

LAND APPLICATION OF BIOSOLIDS: TRENDS AND PERSPECTIVES

Linda S. Lee

Distinguished Professor, Dept. of Agronomy
Professor Environmental Ecological Engineering
lslee@purdue.edu; purdue.ag/lindalee



Thursday, Sept. 11, 2025
Virtual Webinar



Land Application of Biosolids: Trends and Perspectives

Conduit of our wastes
Biosolids, Wastewater



Introduction, Perceptions

Occurrence of Organic Contaminants of Concern (COCs)

COCs in biosolids processing examples

COC Fate in a subset of biosolids-applied sites

Feed Crop Uptake Snapshots: Greenhouse and Field

Perspectives and a Path Forward

EPA Office of Inspector General Report 2022

EPA Unable to Assess the Impact of Hundreds of Unregulated Pollutants in Land-Applied Biosolids

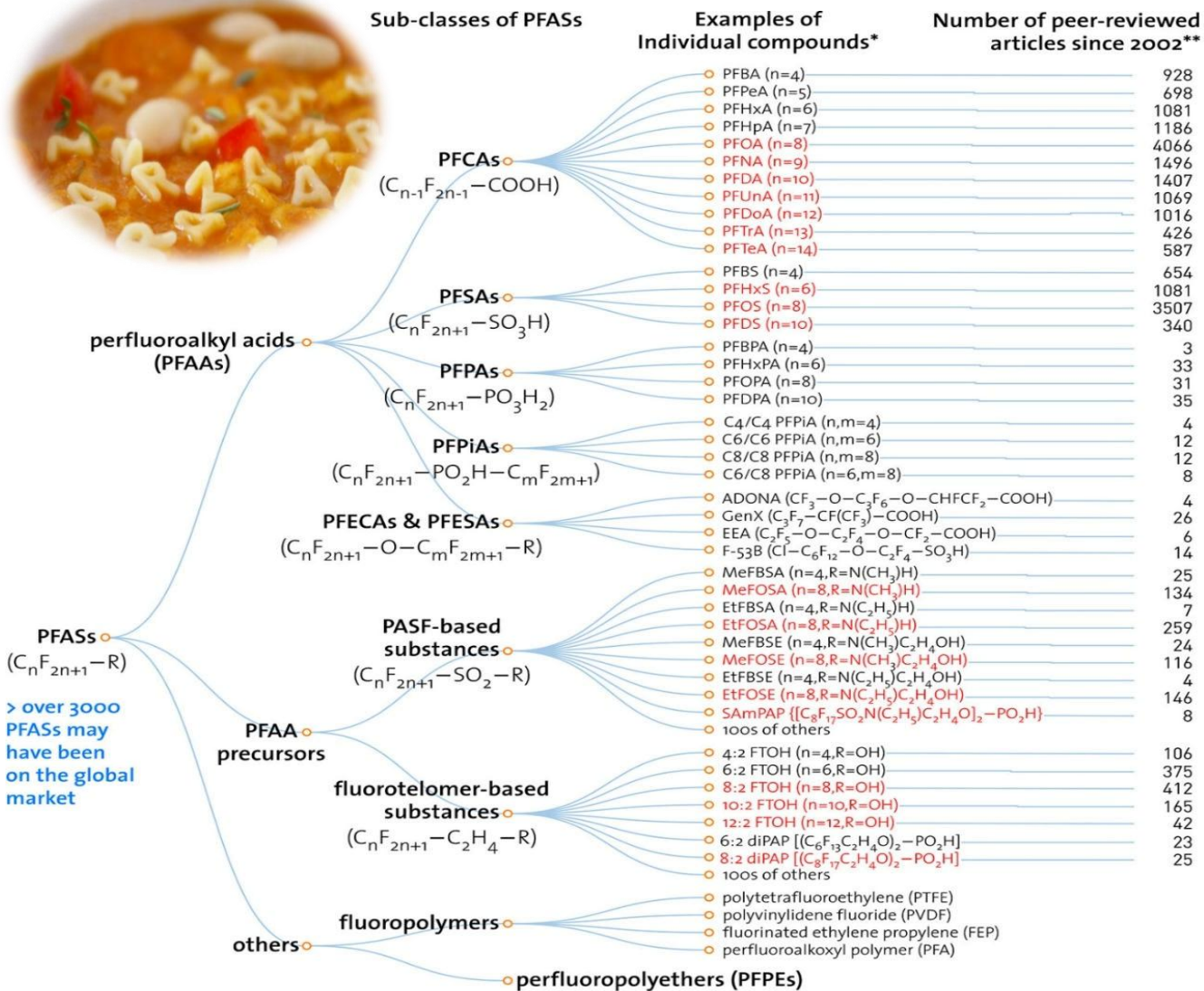
- The EPA identified 352 pollutants in biosolids that cannot be considered for regulation due to either lack of data or risk assessment tools.
- 61 pollutants designated as acutely hazardous, hazardous or priority pollutants plus pharmaceuticals, steroids, etc.

**Response from USEPA Office of Water
and Office of Enforcement and Compliance Assurance on
Draft Report and OIG Evaluation
(Appendix D of OIG Report)**

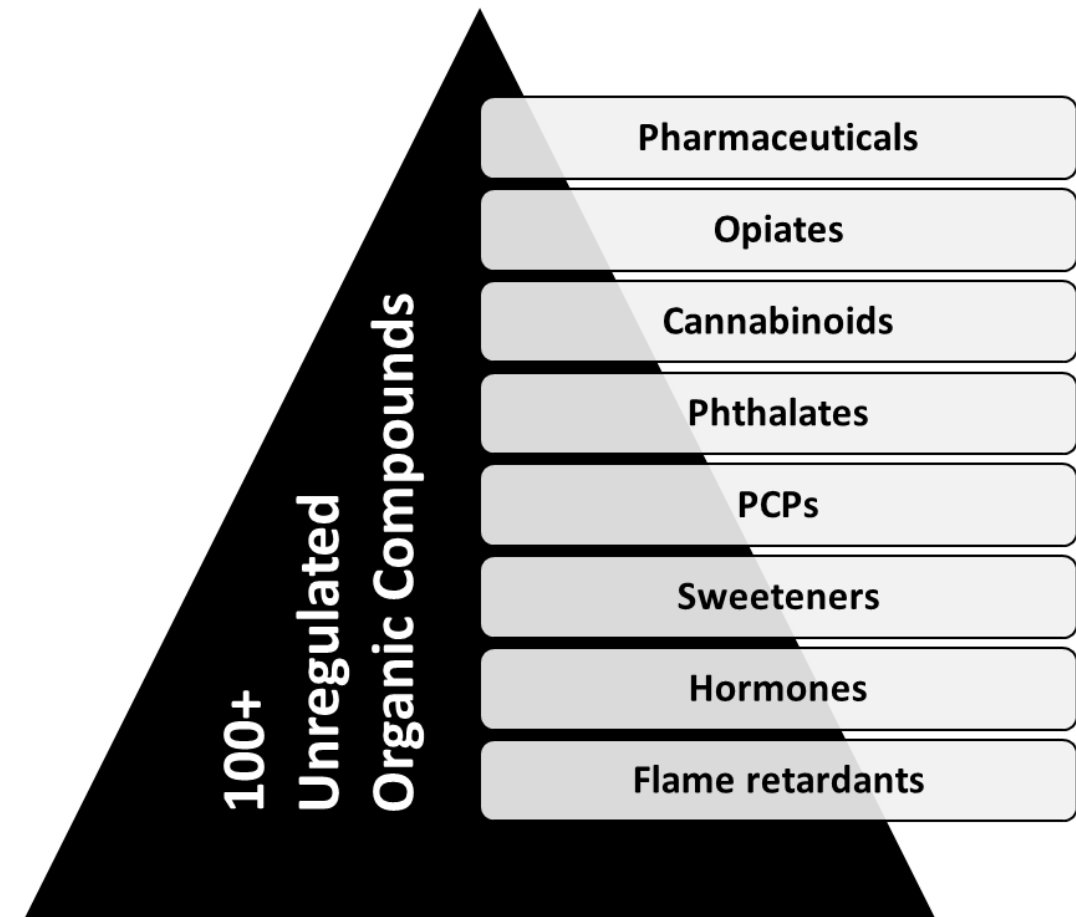
“We are concerned about how the science is presented in the OIG report. It is biased and raises alarm ... and is taken out of context.

.....The presence of a pollutant in biosolids alone does not equate to scientific risk, but the EPA's Biosolids Program is working hard to prioritize its risk assessment work for known but not yet regulated pollutants.”

Per- & Polyfluoroalkyl Substances



Pharmaceuticals & personal care product chemicals (PPCPs) and still others



Reflects Innovations and Societal Use

* PFASs in **RED** are those that have been restricted under national/regional/global regulatory or voluntary frameworks, with or without specific exemptions (for details, see OECD (2015), Risk reduction approaches for PFASs. <http://oe.cd/i1AN>).
 ** The numbers of articles (related to all aspects of research) were retrieved from SciFinder® on Nov. 1, 2016.

MANAGING CHEMICALS OF CONCERN (COCS) IN WASTEWATER AND BIOSOLIDS MANAGEMENT

Multiple PFAS sources – industry, landfills, domestic



Conduit of our wastes



Wastewater Influent



Sorption to Sludge



Effluent discharged to streams or used for irrigation



Biosolids



Land-applied as a soil amendment

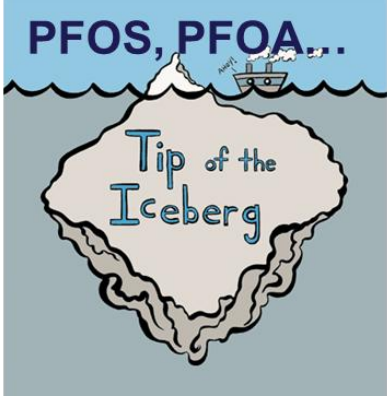


Treatment process with type and treatment stage

COCS entering our WWRFs exit via effluent or sludge partially altered to other compounds or unaltered – most are NOT regulated

Most recent regulatory activity have been focused on PFAS

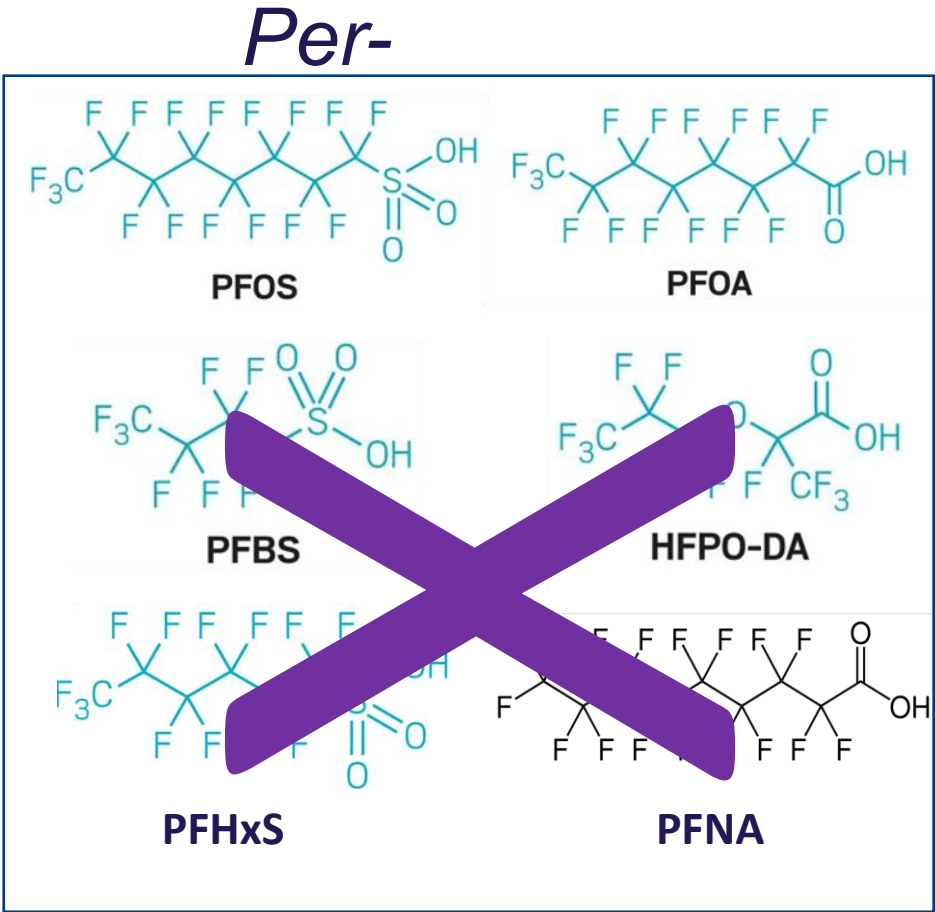
APRIL 10, 2024 U.S. EPA SETS PFAS MAXIMUM CONCENTRATION LEVELS (MCLS) IN PPT (NG/L)



PFAS	MCLG	MCL (enforceable levels)
PFOA (C8)	Zero	4.0 ppt
PFOS (C8)	Zero	4.0 ppt
PFNA (C9)	10 ppt	10 ppt
PFHxS (C6)	10 ppt	10 ppt
PFBS (C4)	1000 ppt	1000 ppt
HFPO-DA (GenX Chemicals)	10 ppt	10 ppt
Mixtures of 2 or more PFNA, PFHxS, PFBS, and HFPO-DA	1.0 (unitless) Hazard Index Σ MCL/measured concentration	

MCLG = maximum contaminant level goals

2025



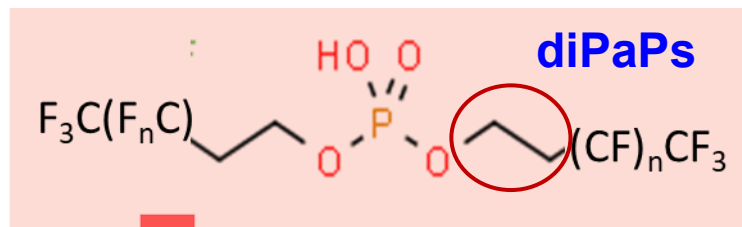
REGULATED OR NOT WE HAVE TO ADDRESS PUBLIC/COMMUNITY PERCEPTION/CONCERNS



PFAS '*news*' has revived/escalated concerns about COCs in biosolids.

PFAS TRANSFORMATION IN SOLIDS PROCESSING IN WASTEWATER TREATMENT PLANTS AND AFTER LAND APPLICATION: A CASE OF MULTIPLICATION

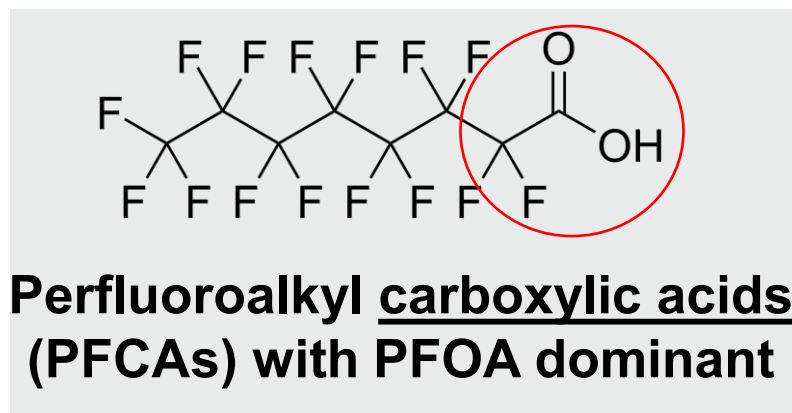
C8 Fluorotelomer-based Example



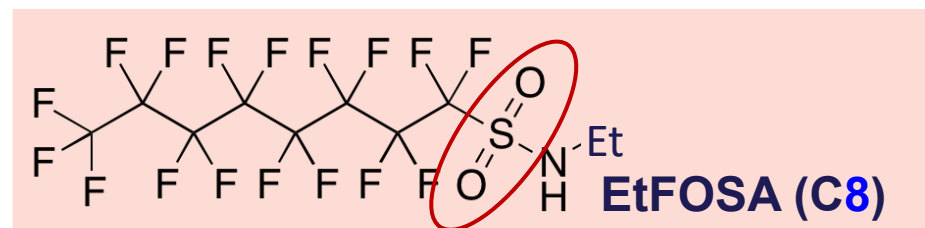
Precursors to PFAAs

*Multiple steps,
pathways, and rates*

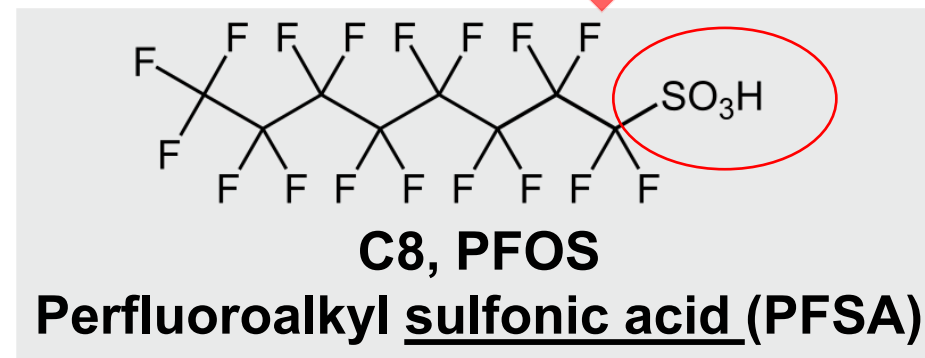
**Intermediates and
Terminal Metabolites
(the PFAAs)
More mobile than
precursors**



C8 Electrochemically-derived Example

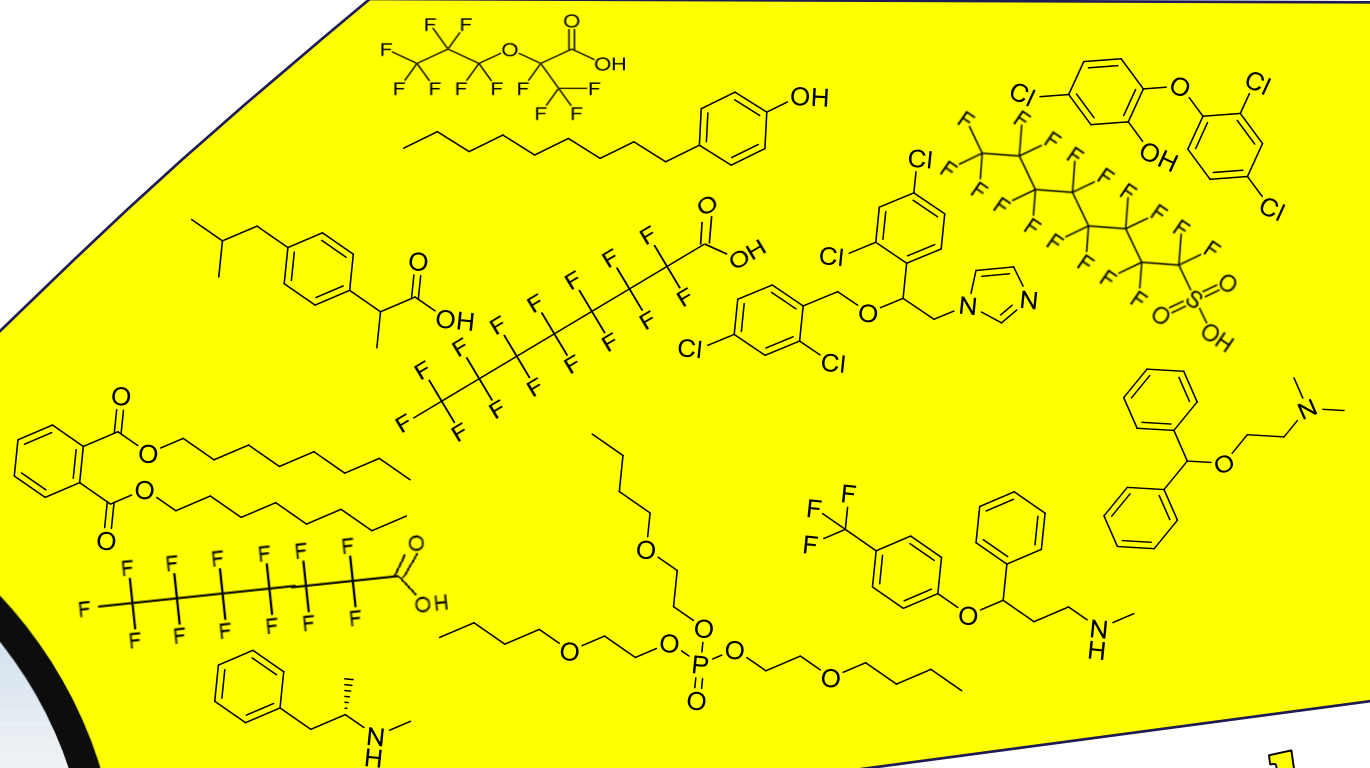
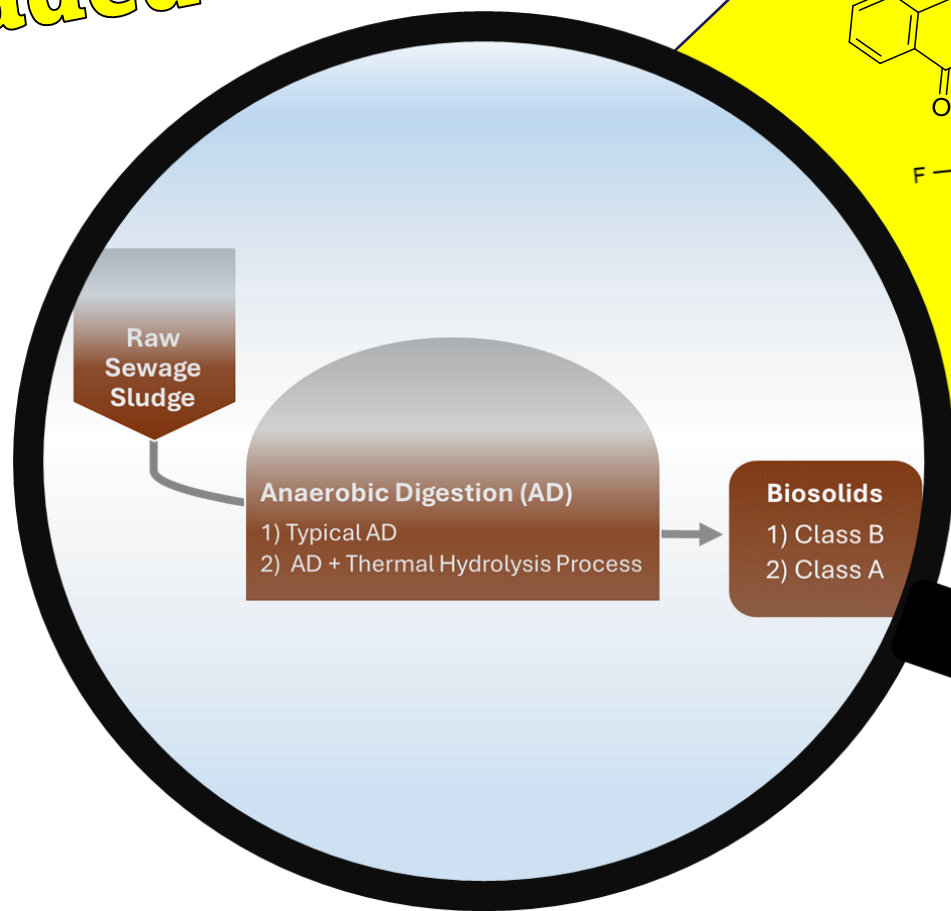


Intermediates



PFCAs + PFSA = Perfluoroalkyl acids (PFAAs)

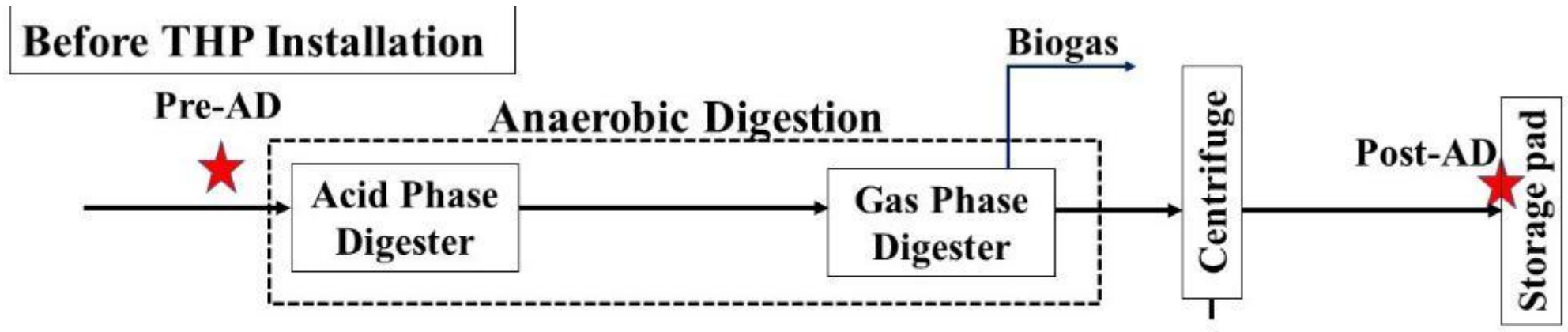
Anaerobic Sewage Sludge Treatment Upgraded



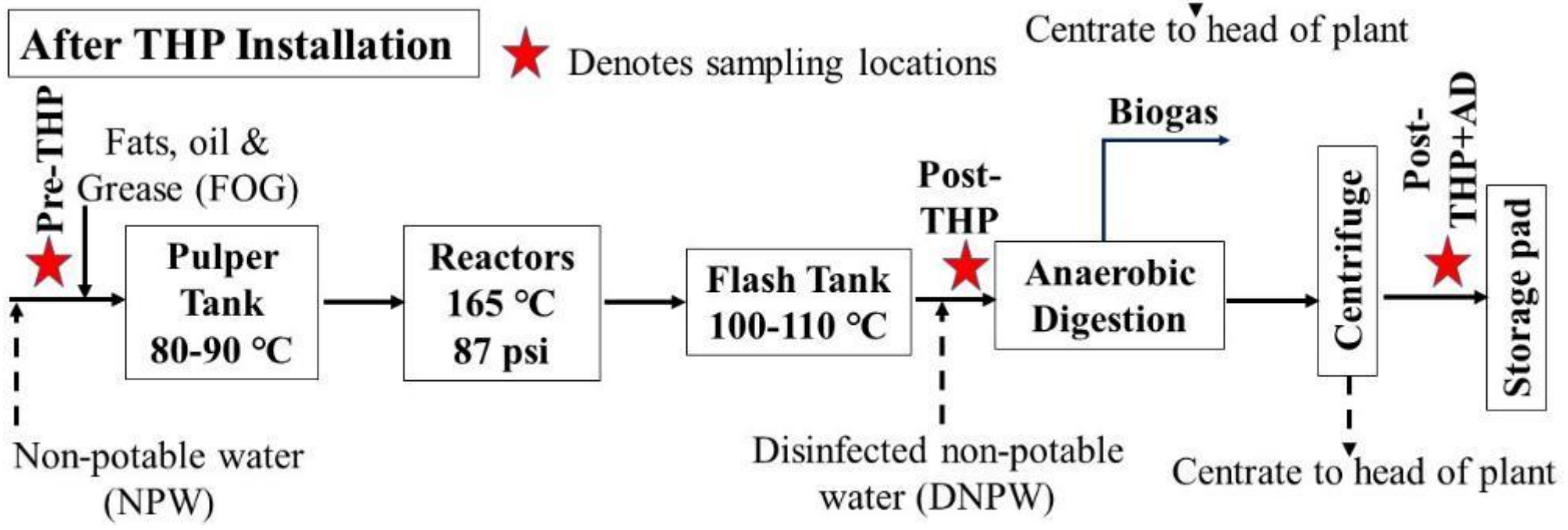
PFAS & Unregulated Organic Chemicals (UOCs) Monitored

PFAS Transformation in Solids Processing to Biosolids for Application

Class B



Class A



Thermal Hydrolysis Process (THP)

Biogas
Production



Sustainable Energy

- ✓ Biogas production is used towards system operation

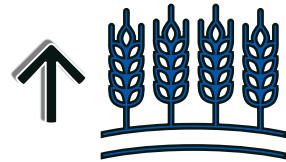
↑ TS %



Transportation

- ✓ Higher Total Solids reduces hauling costs (lower water weight) and the associated greenhouse gasses

Class A
Biosolids



Fertilizer

- ✓ Class A biosolids are land applied as fertilizer
- ✓ The increased biosolid treatment capacity and reduces what may be going to incineration or landfilling

↑ Digester
Loading
Capacity



Incineration



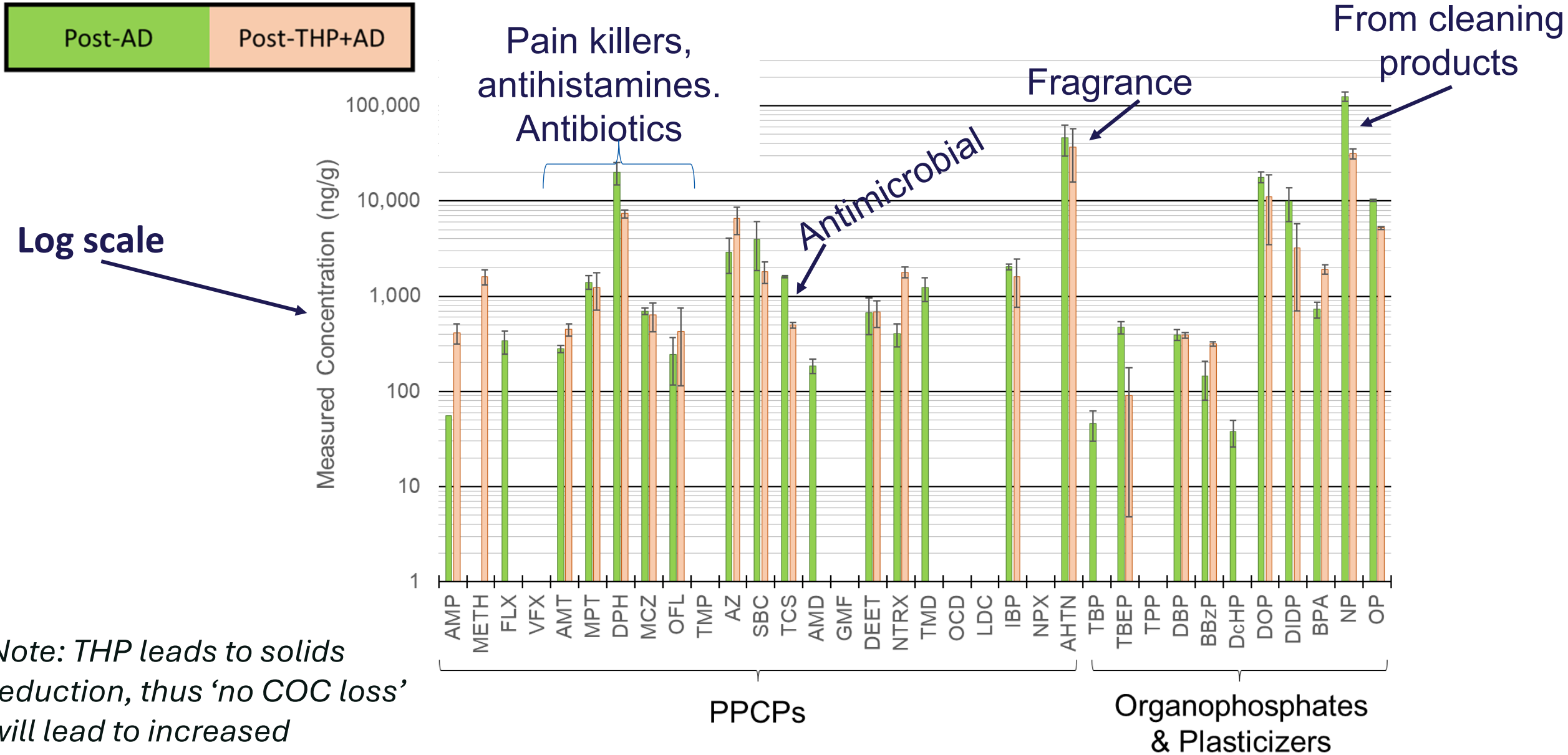
Landfill

↓ Carbon



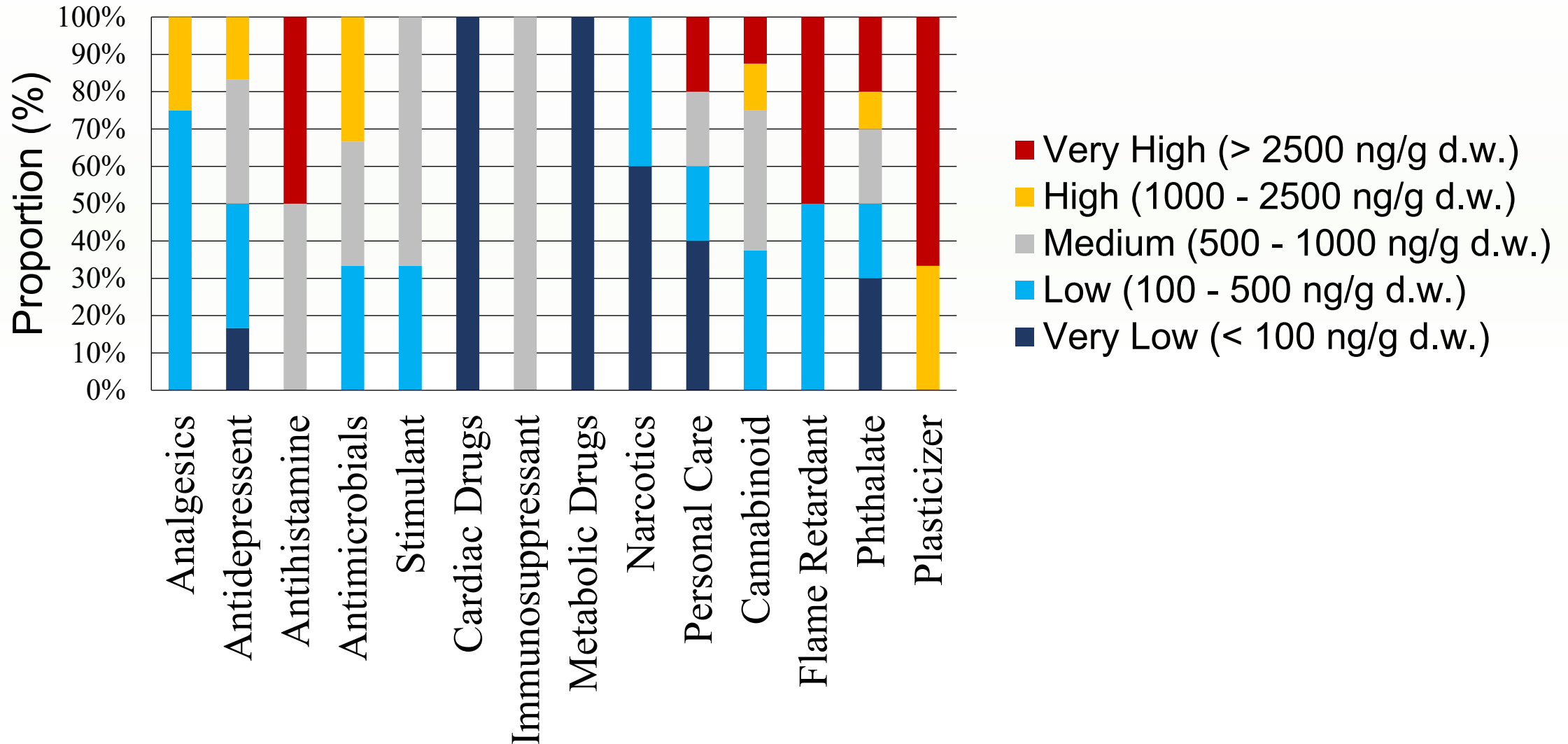
Sustainability

Post-AD and Post THP+AD



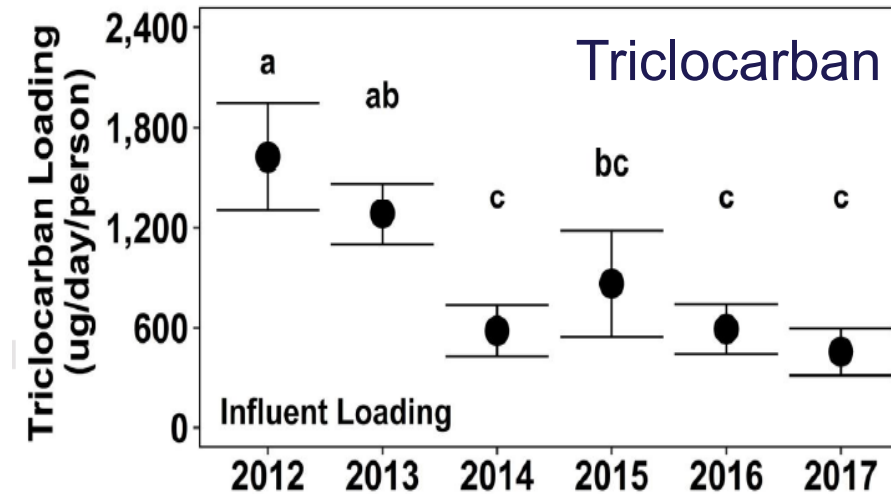
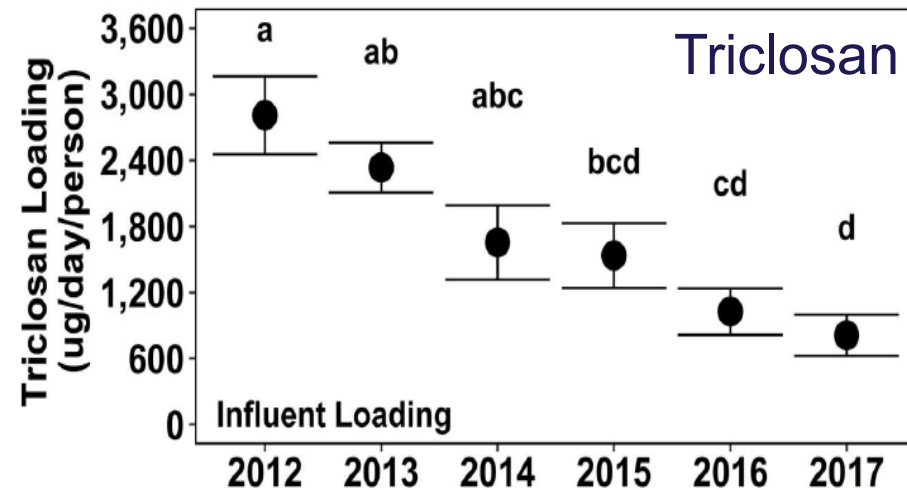
Note: THP leads to solids reduction, thus 'no COC loss' will lead to increased measured concentrations

Class A Biosolids: THP Product Example



We have experienced similar trends with other chemicals

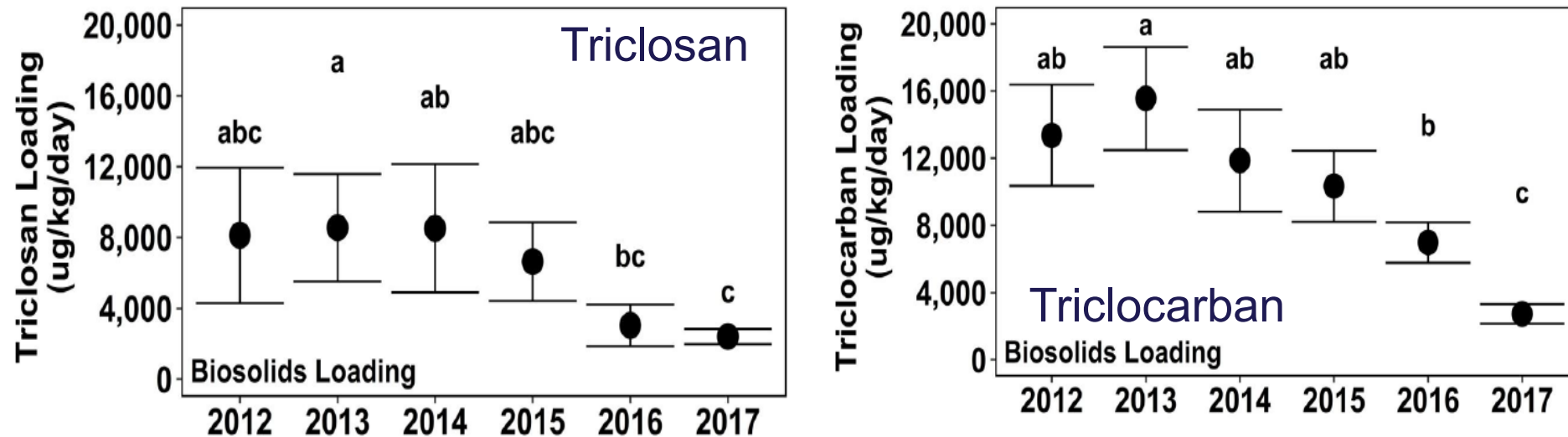
TRICLOSAN & TRICLOCARBAN (ANTIMICROBIAL AGENTS) (INFLUENT: $\mu\text{G}/\text{DAY}/\text{PERSON}$ VS SAMPLING YEAR 2012-2017)



- Fingerprint of regulations and consumer choices
- Consumer choices can lead to some volunteer removal from products prior to regulations
- Regulations in Sept 2016 and Dec. 2017 eliminated their use in many products

We have experienced similar trends with other chemicals

TRICLOSAN & TRICLOCARBAN IN BIOSOLID (ANTIMICROBIAL AGENTS) (BIOSOLIDS LOADING: $\mu\text{G}/\text{DAY}/\text{PERSON}$ VS SAMPLING YEAR 2012-2017)



- Decline over time also observed in biosolids
- Consumer opinions and choices make a difference

Considering Hazards (EPA HCD, Comptox)

Concentration Range

	Very High (> 2500 ng/g d.w.)
	High (1000 - 2500 ng/g d.w.)
	Medium (500 - 1000 ng/g d.w.)
	Low (100 - 500 ng/g d.w.)
	Very Low (< 100 ng/g d.w.)

Hazard Rating

VH - Very High	H - High	M - Medium	L - Low
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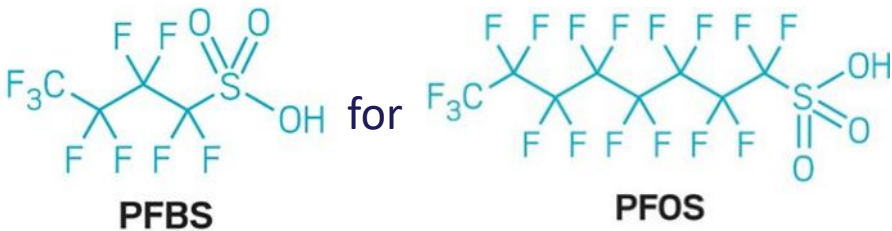
Compound Identification		Human Health Effects		Ecotoxicity (Aquatic)		Fate (QSAR Model)
Name	Class	EDC	Dev	Acute	Chronic	Bioacc
Methylparaben	PCPs	H	H	M	H	L
Tonalide		H	H	H	VH	L
Dicyclohexyl phthalate	Phthalate	H	H	H	H	H
Diethyl phthalate		H	H	H	VH	L
Di-n-octyl phthalate		H	M	H		VH
Dibutyl phthalates		H	H	VH	H	M
Dinonyl phthalate		H	H	H		L
Benzyl butyl phthalate		H	H	VH	VH	H
Di(2-ethylhexyl) phthalate		H	H	H	VH	VH
Diisodecyl phthalate		H	H	VH		L
Bisphenol A		H	H	H	VH	L
Nonylphenol		H	M	VH	VH	H
Octylphenol	Plasticizer	H	H	VH	VH	M
Triphenyl phosphate		H	L	VH	VH	H
Tris(2-butoxyethyl) phosphate		H	H	M	L	L

Name	Class	EDC	Dev	Acute	Chronic	Bioacc.
Acetaminophen	Analgesic	L	L	M	VH	L
Doxepin	Antidepressant	H	H	H		L
Venlafaxine		H	H	H		L
Fluoxetine		L	H	VH	VH	M
Amitriptyline + Maprotiline		H	H	VH	VH	H
Diphenhydramine	Antihistamine	L	L	H	VH	M
Miconazole	Antimicrobial	L	L	VH	VH	M
Triclosan		H	M	VH	VH	L
Cannabigerol	Cannabinoid	H	H	VH		L
delta-8-Tetrahydrocannabinol		H	H	VH		M
Tetrahydrocannabivarin		H	H	VH		M
Caffeine		H	H	L	H	L
Ibuprofen		H	H	L	VH	L
Cannabichromene		H	H	VH		M
Cannabicyclol		H	H	VH		M
delta-9-Tetrahydrocannabinol		H	H	VH		M
Cannabidiol		H	H	VH		M
Cannabinol		H	H	VH		M
Amiodarone	Other Pharm.	I	H	VH	H	M
Hydroxychloroquine		H	H	H	H	L
Simvastatin		L	H	VH		I
Codeine	Narcotics	H	H	H		L
Fentanyl		H	H	VH		L
Methadone		H	H	H	VH	M
Naltrexone		H	H	H		L

- Concerns are for aquatics
- Some pharms, fragrances and plasticizers are of high concern

MOVING ON TO PFAS IN BIOSOLIDS PROCESSING

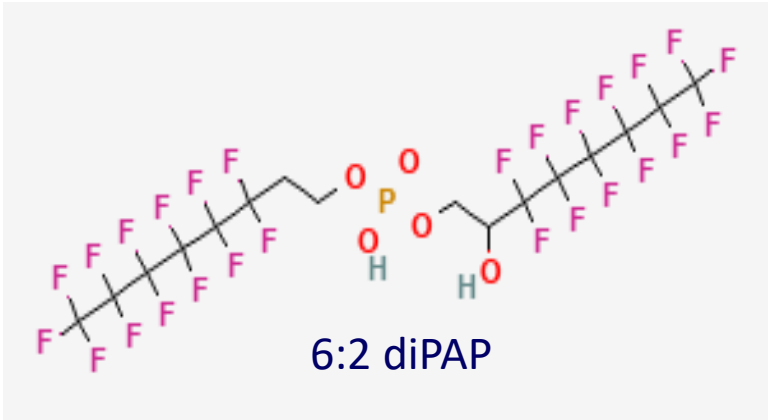
- Short chain homologues such as PFBS substituted PFOS



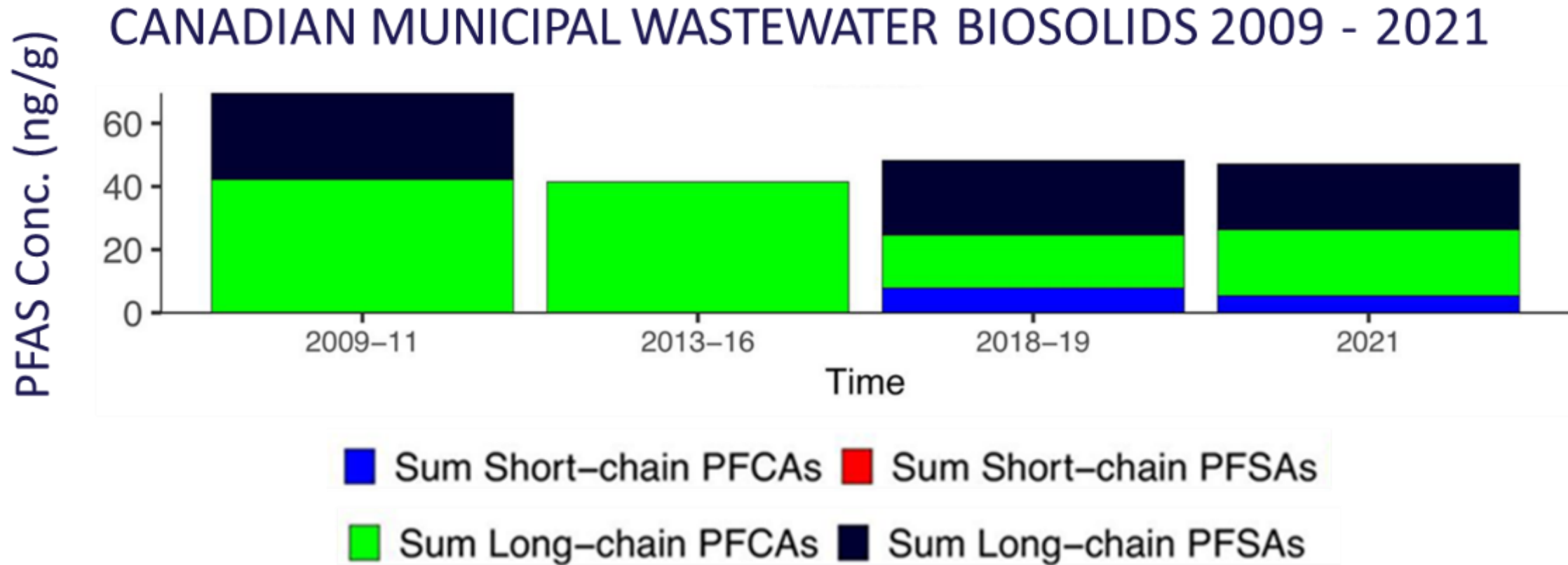
U.S. EPA Chain Length Categories

# of Carbons	4	5	6	7	8	9	10	11	12
PFCAs	Short-chain PFCAs				Long-chain PFCAs				
	PFBA	PFPeA	PFHxA	PFHpA	PFOA	PFNA	PFDA	PFUnA	PFDoA
PFSAs	PFBS	PFPeS	PFHxS	PFHpS	PFOS	PFNS	PFDS	PFUnS	PFDoS
	Short-chain PFSAs		Long-chain PFSAs						

- Precursor example: Fluorotelomer-based substitutes ≤ C6 example replacing C8



PFAS Occurrence in biosolids over time



- Except for PFOS, long- chain PFAS generally decreased over time.
- PFOS did not change with time indicating slow response to phase-outs/regulations and PFOS' overall stickiness.

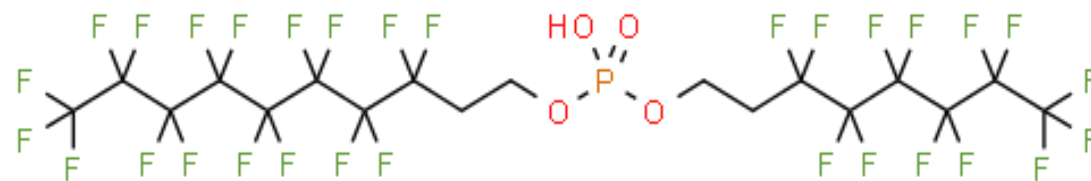
USA EXAMPLE: PFAS IN 2018-2025 BIOSOLIDS (PPB DRY WEIGHT)

	PFAS	~Min	~Max
Perfluoroalkyl acids (PFAAs) -terminal metabolites	PFOS	< LOD	130
	PFHxS	< LOD	50
	PFOA	< LOD	30
	PFHxA	< LOD	30
PFAA precursors/ intermediates	6:2 FTS	< LOD	15
	8:2 FTS	< LOD	30
	MeFOSAA	< LOD	100
	EtFOSAA	< LOD	50
PFAA intermediates	7:3 FTCA	< LOD	100
	5:3 FTCA	< LOD	220
	3:3 FTCA	< LOD	220

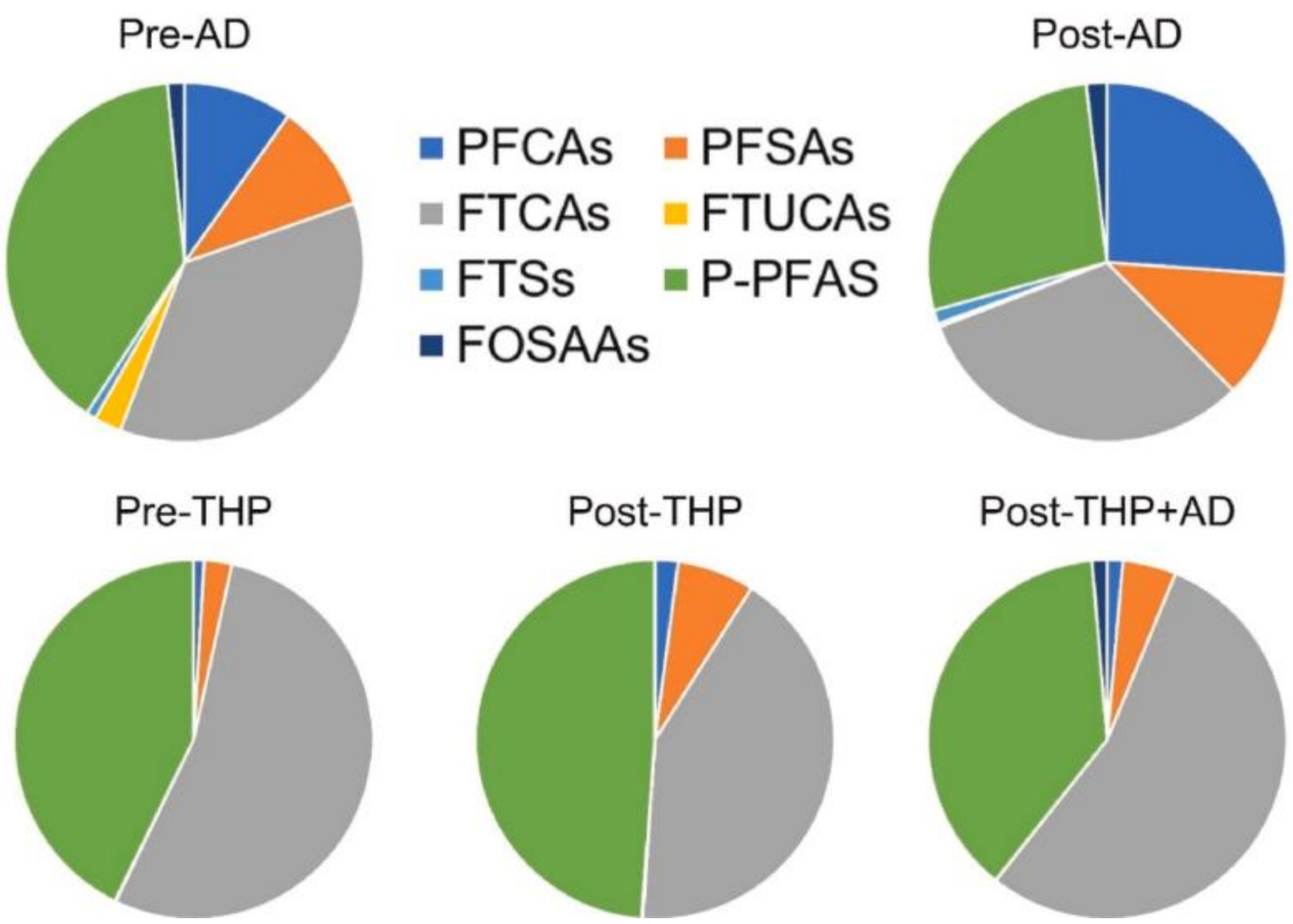
Some Additional Precursors

PFAS	~Min	~Max
6:2 diPAP	10	400
8:2 diPAP	0	200
6:2 PAP	20	340
6:6 PFPI	0	9
6:8 PFPI	0	4.4

- For most utilities, identified precursors are typically at least 75% of the quantifiable PFAS fluorine mole balance



PFAS Transformation Example in Solids Processing to Class B Biosolids



AD: Anaerobic Digestion

- Bulk of the PFAS are precursors and most not measured in the current EPA 40 targets list
- Digestion to remove pathogens, etc. leads to PFAS conversion to PFAAs (orange and blue)

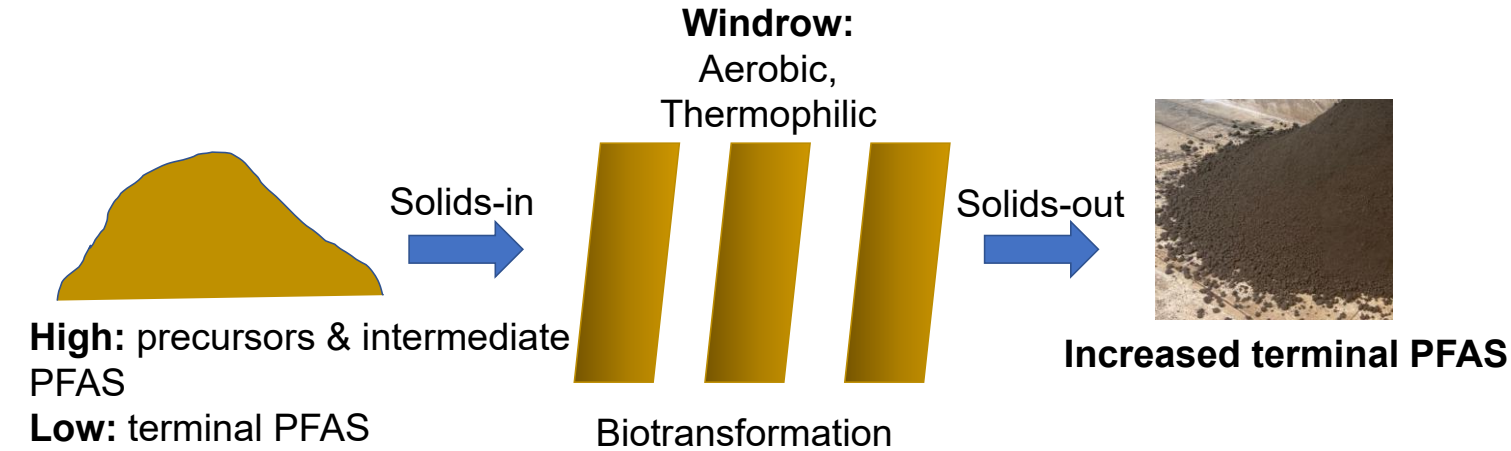
THP + AD

- THP prior to AD reduced/slowed transformation to PFAAs leaving a larger fraction of FTCA intermediates in the final biosolids.

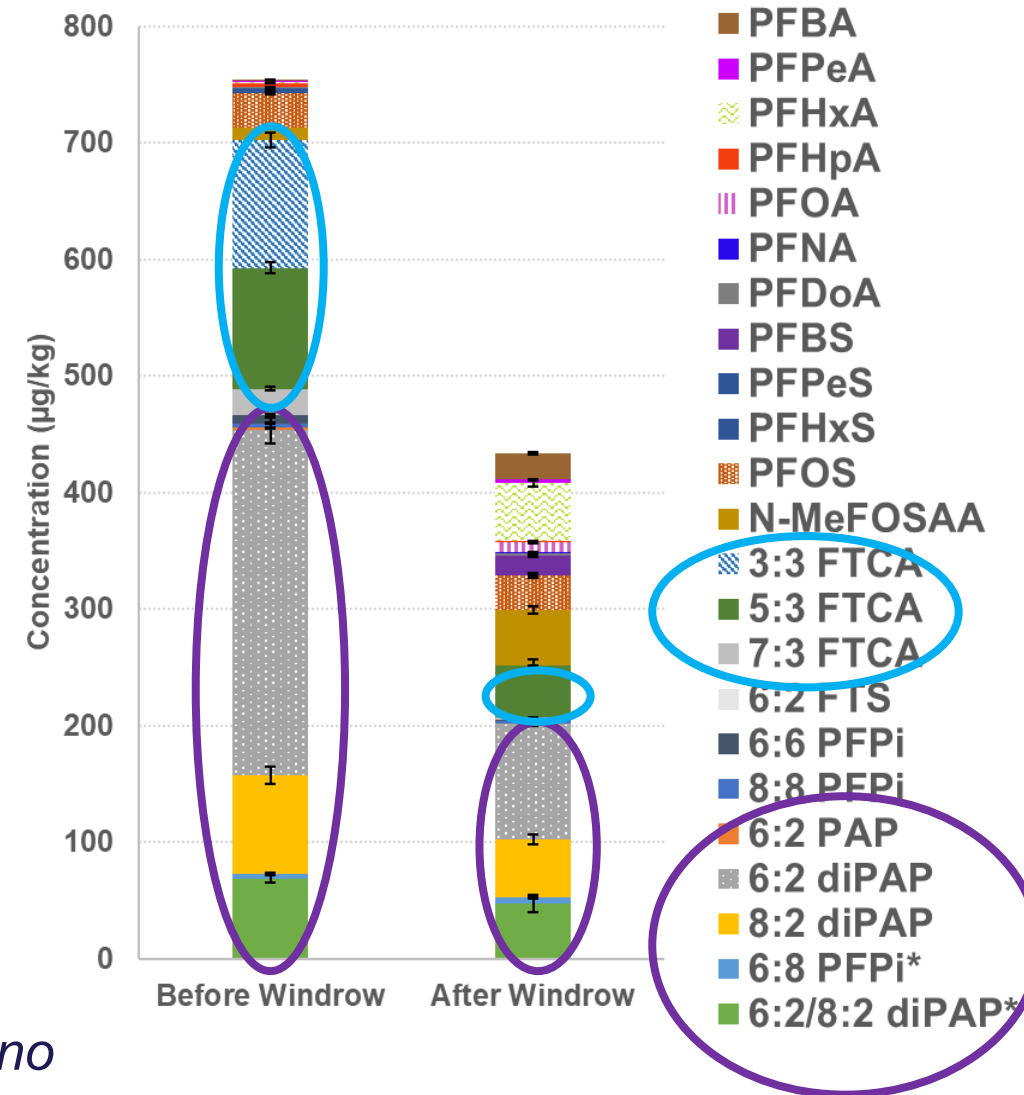
PFAS Transformation in Solids Processing to Biosolids for Application

Solids are from a Municipal WRRF that uses THP + AD Treatment

Windrow Composting

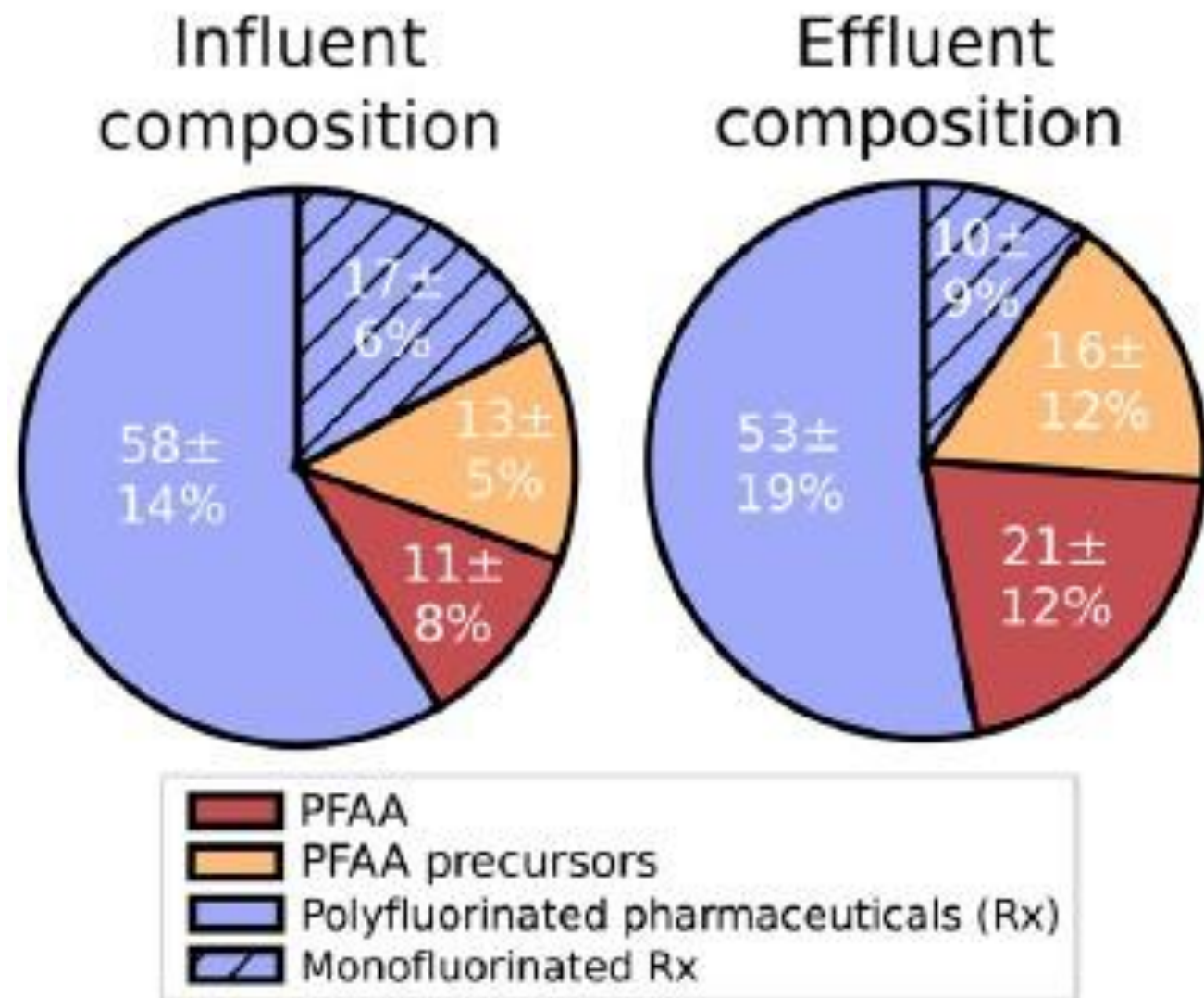


- Overall, moles of PFAS detected decreased by > 40% with windrow composting
- FT precursors, intermediates, and even terminal PFAS decreased after windrow composting
- Possible reasons
 - Expected biotransformation, yes
 - Volatilization, highly likely
 - Heat + mineral-induced oxidation? *Initial data suggests no*

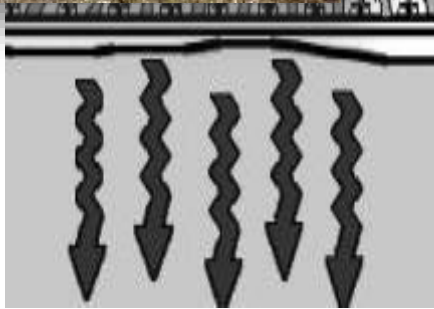


WHAT ABOUT TOTAL ORGANO-FLUORINE SOURCES

- Only about 25% may be PFAS
- Fluorinated pharmaceuticals make up the bulk of organofluorine in wastewater (snapshot from 8 facilities)
- Total organo-fluorine does not change much between influent and effluent



COC FATE AFTER DISCHARGE/APPLICATION TO LAND



Chemical Properties

Soil Properties

**Air-water interface
accumulation (PFAS)**

**Solid Phase
adsorption**

Biotransformation

Plant Uptake

Runoff

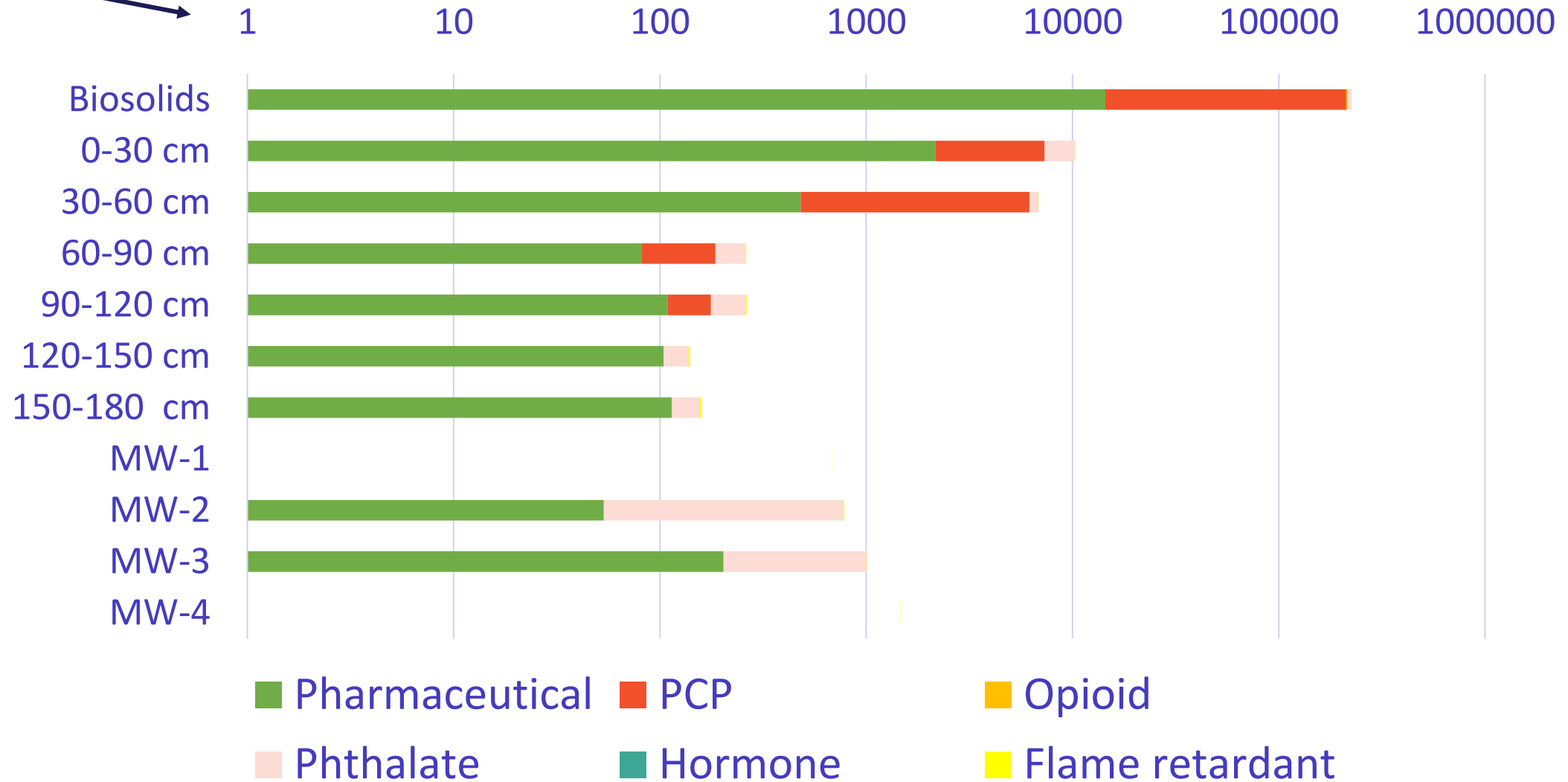
Leaching

Tile-drain discharge

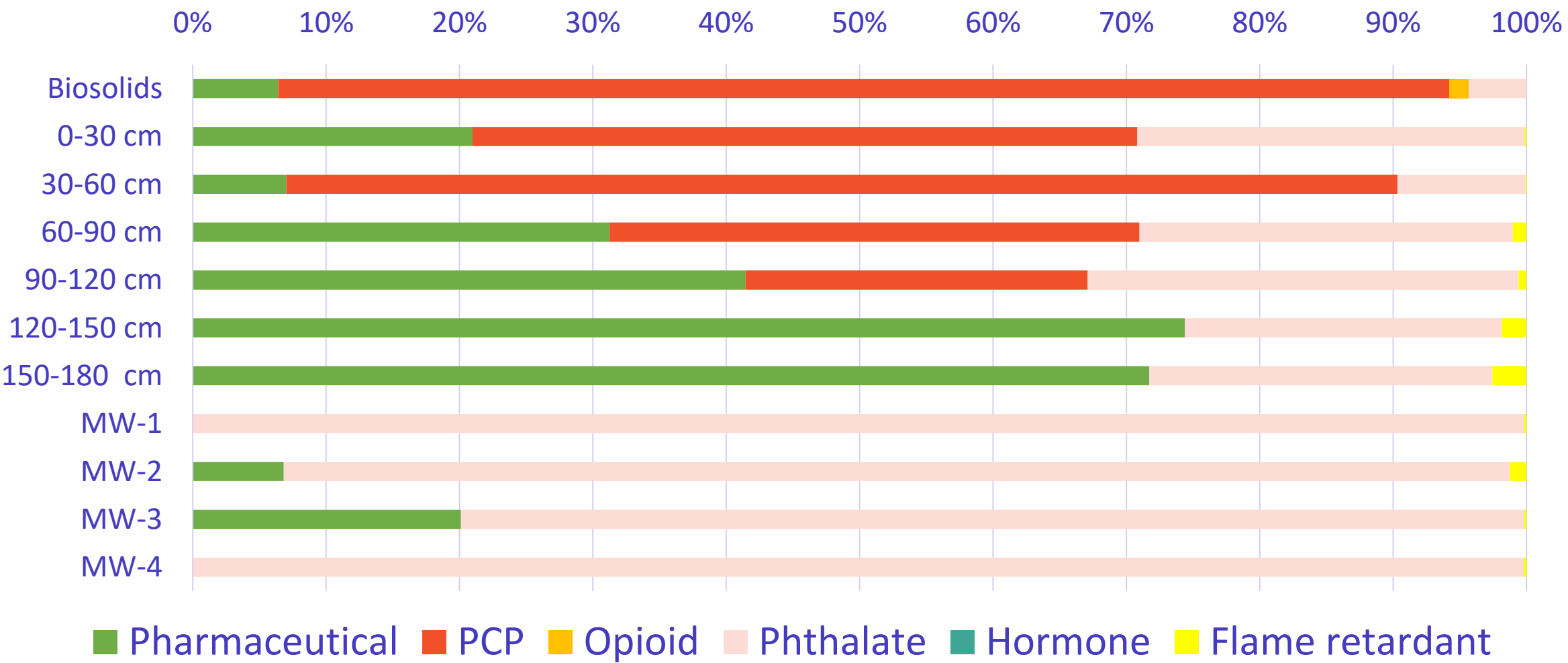
40-Y DEDICATED LAND DISPOSAL SITE (WESTERN USA): NON-PFAS COCS

Log scale

Concentrations (ppb for solids, ppt for Water)



40-Y DEDICATED LAND DISPOSAL SITE (WESTERN USA): NON-PFAS COCS



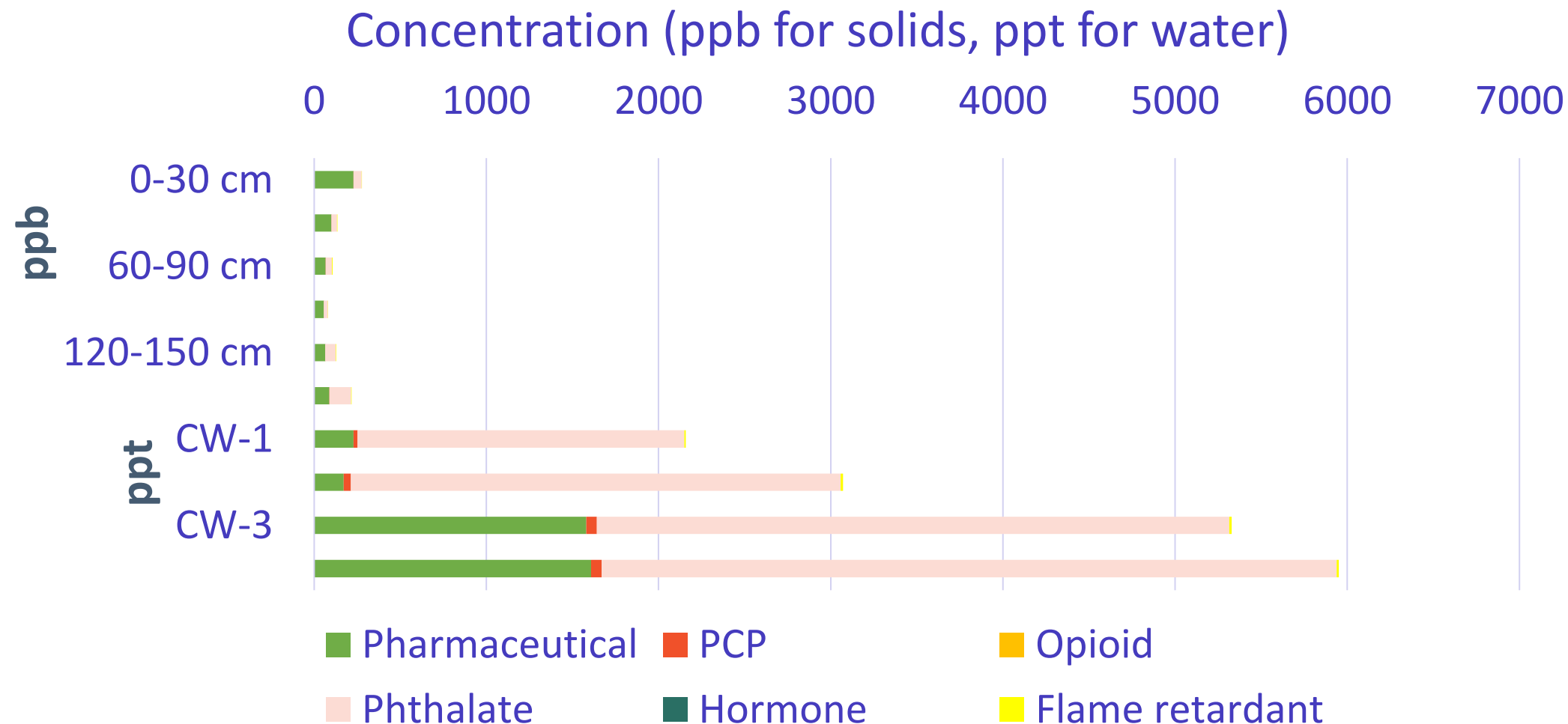


Non-PFAS Mobility at DLD

Monitoring Well Samples

		water W1 (ng/l)	water W2 (ng/l)	water W3 (ng/l)	water W4 (ng/l)	Present in soil	Present in biosolids
Pharmaceuticals	Salicylic acid	n.d.	n.d.	75	n.d.		
	Sebacic acid	n.d.	32	39	n.d.		
	Azelaic acid	n.d.	21	89	n.d.		
PCPs	DEET	18	67	47	21		
	Tributyl phosphate	n.d.	7.9	1.9	4.8		
Phthalates	Benzyl Butyl phthalate	227	297	316	415		
	Di(2-ethylhexyl) phthalate	96	n.d.	155	352		
	Dibutyl phthalates	178	211	168	348		
	Diisodecyl phthalate	10	n.d.	38	22		
Flame retardants	Triphenyl phosphate	1.0	9.8	1.6	3.0		
	Tris(2-butoxyethyl) phosphate	457	434	600	633		
	Total	987	1080	1530	1800		

CONTROL SITE: NON-PFAS COCS



- Pharmaceuticals include some natural occurring ones in nature)
- PCP includes DEET

Persistence of Non-PFAS COCs one year post biosolids application, dry farm

■ Pharmaceuticals

■ PCPs

■ Opioids

■ Phthalates

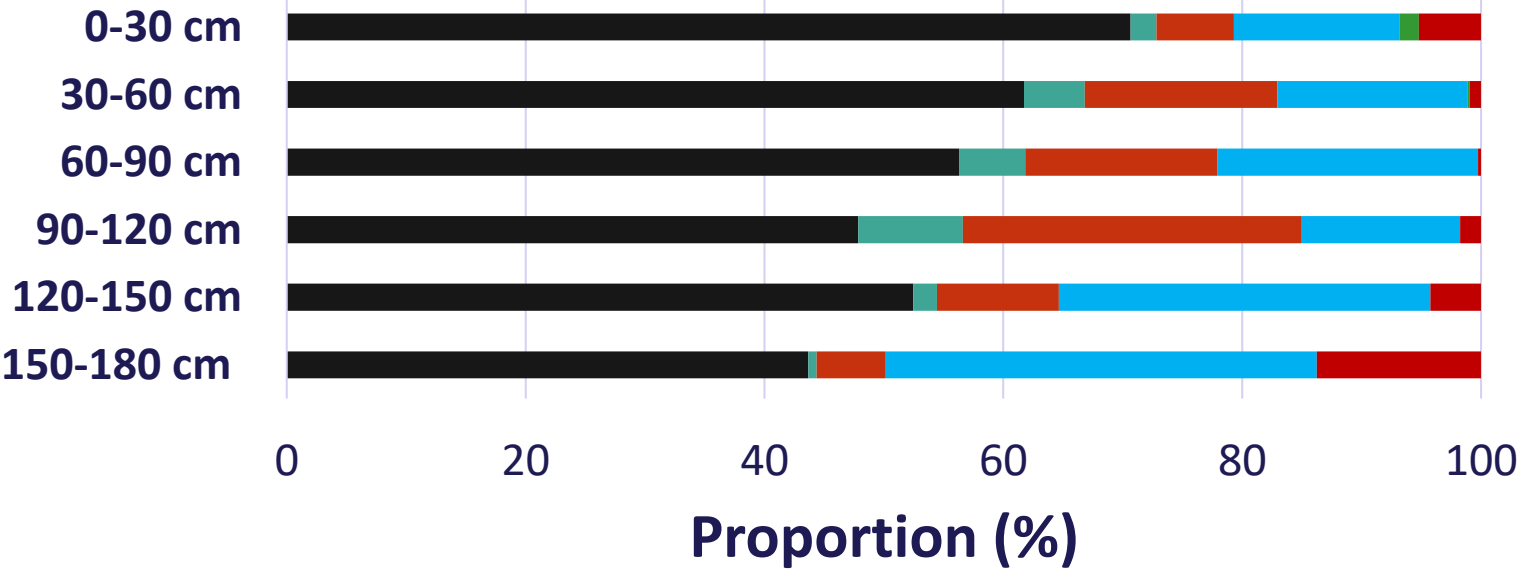
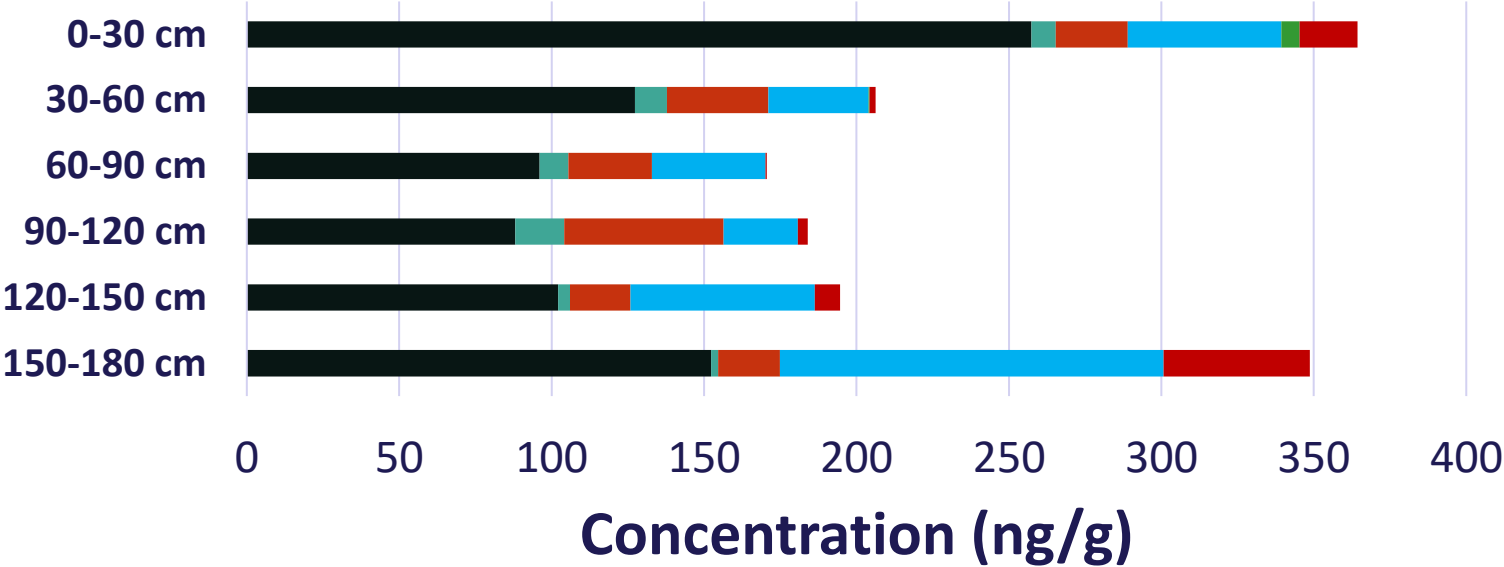
■ Sweeteners

■ Cannabinoids

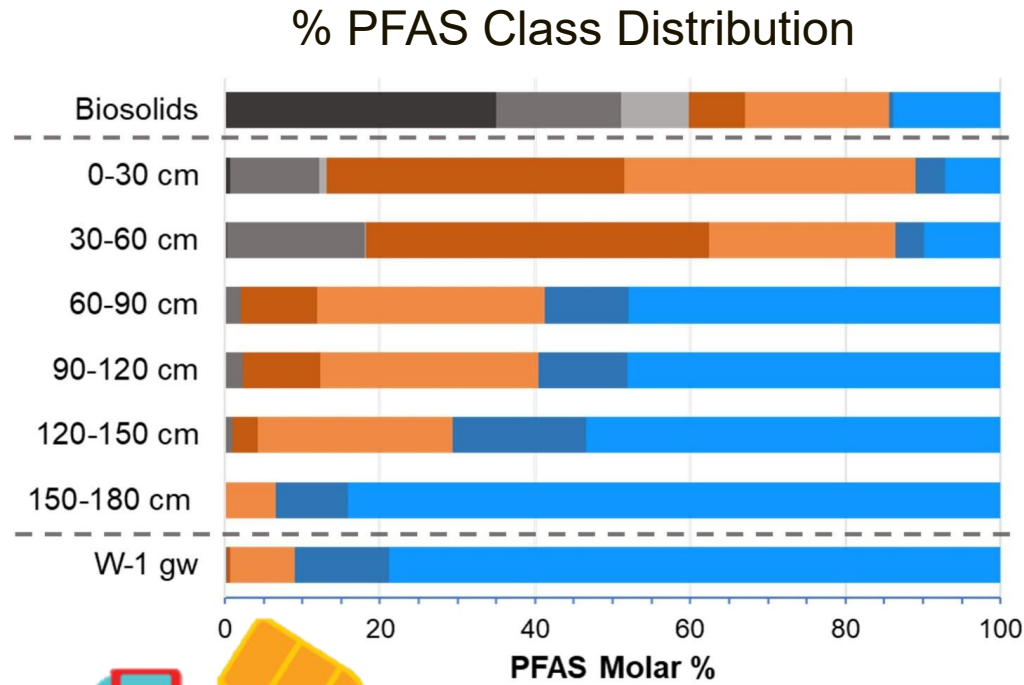
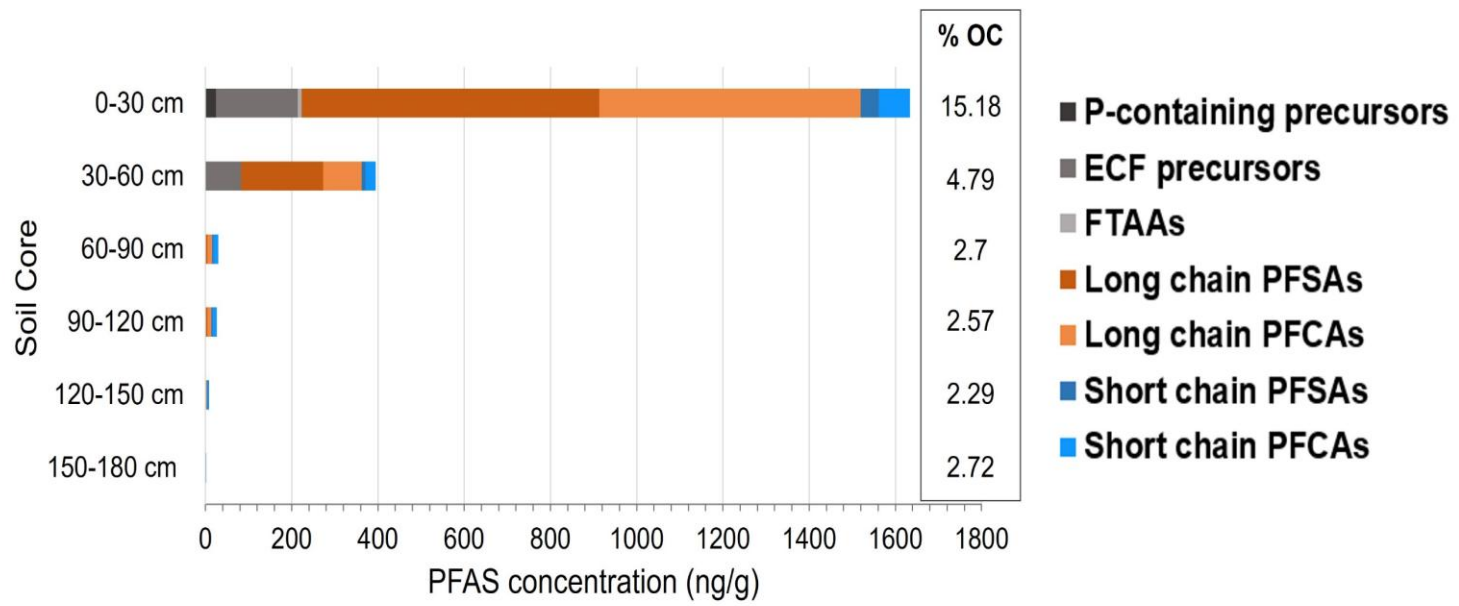
■ Hormones

■ Flame retardants

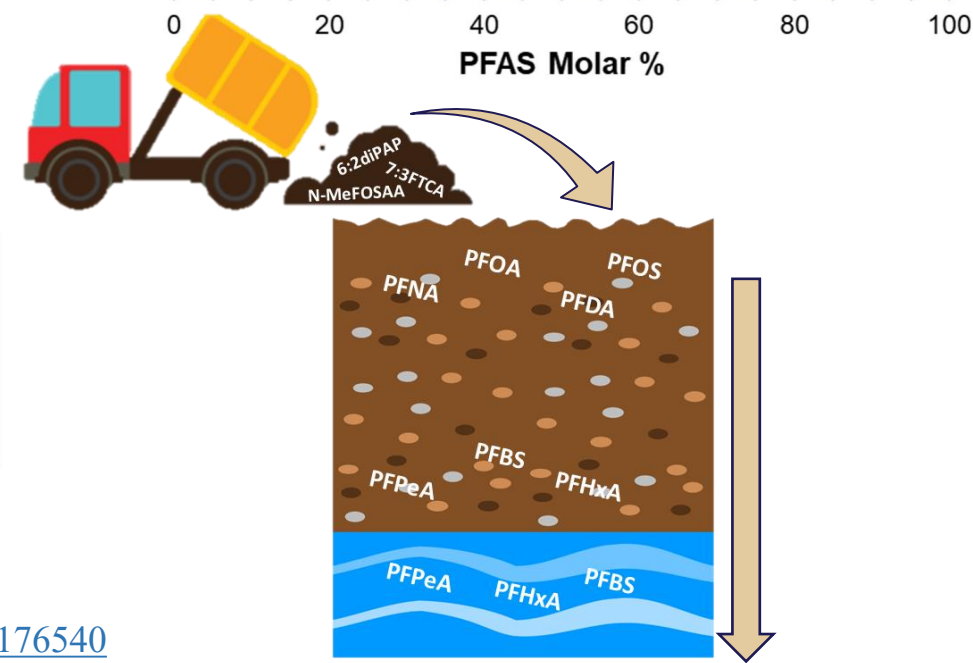
■ Rubber derivatives



40-Y DEDICATED LAND DISPOSAL SITE (WESTERN USA)

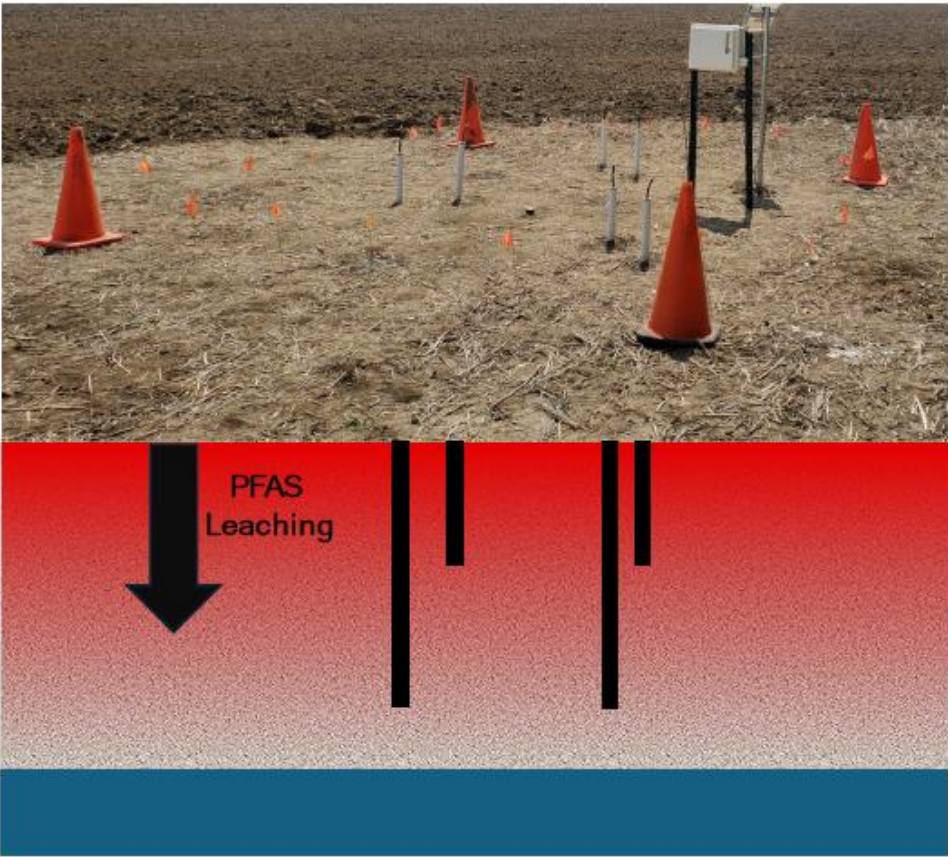
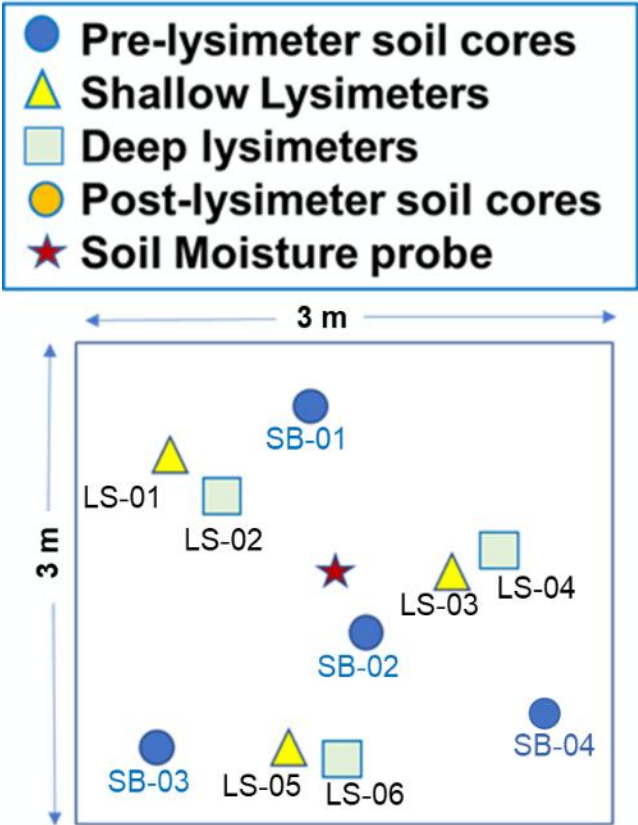
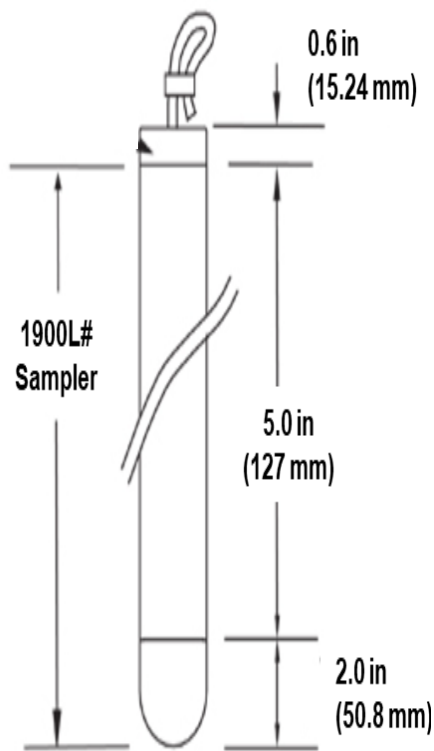


- Repetitive (annual) and high rate of biosolids application leads to PFAS and organic carbon (% OC) accumulation in soil over time
- Most precursors breakdown to PFAAs within 1 year
- Long-chain PFAS retained in the upper soil profile
- Short-chain PFAAs dominate what is getting to groundwater
- Interestingly, only PFOA exceed new EPA MCLs



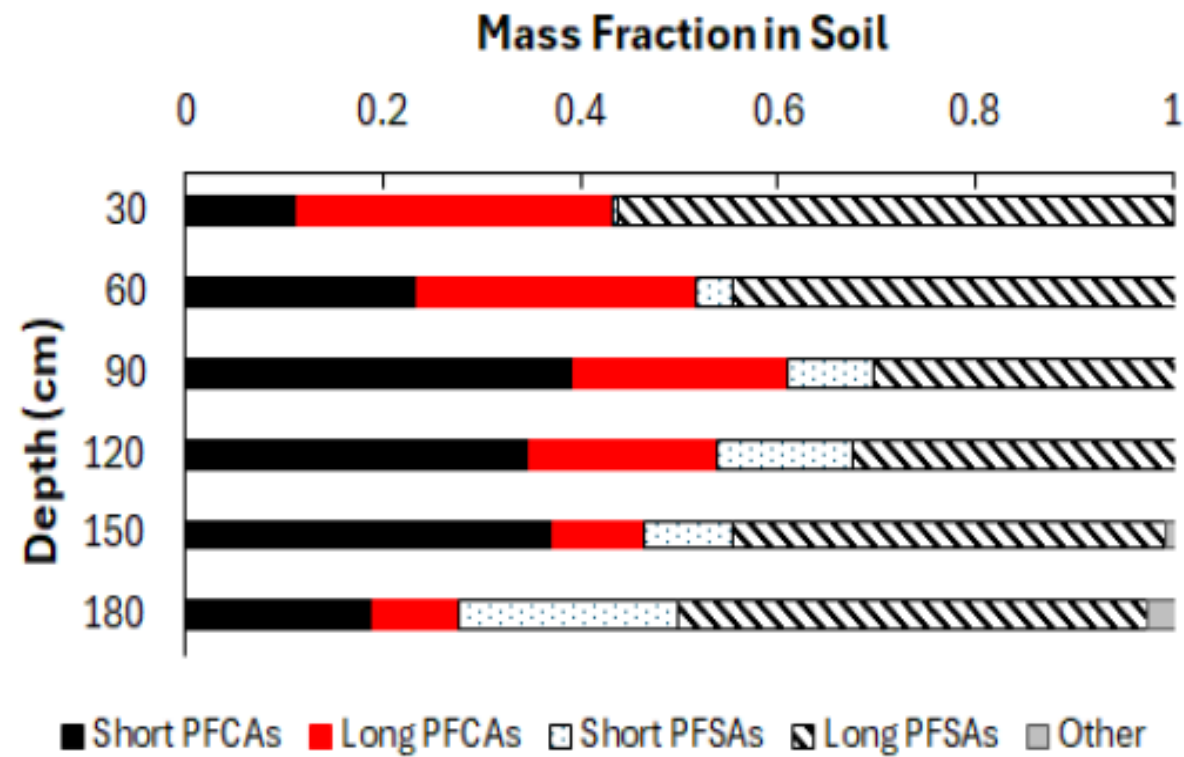
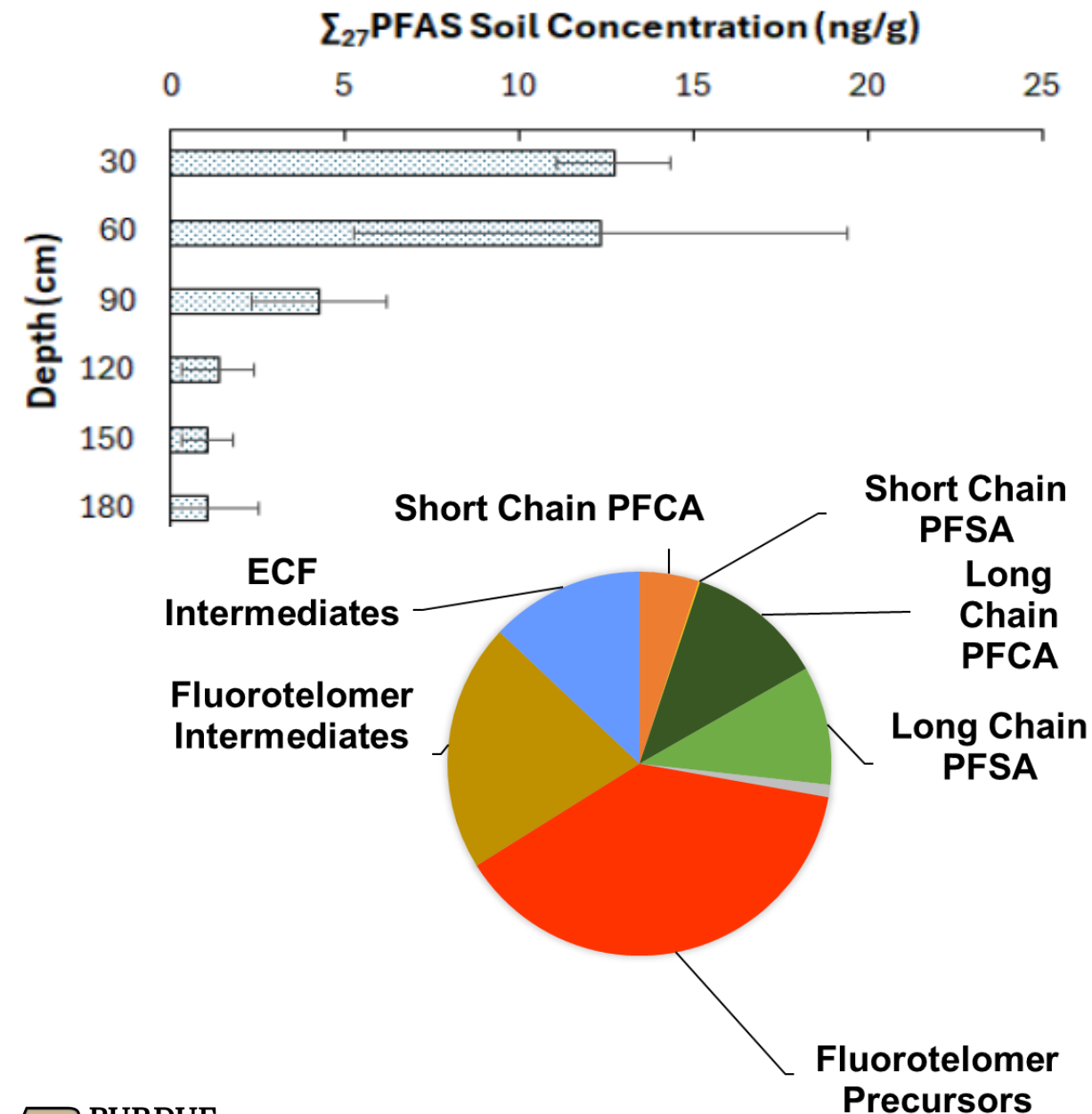
BIOSOLID-APPLIED MIDWEST AGRICULTURAL SITE

- Agricultural Field - ~40 y of land-application
- Agronomic rate for corn biannually, last app 2022
- Crops cleared so transpiration not a factor
- Depth to water table: 4.3 m (*pre-installation river flooding*,
- 3-mo study with 2 ft & 4-ft suction cup lysimeters
- 58 PFAS targeted



Depth (cm)	% OC	Type, % Clay
0-60	2	Loam, 15% clay
60-110	2.3	Silt loam, 20%
110-180	< 1.5	Loam, 17%

40-Y BIOSOLIDS-APPLIED MIDWEST AGRICULTURAL SITE: PFAS IN SOILS & BIOSOLIDS

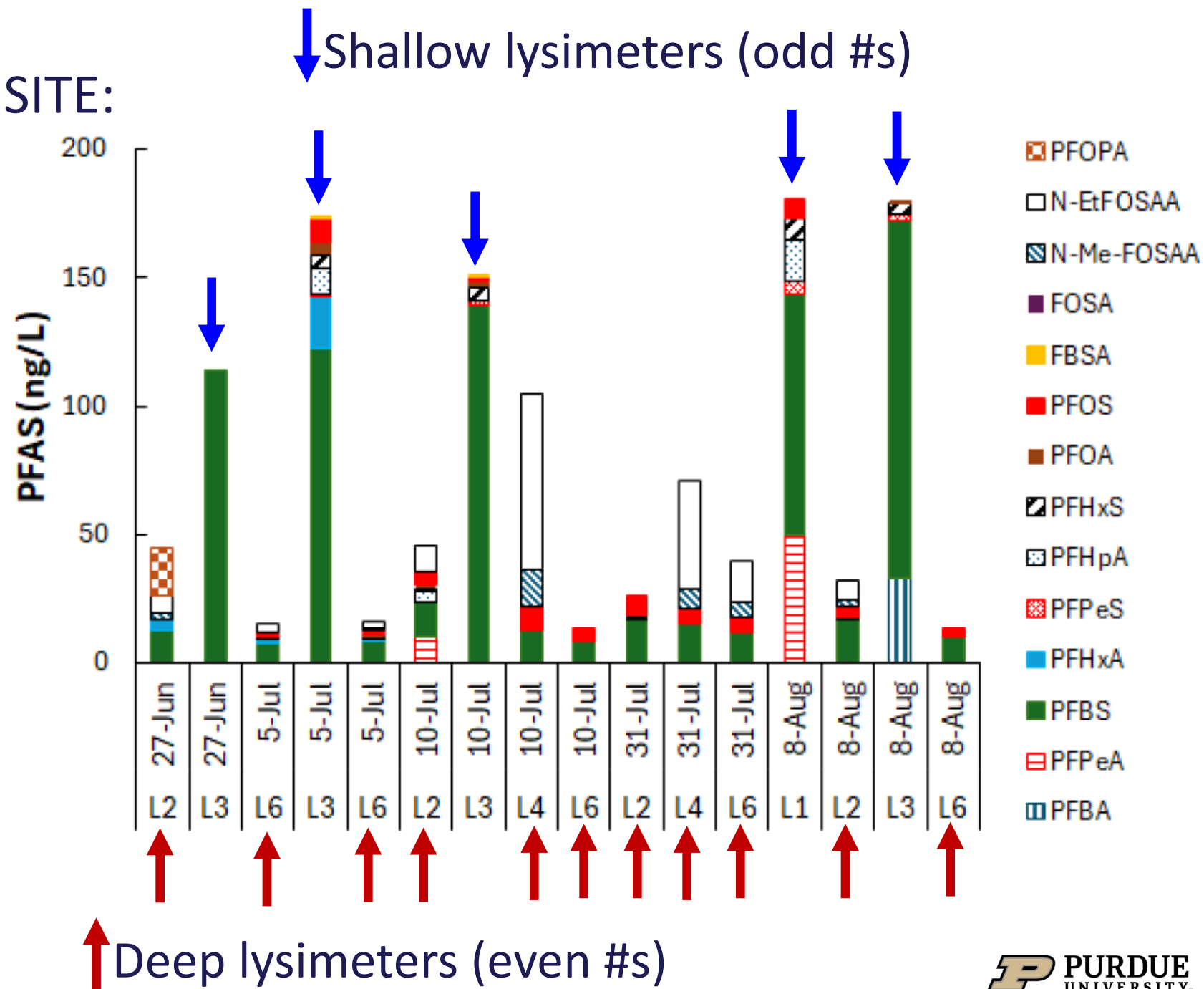
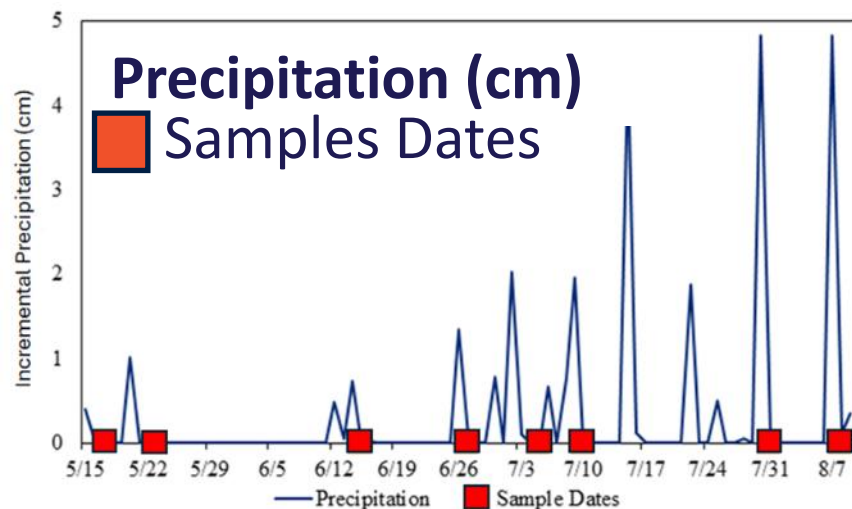


- Precursors degrade, most < 1-y post application (not ECFs)
- Long-chain PFAS retained in the upper soil profile

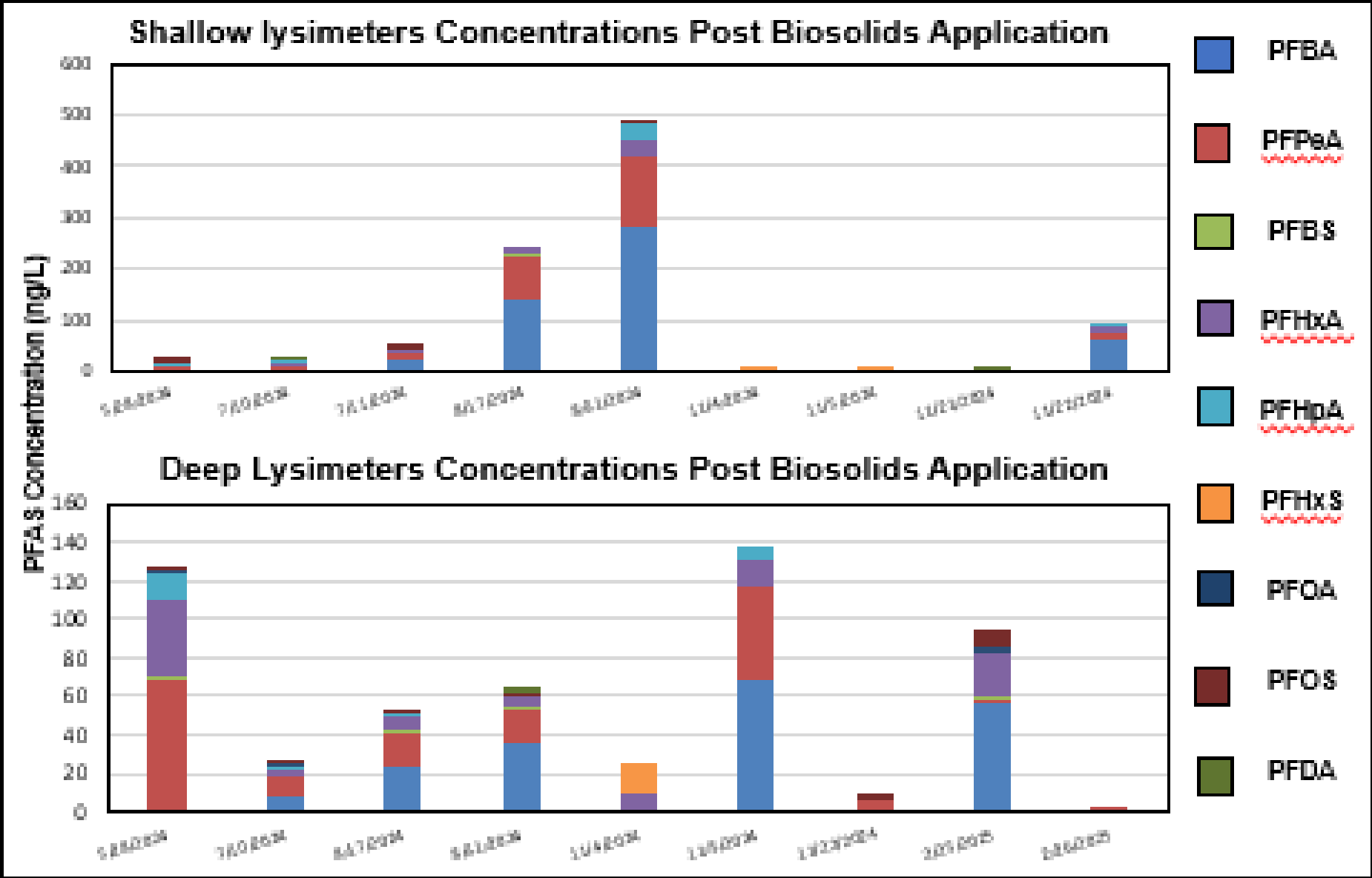
WRF 5214 (CDM Smith, Purdue)



40-Y BIOSOLIDS-APPLIED MIDWEST AGRICULTURAL SITE: LEACHING TRENDS

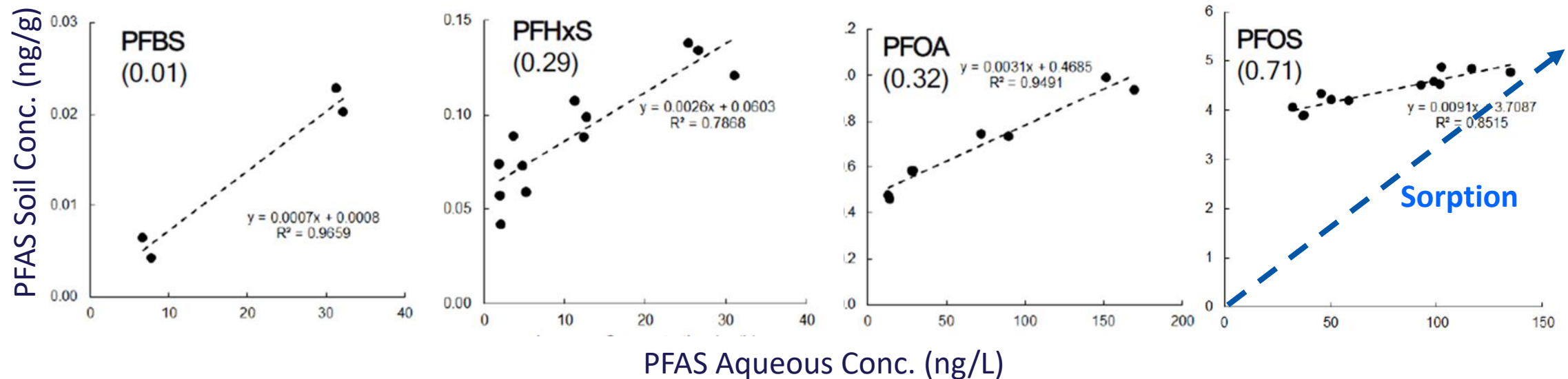


BIOSOLIDS-APPLIED MIDWEST AGRICULTURAL SITE: FIRST TIME APPLICATION LEACHING TRENDS



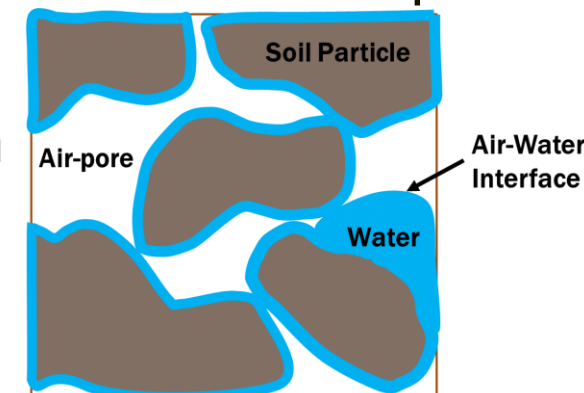
SOIL FACTORS IMPACTING TRANSPORT: DESORPTION HYSTERESIS

Desorption Isotherms from Historic Biosolids-applied soil

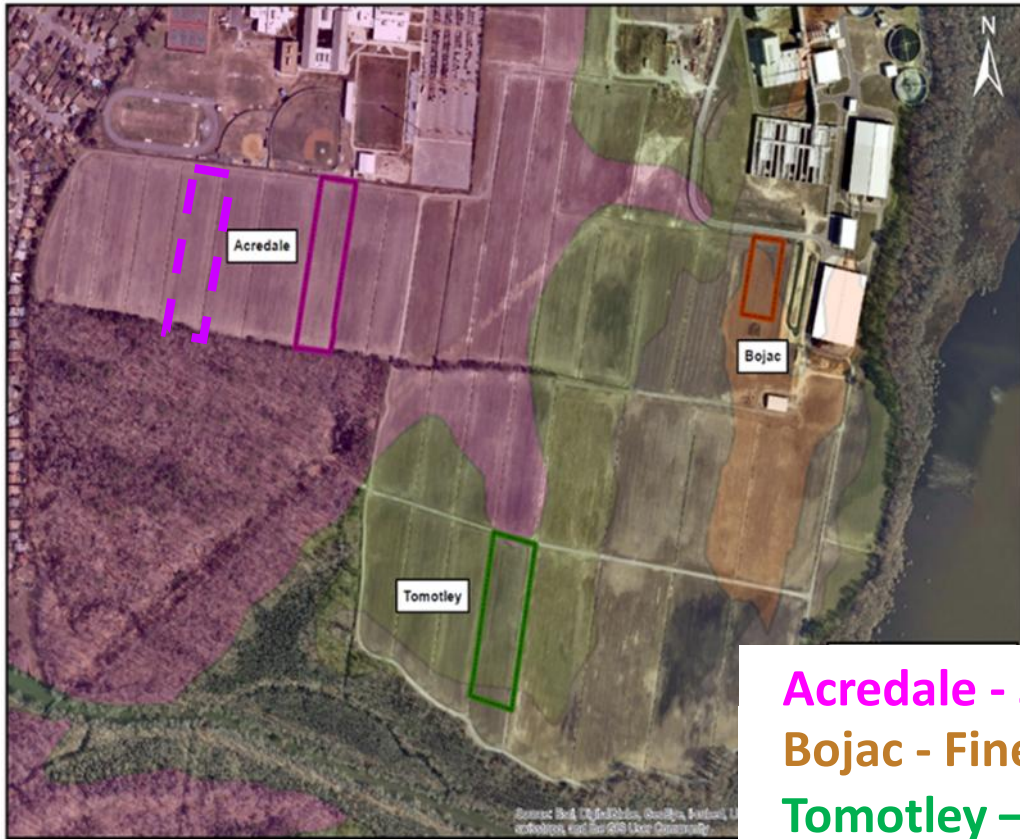
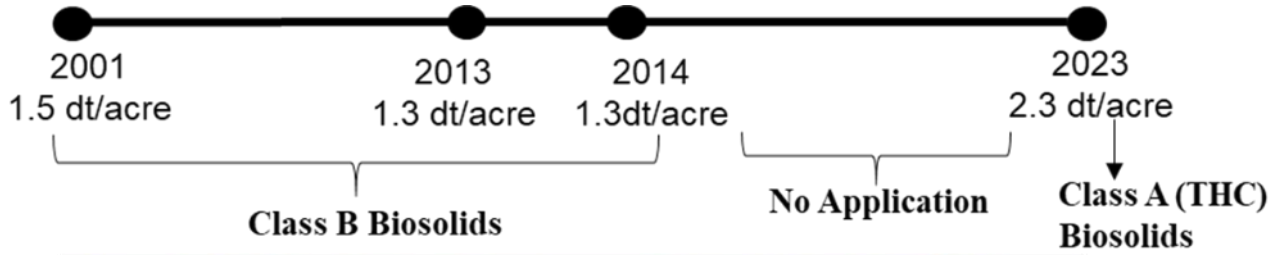


Desorption hysteresis - chemical release (desorption) does not mirror chemical adsorption

- Desorption hysteresis – increases with increasing chain length
- Air-water interfacial retention – increases with increasing chain length
 - Contributes to longevity of long-chain PFAS in the soil profile
 - Decreases movement to groundwater

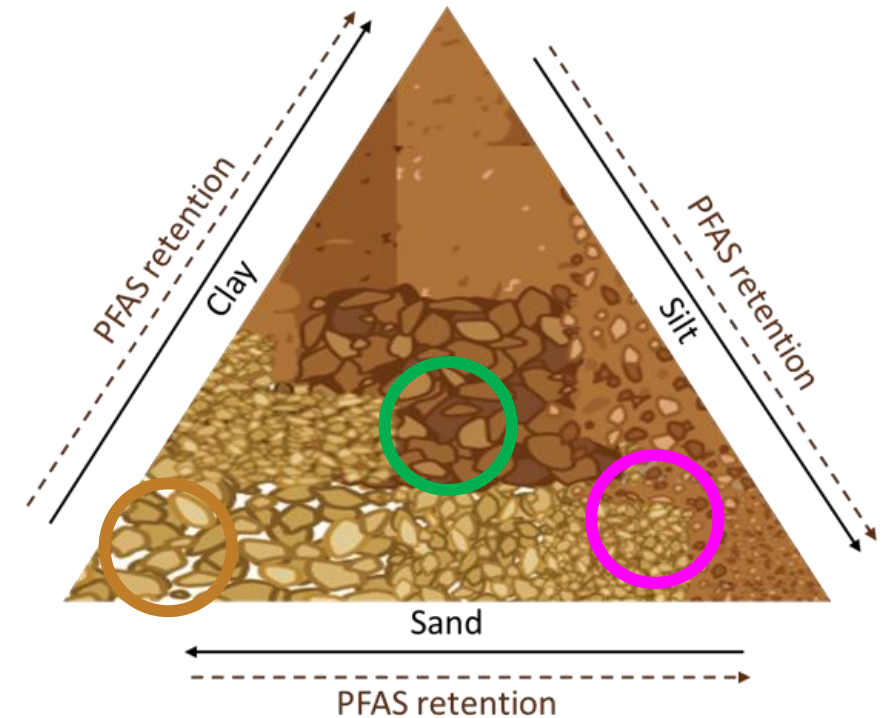


EAST COAST USA STUDY SITE



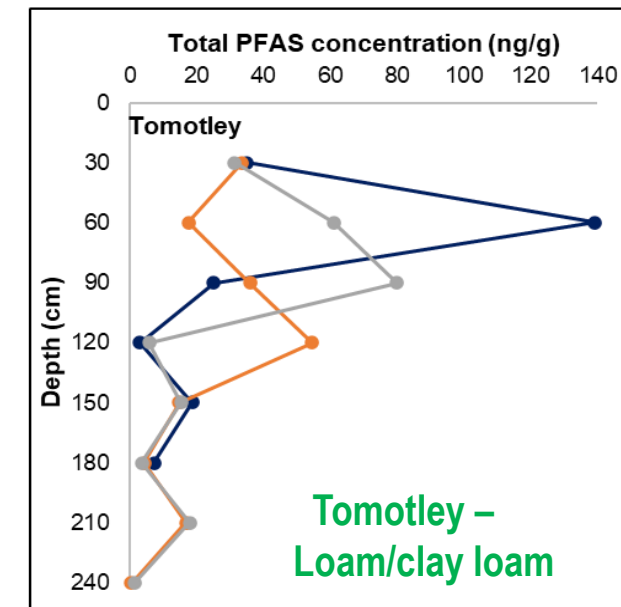
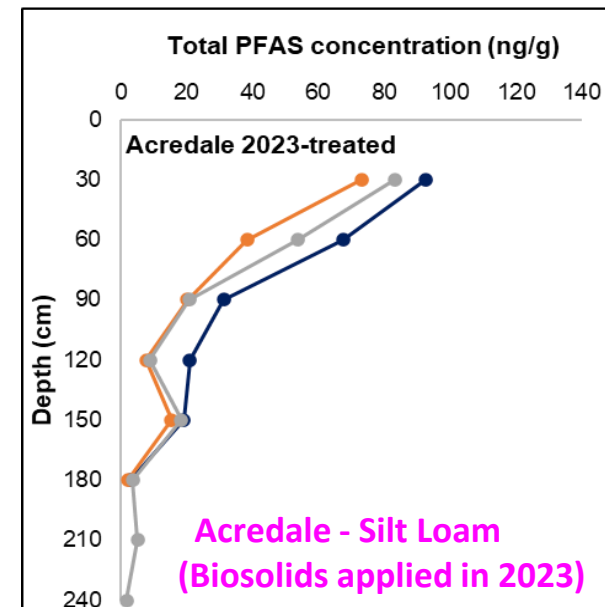
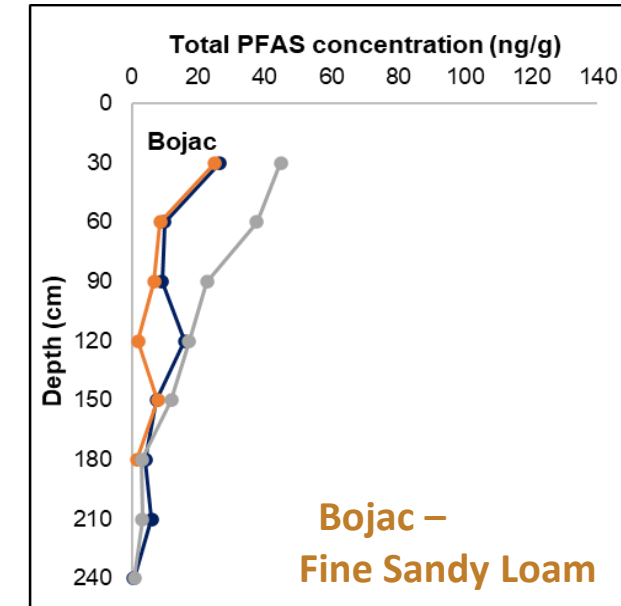
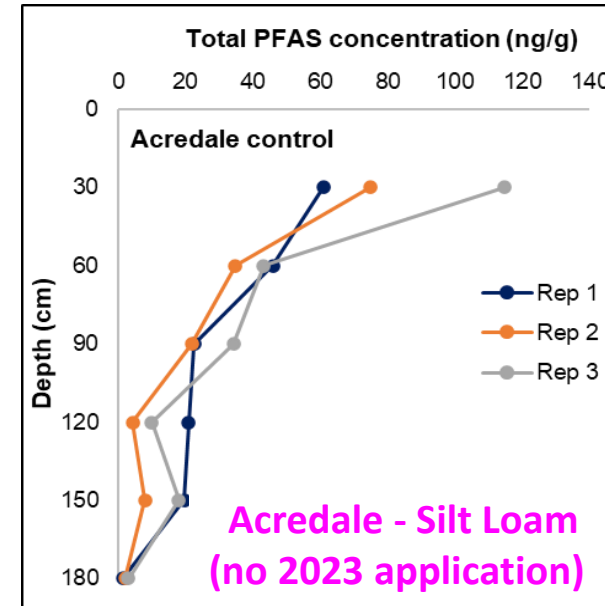
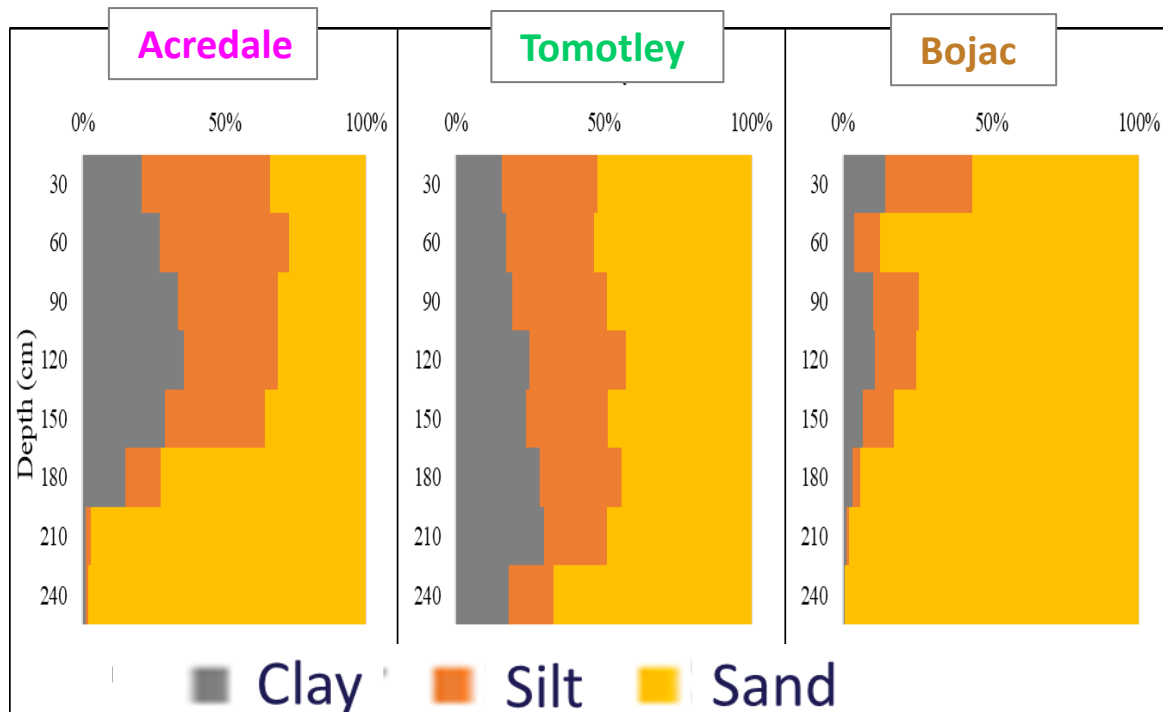
Acredale - Silt Loam
Bojac - Fine Sandy Loam
Tomotley – Loam/Clay loam

- Corn/soybean rotations
- Municipal biosolids: Class B to Class A
- 3 soil types
- Organic carbon ~ 2% in surface soils decreased with depth, Lower average in Bojac Sandy loam



EAST COAST USA STUDY SITE

- PFAS profiles in cores taken prior to 2023 application were not statistically different
- PFAS longevity
- Sandy soil – less retention
- Finer texture soil, greater retention



EAST COAST USA STUDY SITE

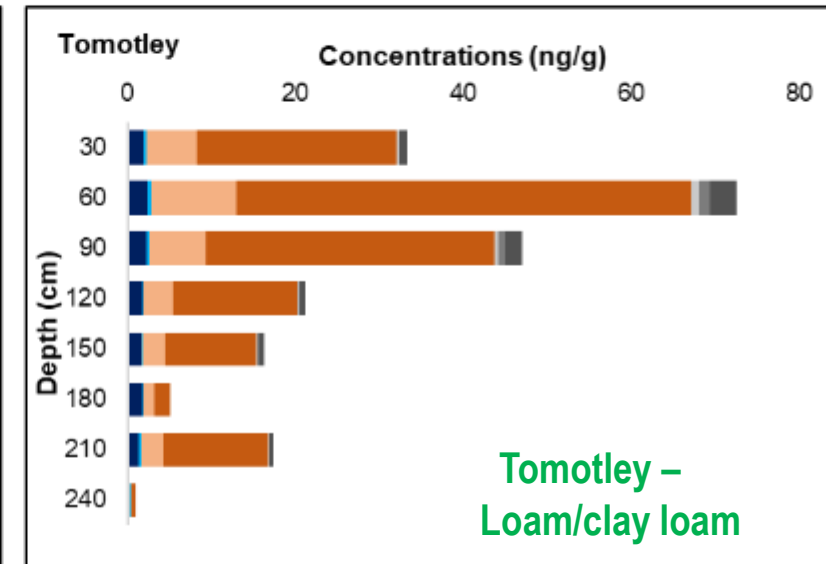
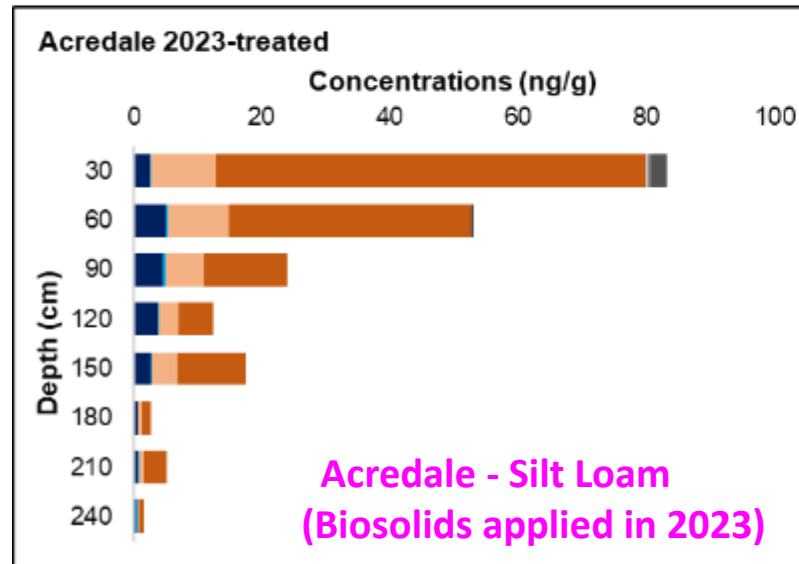
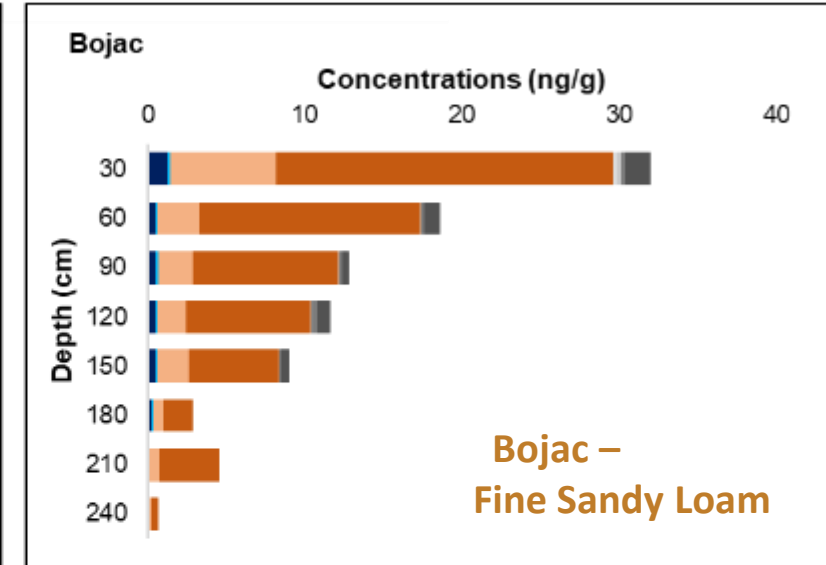
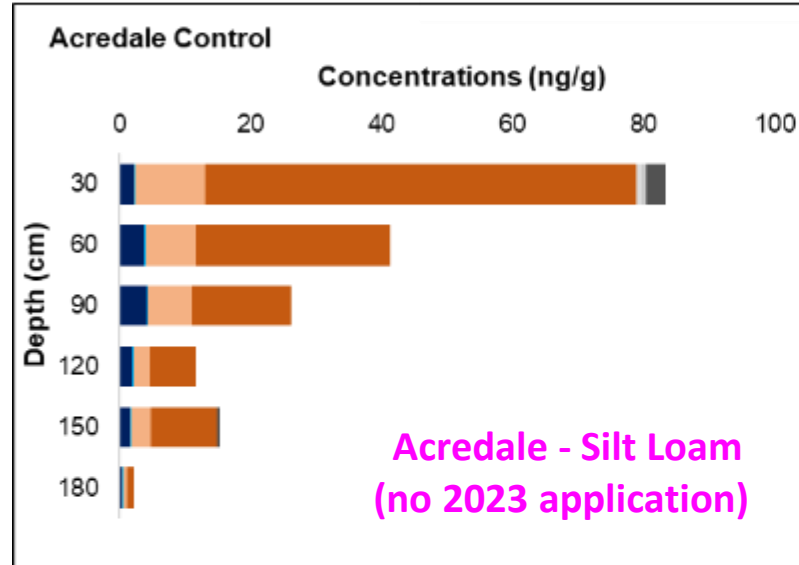


- Long-chain PFAS dominated the upper soil profile
- PFAS in surface soil subject to runoff and available for uptake
- Less PFAS residing in the soil for the loam and sandy loam

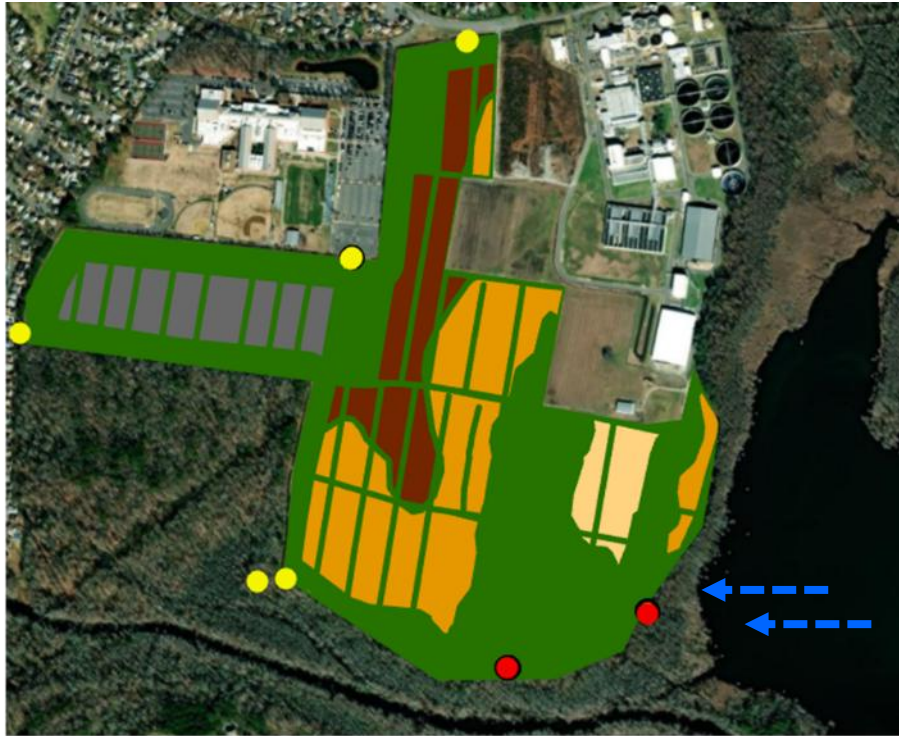
Acredale - Silt Loam

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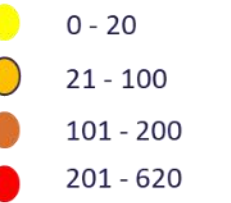


EAST COAST STUDY SITE: OTHER PFAS SOURCES



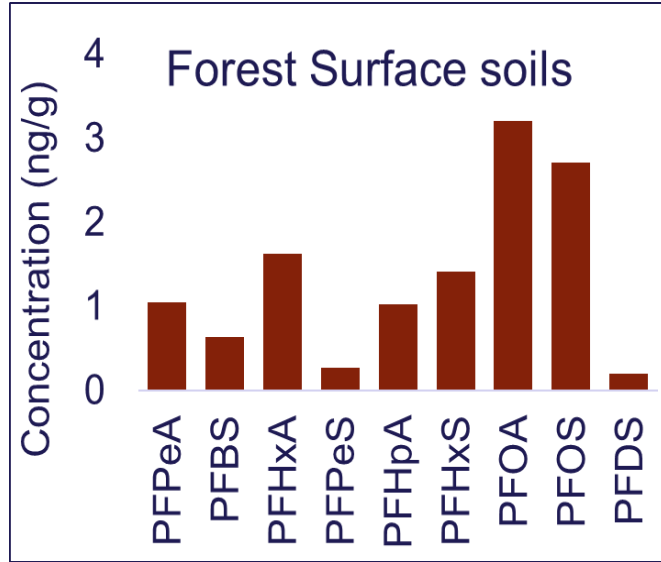
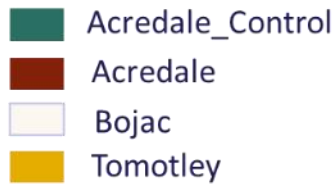
Monitoring Wells

Total PFAS (ng/L)

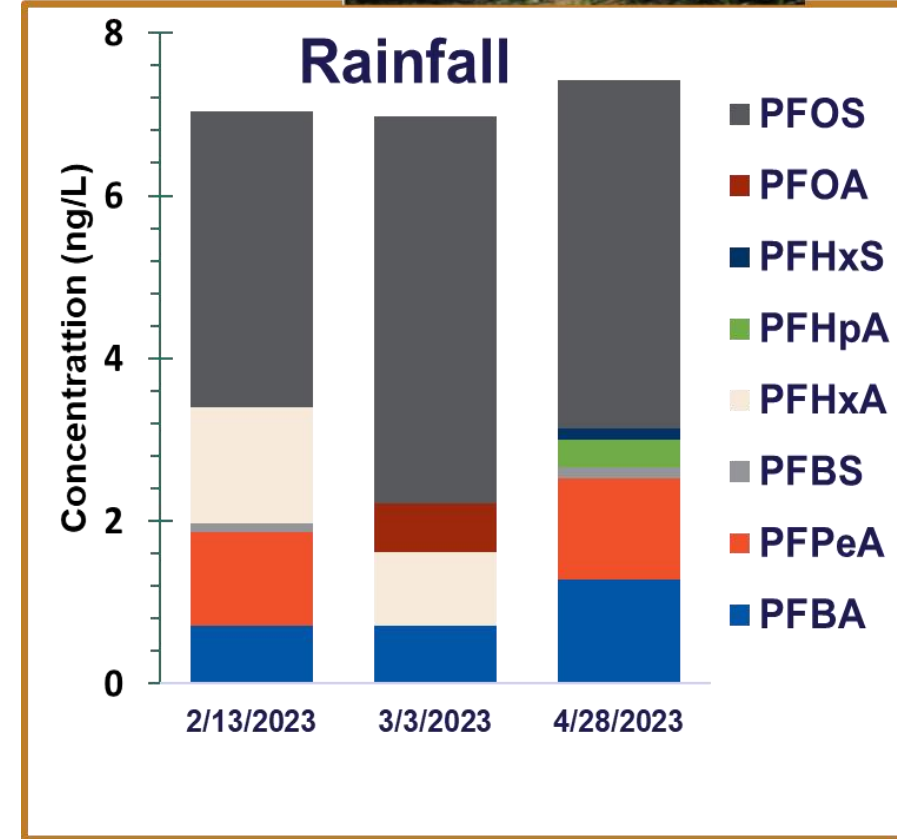


Plots

Soil Type

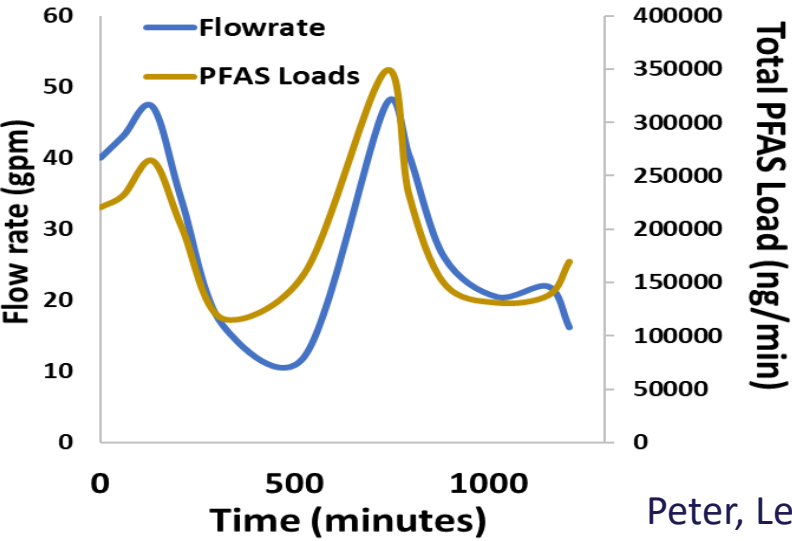


Wet/dry deposition

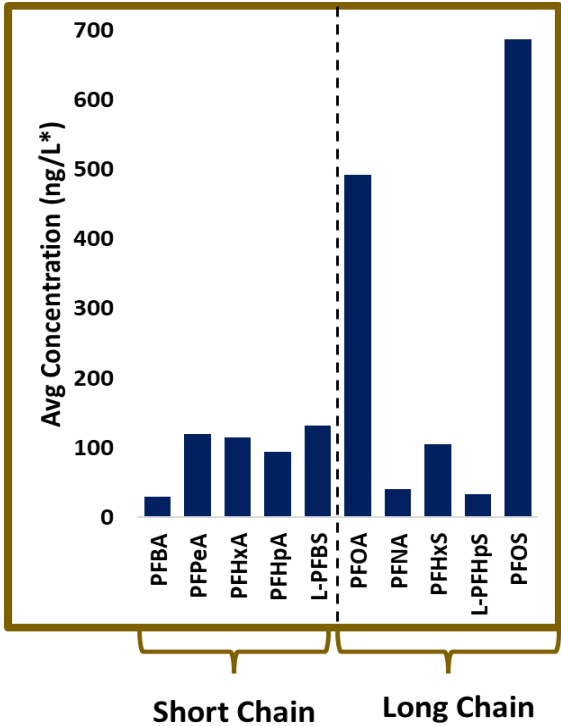


- Assessment complicated near the lake
- Military base, airport within 5-mi radius

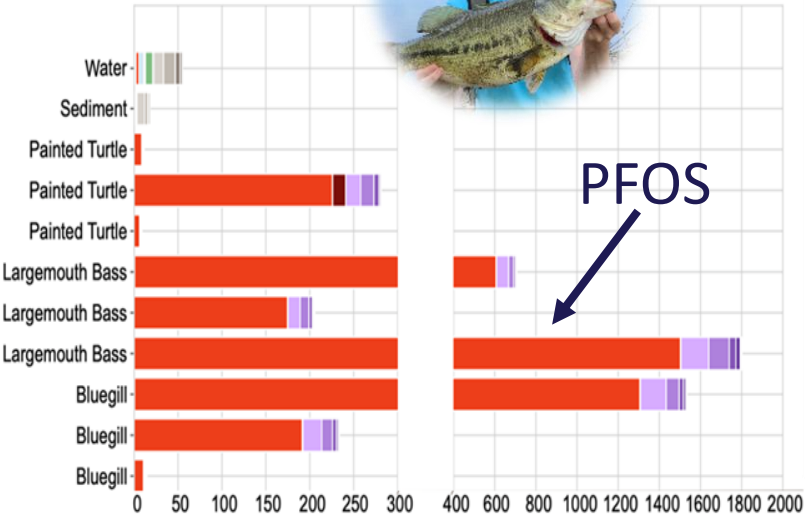
WHAT ABOUT IMPACT OF RUNOFF FROM LAND-APPLICATION?



Peter, Lee et al.



USGS 104B



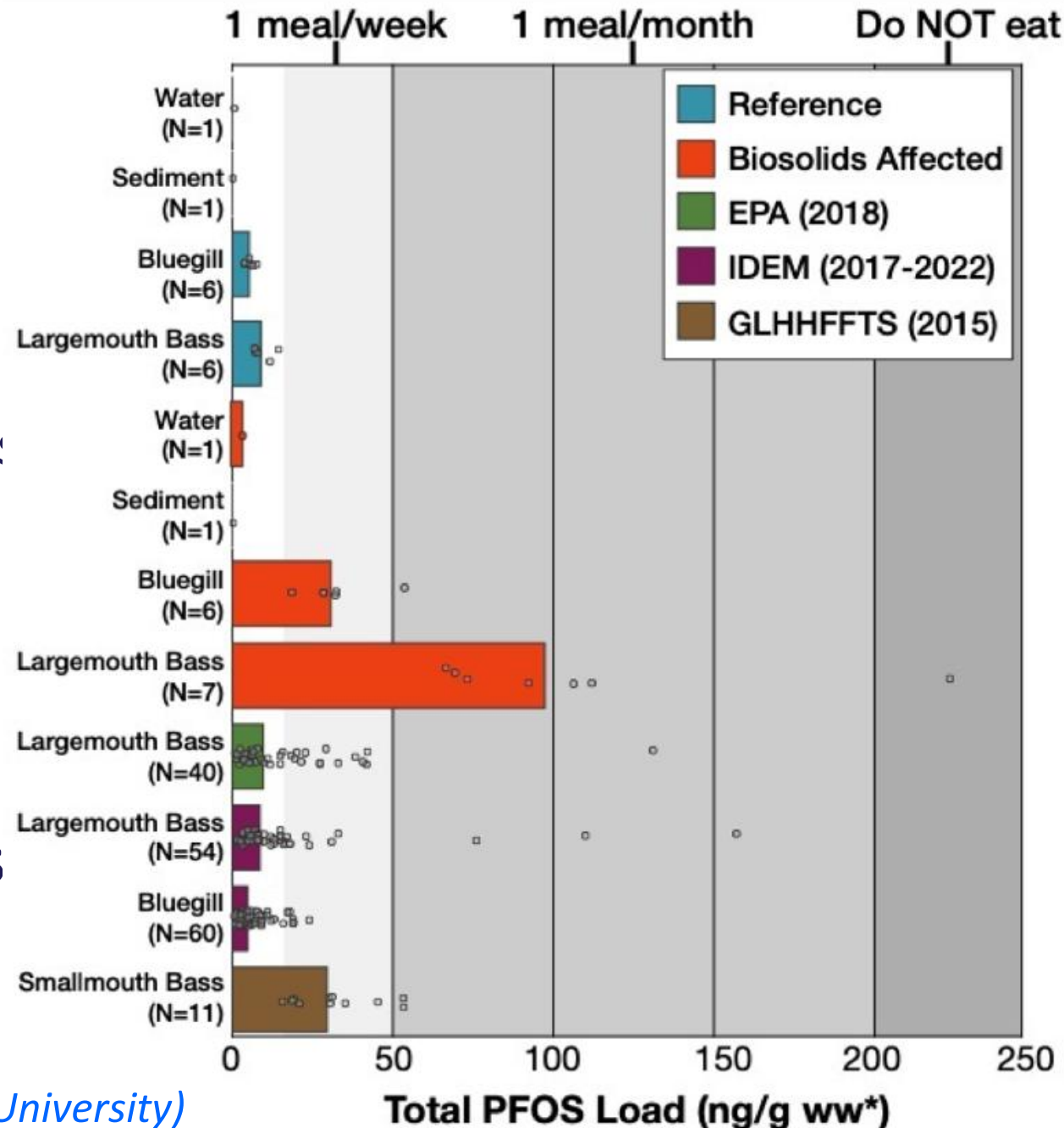
PFAS (ng/g)* Hoskins et al.

With high PFAS retention in surface soils, runoff into streams, wetlands, and farm ponds may be a significant PFAS exposure pathway to fish, wildlife, and grazing cattle, and subsequently, humans

Do not cite, Manuscripts in preparation, Peter, Lee et al. and Hoskins, Sepulveda, et al.

FARM POND FISH MUSCLE FROM BIOSOLID SITE HAD HEAVY PFAS LOADS

- Greater PFOS burdens at **Biosolids Site** than at **Reference Site**
- **Reference site** resembled concentrations in larger monitoring efforts
- Some perspective:
 - At geometric mean for **Biosolids Site**
 - **ONE 8 OZ MEAL OF BASS = 30 DAYS of DRINKING WATER AT 27,840 X HIGHER THAN EPA INTERIM GUIDELINE FOR PFOS**



CROP UPTAKE: PFAS EXPOSURE AND BIOACCUMULATION



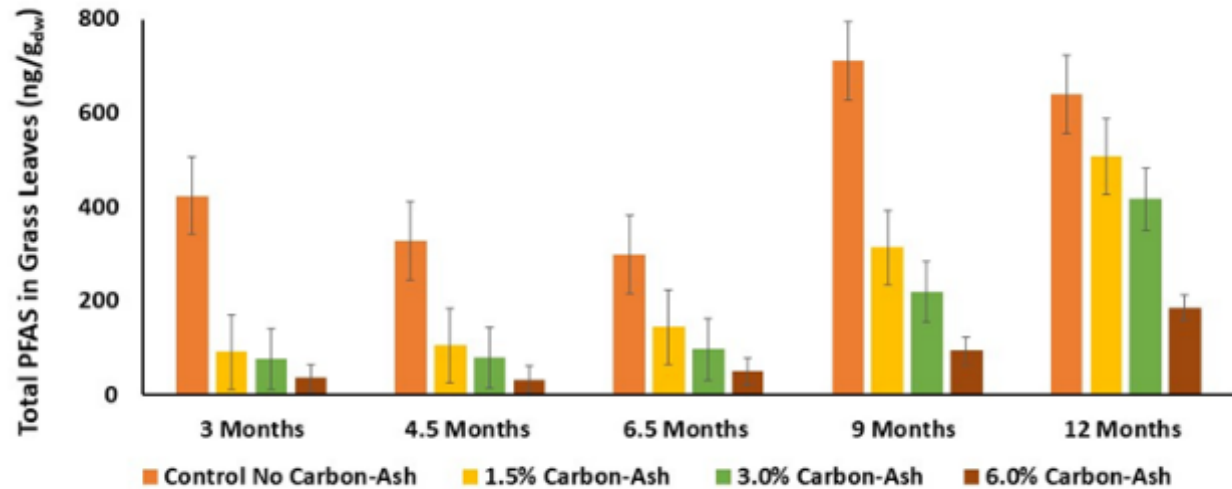
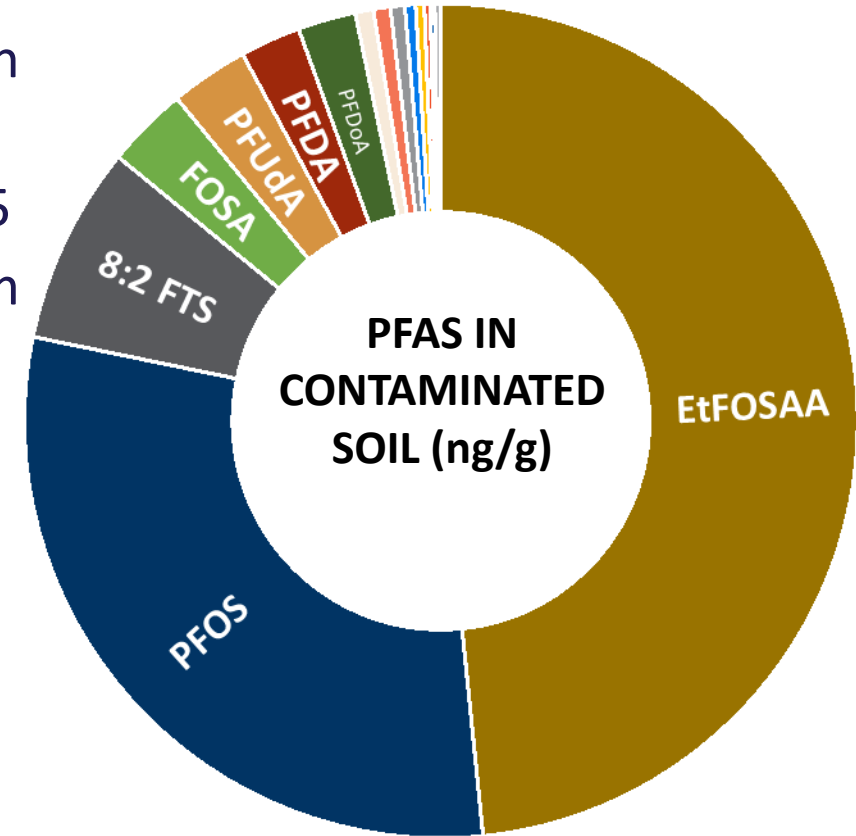
- **PFAS (particularly long chain like PFOS) bioaccumulates in meat and milk**
- Irrigation with PFAS-contaminated water
- PFAS-containing biosolids-based fertilizer
- PFAS in the feed
 - PFAS uptake is greatest into leaves and stalk
 - PFAS accumulate in grasses (hay)
 - PFAS uptake is small into fruit (e.g., tomatoes), seeds (e.g., soybeans, corn kernels and cob)
- Drinking PFAS-contaminated water
- Only Maine has guidance; currently
 - 3.4 ppb in meat
 - 211 ppt in milk
 - Expect 5x reduction in milk guidance



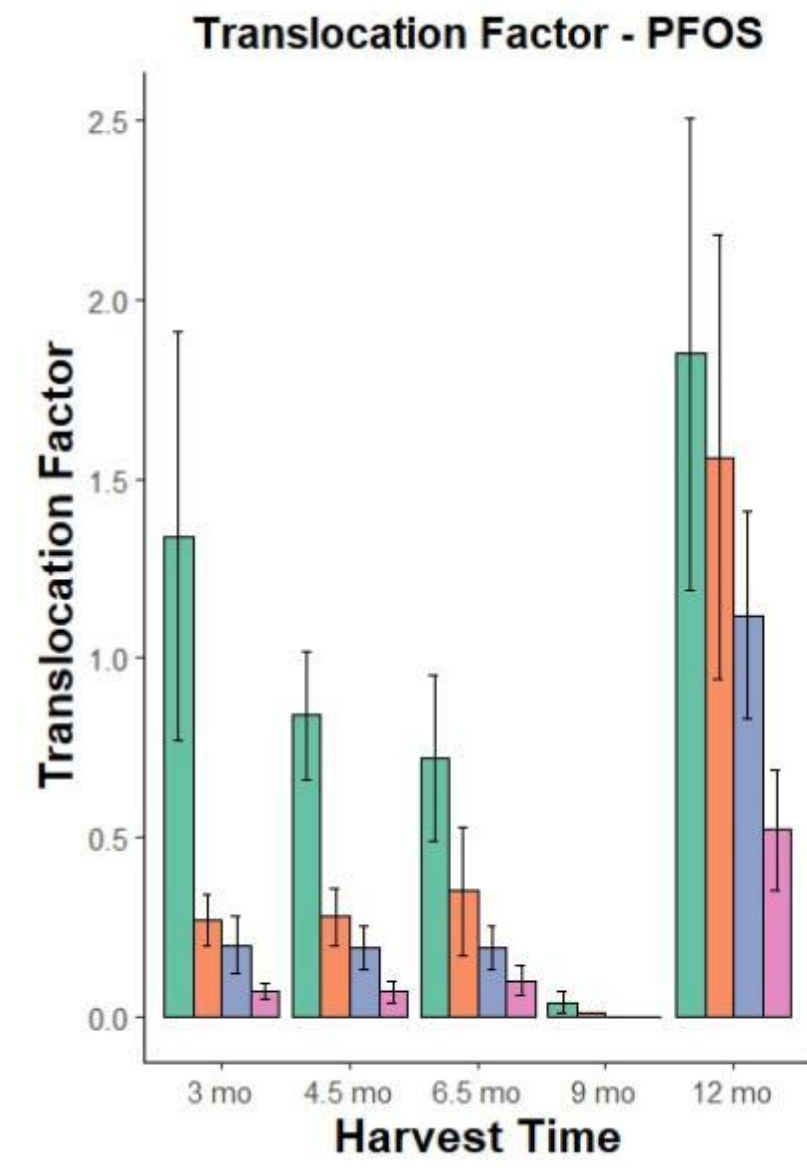


Grass Greenhouse Studies: Uptake and Mitigation

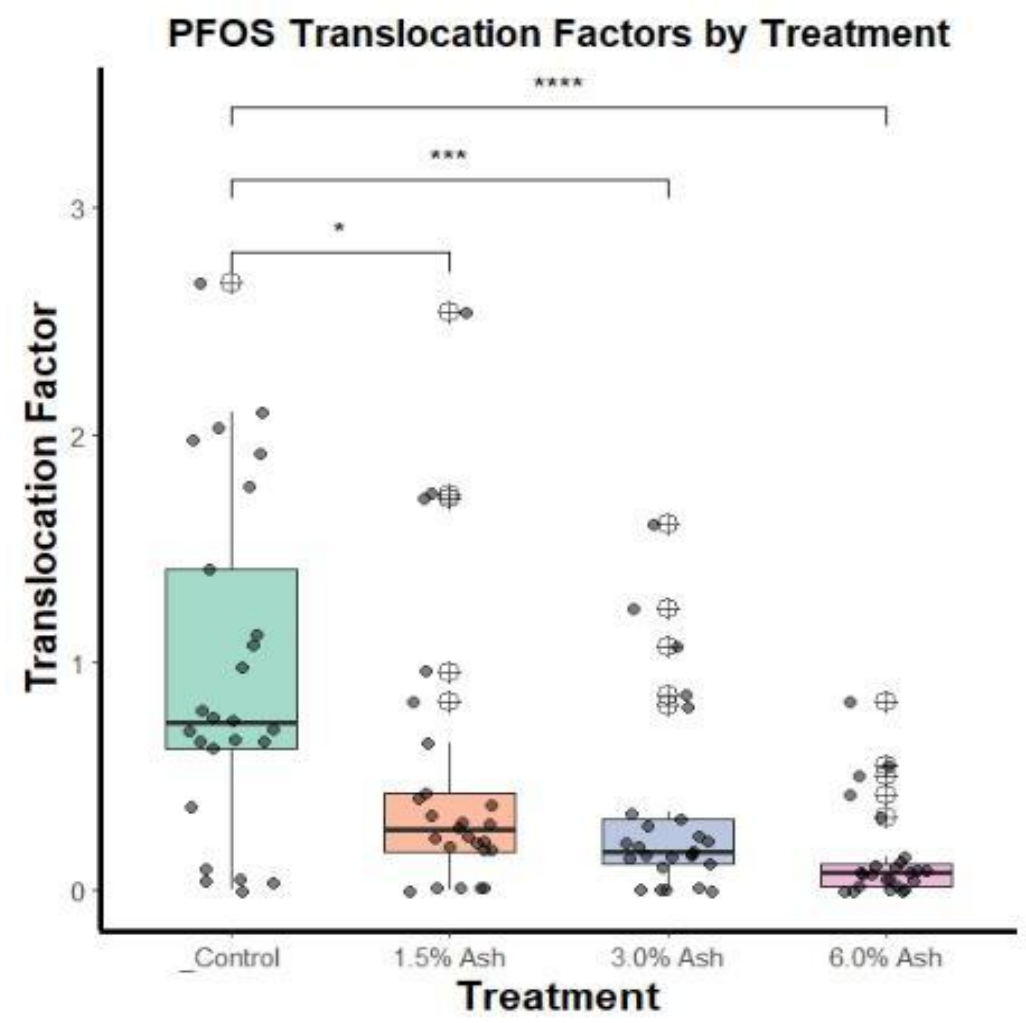
- Contaminated agricultural soil from Maine - $\sum$ PFAS = 840 ng/g with most being PFOS and PFOS precursors (Me- & EtFOSAA, FOSA)
- Biochar (**wood chips ash**) mixed at 3 rates (1.5%, 3% and 6%); n= 5
- Grass mix (40% Tall Fescue, 30% Orchard grass, 20% Festulolium and 10% Timothy) – typical feed grasses for cattle
- Soil seeded in March 2024
- Pots watered as needed weekly
- 4 Grass leaves harvest: 3, 4.5, 6 and 7.5 months....



Grass Greenhouse Studies: Uptake and Mitigation

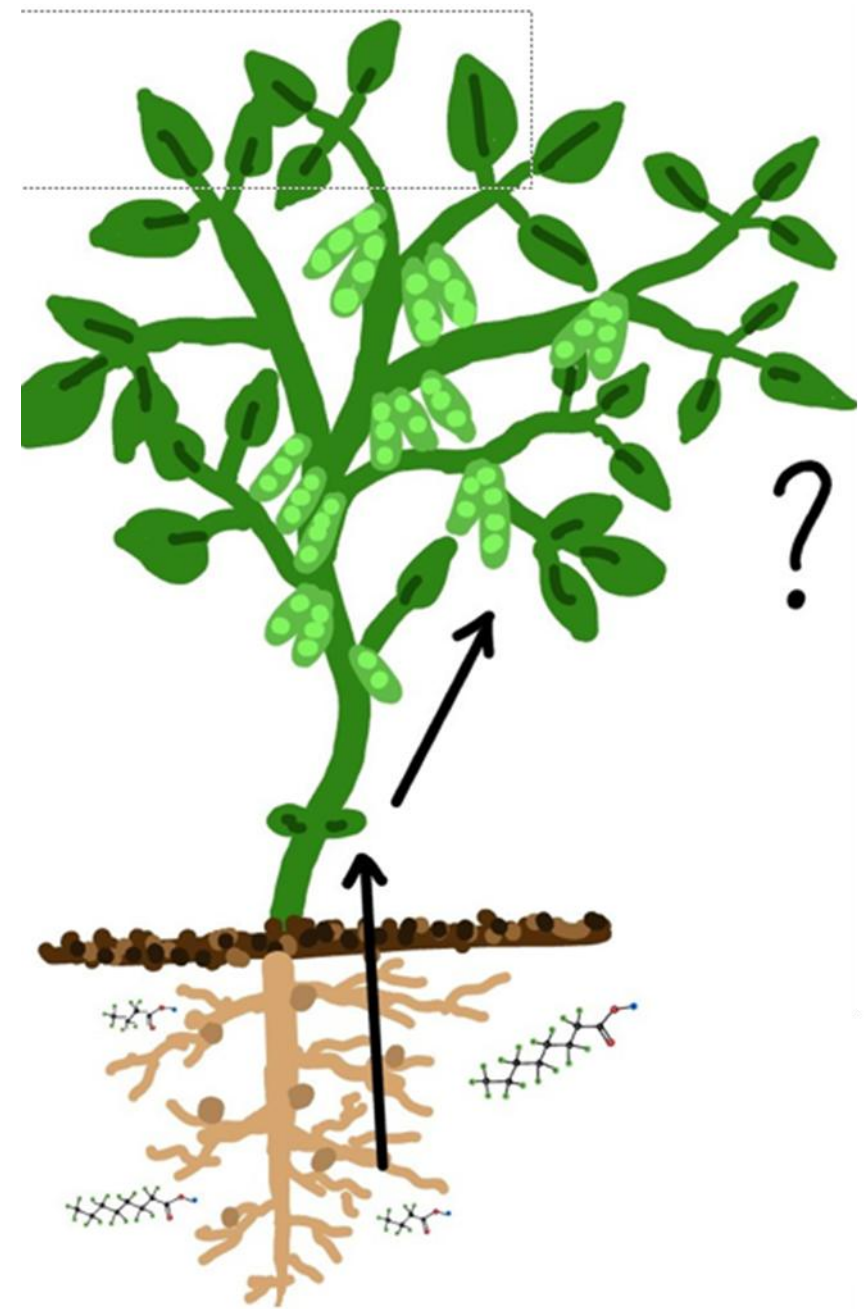
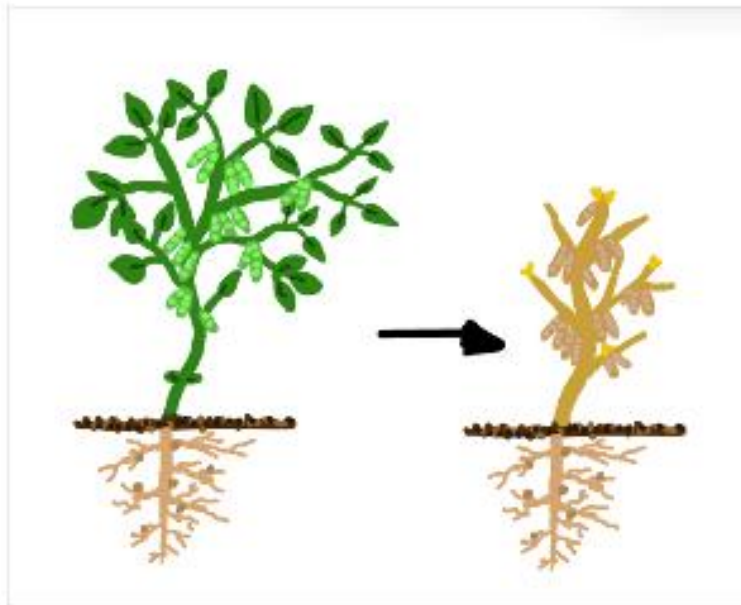
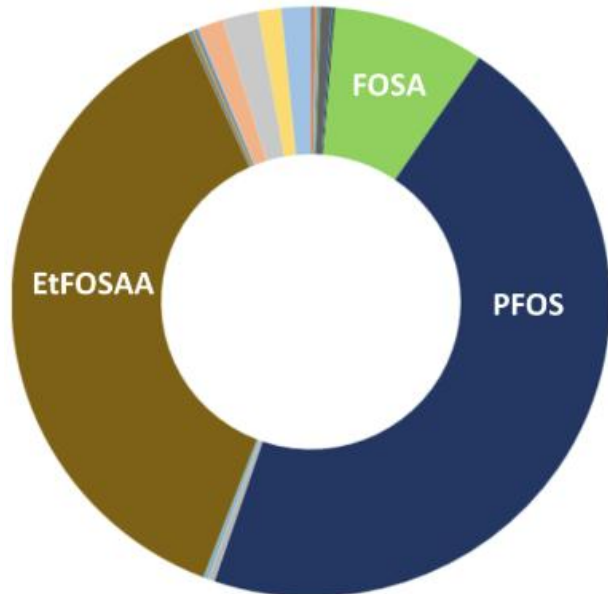


- High carbon wood ash reduced uptake for all harvests over one year
- Increased variability with time
- Field trials in process

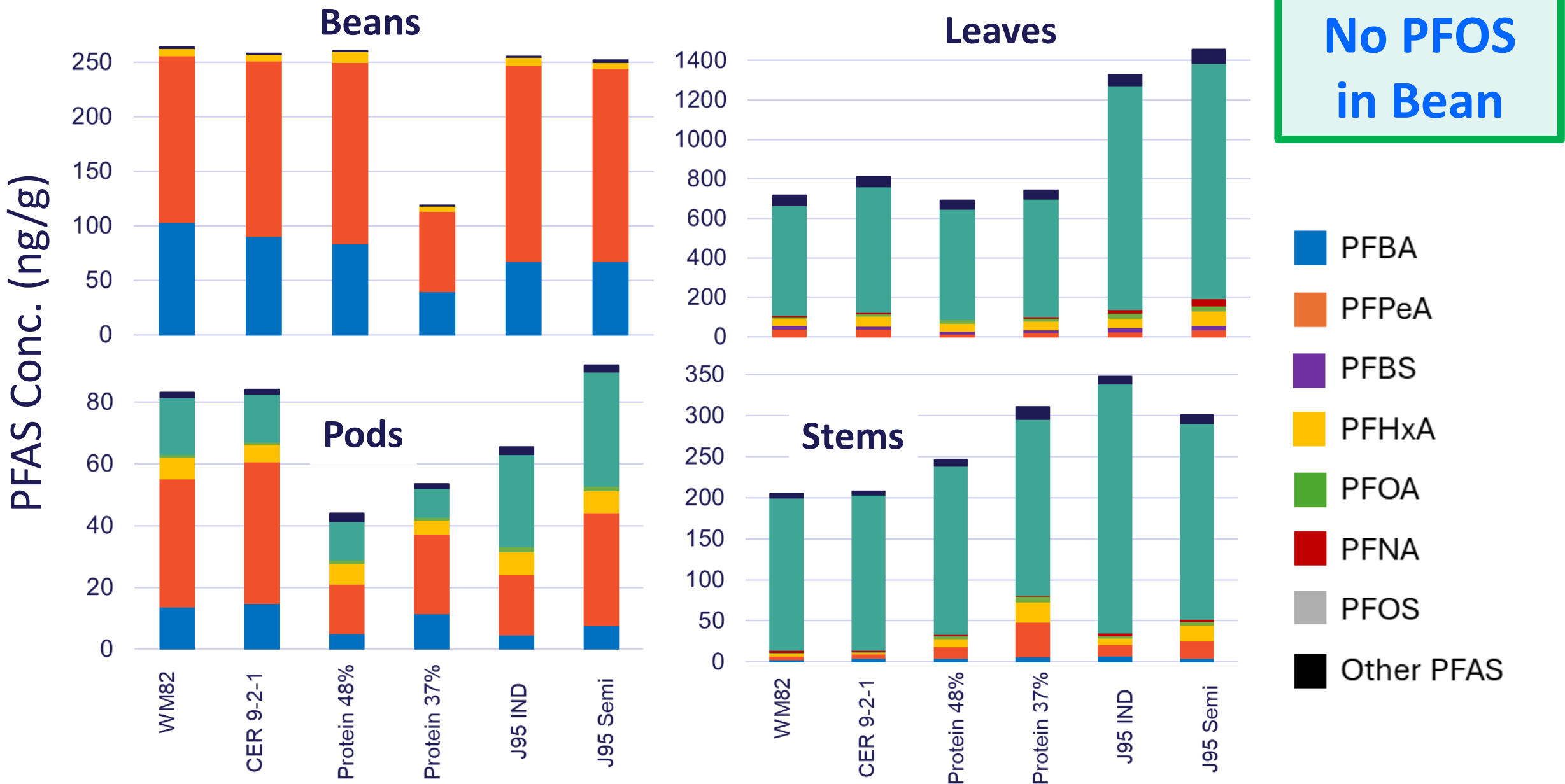


CROP UPTAKE HIGHLIGHTS: SOYBEANS

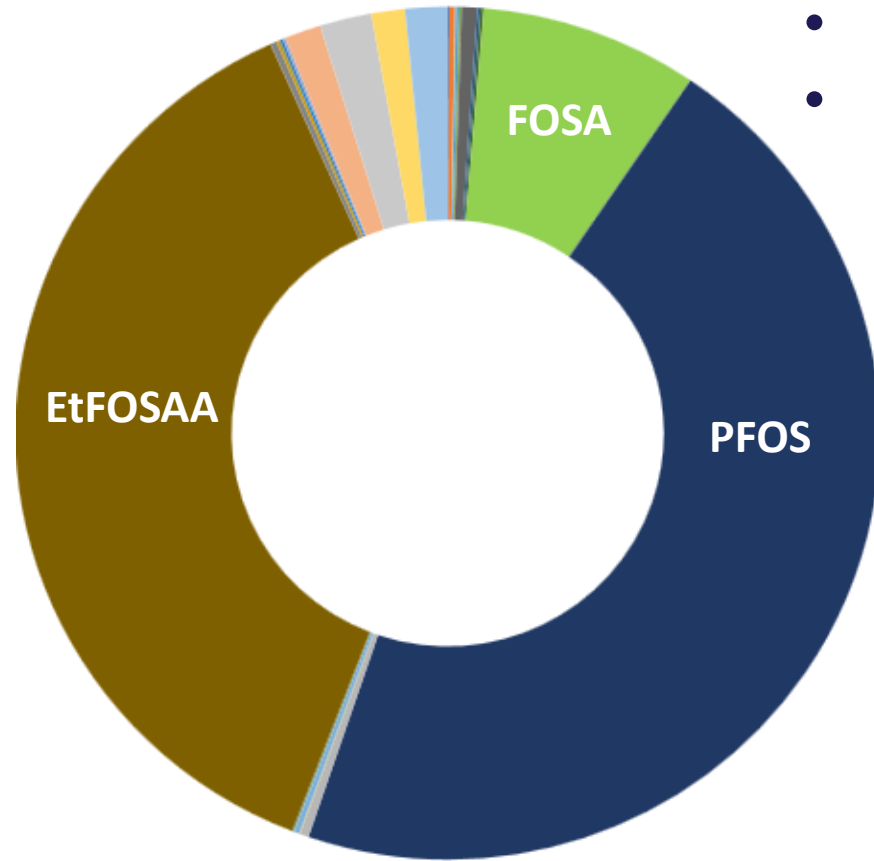
- Soils from years of **paper mill biosolids**-applied fields
- Soils had Σ PFAS ~2800 ppb, most are PFOS + ETFOSAA
- All pots were amended with Milorganite Biosolids at a rate of 1% for PFCA precursor addition
- 6 different soybean varieties grown through senescence
- No PFOS or ETFOSA in the bean
- Only primarily PFBA, PFPeA & PFHxA
- Higher % protein, higher uptake



UPTAKE - SOYBEAN PLANTS: LEAVES > STEMS > PODS

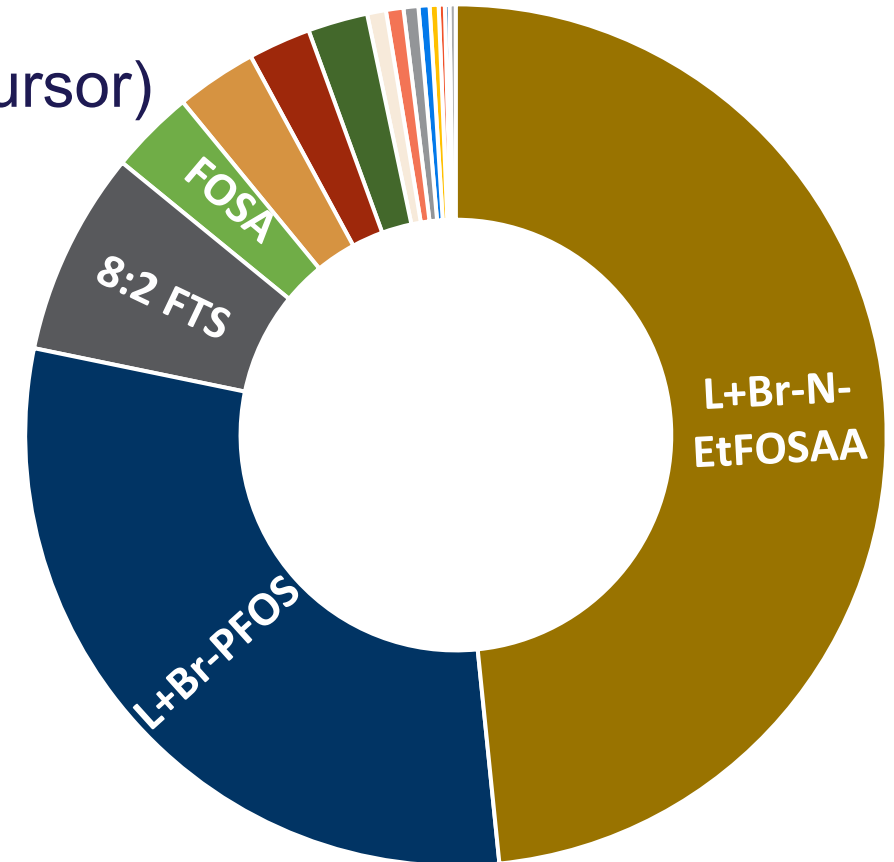


PFAS-Contaminated Soil (Municipal versus Industrial Signature)



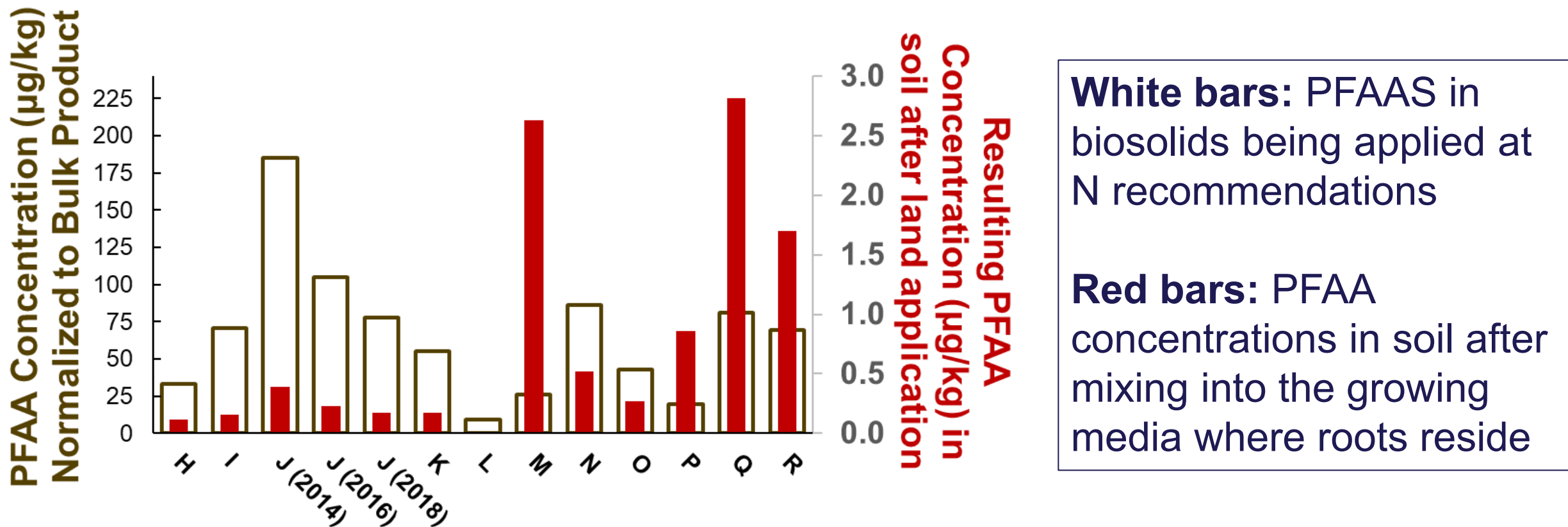
Received only papermill biosolids

- Dominated by:
- EtFOSAA (PFOS precursor)
 - PFOS



Received papermill and municipal biosolids -
also impacted by paper mill discharge into WWTPs

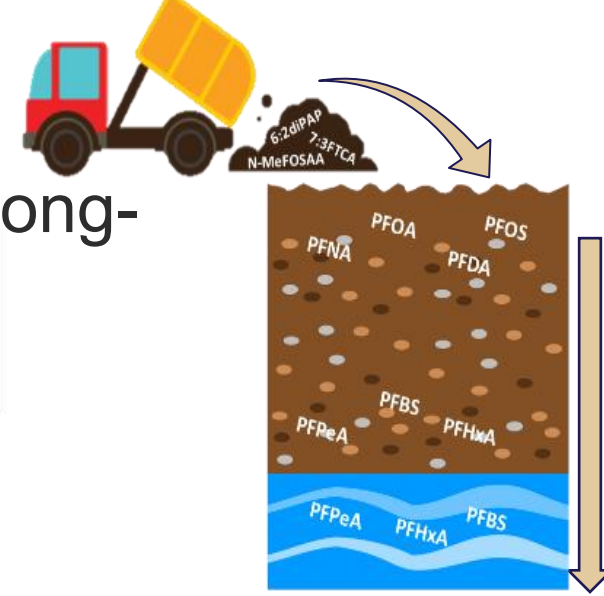
Putting PFAS in Biosolids in Perspective (*Example*)



- Soil amendments/fertilizers are often applied based on N requirements
- Products low in N could lead to higher PFAS loads with a single application

Synopsis and Insights

- Most non-PFAS unregulated COCs are not a short-term or long-term concern
- Our biggest PFAS issues are legacy (prior applications)
- PFAS precursors (many not measured) biotransform to measured PFAS after land application
- PFAS migration from soil to groundwater is dominated by short-chain PFAS
- Long-chain PFAS predominantly persist (with longevity) in the upper profile, thus subject to continual runoff – optimize application strategies and buffer zones, ALSO long chain are decreasing
- Fields under tile-drainage, direct transport to groundwater is minimized (*not shown*)



Perspectives and Path Forward towards a Circular Economy

- Banning land application places a **heavy burden on public municipalities and can lead to numerous unintended consequences**
- **Control sources** contributing to PFAS levels in biosolids (e.g., **pretreatment** of influent from industry or landfills with *high* PFAA levels)
- **Focus on regulating nonessential PFAS uses** to reduce PFAS loads coming into our WRRFs
Domestic sewage typically contributes the largest PFAS mass load coming into our treatment plants and most PFAS mass exits via effluent to surface waters not biosolids.
- Continue research on fate, effects, **application optimization**, & treatment/mitigation strategies
- **Innovate biosolids processing** to reduce PFAS in biosolids
- Consider **adaptable plans for PFAS-contaminated lands**



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THANK YOU! QUESTIONS?

Acknowledgements



Purdue Research Group Contributors: Your Choi, Roy Ruiz-Alvarez, Lynda Peter, Caroline Alukkal, Ariana Lazo, Elijah Openiyi, Jamie Klamerus, Angelly Garin

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