Characterizing the Retention of UPW Filters Using a Polydispersed Silica Challenge

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Introduction

- Particles present in ultrapure water can deposit onto the wafer surface during semiconductor manufacturing processes resulting in decreased yield and reduced circuit reliability.
- While UPW filters are effective in reducing the concentration of particles, the unrelenting path toward smaller feature sizes increases the challenge for the filters to capture smaller particles.
- Several test methods have been published over the past 5 years that seek to quantify filter removal efficiency for particles smaller than 50 nanometers.
- SEMI C79 was introduced in 2013 as a guideline for evaluating the effectiveness of UPW filters in capturing particles 15 nm and smaller utilizing relatively mono-dispersed silica particles.
- This paper reviews the expansion of this test method to measure particle retention of UPW filters using a poly-dispersed challenge that more closely mimics "real life" particle size distributions typically found in UPW systems.

Outline

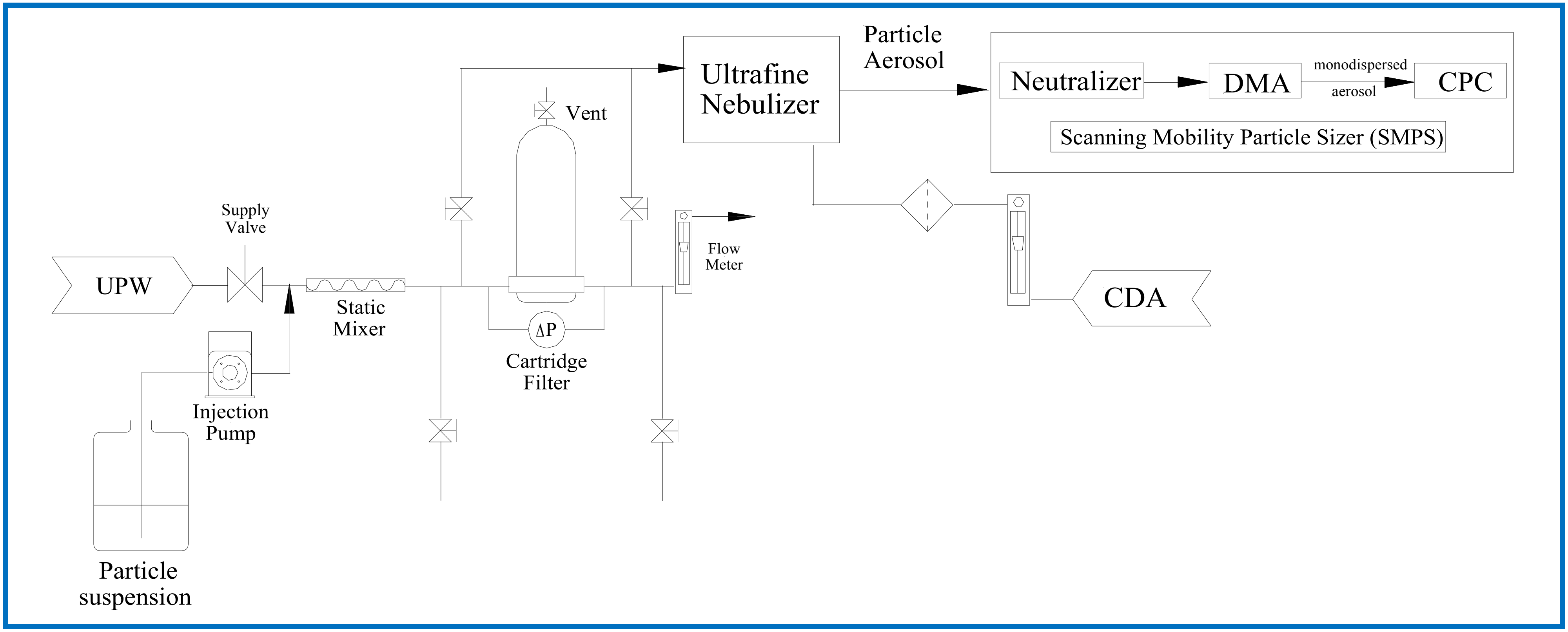
- SEMI C79-0113, "Guide to Evaluate the Efficacy of Sub-15 nm Filters Used in Ultrapure Water (UPW) Distribution System", method review.
- Areas of investigation and enhancements.
- Expanded particle size distribution (PSD) methodology.
  - Filters tested
  - Retention results
  - Observations
- Recommendations
- Future Activities

SEMI C79-0113

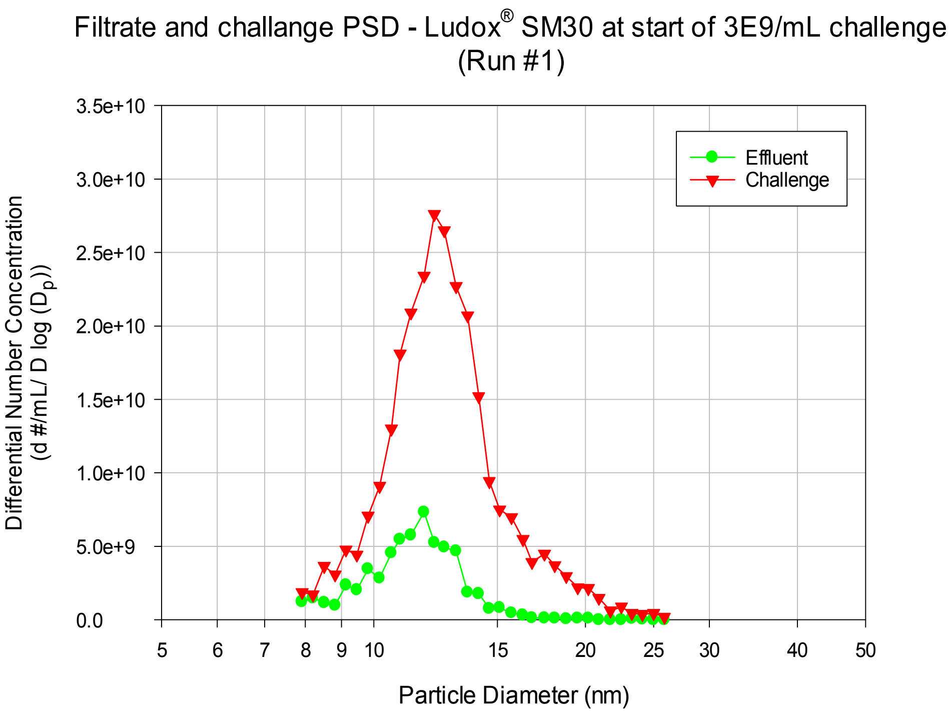
- Generally used for testing cartridge filters; also applicable to UF modules.
- Flow rate is established as a function of filter surface area (face velocity of 0.8 cm/min for cartridge filters; 0.6 cm/min for UF).
- The challenge suspension contains colloidal silica particles with a mean size between 5 and 15 nanometers.
- The suggested challenge concentration is 5E+09 particles per mL at the filter.
- The filter is challenged to a minimum of one monolayer (typically 4 - 6 hours).
- Particle concentrations are measured by two methods:
  - Grab samples are taken for off-line concentration analysis via inductively-coupled plasma mass spectrometry (ICP/MS).
  - Continuous measurement of filtrate particle concentration.*

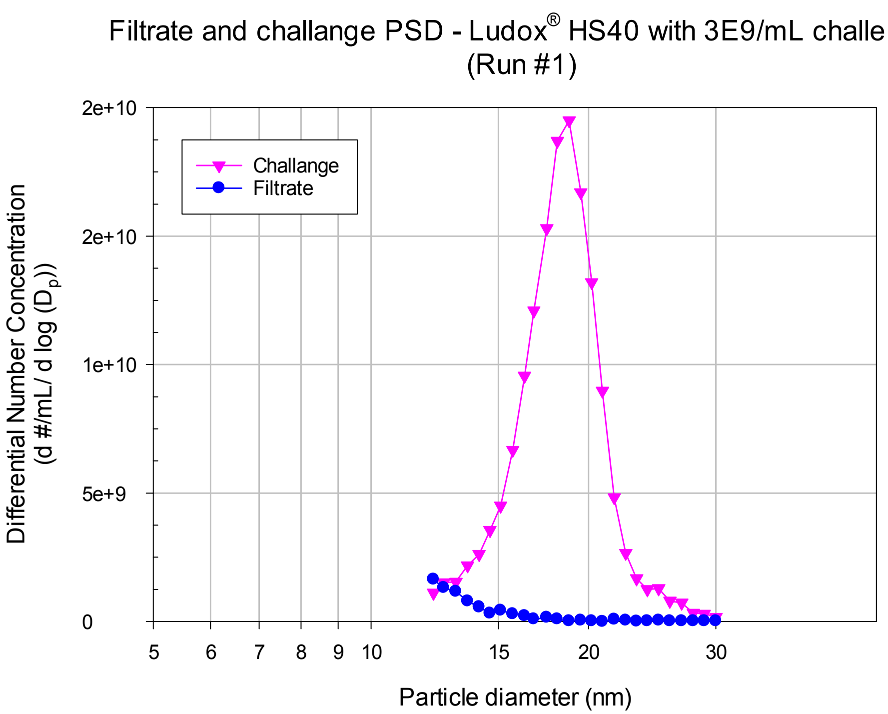
*In this paper, all particle concentration data presented was generated using a Liquid Nanoparticle Sizing system (LNS).

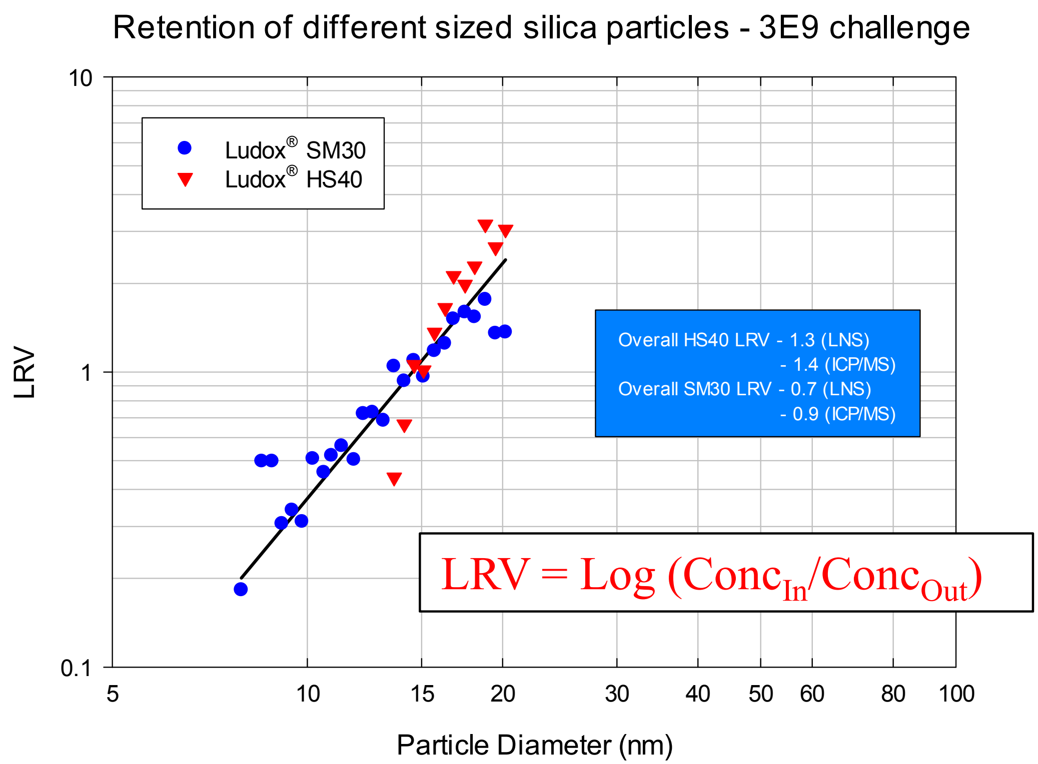
SEMI C79 Test Schematic



Filter retention as a function of particle diameter.







Test method work conducted in support of developing SEMI test guidelines for measuring retention of 5-15 nm filters used with UPW.

Areas of Investigation and Potential Enhancement to SEMI C79

- Reduce the challenge concentration.
- Select a challenge particle size distribution (PSD) that better reflects the “typical” PSD's expected in UPW (log-log slope of -2 to -4).

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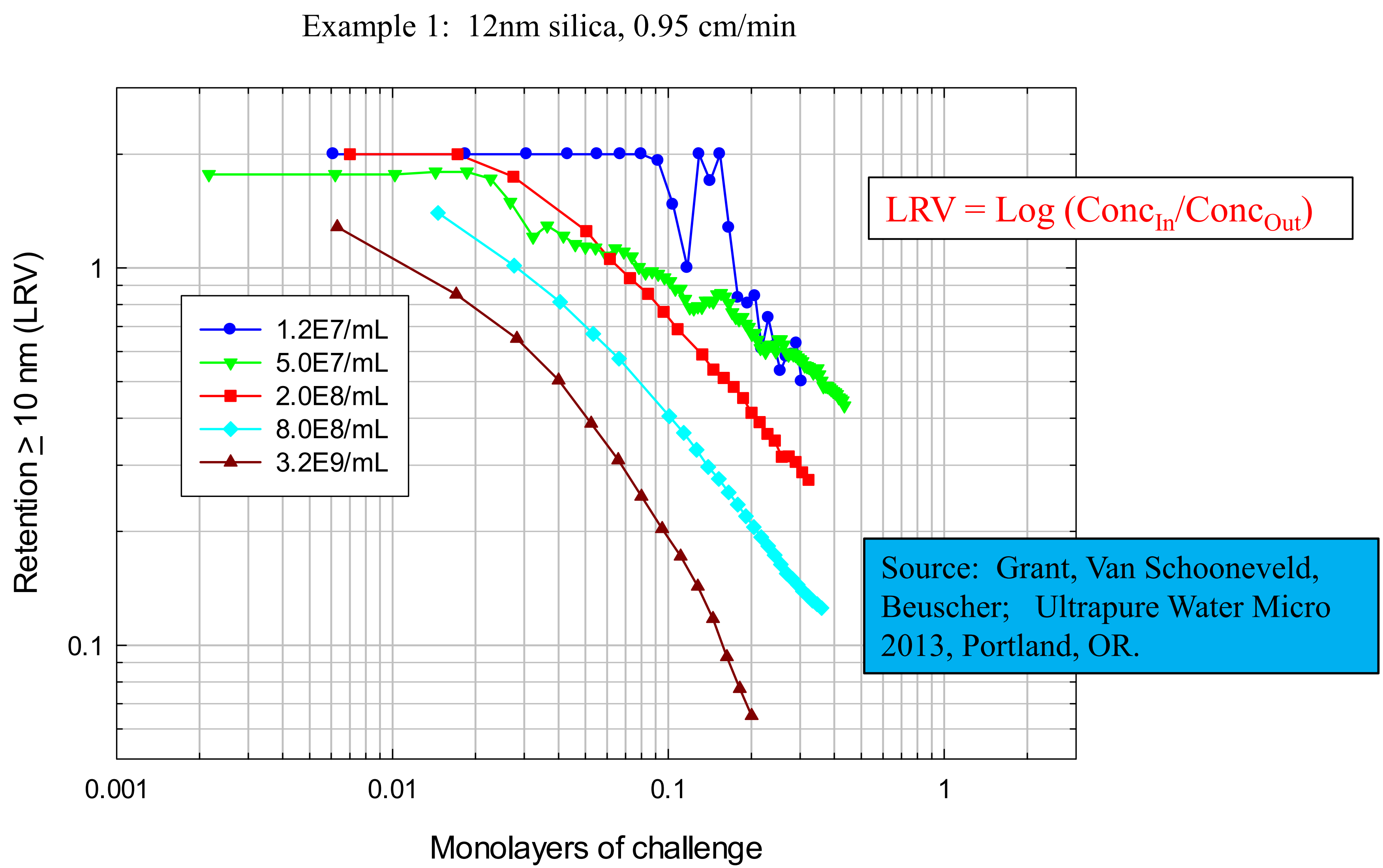
The effect of concentration on retention



Retention decreases with increased challenge concentration.

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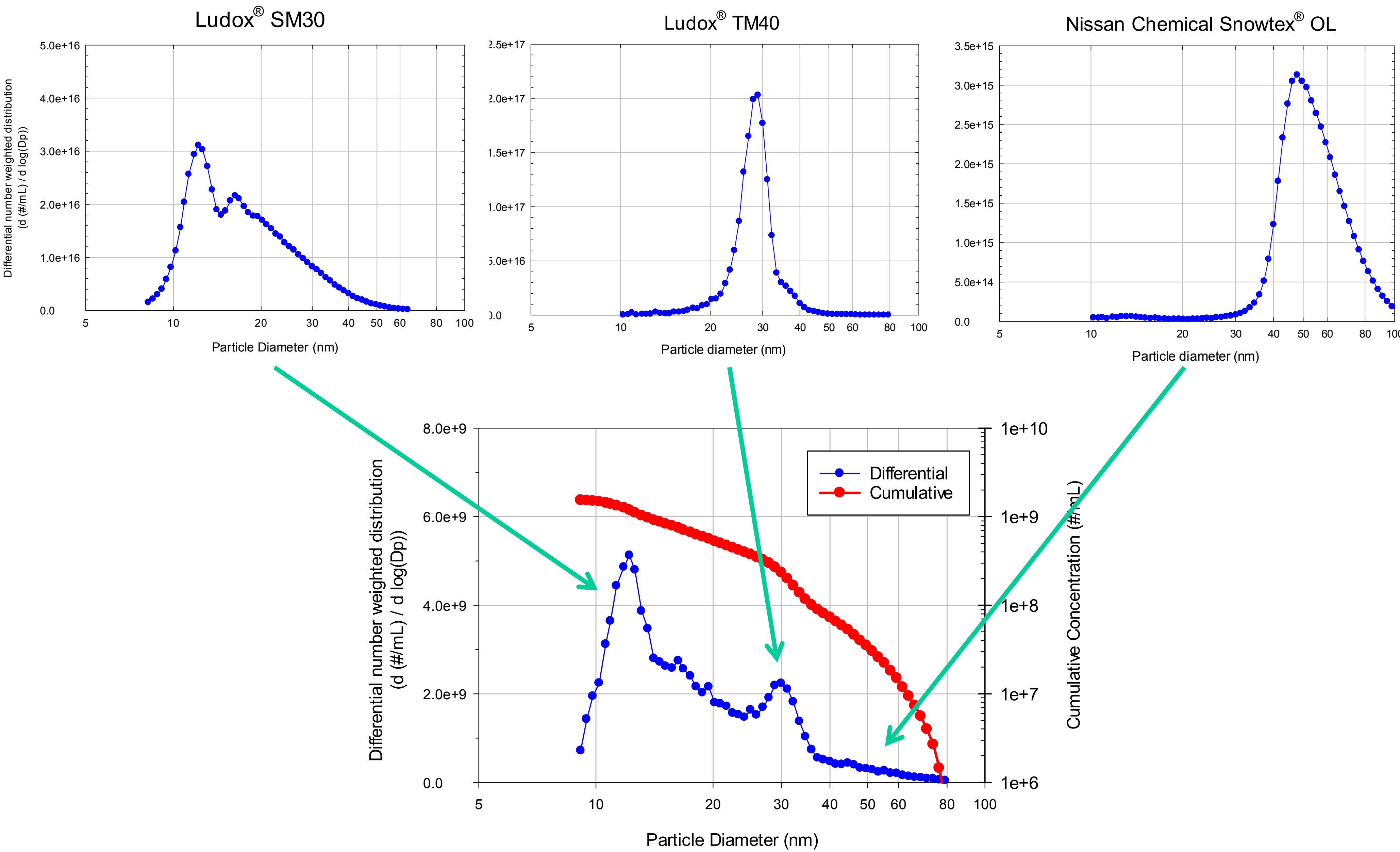
Area Weighted Silica (AW Si)



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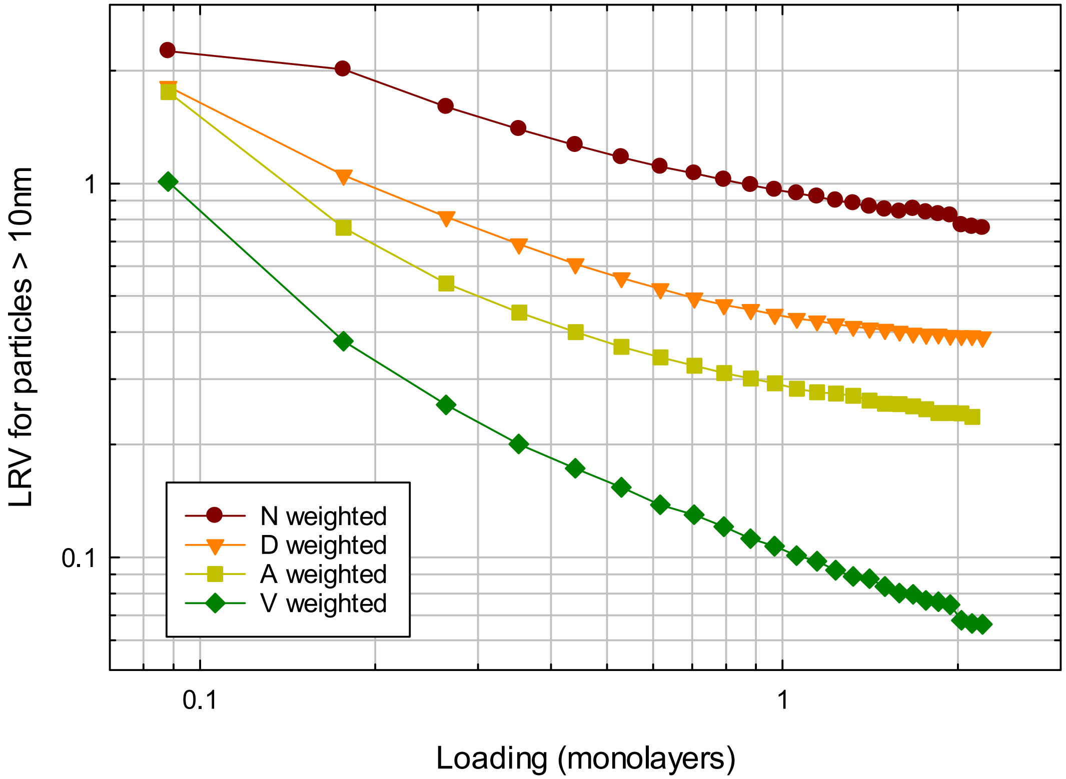
Challenge Weightings

- Number Weighted: $\sum N_{SM30} = \sum N_{TM40} = \sum N_{SnowOL}$
- Diameter Weighted: $\sum D_{SM30} = \sum D_{TM40} = \sum D_{SnowOL}$
- Area Weighted: $\sum A_{SM30} = \sum A_{TM40} = \sum A_{SnowOL}$
- Volume Weighted: $\sum V_{SM30} = \sum V_{TM40} = \sum V_{SnowOL}$

Effect of PSD Weighting on Retention



- Overall retention can be effected by the PSD weighting factor.
- “Real world” distribution is likely between area-weighted and volume-weighted.
- Area-weighted was selected due to better counting statistics for the larger particles as compared to the volume-weighted challenge.

Enhanced SEMI C79 Testing

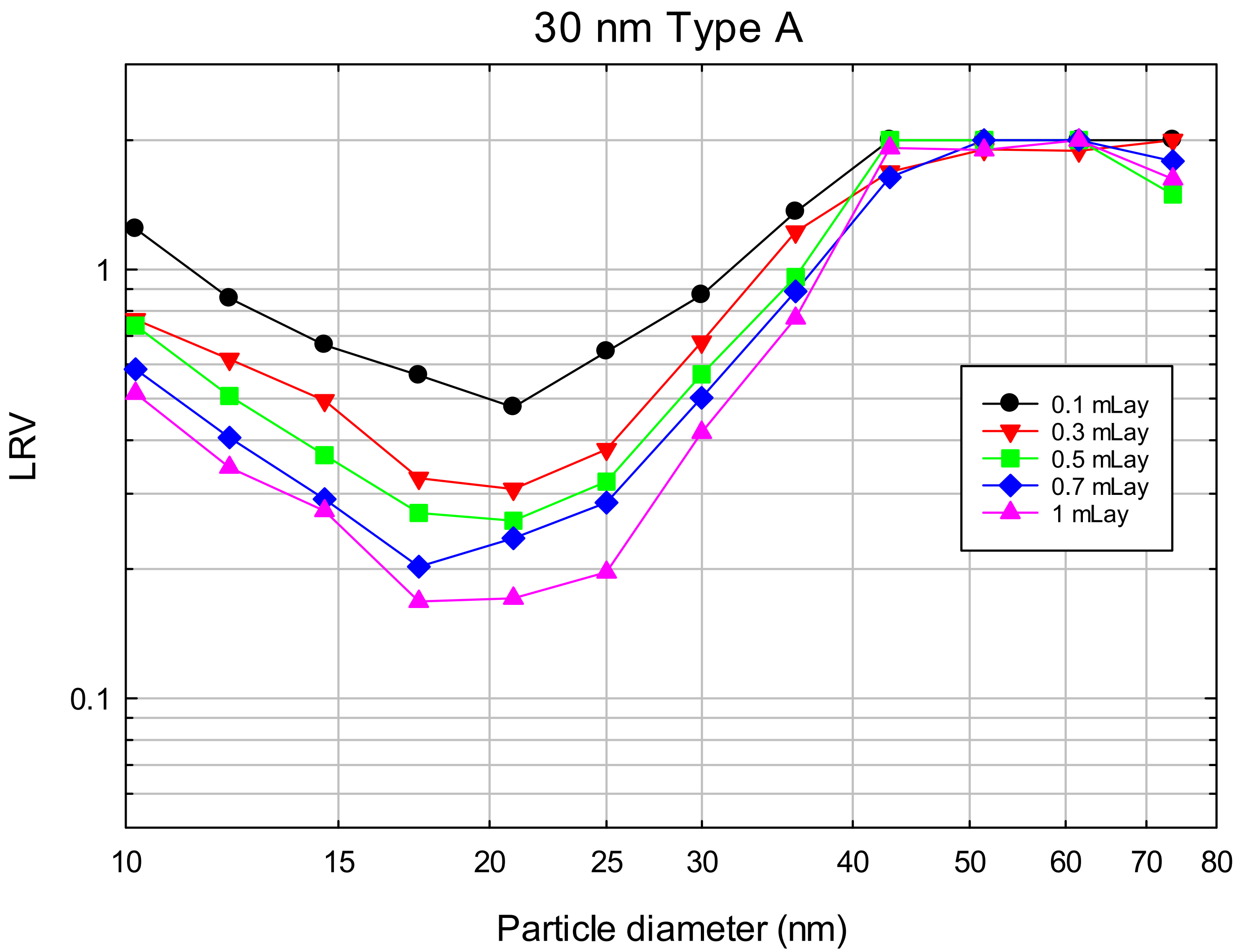
- Three silica particles with modes of 12, 30 and 50 nanometer are used.
- The concentration of each particle type is adjusted to achieve equal area concentrations.
- The challenge concentration is reduced to 1.5E9 particles per mL $\geq$ 10 nm.
- LRVs up to 2 can be measured for particles ranging from 10 to 70 nm.
- Filter face velocity during the challenge is 0.8 cm/min.
- Typical loading is to 1.25 monolayers (based on projected particle cross-sectional areas assuming perfectly spherical particles).
- Extended testing to 10 monolayers can be used to assess the effect of long-term loading conditions.

Filter Media Tested

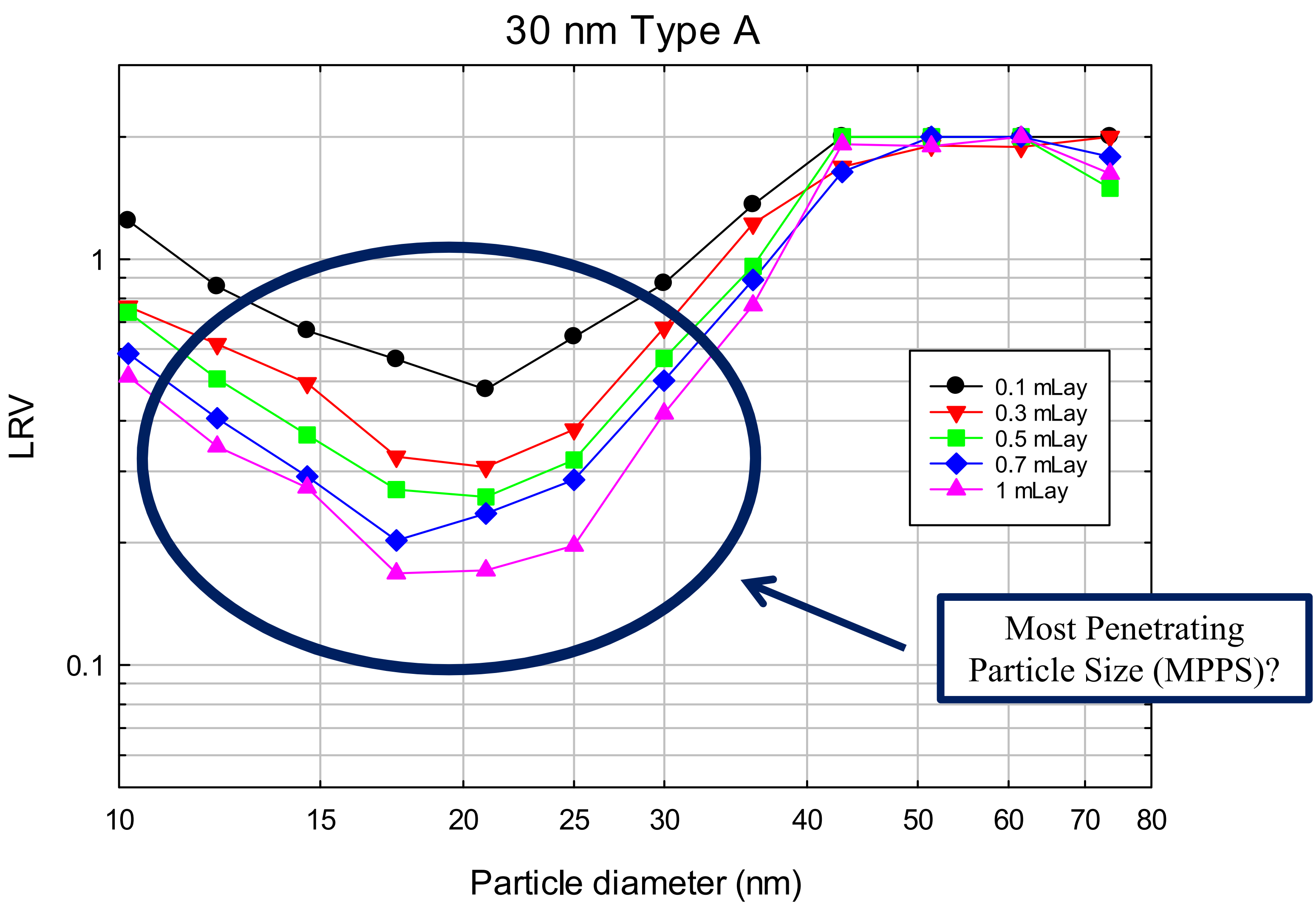
- Three filter types tested:
  - Polyarylsulfone (PAS)
  - Polytetrafluoroethylene (PTFE)
  - Charge modified Nylon 6,6
- All filters in this study were hydrophilic.
- Filters have been randomly designated as Types A – C.
- Multiple retention rating tested ranging from 20 to 100 nm.

Filter Retention Data

Retention Example – AW Si

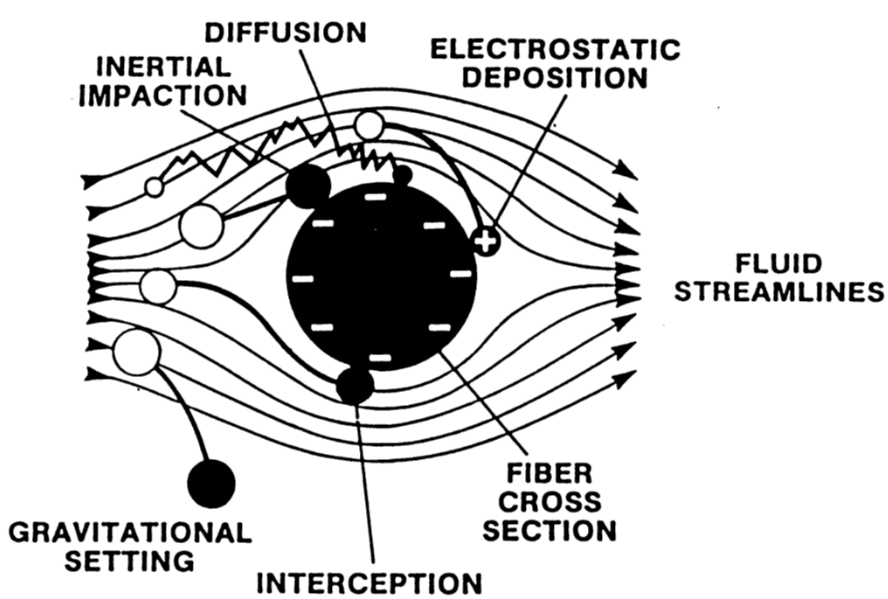


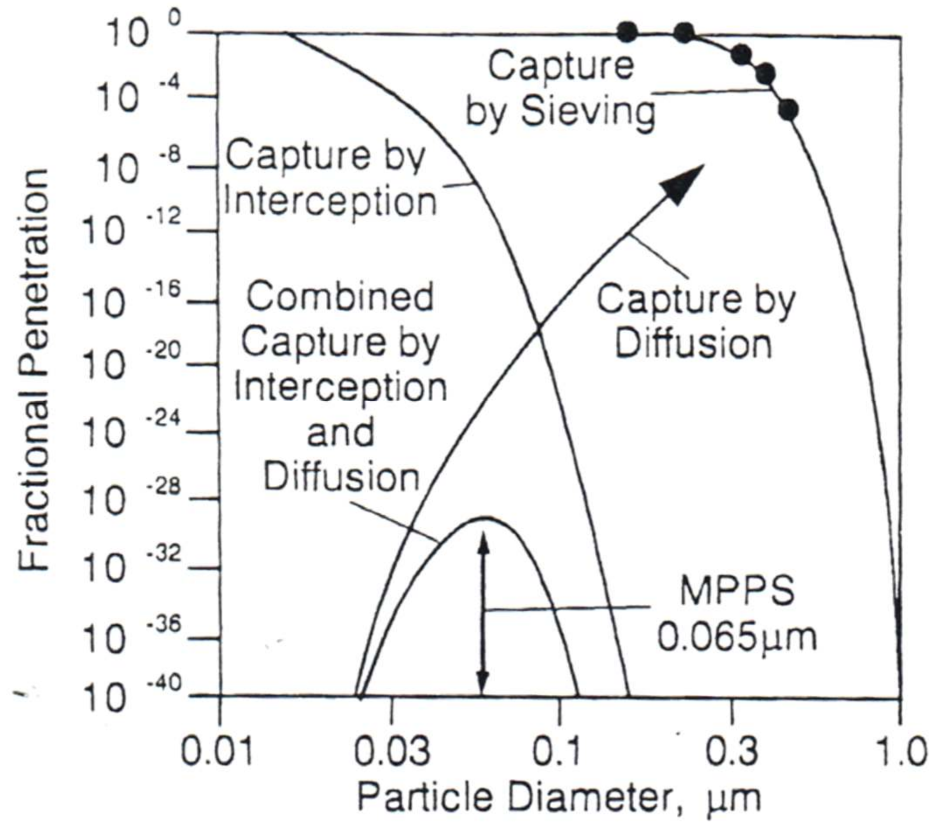
Retention Example – AW Si



Particle capture mechanisms beyond sieving

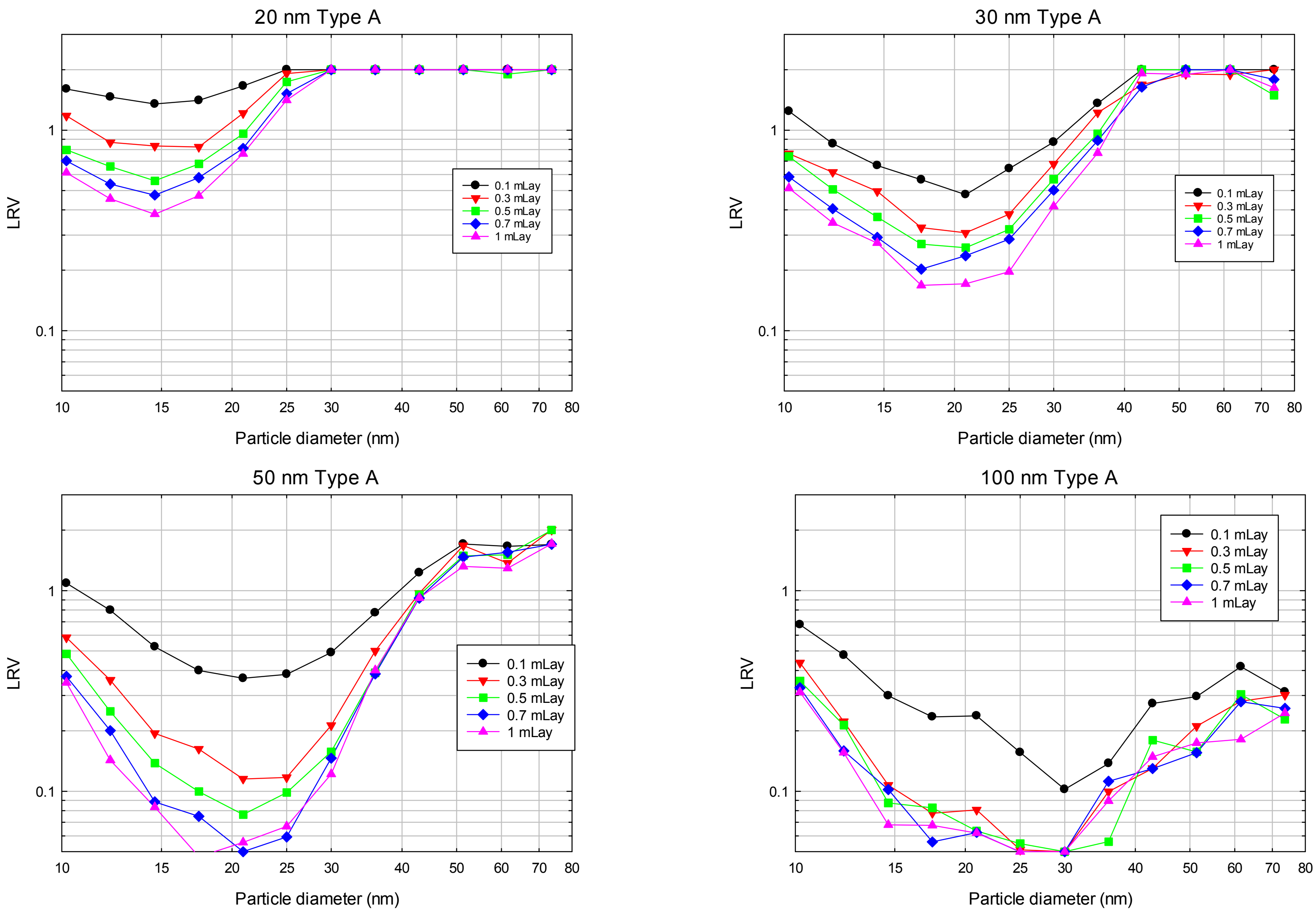
- Under certain conditions, particle capture can result from additional mechanisms:
  - Diffusion
  - Interception
  - Electrostatic attraction





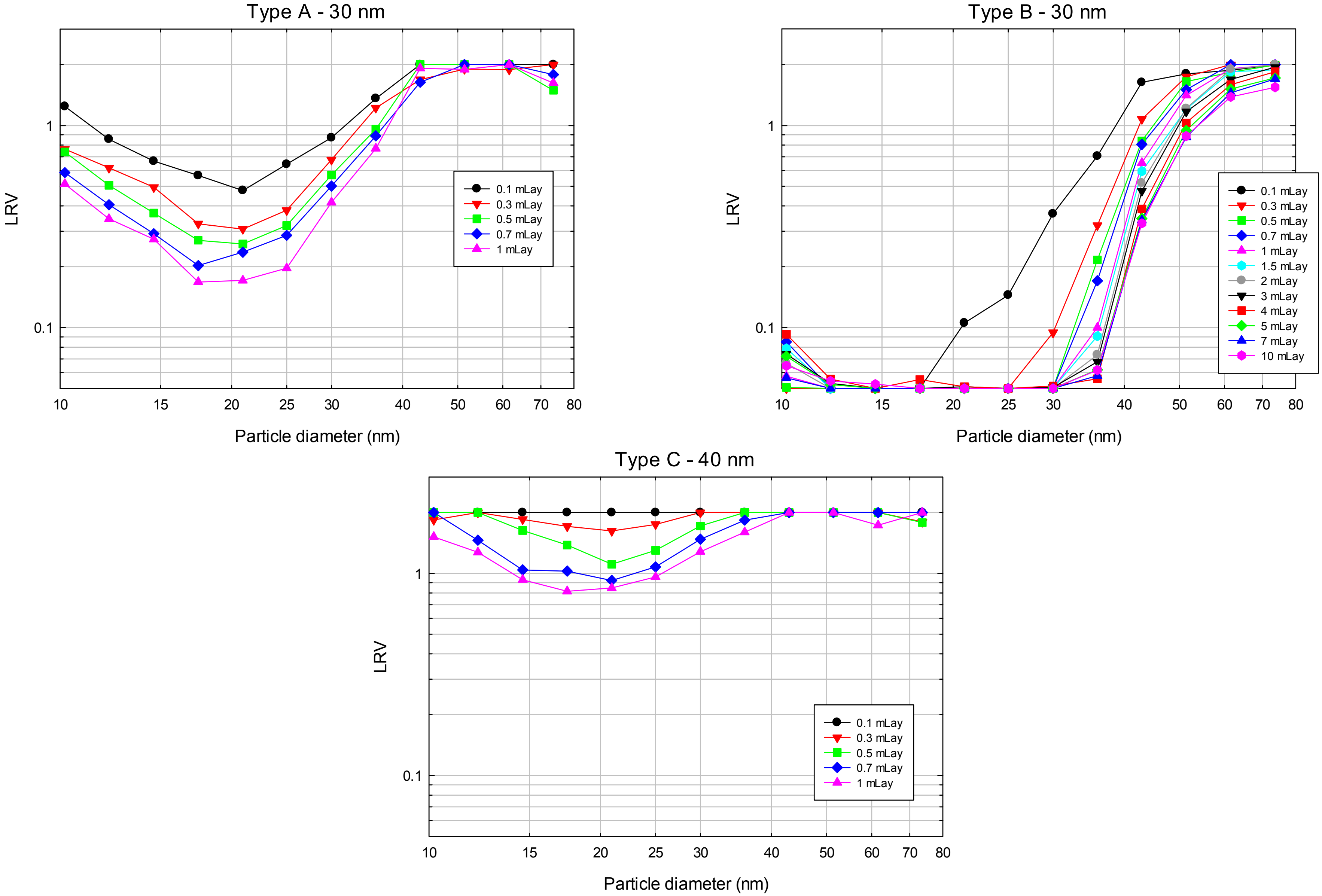
Source: Grant, Liu, Fisher. Journal of Environmental Science, July/August 1989

Influence of Filter Retention Rating



- MPPS may be present over a range of retention ratings.
- MPPS appears to be correlated with retention rating.

Influence of Filter Media



- A MPPS has been observed in multiple media.

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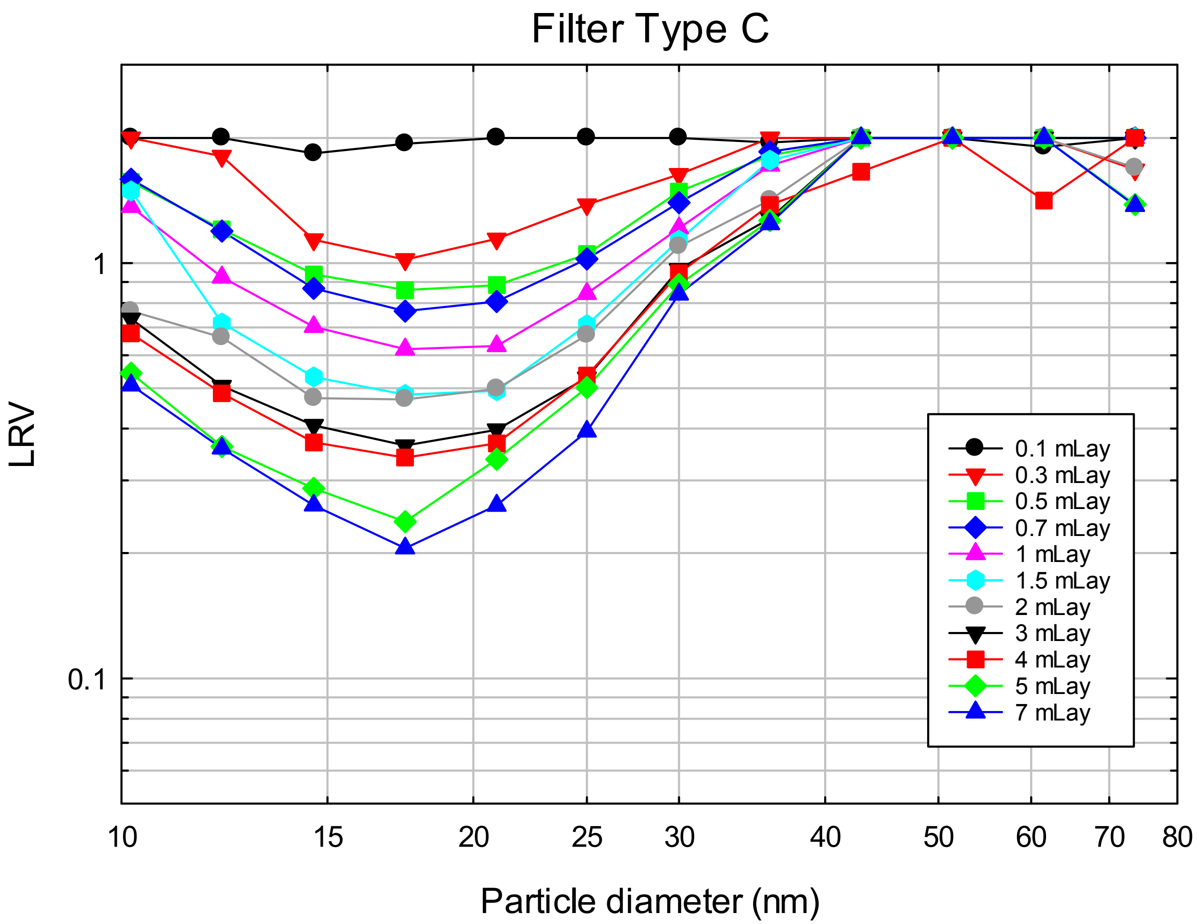
Influence of Loading



- Particle loading decreases the retention at the MPPS.
- MPPS appears to be stable with loading.

Observations

- The particle size distribution (PSD) of the challenge can influence the retention of a filter.
- A most penetrating particle size (MPPS) was observed with multiple filters types and ratings.
- Retention at the MPPS decreased with filter loading.
- Using a poly-dispersed challenge can provide additional insight into the retention capabilities of a filter compared to a mono-dispersed challenge.

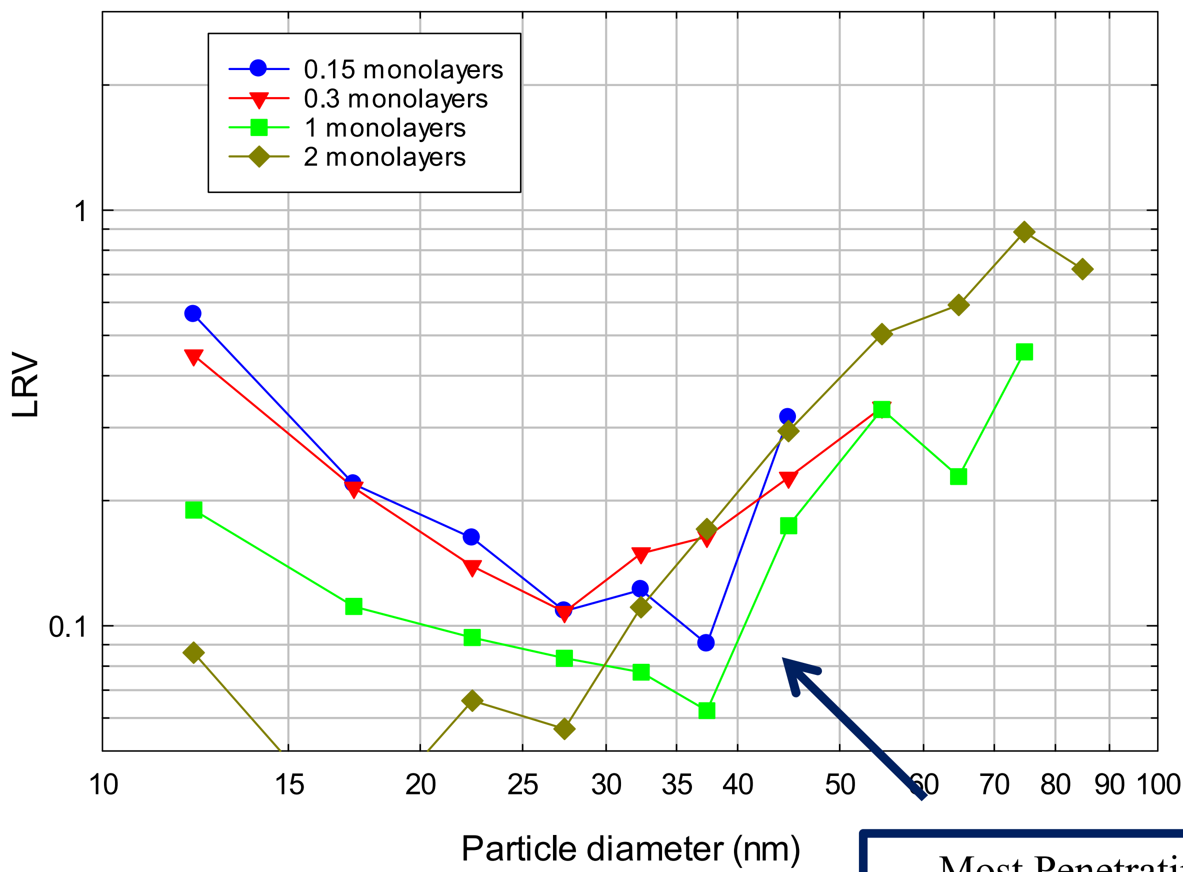
Recommendations

- Consider updating SEMI C79 by:
  - Adding to or replacing the mono-dispersed silica challenge with an area-weighted silica challenge.
  - Reducing the target challenge concentration at the filter to 1.5E9/ml ≥ 10 nm.

Future Activities

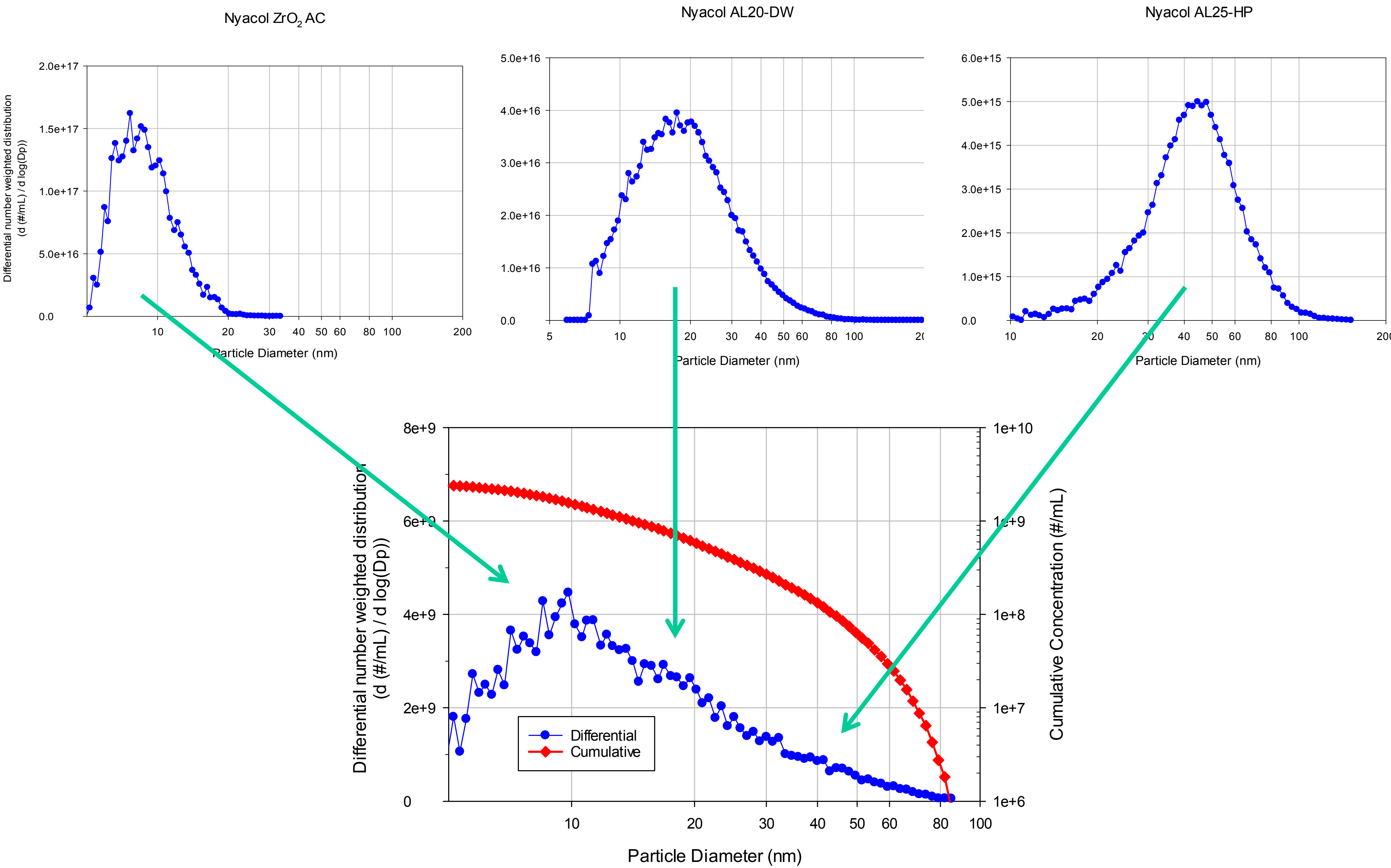
- Continue testing filters using area-weighted silica in an effort to understand the retention mechanisms associated with the most penetrating particle size.
- Extend the size range of the AW Si challenge by adding a 5 to 10 nm silica particle to the challenge.
- Investigate the influence of other particle types and charges on filter retention and the presence of a MPPS.

Polydispersed Polystyrene Latex

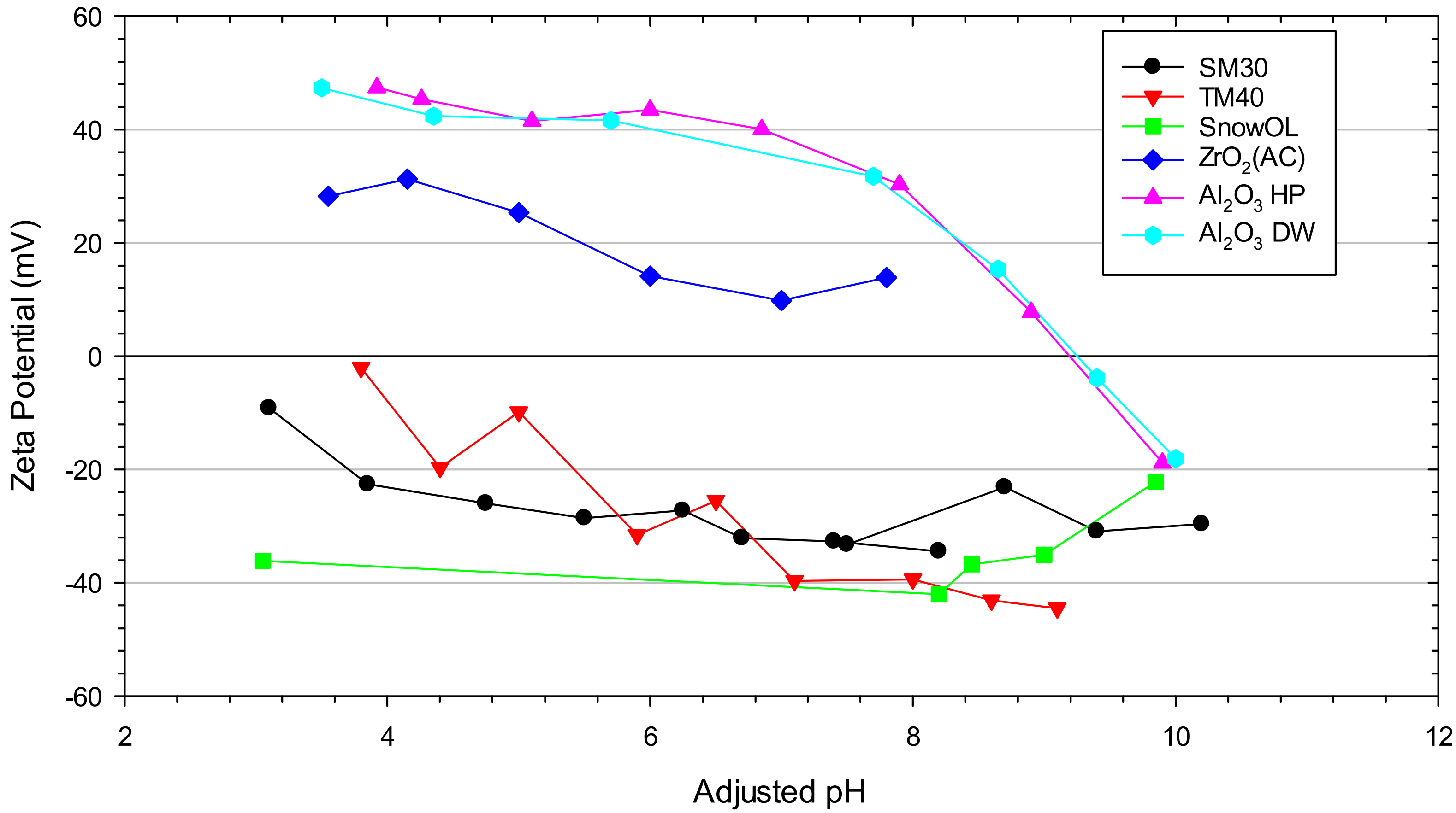


Most Penetrating Particle Size (MPPS)?

Positively Charge Particle (AW++)



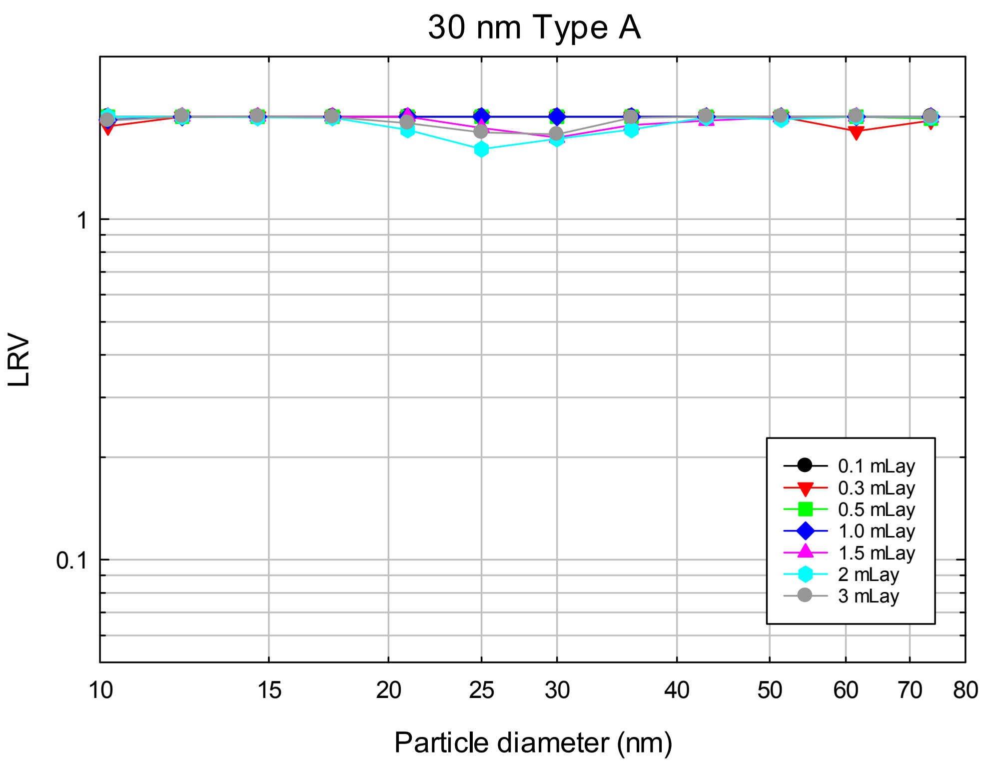
Particle Charge (Zeta Potential)



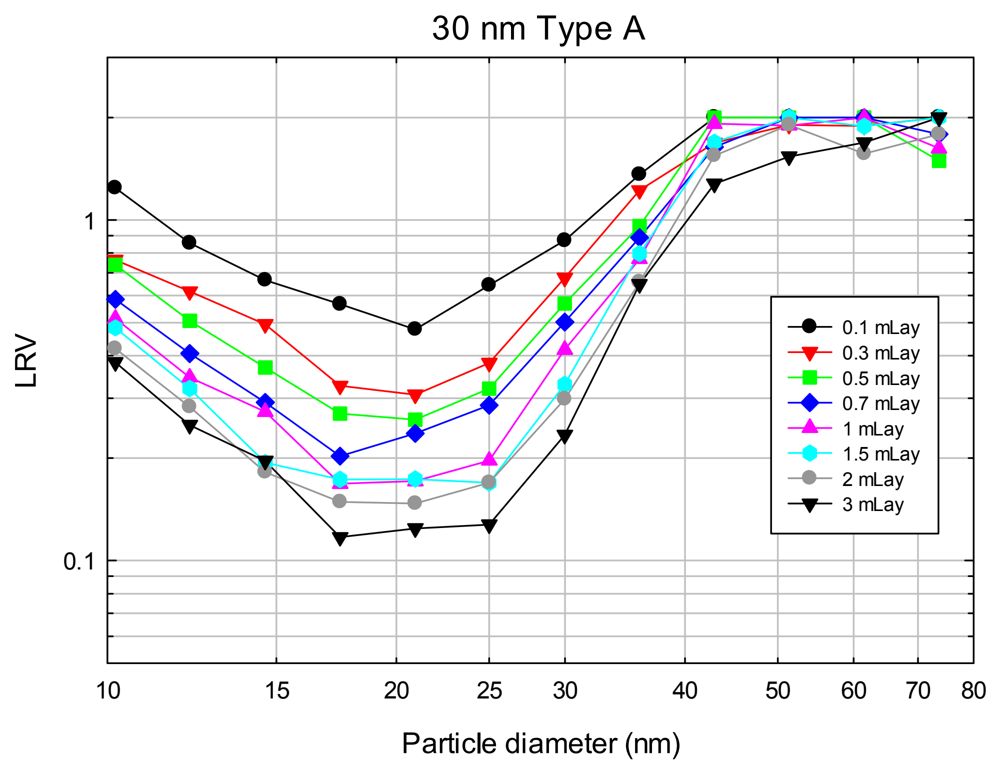
- Charges on all three silica particles are strongly negative at the pH of UPW.
- Charges on both alumina particles are strongly positive at pH of UPW.
- The charge on the zirconia particles is moderately positive at pH of UPW.

Effect of Particle Charge on Retention

Area Weighted ZrO_2/Al_2O_3 – Positive



Area Weighted SiO_2 – Negative



- Particle charge can have a significant effect on retention

Closing thought…

We still have some interesting and challenging work to do in building a comprehensive understanding of retention of sub-50nm particles in UPW.

Thank you for your attention!