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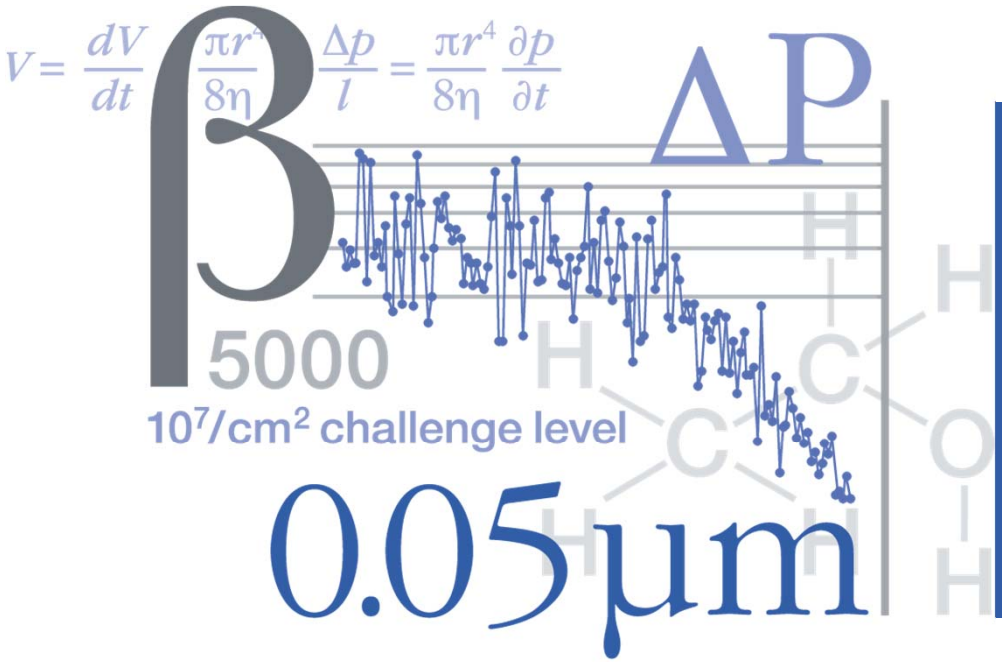
2018 Amsterdam



Extending filter life time for Ceria CMP Filtration

SLS Global Technical Support

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April 2018

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Pall GmbH

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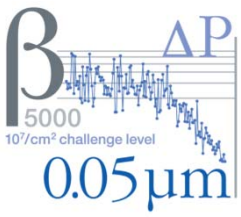
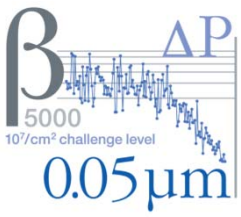


Outline

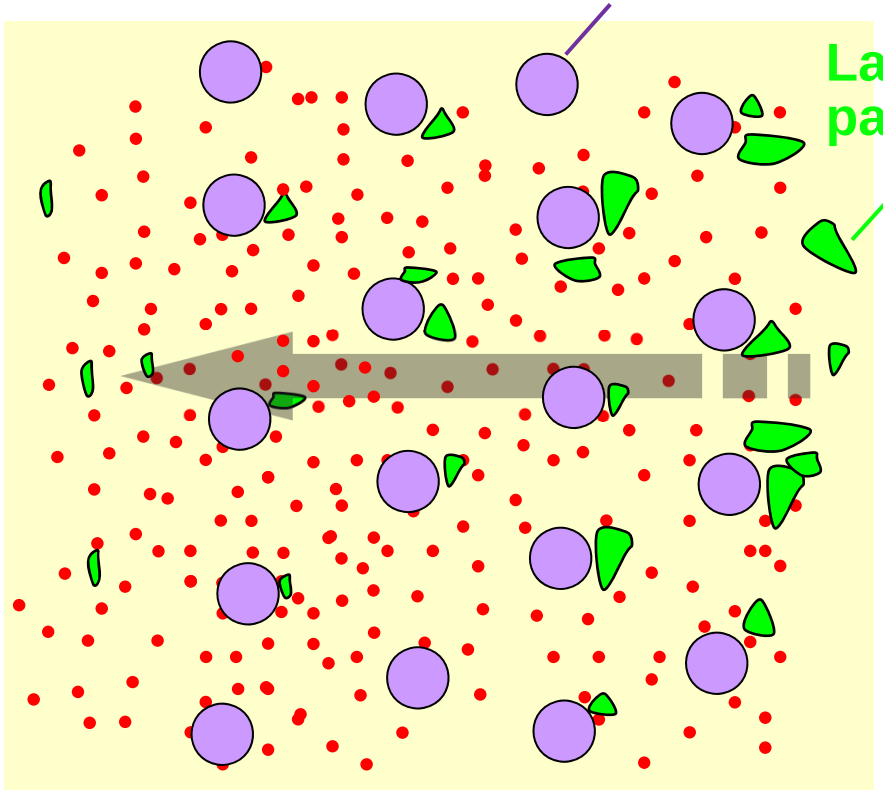
- Introduction and Motivation
- Depth vs Membrane Filtration
- Test outline (3 Scenarios)
- Results
- Conclusions



Introduction and Motivation

- Microscratch reduction is the dominant driver for extensive filtration
- Different scenarios are established to ensure adequate CMP quality and LPC reduction
- As CMP Slurries are very complex and multi-compound mixtures filtration needs to be adapted
- Not all interactions between filters, abrasive particles and additives are fully understood
- The need for tighter filtration and improved cut-off properties lead to introduction of membrane based filter media
- One major drawback of those devices can be limited lifetime due to uncontrolled build up of filter cake
- As a consequence the PSD of the slurry could be negatively impacted, and filter life time dramatically reduced
- This report shall provide some data to understand those interactions and provide a possible treatment option to extend filter life time for Ceria CMP slurries

Depth filter Media loading Mechanism



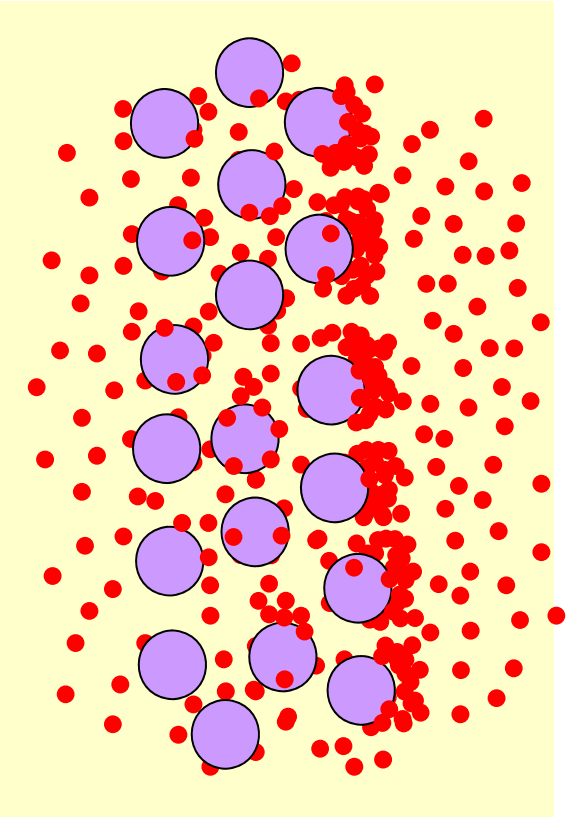
Particle removal happens INSIDE and **over** the depth of the filter medium

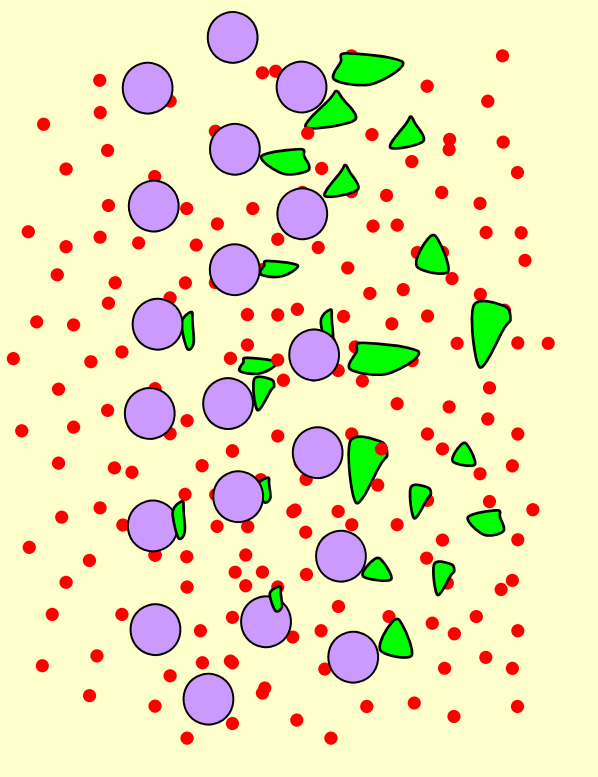
Large inner surface and statistical interaction with particles

Depends on interception, inertia and diffusion

Removal is determined by filter media over large range of pressure drop

Membrane Filter Media loading mechanism





In relation to the size of primary particles of slurry, membrane filters are not ONLY surface filters

Increasing pressure drop indicates build up of first layers of a filter cake

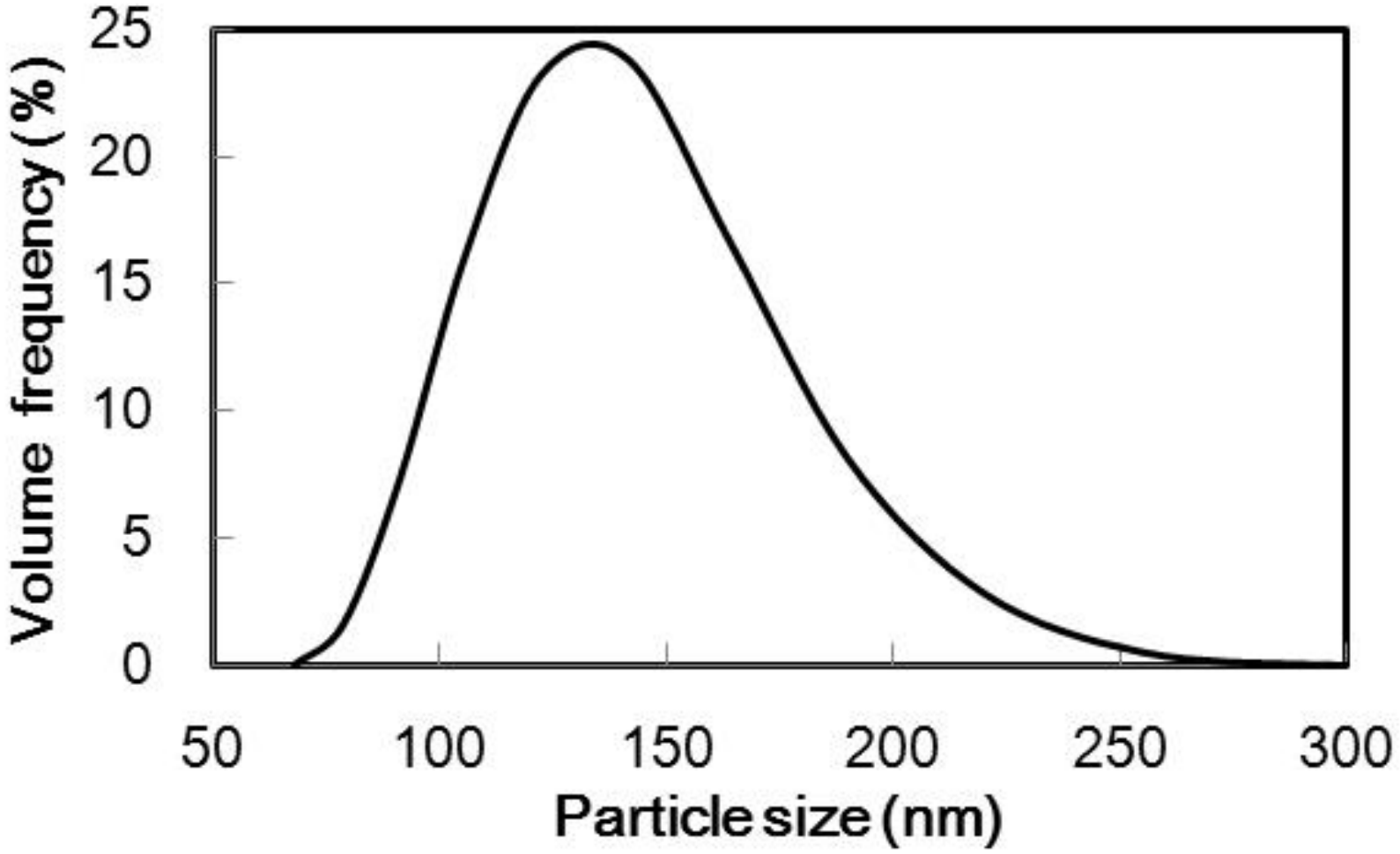
Removal is then determined more and more by the properties of the cake layer

Transition from media-controlled to cake-controlled removal can go very fast

Undesired removal of primary particles and self accelerating mechanism

Ceria Slurry Characterization

$D_{50:3}$ about 140 nm, solid concentration 1.7 %, pH 5.0



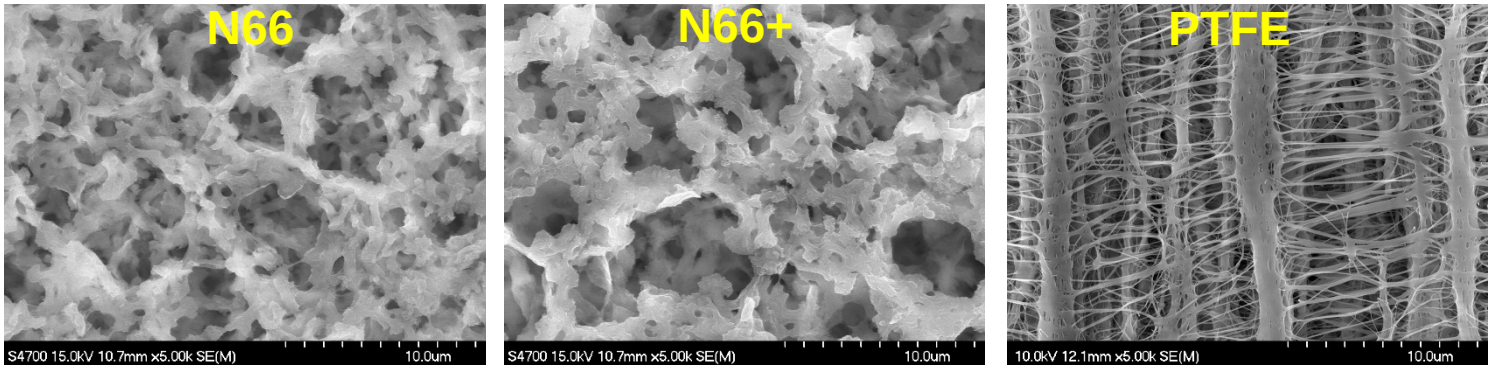
Particle size distribution in volume by DLS.

Filter Media Characterization

- all filters rated at 0.45 µm, porous membranes, 47 mm Φ disk filter
- Effective surface area 13.9 cm^2

Materials (6 kinds below)

1. Polypropylene (noted as PP), hydrophobic
2. PTFE (hydrophobic)
3. Hydrophilic polyether sulfone (noted as PES)
4. Surface modified PVDF to hydrophilic (noted as PVDF)
5. Nylon 6,6 (noted as N66), hydrophilic
6. Charged modified Nylon 6,6 (Noted as N66+), hydrophilic

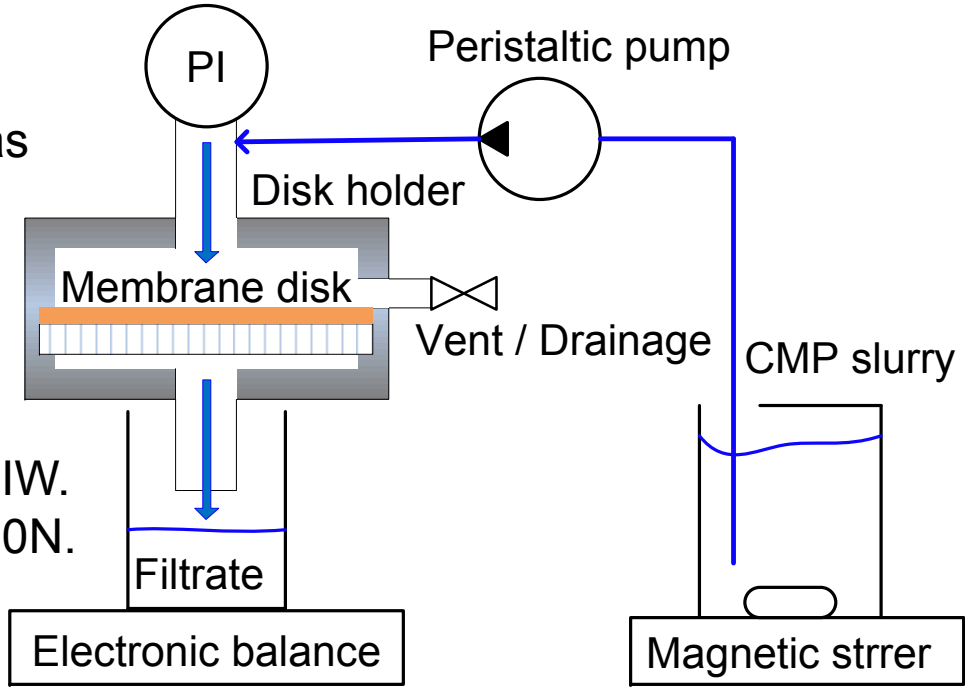


SEM images of some of the test filters

Test setup

Filtration

- A test filter was installed to the disk holder
- The filter was pre-wetted by alcohol in the case of hydrophobic filter
- The filter was flushed by DIW
- DIW was completely drained by pressurized air
- Flow rate: 5 mL/min
- Filtrate was sampled taken each 30 mL
- Inlet pressure (= dP) with filtration time was monitored.

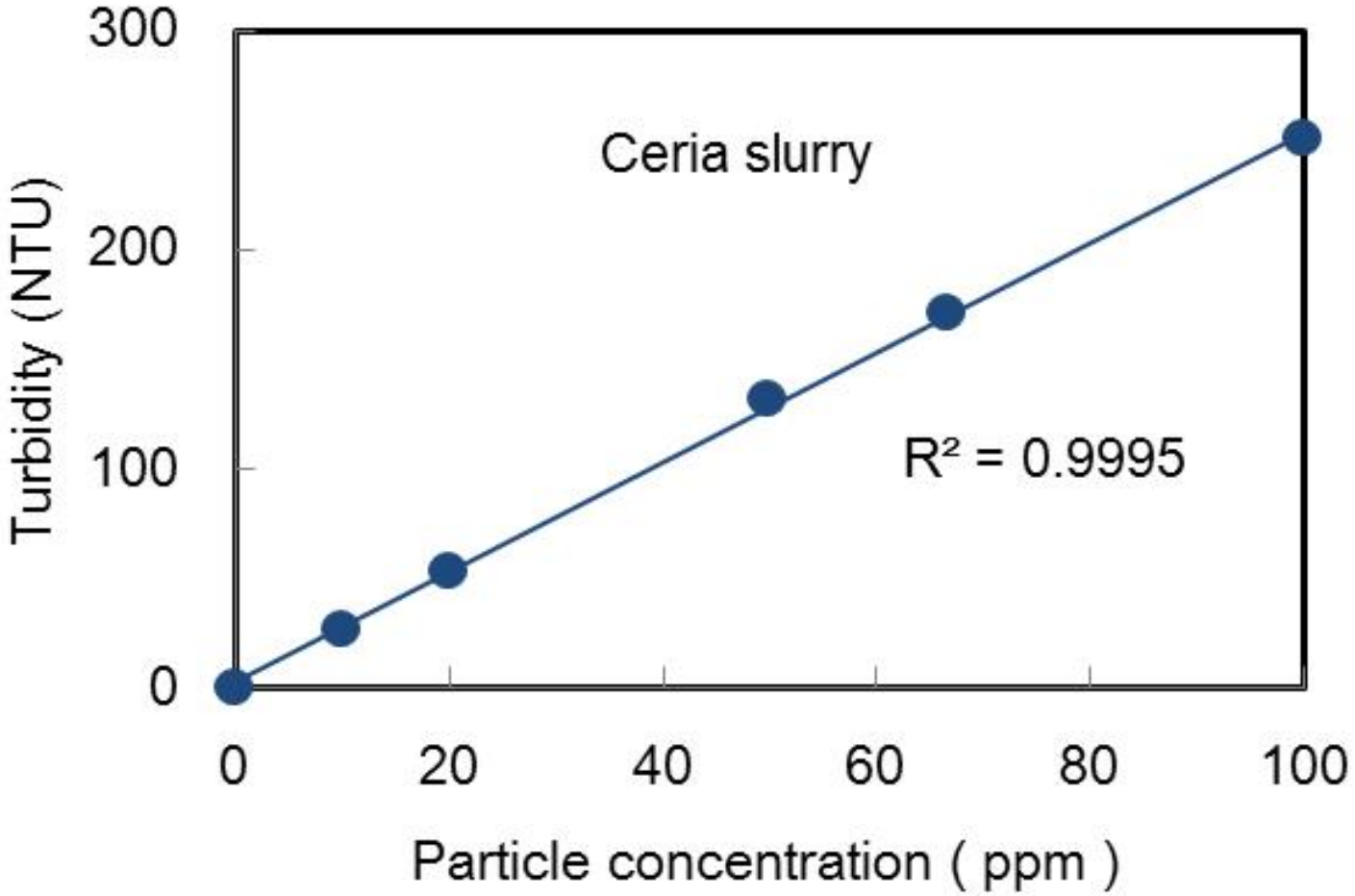


Constant flow filtration equipment

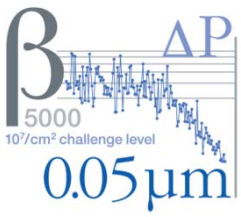
Turbidity measurement

- Filtrate, feed samples were diluted by DIW.
- Turbidity measurement with HACH, 2100N.
- Relative turbidity was calculated.
- Relative turbidity
  = (Filtrate turbidity/ Feed turbidity) x 100

Relationship of turbidity and particle concentration



Linear relationship of turbidity with abrasive particle concentration



Test Scenario 1

- all 6 Filter Media were used for this test
- Filtrate sample was taken every 30 mL
- Inlet pressure(= dP) with time was monitored
- Undiluted slurry was used (after prewetting for hydrophobic filters)
- 60 mL total filtrate was produced for all media
- Up to 800 mL filtrate was produced for N66 for extended exposure

Test Scenarios 2 and 3

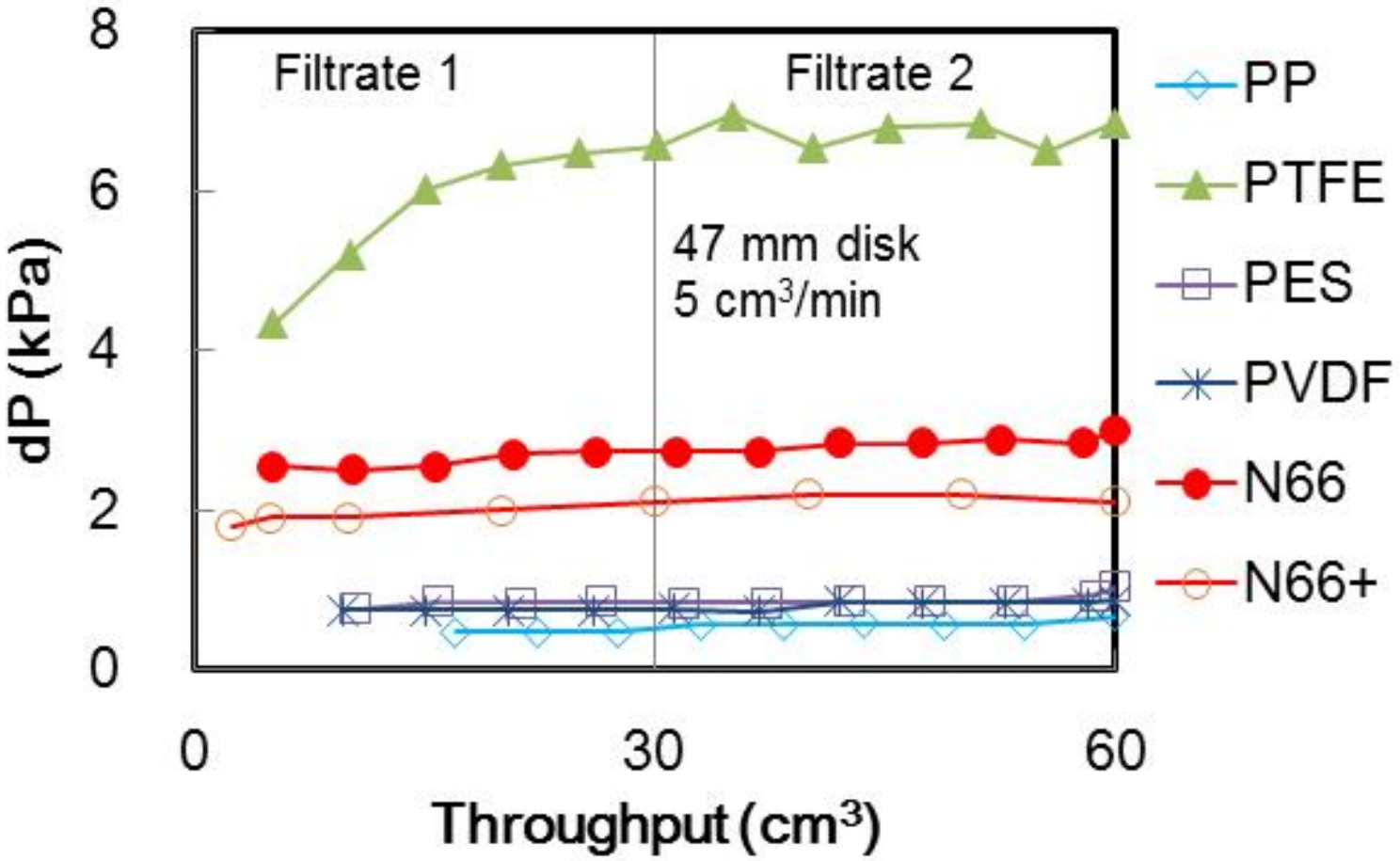
Scenario 2

- Only Nylon product family was used for this test.
- Charged and non-charged, both hydrophilic
- DIW wash after 60 mL Filtrate was executed
- Continue slurry filtration in direct sequence with flush

Scenario 3:

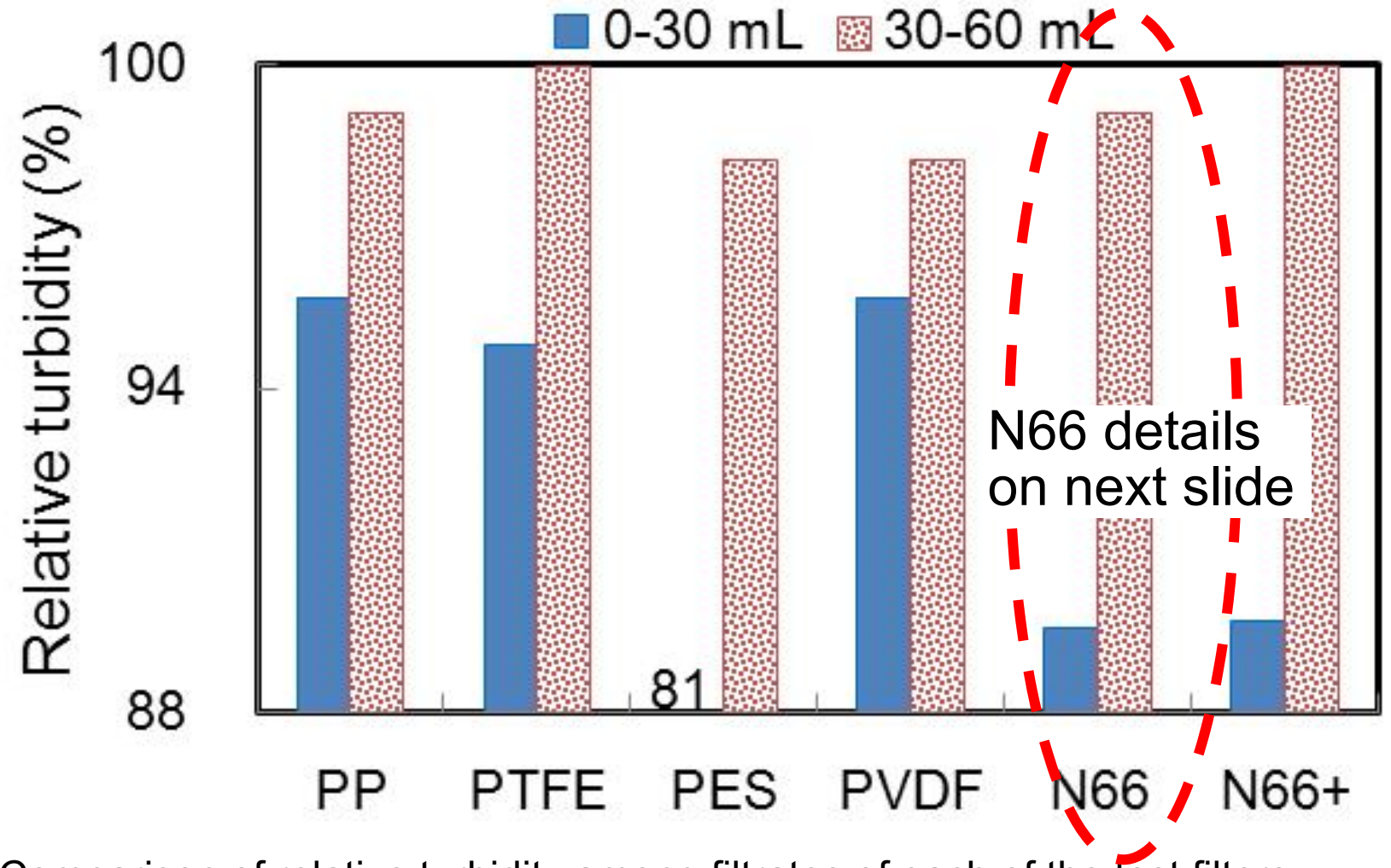
- Preconditioning of membrane disks with diluted slurry
- 10 and 100 fold dilution ratio were applied
- Only non-charged Nylon media was used

Scenario 1: Influence of membrane material - dP -



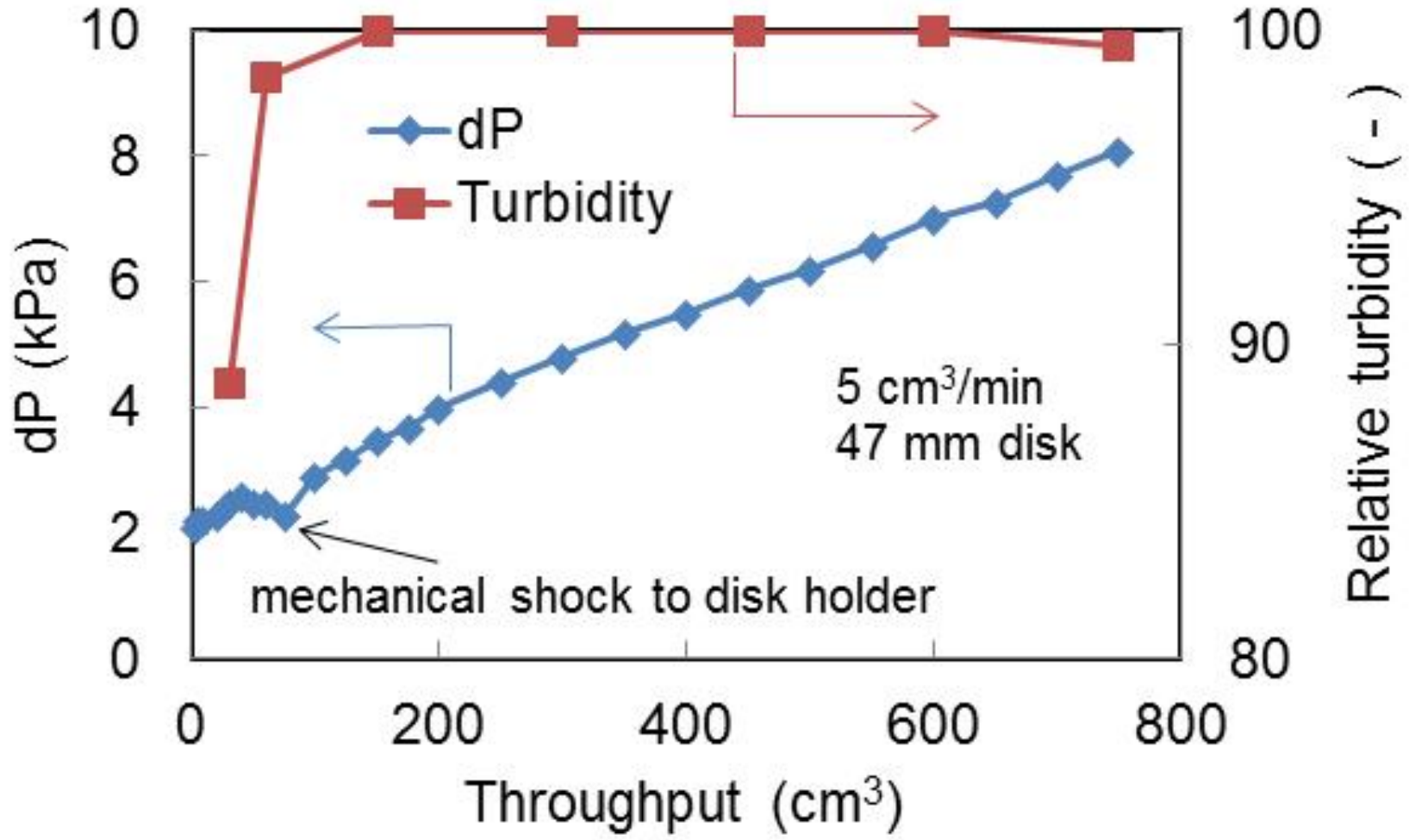
dP of test filters of various materials with filtration time
Flow rate 5 cm^3/min, 47 mm disk.

Scenario 1: Influence of membrane material - Turbidity -



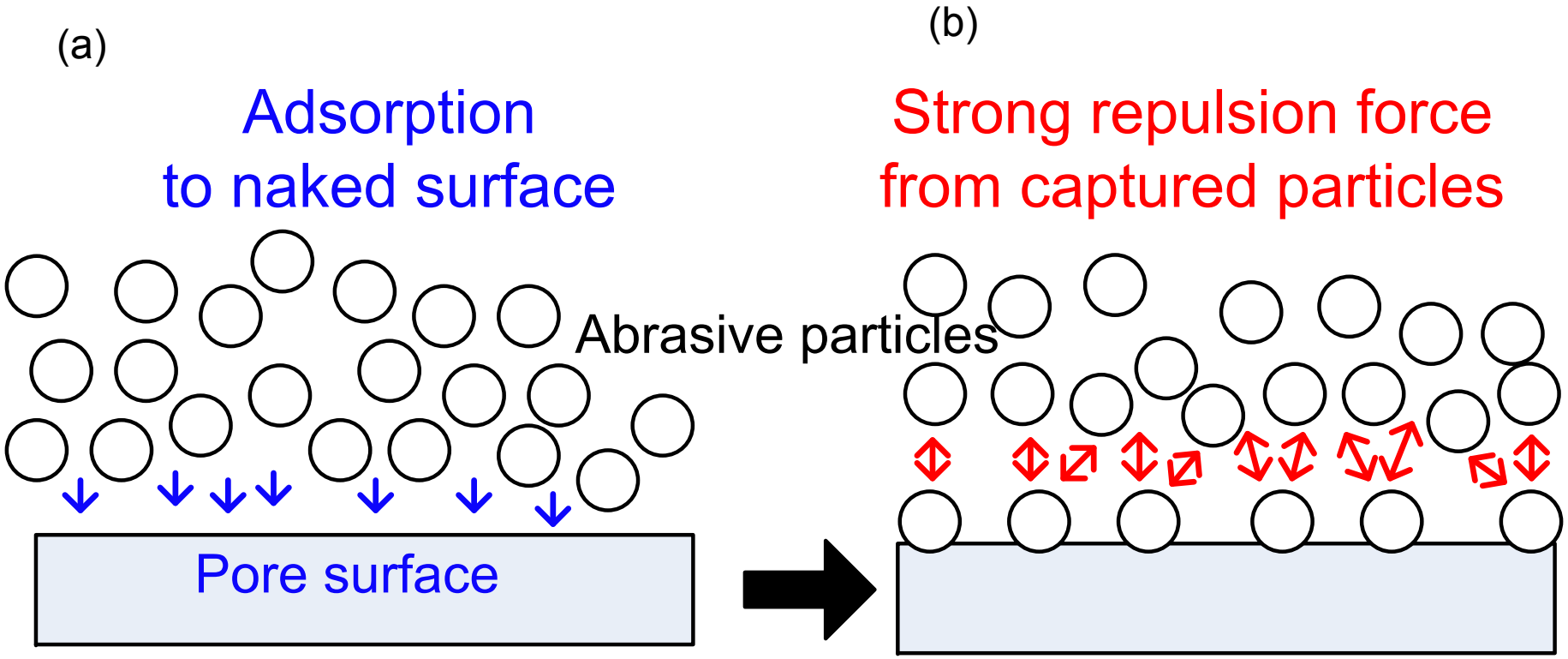
Comparison of relative turbidity among filtrates of each of the test filters

Change of relative turbidity of N66 filter filtrate with larger throughput



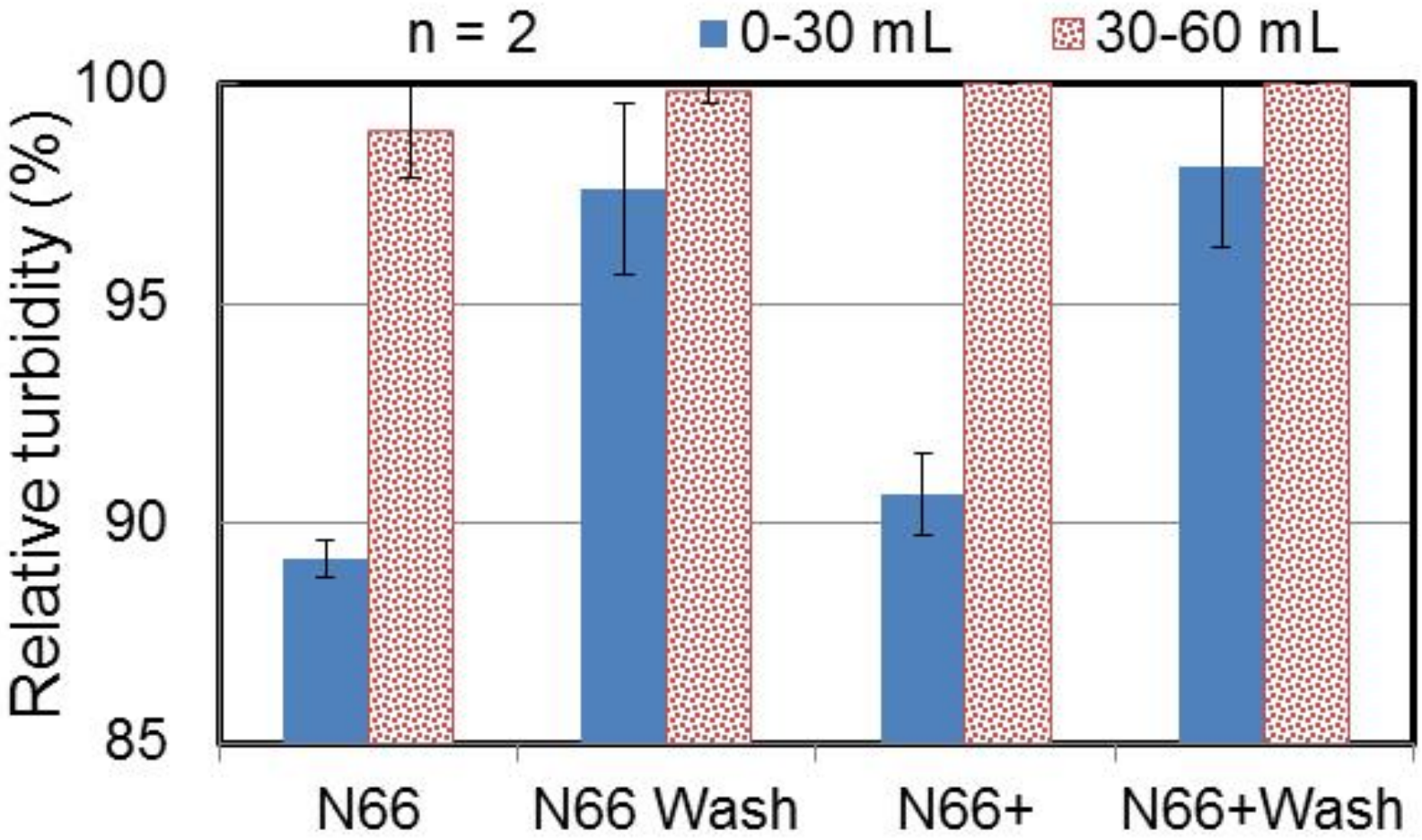
Relative turbidity, dP of N66 filter filtrate with total throughput 800 cm^3.
Flow rate 5 cm^3/min, 47 mm disk.

Interpretation Scenario 1:



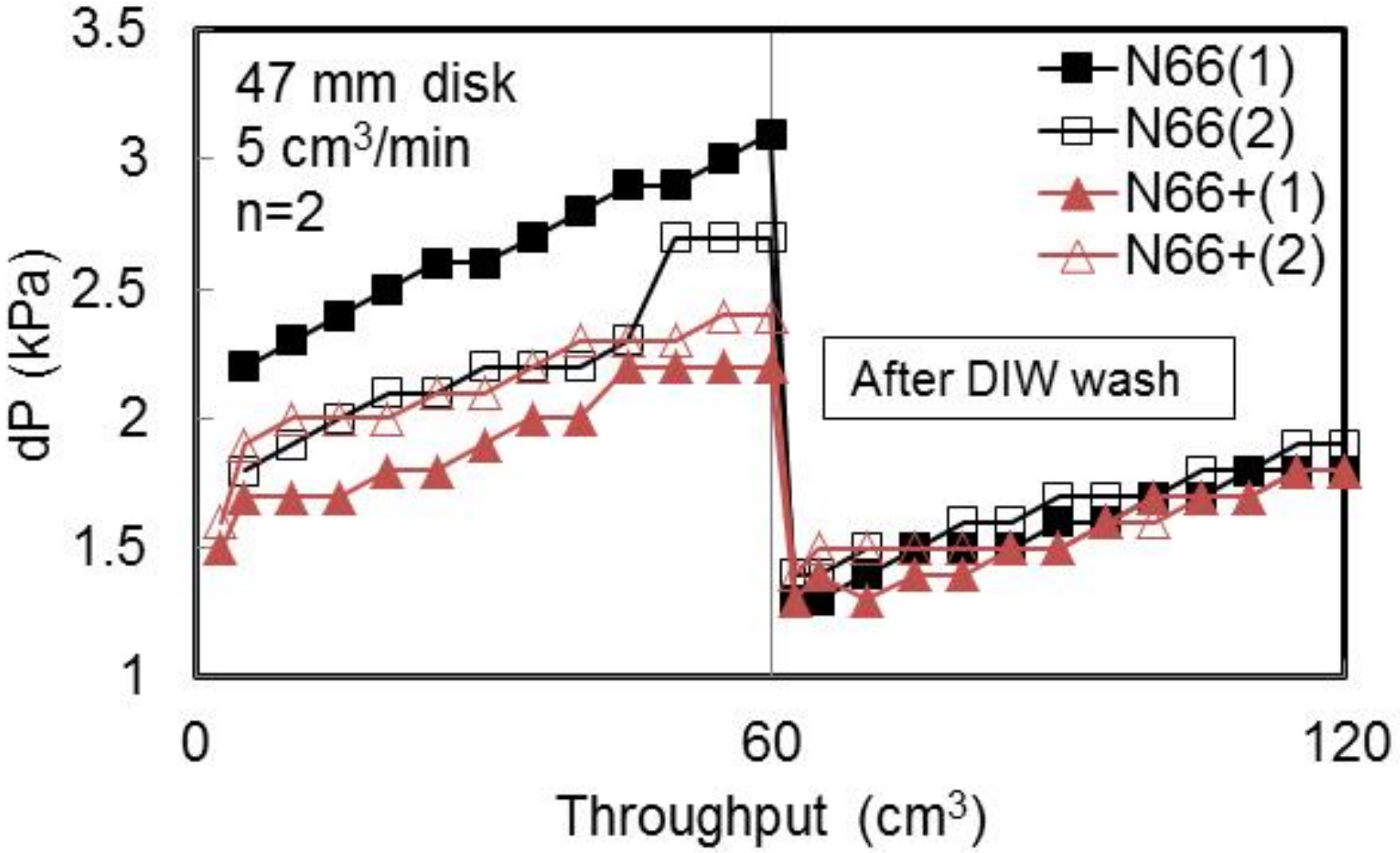
Schematic of abrasive particle adsorption and repulsion at pore surface.
(a) Adsorption at startup
(b) Repulsion of abrasive particles by abrasive particles trapped on the surface

Scenario 2: Effect of DIW wash after ceria slurry filtration - Relative turbidity -



Comparison of relative turbidity of N66, N66+ filtrates before and after DIW wash. Error bars indicate max. and min. value.

Scenario 2: Effect of DIW wash after ceria slurry filtration - dP -



(a) Deviation of dP shift without DIW wash

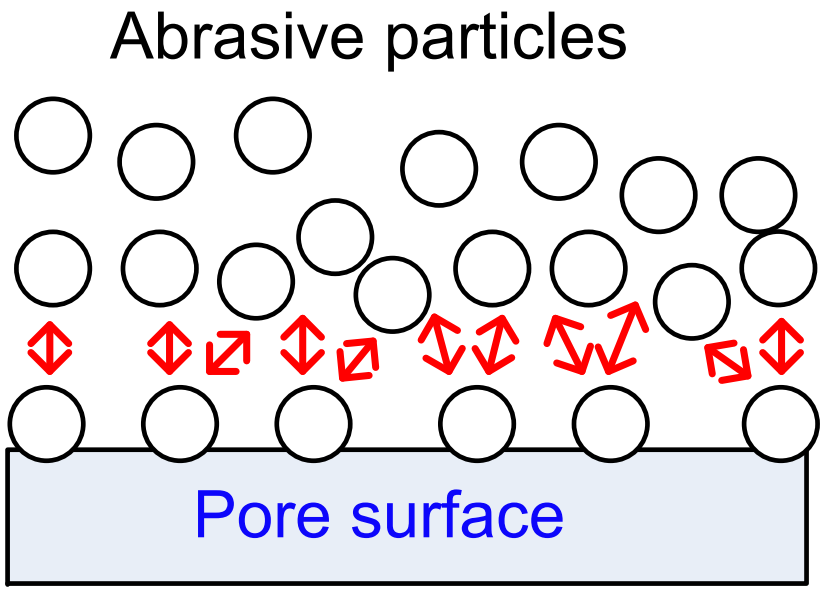
(b) Comparison of dP shift before and after DIW wash

Interpretation Scenario 2:

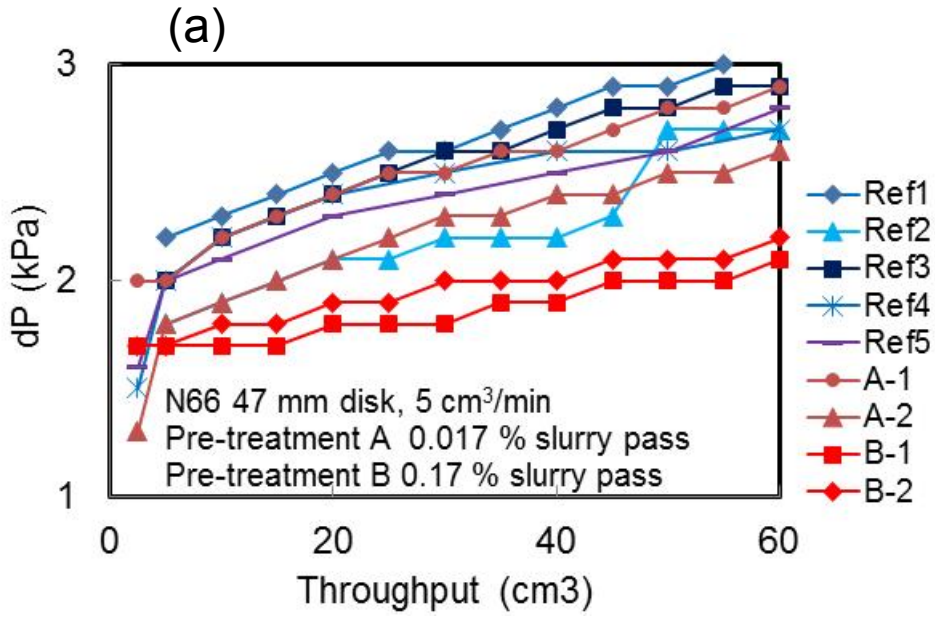
Comparing to virgin Nylon membrane at startup, N66 membrane at re-start after DIW wash showed:

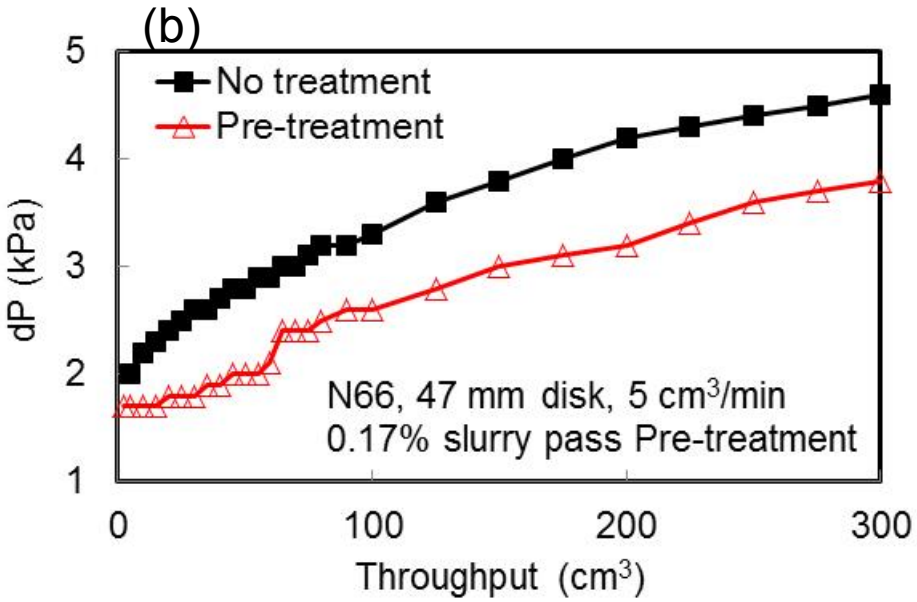
- Lower starting dP
- Smaller deviation in dP over filtrate volume
- Faster recovery of filtrate quality

DIW wash after filtration would cover membrane surface with adequate number of primary particles.
1) dP recovery
2) Repulsion force to dispersed abrasive particles by captured particles
→ less dP increase, dP deviation



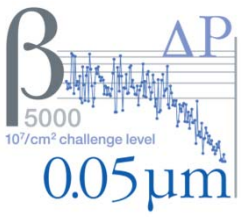
Scenario 3: Effect of filter pre-treatment in ceria slurry filtration





(a) Comparison of virgin membranes (n=5) and pre-treatment (n=2, A = 100x dilution slurry, B = 10x dilution slurry).

(b) Comparison of larger throughput between virgin membrane and pre-treatment with 10x dilution



Conclusions

repulsive force by primary particles on membrane pore surface would dominate slurry filtration except for startup.
→ virgin surface property of membranes is less relevant

DIW wash after producing a small quantity of filtrate would create membrane surface with significant number of primary particles.
1) dP recovery 2) Repulsion force to dispersed abrasive particles by captured particles → less dP increase, dP deviation

pre-treatment with contacting diluted slurry would create similar surface condition as above.

Filter lifetime can be extended by flushing or pre-treatment.

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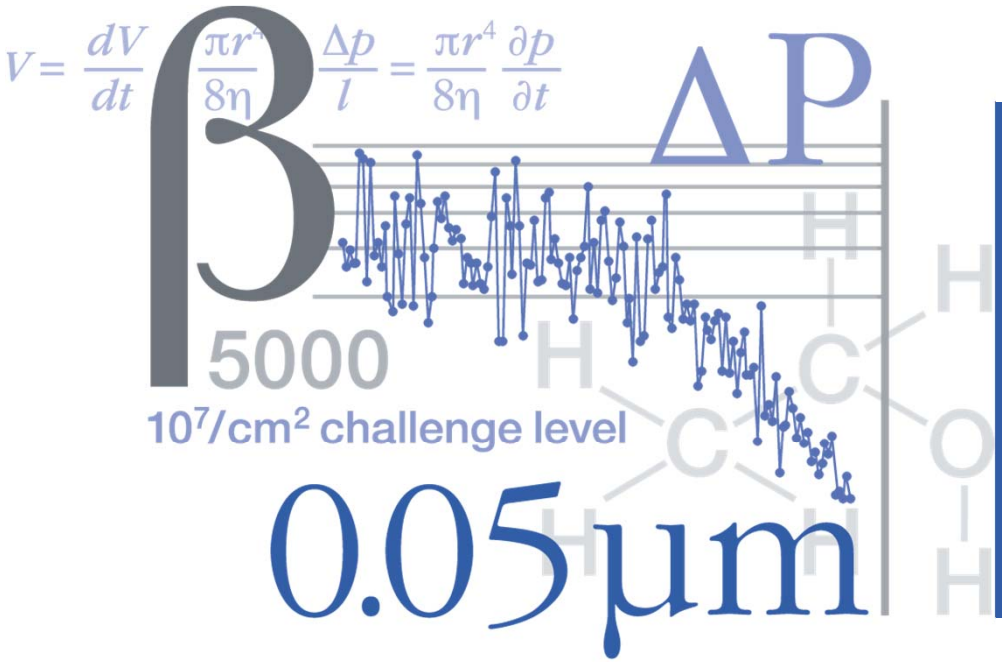

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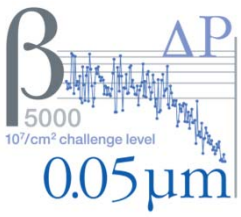
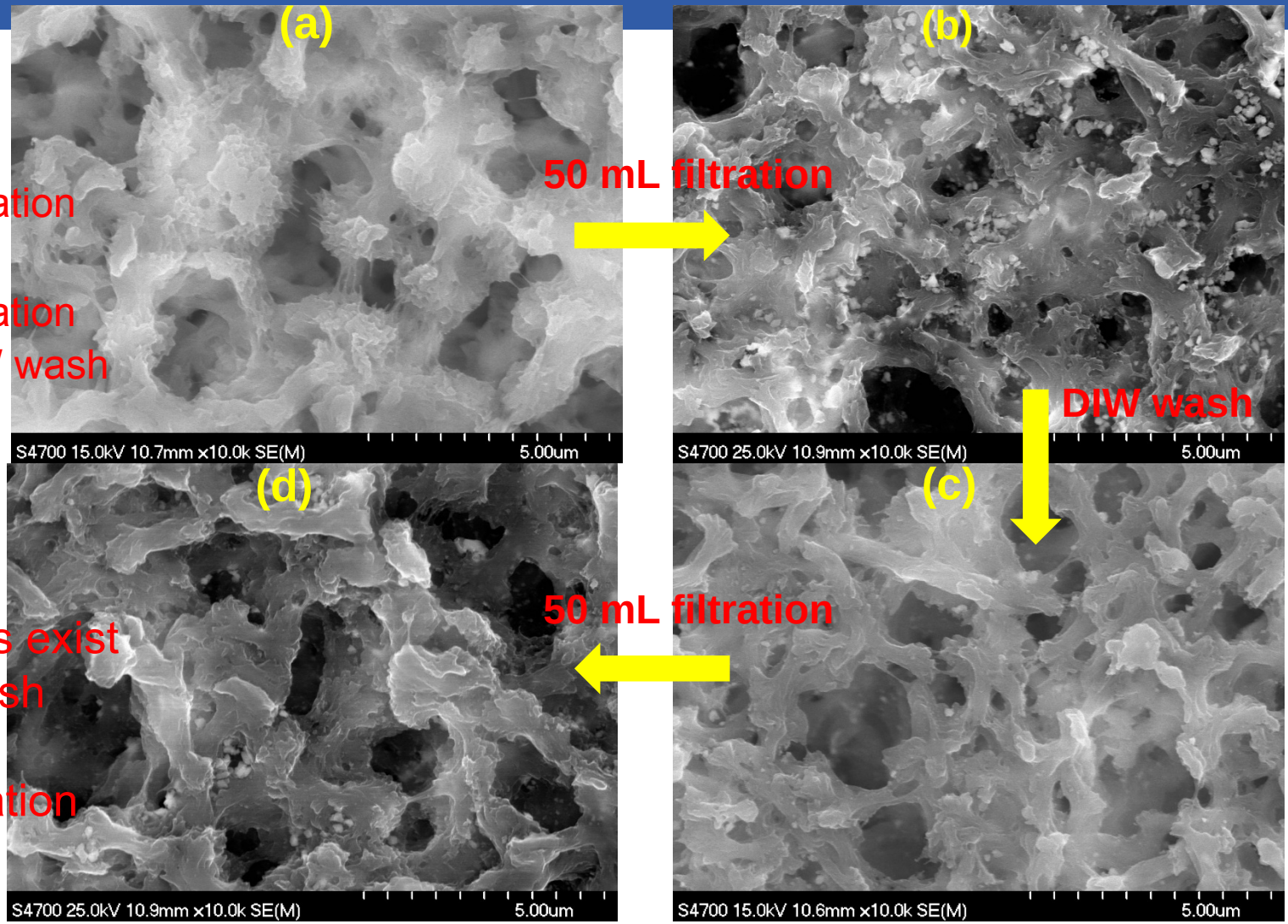


Appendix

Test 2 Effect of DIW wash after ceria slurry filtration - SEM observation of N66 membrane surface -

(a) No use
(b) 50 mL filtration
(c) DIW wash
(d) 50 mL filtration
after DIW wash



Less particles exist after DIW wash and filtration than just filtration

SEM images of N66 membrane surface after ceria slurry filtration, DIW wash.