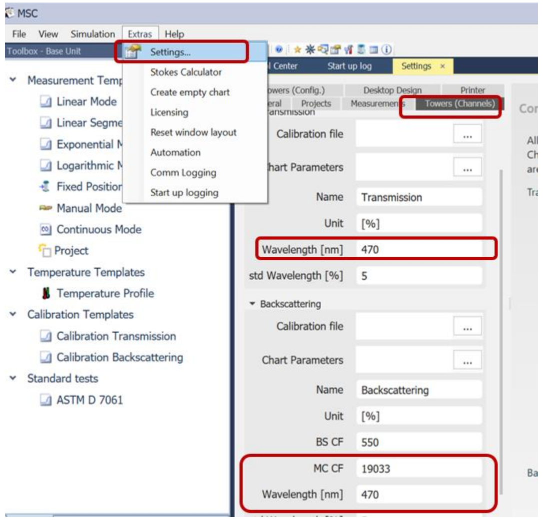


Increased sensitivity to small size particles by modified wavelength light source

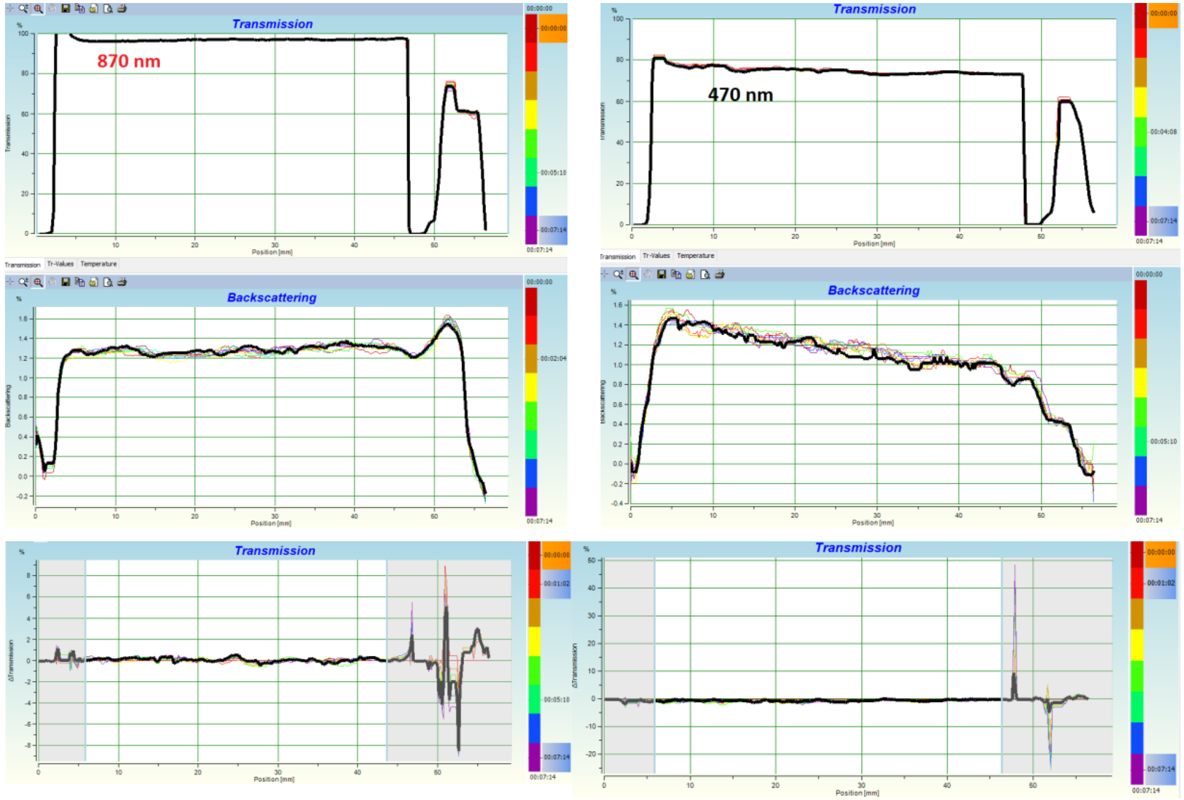
- 870 nm LED light source replaced with 470 nm.
- Changes made to LED light path slit.
- Software/firmware updates for automated detection of the measurement tower type.

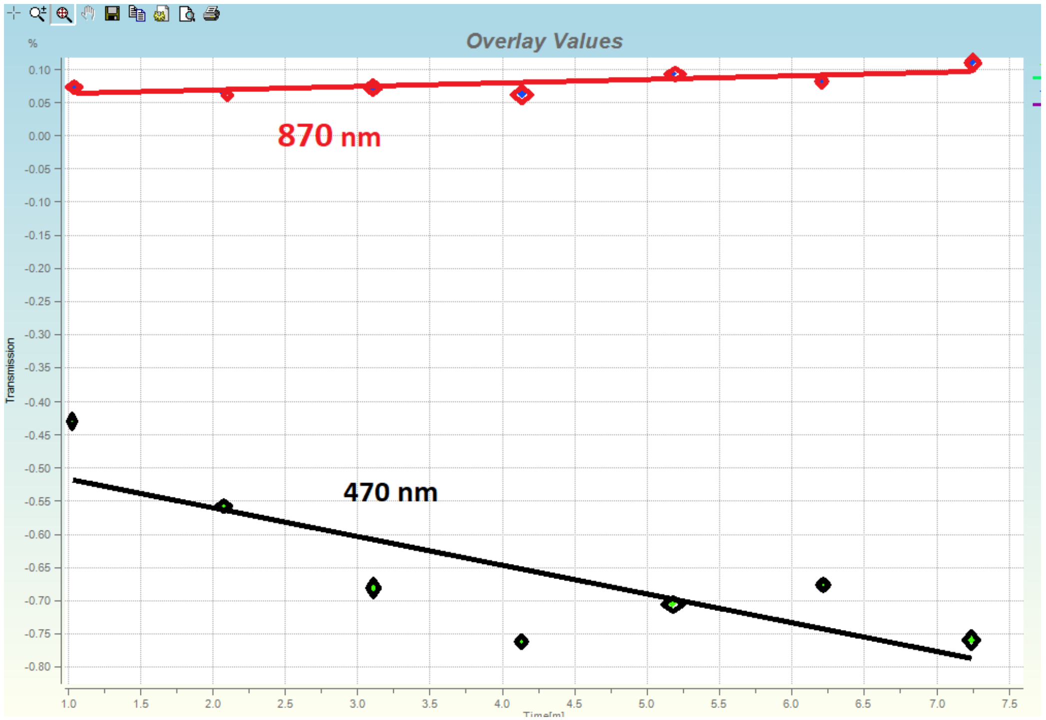






Increased sensitivity to small size particles by modified wavelength light source





→1. Same sample of SiO_2 (particle size around 60 nm) with 470 nm tower shows stronger affect on transmission intensity in comparison to 870 nm tower.

→2. 470 nm tower scans of the same sample show decrease in transmission intensity in just in 7 min, while 870 nm tower has much less change.

Increased sensitivity to small size particles by modified wavelength light source.

Polystyrene microsphere suspension in water, density 0.998 g/cm³, viscosity 1.002 mPas, absolute values of transmission and backscattering measured every 13 sec for 40 seconds.

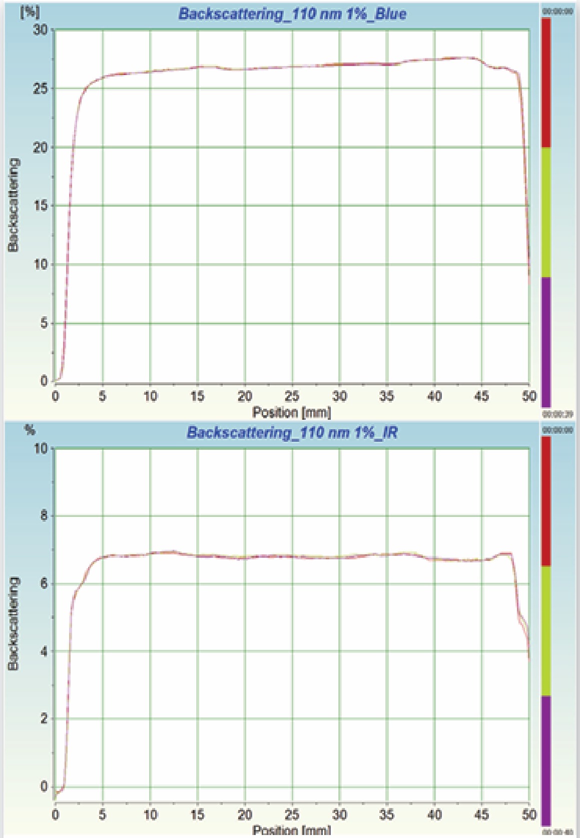
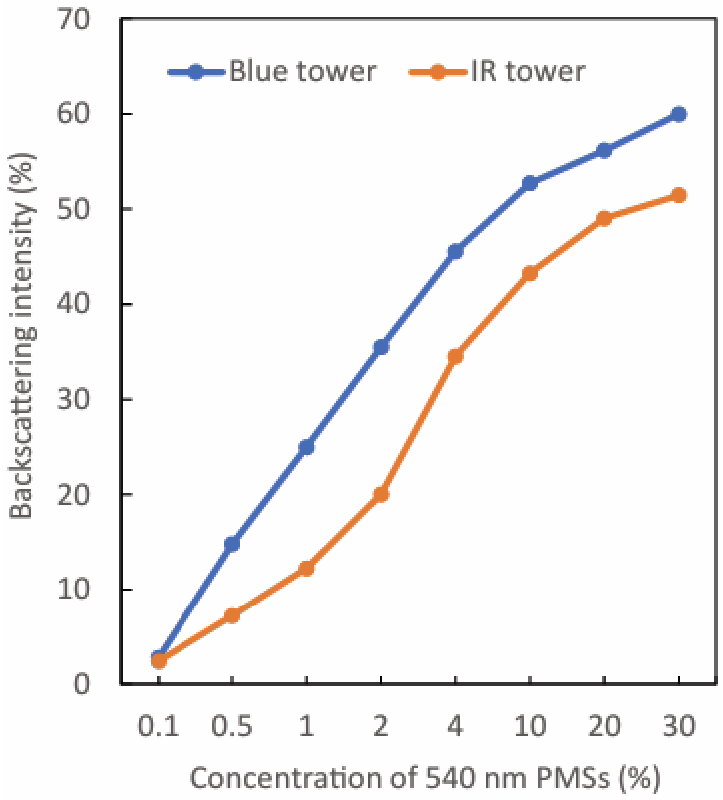
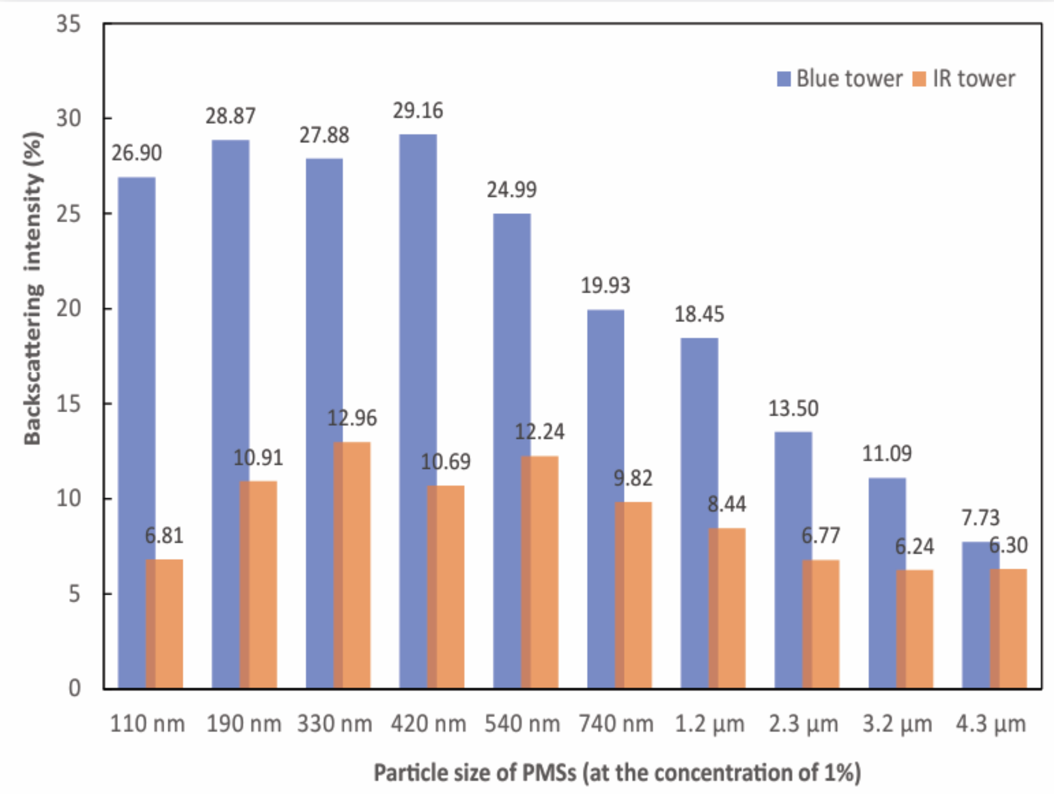


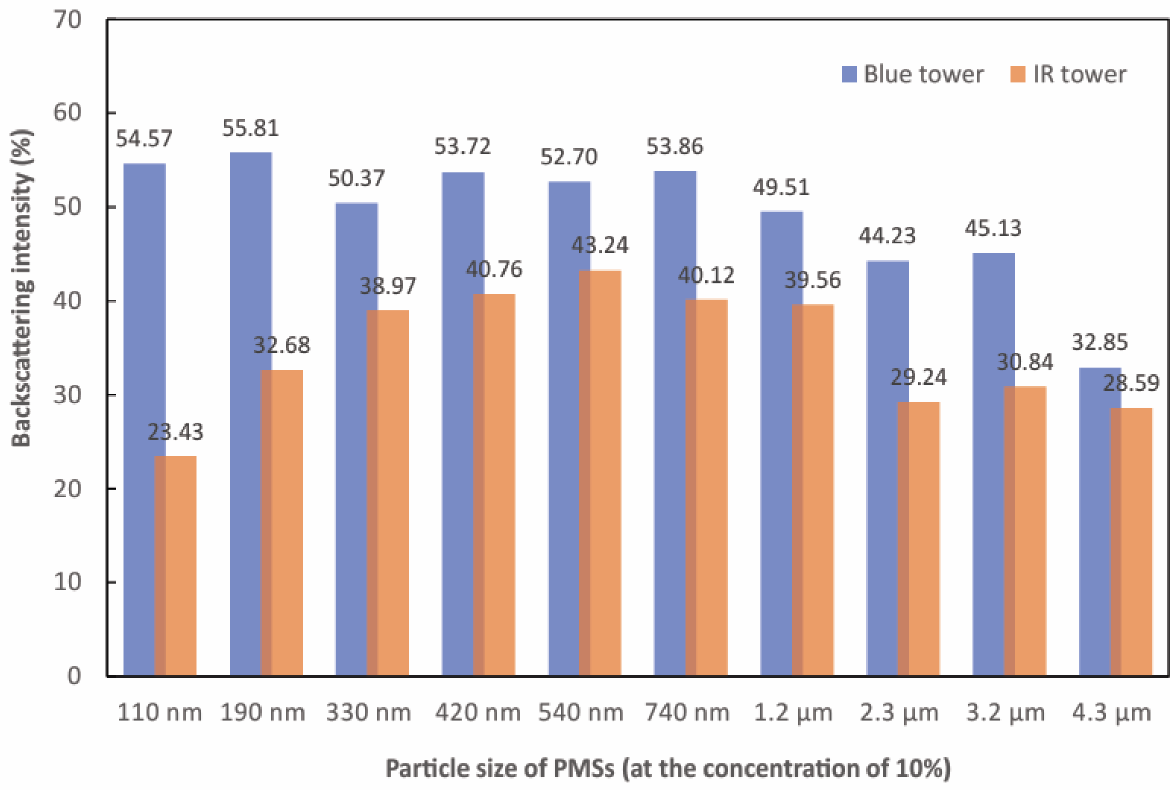
Fig. 3: Backscattering diagrams of 110 nm PMS at the volume concentration of 1% from Blue tower and IR tower, respectively

Table 1: Properties of the polymer microsphere suspensions

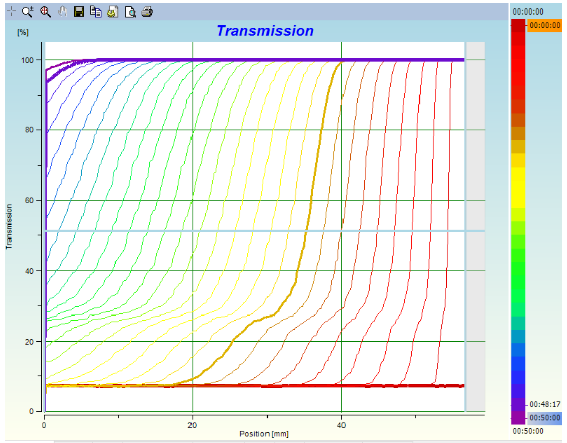
Particle size	Volume concentration (%)							
110 nm	-	-	1	-	-	10	-	-
190 nm	-	-	1	-	-	10	-	-
330 nm	-	-	1	-	-	10	-	-
420 nm	-	-	1	-	-	10	-	-
540 nm	0.1	0.5	1	2	4	10	20	30
740 nm	-	-	1	-	-	10	-	-
1.2 µm	-	-	1	-	-	10	-	-
2.3 µm	-	-	1	-	-	10	-	-
3.2 µm	-	-	1	-	-	10	-	-
4.3 µm	-	-	1	-	-	10	-	-

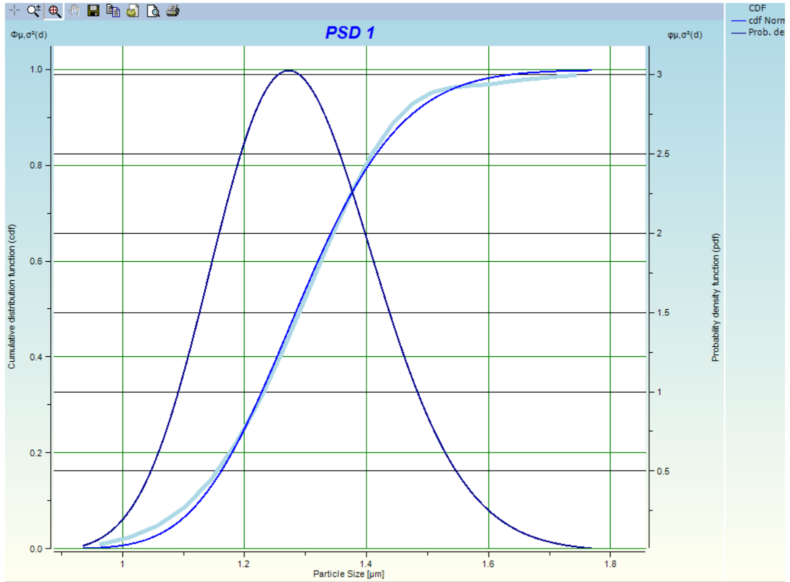


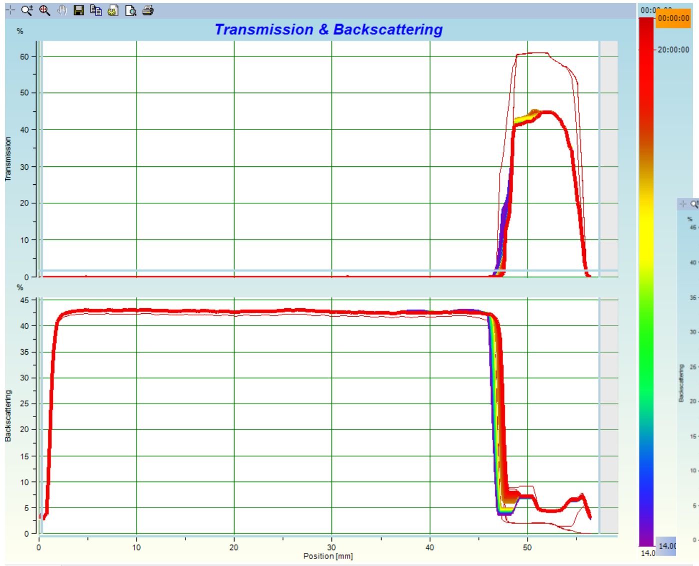

Blue tower
IR tower
Backscattering intensity (%)
35
30
25
20
15
10
5
0
26.90
6.81
28.87
10.91
27.88
12.96
29.16
10.69
24.99
12.24
19.93
9.82
18.45
8.44
13.50
6.77
11.09
6.24
7.73
6.30
110 nm
190 nm
330 nm
420 nm
540 nm
740 nm
1.2 µm
2.3 µm
3.2 µm
4.3 µm
Particle size of PMSs (at the concentration of 1%)

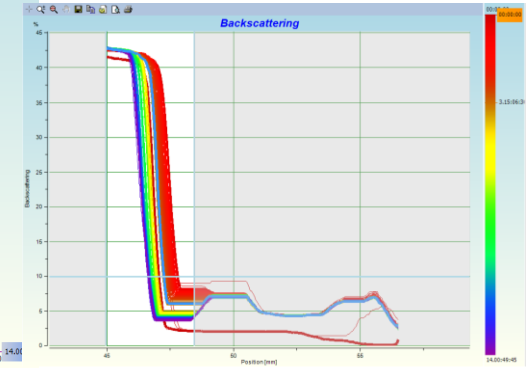

Blue tower
IR tower
Backscattering intensity (%)
70
60
50
40
30
20
10
0
54.57
23.43
55.81
32.68
50.37
38.97
53.72
40.76
52.70
43.24
53.86
40.12
49.51
39.56
44.23
29.24
45.13
30.84
32.85
28.59
110 nm
190 nm
330 nm
420 nm
540 nm
740 nm
1.2 µm
2.3 µm
3.2 µm
4.3 µm
Particle size of PMSs (at the concentration of 10%)

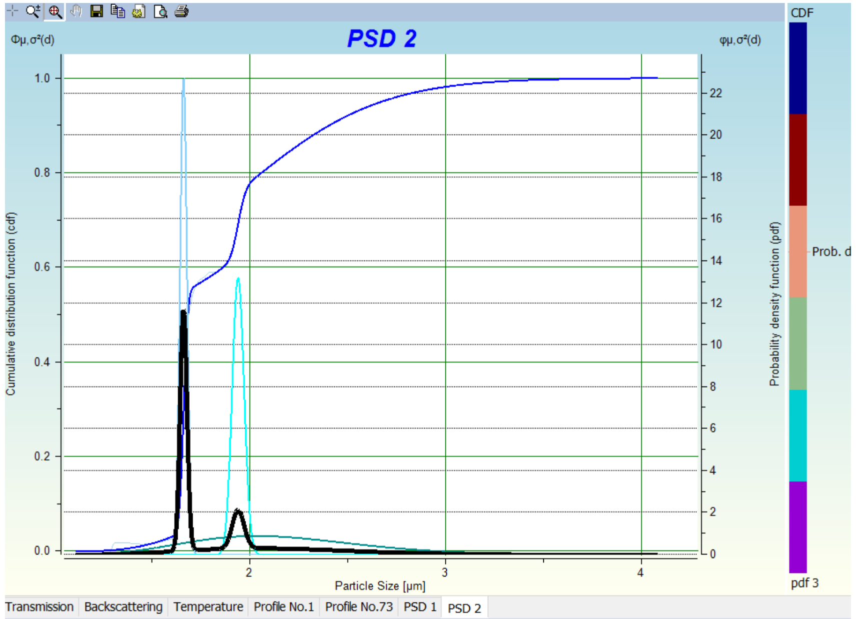
Calculation of average particle size and particle size distribution based on particle migration due to density difference.





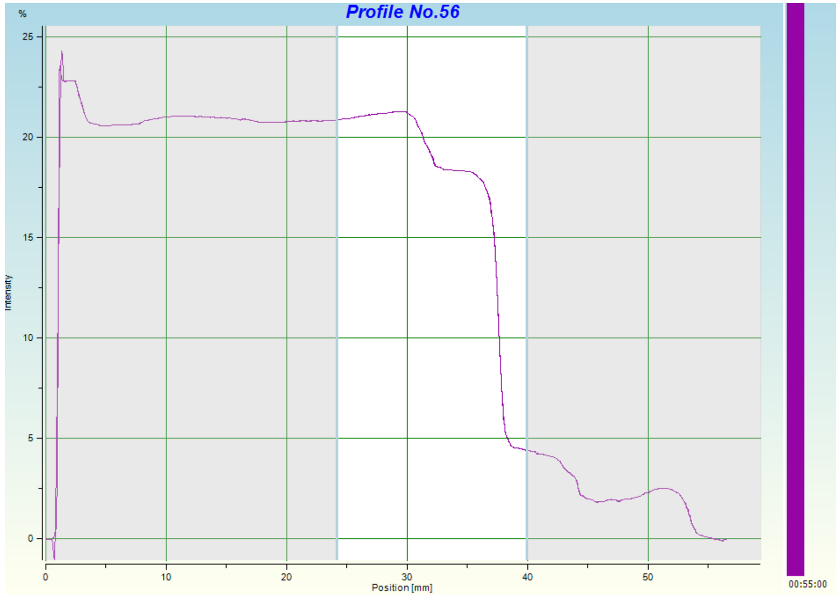


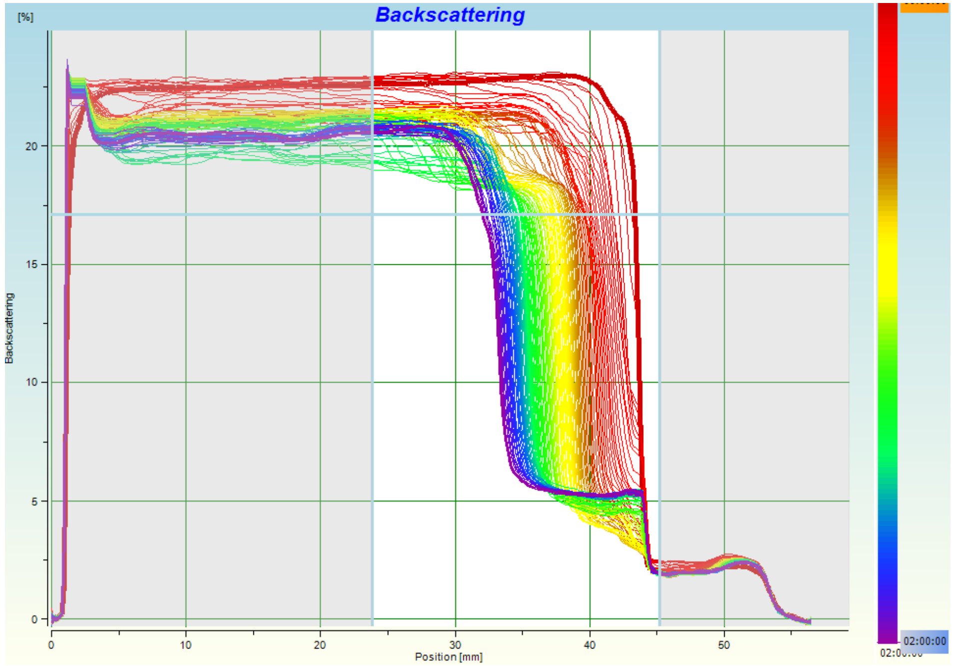


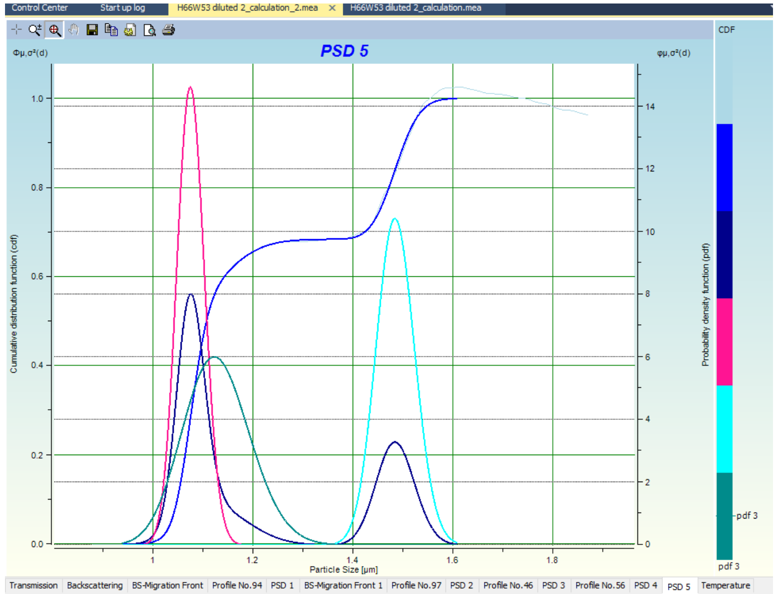


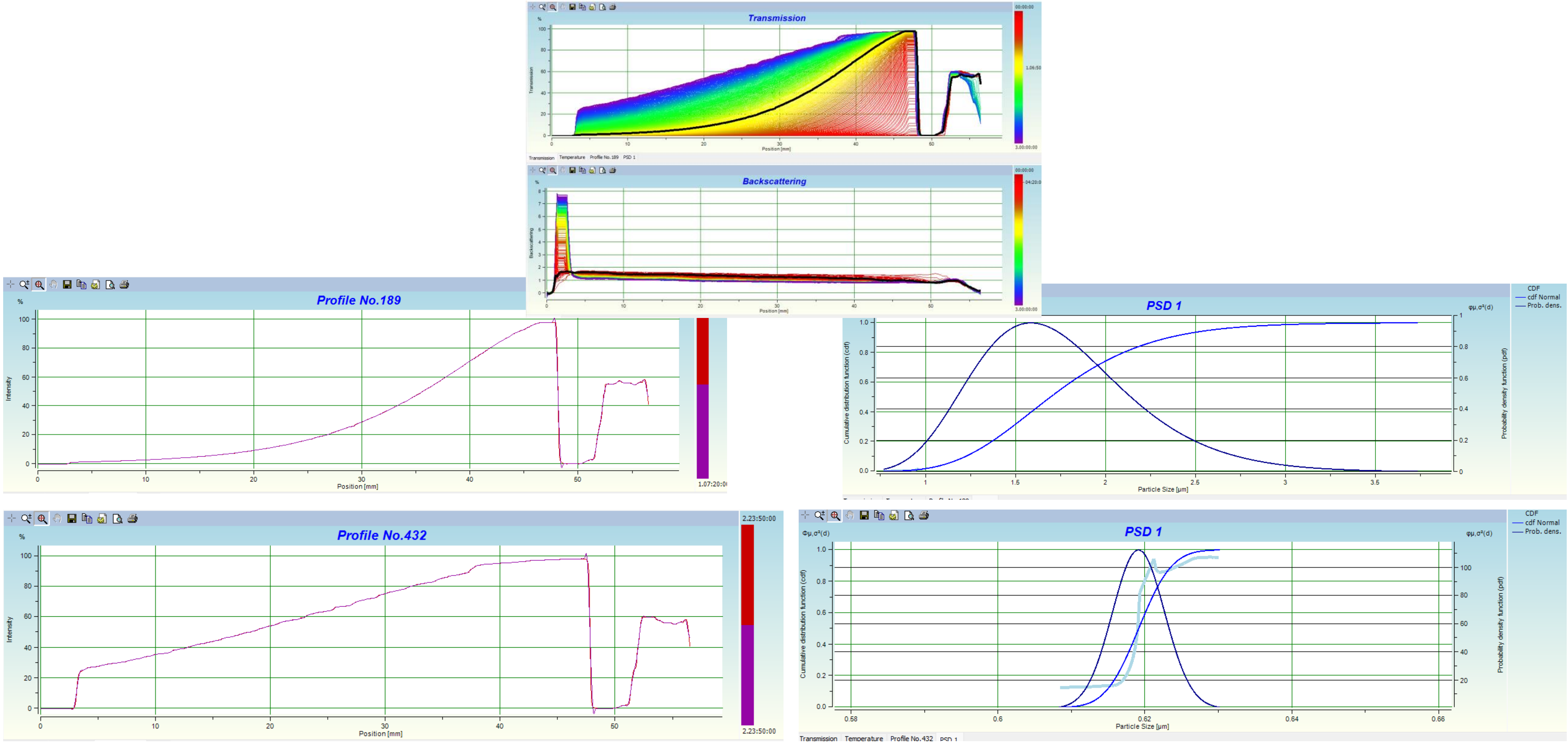
Coating sample with different size TiO_2 powder

- White paint sample diluted 200 times with water
- Goal: determine particle size distribution









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