ULTRAPURE MICRO 2023

Identification and Management of Potential Particle Precursors from Mixed Bed Ion Exchange

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This type of study can not be done alone!

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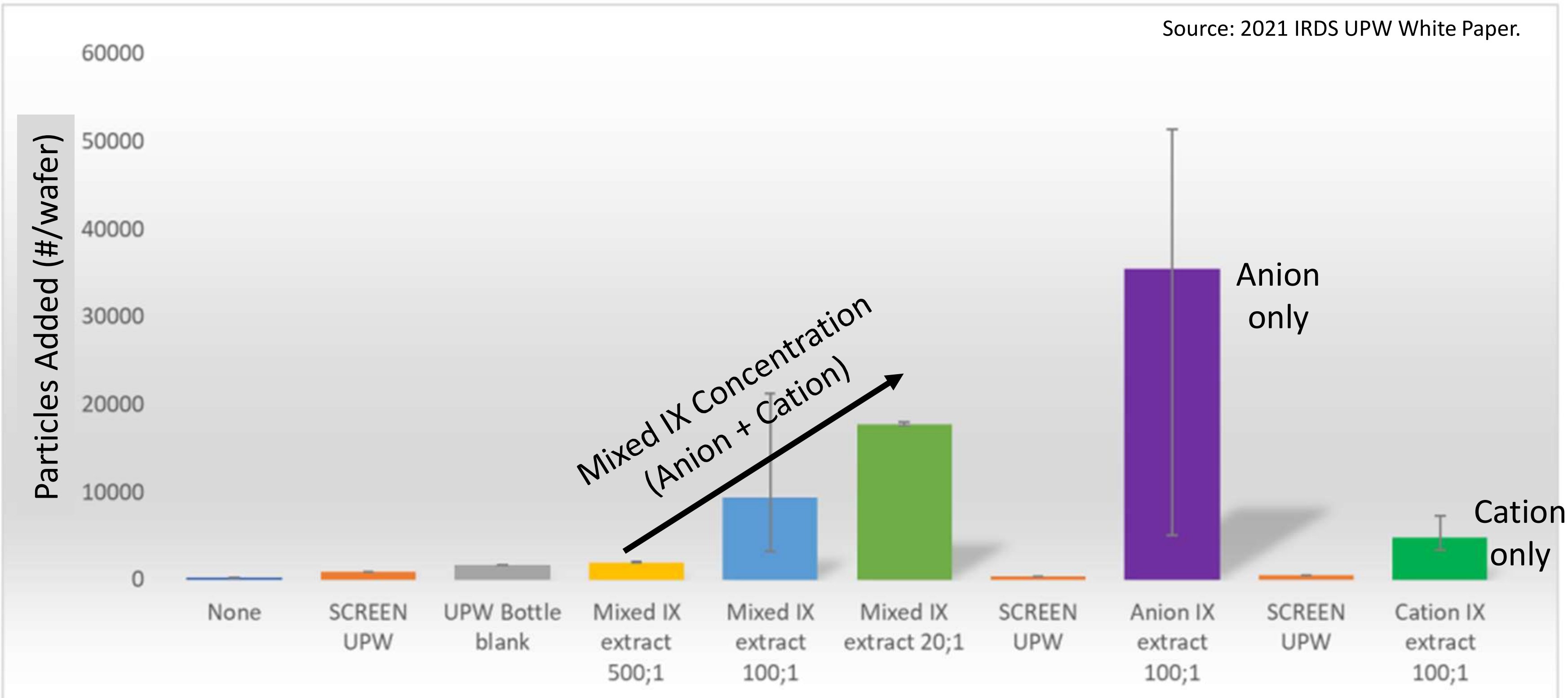
What We Know and a Problem Statement

- Particle precursors (dissolved compounds) are present in UPW and pose a risk of depositing on wafers.
- Mixed bed (MB) ion exchange (IX) is a known potential source of particle precursors.
- To mitigate the risk, the SEMI UPW Task Force is developing a particle precursor challenge based on IX resin to evaluate sub-15 nm filters used for ultrapure water (UPW).
- Size, composition and reactivity data from extract is needed to identify potential particle precursors and to develop organic particle precursor challenges.

Implications for the industry: Wafer Particle Adders



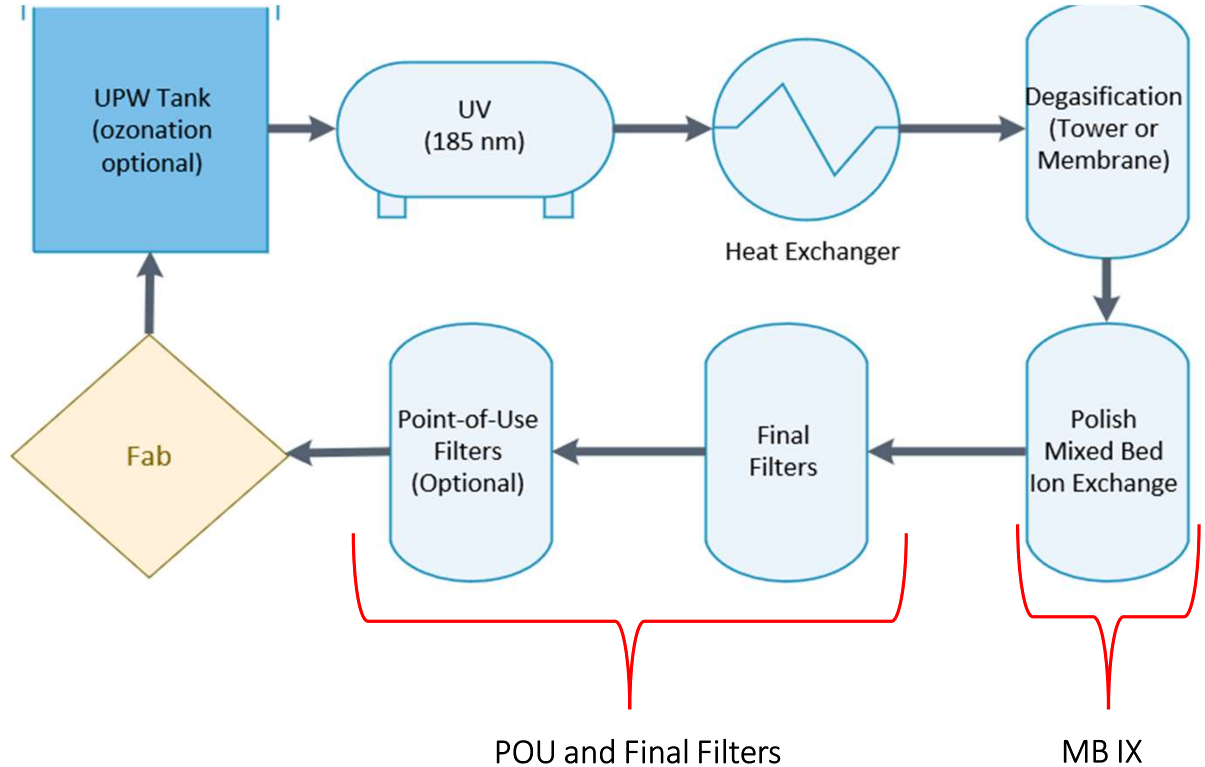
Comparing particles added to the wafer after rinsing and drying with different IX extracts

Why Focus on IX?

- MB IX has large surface area
- MB IX releases trace organics ("leachates")
- Leachates can arrive on the wafers

Typical UPW polish plant with MB IX

adapted from SEMI F61

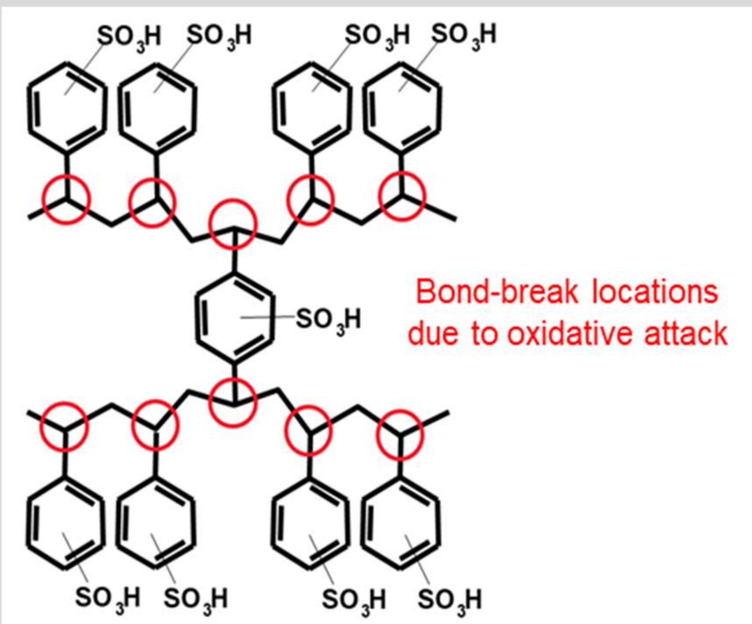


IX Resin Decomposition and Potential Particle Precursors

Source: LANXESS

Strong Acid Cation (SAC) Resin

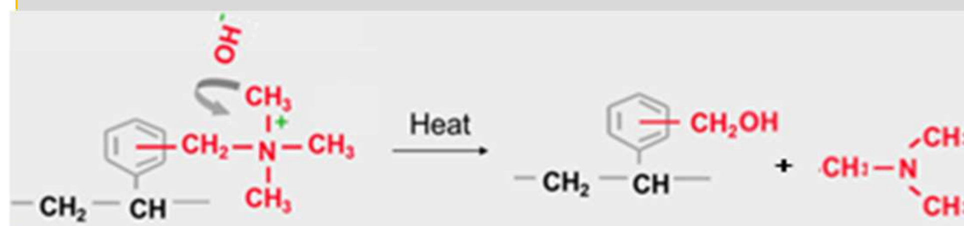
SAC chain scission produces oligomers and polymers

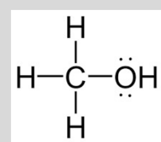


Polymers are an obvious potential source of particles

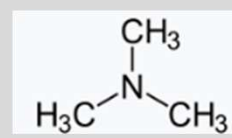
Strong Base Anion (SBA) Resin

SBA amine oxidation produces small molecules

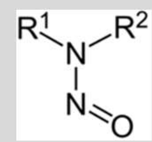




(Methanol)



(TMA)



(NDMA)

Small molecules are a less obvious source of particles

James R. Stahlbush et. al., Reactive Polymers, 13 (1990) 233-240

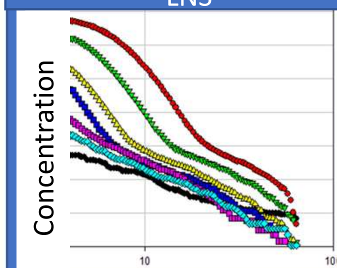
Technical Objectives

1. Extract “useful” concentrations of organics from isolated SAC and SBA resins

Extract organics from Ion Exchange Resin (UPW 80ºC, 7 days)

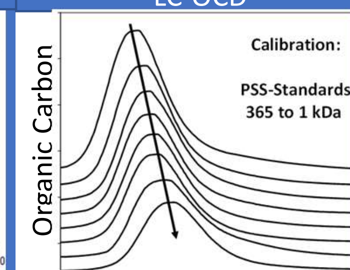
2. Characterize the size, chemical composition and reactivity of organic particles and particle precursors
   - Liquid Nanoparticle Sizing (LNS)
   - Size exclusion Liquid Chromatography with organic carbon detection (LC-OCD)
   - Fourier Transform infrared (FTIR)

LNS



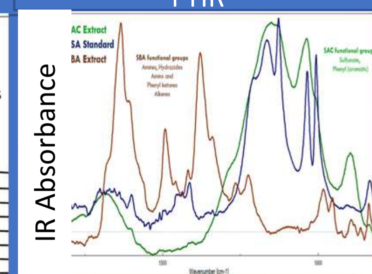
Particle size (nm)

LC-OCD



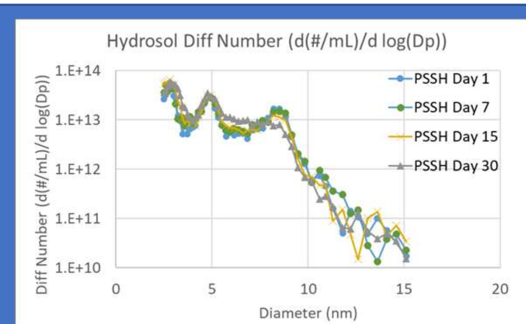
Retention Time (min)

FTIR



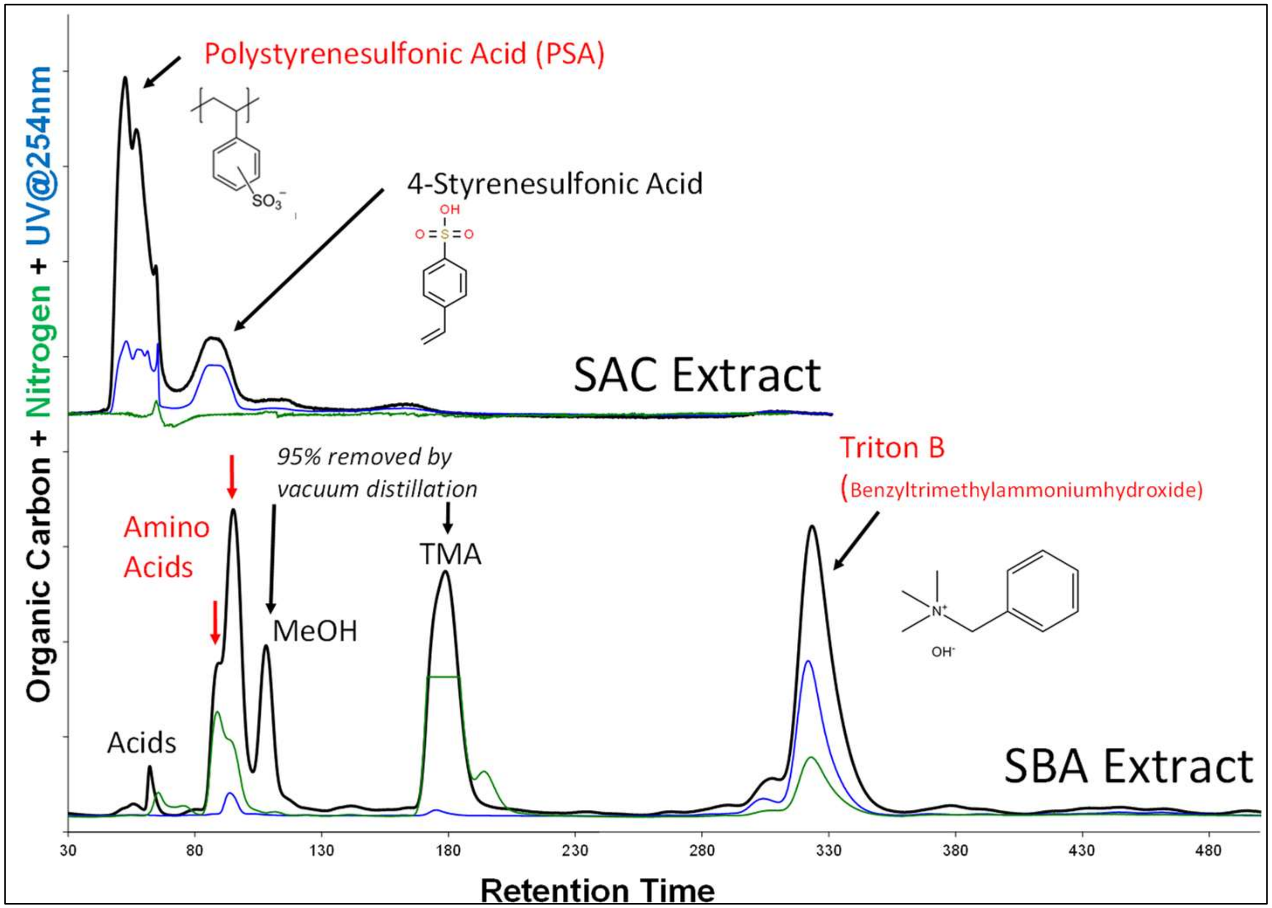
Wavenumber (cm-1)

3. Provide knowledge to develop nanoparticle challenges for sub 15 nm filter devices



LNS of 3, 5 and 8 nm particle challenges

Potential Particle Precursors according to LC-OCD



Compounds highlighted in red are Potential Particle Precursors

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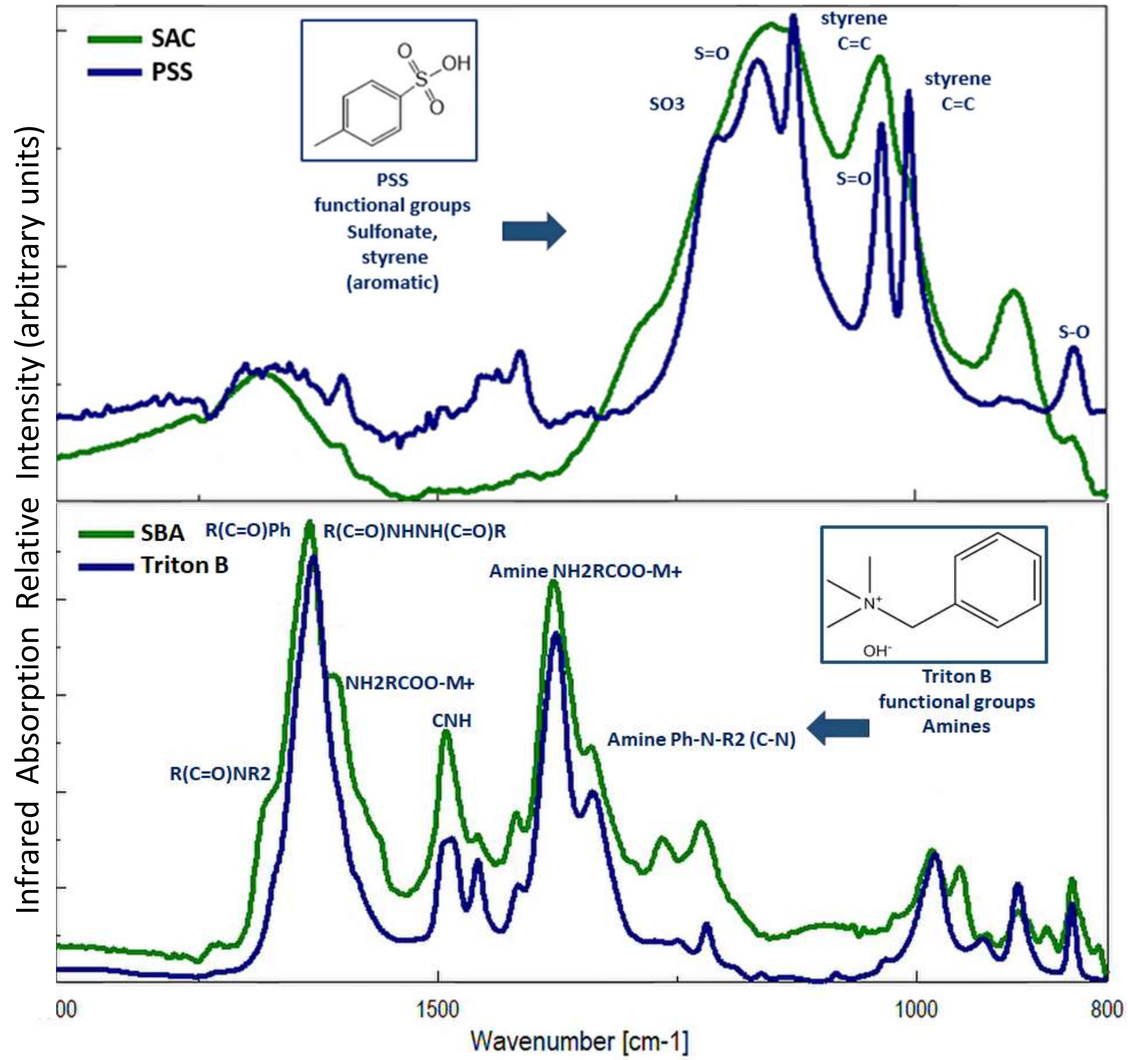
Potential Particle Precursors according to FTIR

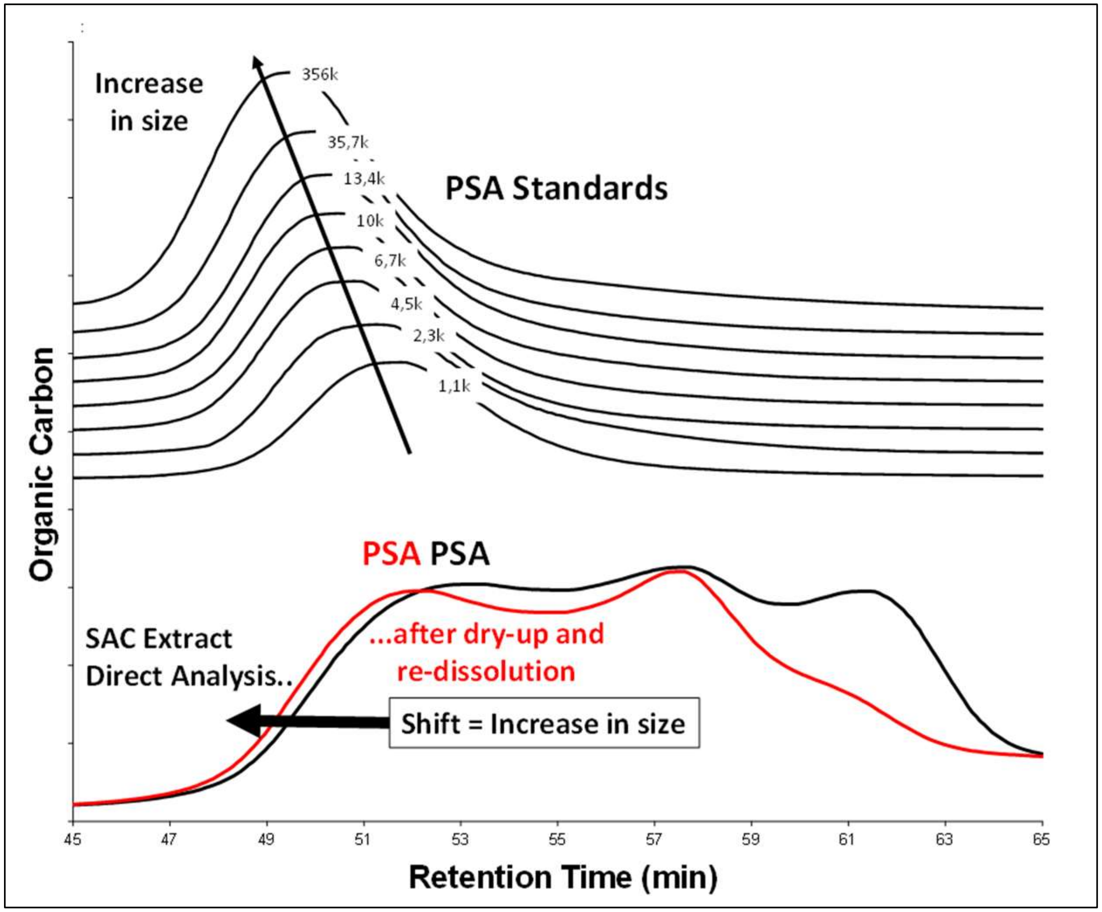
SAC Extract shows organic sulfonate and styrene peaks like PSS standard.

SBA Extract shows organic nitrogen peaks like Triton B standard

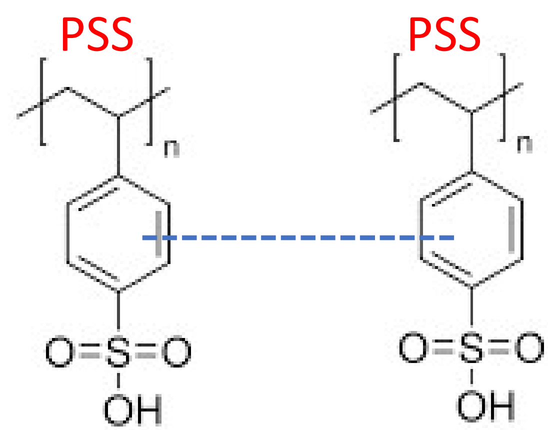


SAC extract contains polydisperse PSS and SBA extract contains small organic nitrogen molecules. All are Potential Particle Precursors

Particle Formation Pathway 1: “Pi-stacking”



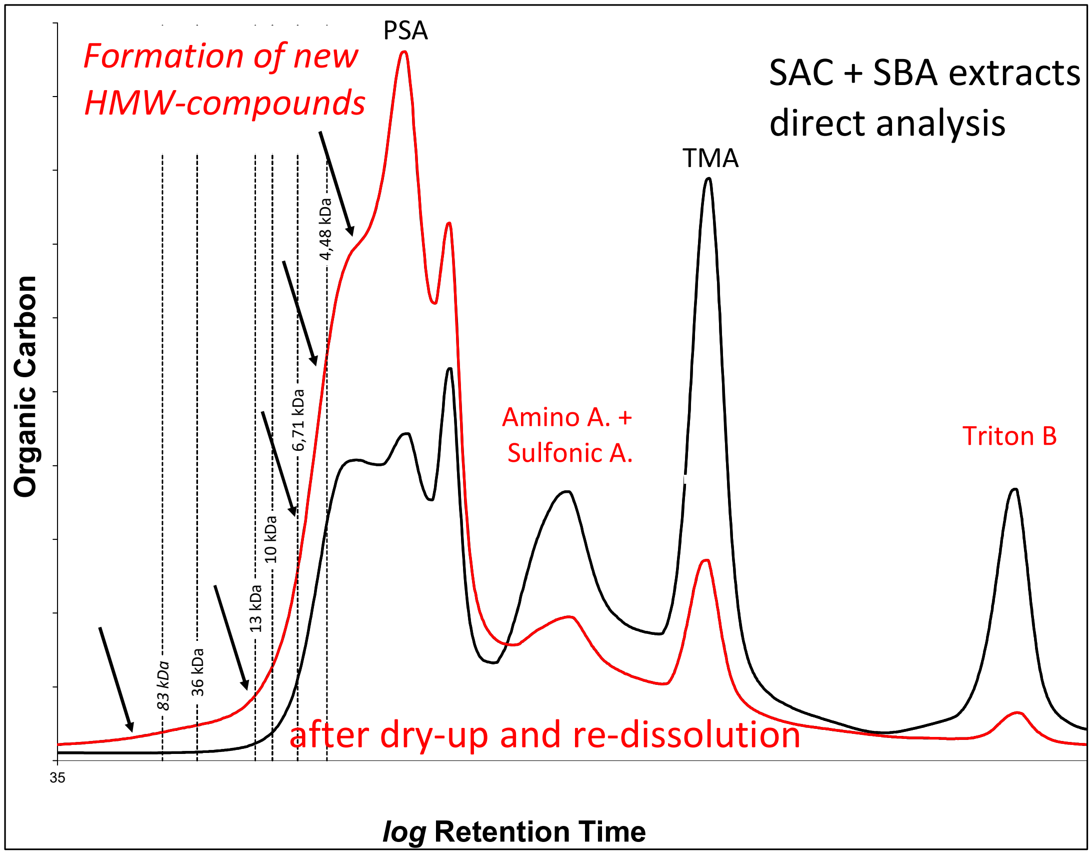
- SAC extracts were dried and re-dissolved.
- New HMW material was formed.



- PSSs of SAC were probably subject to “Pi-stacking” and subsequent aggregation.

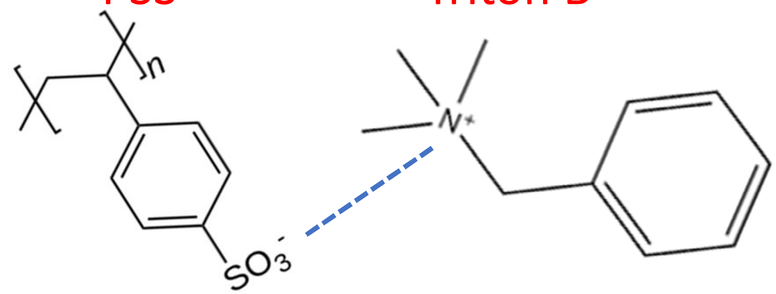
Potential pathway to particle formation: PSS “Pi-stacking”

Particle Formation Pathway 2: “Ion pairing”



- SAC+SBA extracts were dried and re-dissolved.
- New HMW material was formed.

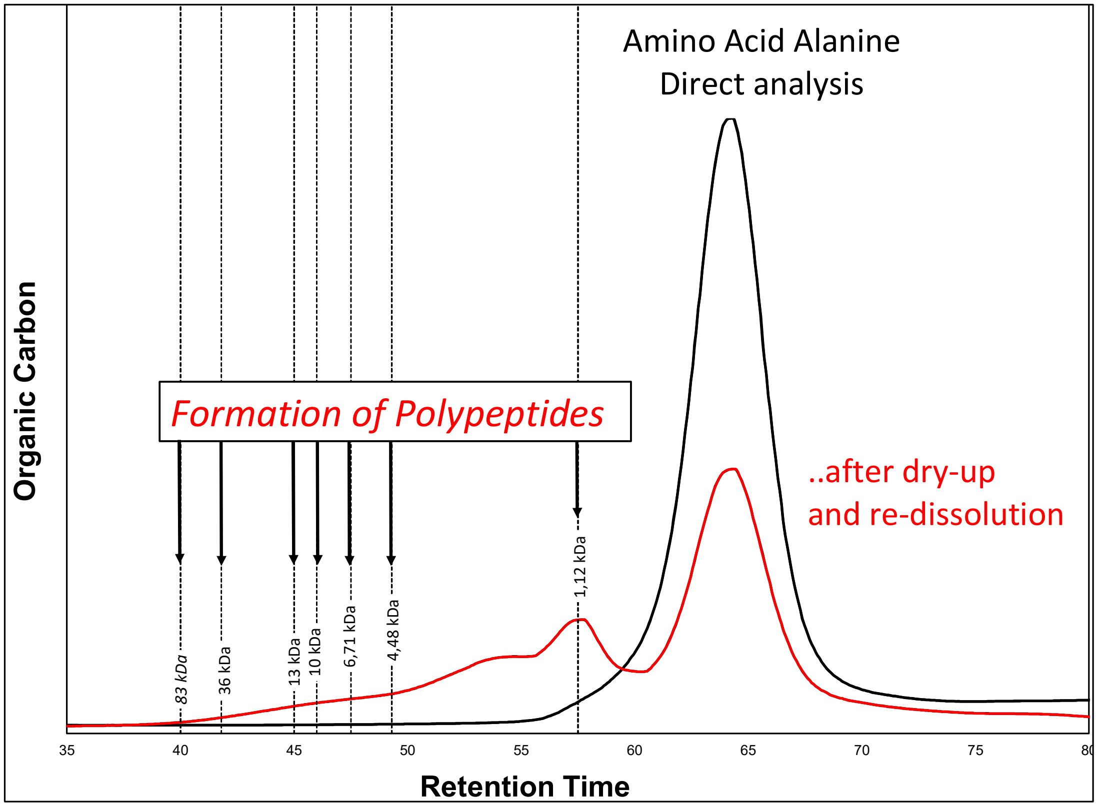
PSS Triton B



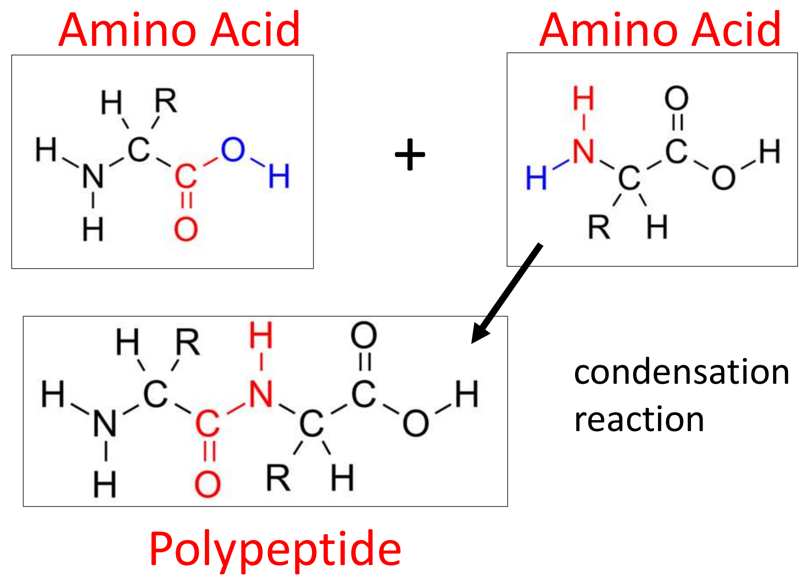
- PSS from SAC reacted with Triton B from SBA to produce ion-pairs which subsequently aggregated

Potential pathway to particle formation: PSS – Triton B “Ion pair complexes”

Particle formation Pathway 3: "Condensation"



- Amino Acid was dried and re-dissolved.
- New HMW material was formed.

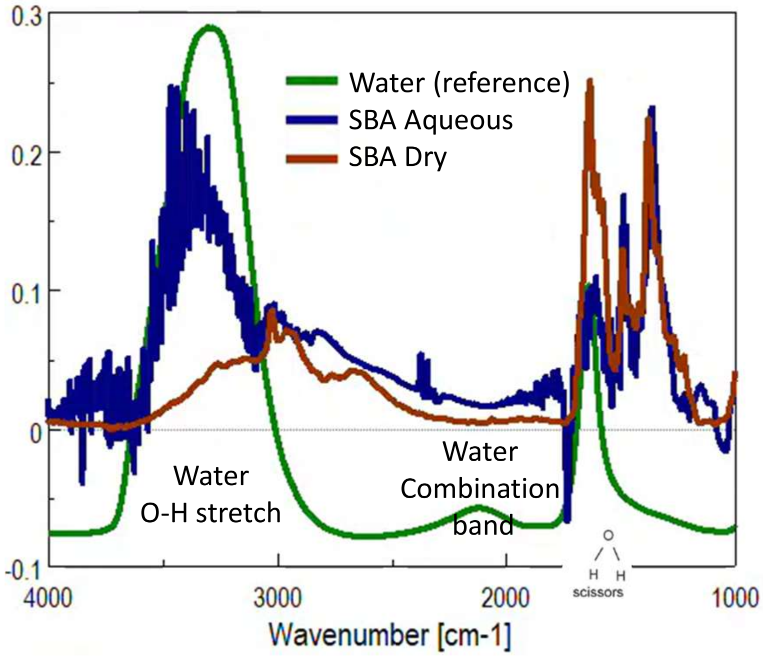


- Amino Acids from SBA polymerize to form polypeptides

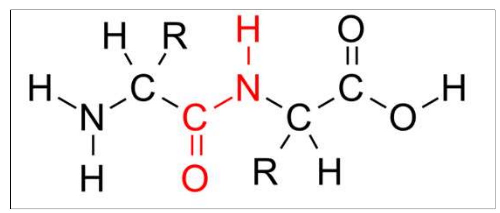
Potential pathway to particle formation: Amino Acid condensation polymerization

"Condensation" Pathway Confirmed by FTIR

Amide search and functional group classification for wet and dry SBA



SBA extract hydration state	Search returns amide classification ?	Functional group classification includes amide IR Bands?
Aqueous	No	No
Dry	Yes, amides and polyamides	Yes, C=O stretch "Amide I" CNH bend "Amide II" CN stretch of amide



Amide bonds form polypeptide links

IR and LC-OCD data indicate condensation reaction of amino acids to form amide bonds and HMW polypeptides

Potential pathway to particle formation: Amino Acid condensation polymerization

Summary and Take-aways

- MB IX in the polish loop releases trace levels of dissolved organics a.k.a. potential particle precursors.
- SAC extract contains monomeric and polymeric PSS and is an obvious source of potential particle precursors.
- SBA extract contains low molecular weight organic nitrogen which is not an obvious particle precursor.
- SAC and SBA extracts can react upon drying to form new high-molecular weight material.
- We proposed three pathways to transform potential particle precursors to particles on wafers.
    1. "Pi-stacking" of PSS from SAC
    2. "Ion pairing" of anionic PSS from SAC with cationic Triton B from SBA
    3. "Condensation polymerization" of amino acids from SBA
- These findings shed new light on the potential impacts of the polishing MB, and the development of particle and particle precursor challenges that could be used to evaluate sub-15 nm filter devices for UPW.