# 2024 CHLORINATED CONFERENCE

Technologies to Support the Quality Control of PFAS Immobilization and Minimize Uncertainty

**Dr. Matthew Askeland** Andrew Mitchell Tim Dowle



Session I9 June 06, 2024 (Mile High Ballroom, Rooms 4a-4c)





### Why immobilize? – distinct considerations for source and diffuse zones.





## Why immobilize? – suitability amongst other treatment options (mg/\$)



Matthew Askeland, ADE, Federal Contaminated Sites National Workshop, 16th November 2021 RPIC.





## Why immobilize? – benefits as compared to other treatment options

- Drenning et al. (2023) used a probabilistic cost-benefit analysis for evaluating PFAS remediation options.
- It demonstrated that remediation of PFAS hotspots and bulk material on the rest of site (diffuse zone) present different needs.
- Maximum value was presented where the bulk material is addressed by immobilisation or solidification.

Science of The Total Environment Volume 882, 15 July 2023, 163664	Four Engineers	PFAS Remediation Alternatives	Alt 0	Hotspot	& Rest of site
		= excavation			
Comparison of PFAS soil remediation		= stabilisation/ solidification	Alt 1		
benefit analysis		= landfilling	Alt 2	<b>A</b>	Ĺø 🗸
P. Drenning. <sup>a</sup> A 🖾 , Y. Volchko. <sup>a</sup> , L. Ahrens. <sup>b</sup> , L. Rosén. <sup>a</sup> , T. Söderqvist. <sup>c</sup> , J. Norrman. <sup>a</sup>		= stabilisation with activated carbon	Alt 3	<b>A</b>	
+ Add to Mendeley 🚓 Share 🍠 Cite		= phytoremediation	Alt 4	<b>▲ ८</b>	•
https://doi.org/10.1016/j.scitotenv.2023.163664 7 Get rights and co	ontent 7	= incineration			778
Under a Creative Commons license 🛪 🔹 o	open access	: soil washing	Alt 5		





## **Case Study Overview**



Australian Airport with multiple source zones (high PFAS concentration, lower volume) and significant diffuse contamination (low concentrations, few million cubic meters).



Desire to explore options to manage heavy clay material on site in light of major upcoming capital works program.



Landfill or thermal options (160 – 400 \$AUD/ton) both cost prohibitive and unsustainable for managing high volumes of relatively low PFAS concentration soils.



Soil-sorbent blend quality and soil-sorbent interactions often overlooked, presenting uncertainties that may impact immobilisation outcome.



**Objective:** Employ a large-scale trial to assess whether immobilisation was a viable alternative for the treatment of large volumes of diffuse PFAS contaminated clayey material.





### **Example Project Process**







#### **Case Study Project Process**









## **Sorbent Selection by SSQM**

- Sorbent Standardized Quality Measure (SSQM) fills a need for a cross comparable method for selecting a sorbent.
- Considers a range key PFAS species of concern.
- Takes into account matrix effect.
- Suitable for a variety of sorbents.









## Sorbent Standardized Qualities Measure (SSQM)

- SSQM and MSQM measure performance for PFAS species of interest **Provides Direct Measure**.
- Makes use of sequential sorb and desorb step for 6 PFAS species.
- Standardized: PFAS species and concentration, volume, time, sorbent mass (2% w/w), (soil mass for MSQM)
- **SSQM** (**MSQM** Matrix) = Matrix interference







#### SSQM used for Australian Airport Soil/Sorbents

- 16 sorbents and a control (quarts sand) tested with SSQM in duplicate.
- 2 sorbents progressed to MSQM testing (quarts sand).
- Both selected sorbents performed similarly.
- Mixed Mineral selected from the two sorbents based on cost and merit (literature available on that specific product).

$\square$	SSQM Output	Percentage Sorbed (%)	Percentage Desorbed (%)	Performance Quotient	Net Removal (%)	SSQM Score
	F-100A	43	2.0	39	76	58
	F-100B	41	2.0	28	80	54
	F-400A	99	1.0	98	97	98
	F-400B	83	2.0	43	84	64
	PS900A	99	1.0	100	99	100
	PS900B	99	1.0	100	99	100
	PS1300A	99	1.0	100	99	100
	PS1300B	99	1.0	100	99	100
	R100A	99	1.0	100	99	100
	R100B	99	1.0	100	99	100
	S1A	53	12.0	33	97	65
	S1B	49	13.0	25	96	61
5	MSQM Output	Percentage Sorbed (%)	Percentage Desorbed (%)	Performance Quotient	Net Removal (%)	MSQM Score
	RB100 + Soil	100	1	100	99	100
	PAC13 + Soil	100	1	100	99	100







## **Bench Scale Trial**

- Adequate number of samples collected from each bulk stockpile for characterisation.
- Sub-Stockpiles SPW and SPS selected for trial and amalgamated (~8000 ton).
- Representative soil dosed at 0, 0.5, 1, 1.5, 2, and 5% w/w.
- Academic and realistic method used to assess sample preparation and blending method impact on specification.
- Soils analysed for PFAS leachability at pH 5 and pH 7.









### **Bench Scale Trial Outcomes**

- Suitable PFAS leachability reduction demonstrated by 1% and 2% w/w applications.
- RemBind Treatment outcome below all target criteria.
- No significant difference seen between academic/realistic methodology.

Stockpiles		Average Total Concentration		Average Leachable Fraction at pH 5		Average Leachable Fraction at pH 7	
<b>RemBind Application</b>	RemBind Dose	PFHxS + PFOS	Standard	PFHxS + PFOS	Standard	PFHxS + PFOS	Standard
Approach	(%)	(mg/kg)	Deviation	(µg/L)	Deviation	(µg/L)	Deviation
	0	0.0513	0.005	1.390	0.349	1.668	0.565
	0.5	0.0117	0.004	0.010*	0.111	0.093	0.059
CDW (Realistic)	1	0.0035	0.001	0.010	0.000	0.052	0.059
SPW (Realistic)	1.5	0.0028	0.003	0.010	0.000	0.010	0.000
	2.0	0.0018	0.000	0.010	0.000	0.010	0.000
	5	0.0017	0.001	0.010	0.000	0.010	0.000
	0	0.0897	0.015	1.403	0.266	2.917	0.324
	0.5	0.0635*	0.031	0.078*	0.110	0.247	0.240
SPW (Academic)	1	0.0267	0.009	0.062	0.034	0.010*	38.72
	1.5	0.0143	0.002	0.053	0.027	0.010*	0.054
	2.0	0.0140	0.006	0.060	0.012	0.010	0.000
	5	0.0055*	0.091	0.022	0.009	0.010	0.000
	0	0.0417	0.004	1.003	0.647	1.233	0.488
	0.5	0.0073	0.002	0.010	0.000	0.010	0.000
CDC (Poplictic)	1	0.0032	0.000	0.010	0.000	0.010	0.000
SPS (Realistic)	1.5	0.0028	0.000	0.010	0.000	0.010	0.000
	2.0	0.0015	0.000	0.010	0.000	0.010	0.000
	5	0.0017	0.001	0.010	0.000	0.010	0.000
	0	0.0530	0.001	0.062	0.012	1.223	0.316
CDC (Assistantia)	0.5	0.0233	0.001	0.010	0.000	0.010	0.000
	1	0.0200	0.005	0.010	0.000	0.010	0.000
SPS (Academic)	1.5	0.0210	0.016	0.010*	1.636	0.010	0.000
	2.0	0.0100	0.004	0.010	0.000	0.032*	0.031
	5	0.0043	0.000	0.010	0.000	0.010	0.000





APAM Level 1 Soil, APAM Level 2 Soil, APAM Level 3 Soil, APAM Level 3 Soil, APAM Level 4, APAM Level 5 Soil \* The calculation of this value excluded true outliers that were clearly erroneous or not representative of the of the other replicates in the data group.





## **Pilot Scale Methodology**

- Soil treatment involved the addition of 1-2% RemBind<sup>®</sup> to 8000 tons of stockpiled soil.
- Three different blending methods to assess their ability to apply the sorbent evenly and efficiently; an excavator with sieve bucket, a pug mill, a portable trommel screen.
- Two treatment approaches, "single pass blend and dose", as well as a "double pass" dose and blend then a second blend.
- Over 24 stockpiles of 100 2000 tons each included in testing factorial and treated in the trial.







## **Pilot Scale Methodology**

- Each stockpile characterised pre- and post- treatment.
- Post treatment characterisation undertaken 24 hours after treatment.
- In total, more than 500 soil samples were collected from 24 stockpiles including 50 QAQC samples to enable high resolution assessment.
- The Sorbent Application Uniformity Test (SAUT) was used to assess the accuracy and precision of the mixing processes by assessing the distribution throughout the material.









## **Pilot Scale Outcomes**

- Validation demonstrated PFAS leachability was reduced from Sum PFAS ranging from 0.052 -2.346 µg/L to less than the limit of detection (0.001 µg/L).
- PFAS leachability reduction was noted for shorter (where present) as well as long chain congeners.
- The reduction in leachable fractions for PFOS + PFHxS was found to be >99 % in treated soils.

Stockpile	PFAS Congeners	Leachable Fraction Pre -Treatment (μg/L)	Leachable Fraction Post -Treatment (µg/L)	Immobilised (%)			
Excavator Blended Stockpiles							
1%-E-E1 (ST-P1)	PFOS + PFHxS	1.077	( <lod)< th=""><th>~100</th></lod)<>	~100			
1%-E-E2 (ST-P2)	PFOS + PFHxS	0.065	( <lod)< th=""><th>~100</th></lod)<>	~100			
1%-E-E3 (ST-P3)	PFOS + PFHxS	1.901	0.023	98.79			
1%-E-B (ST-BE2)	PFOS + PFHxS	0.707	( <lod)< th=""><th>~100</th></lod)<>	~100			
2%-E-E1 (ST-P4)	PFOS + PFHxS	1.464	( <lod)< th=""><th>~100</th></lod)<>	~100			
2%-E-E2 (ST-P5)	PFOS + PFHxS	0.385	( <lod)< th=""><th>~100</th></lod)<>	~100			
2%-E-E3 (ST-P6)	PFOS + PFHxS	0.989	( <lod)< th=""><th>~100</th></lod)<>	~100			
2%-E-B (ST-BE1)	PFOS + PFHxS	1.035	( <lod)< th=""><th>~100</th></lod)<>	~100			
Pugmill Blended Stoc	kpiles						
1%-P-E1 (ST-E1)	PFOS + PFHxS	1.439	0.007	99.51			
1%-P-E2 (ST-E2)	PFOS + PFHxS	0.554	0.001	99.82			
1%-P-E3 (ST-E3)	PFOS + PFHxS	0.639	0.001	99.63			
1%-P-B (ST-BP1)	PFOS + PFHxS	0.887	( <lod)< th=""><th>~100</th></lod)<>	~100			
2%-P-E1 (ST-E4)	PFOS + PFHxS	0.608	( <lod)< th=""><th>~100</th></lod)<>	~100			
2%-P-E2 (ST-E5)	PFOS + PFHxS	1.318	( <lod)< th=""><th>~100</th></lod)<>	~100			
2%-P-E3 (ST-E6)	PFOS + PFHxS	0.073	( <lod)< th=""><th>~100</th></lod)<>	~100			
2%-P-B (ST-BP2)	PFOS + PFHxS	1.541	( <lod)< td=""><td>~100</td></lod)<>	~100			
Trommel Blended Stockpiles							
1%-T-E1 (ST-T4)	PFOS + PFHxS	0.138	( <lod)< th=""><th>~100</th></lod)<>	~100			
1%-T-E2 (ST-T5)	PFOS + PFHxS	0.174	( <lod)< th=""><th>~100</th></lod)<>	~100			
1%-T-E3 (ST-T6)	PFOS + PFHxS	0.648	( <lod)< th=""><th>~100</th></lod)<>	~100			
1%-Т-В (ST-ВТ2)	PFOS + PFHxS	0.390	( <lod)< th=""><th>~100</th></lod)<>	~100			
2%-T-E1(ST-T3)	PFOS + PFHxS	0.139	( <lod)< th=""><th>~100</th></lod)<>	~100			
2%-T-E2 (ST-T2)	PFOS + PFHxS	0.137	( <lod)< th=""><th>~100</th></lod)<>	~100			
2%-T-E3 (ST-T1)	PFOS + PFHxS	0.130	( <lod)< th=""><th>~100</th></lod)<>	~100			
2%-T-B (ST-BT1)	PFOS + PFHxS	0.025	( <lod)< th=""><th>~100</th></lod)<>	~100			







## **Pilot Scale Outcomes**

- Reuse target of below 0.4 μg/L PFOS + PFHxS achieved.
- Retrospectively, all results below drinking water criteria (0.070 µg/L).
- All but 1 outcome below Australian Draft PFOS Default Guideline Value for Freshwater ecosystems (9.1 ng/L), assessed here as PFOS + PFHxS due to comparable toxicological profiles.

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2%-P-B (ST-BP2)	PFOS + PFHxS	1.541	( <lod)< td=""><td>~100</td></lod)<>	~100			
Trommel Blended Sto	ckpiles						
1%-T-E1 (ST-T4)	PFOS + PFHxS	0.138	( <lod)< td=""><td>~100</td></lod)<>	~100			
1%-T-E2 (ST-T5)	PFOS + PFHxS	0.174	( <lod)< td=""><td>~100</td></lod)<>	~100			
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2%-T-B (ST-BT1)	PFOS + PFHxS	0.025	( <lod)< th=""><th>~100</th></lod)<>	~100			







## **Quality Control – SAUT**

- Dye based method matrix corrected calibration curve to quantify the mass of sorbent in a soil.
- Used to ensure actual sorbent dose lies within an acceptable range based on the % w/w specification from bench trial.
- Provides quick evidence of success and material treatment uniformity and quality.
- Used at various stages in treatment process for identification of non-conformities and rectification.
- Used alongside leachability testing.









## **Quality Control – SAUT**

- The SAUT method was found to be suitable as a QA/QC method to be used in tandem with PFAS analysis.
- Demonstrated that some sorbent-soil blending technologies performed better for the soil type when assessed alongside ASLP results.
- Trommel producing the most uniform and accurate blends.



Mixing Equipment	Mean Actual RemBind (%)	Mean Accuracy (%)	Uniformity - RSD (%)	Blend Quality
1% RemBind Specified Application				
Excavator (E)	0.35	35.05	67.13	Unacceptable*
Pugmill (P)	0.86	86.43	69.52	Acceptable
Trommel (T)	1.23	122.68	51.09	Good
2% RemBind Specified Application				
Excavator (E)	1.21	60.50	85.31	Acceptable
Pugmill (P)	0.67	33.70	100.08	Unacceptable*
Trommel (T)	2.58	128.76	30.63	Good

Note to Table: \* represents data that demonstrates loss of sorbent fraction as dust that biased the methodology, in that blending was found by SAUT to be low quality but PFAS leachability testing demonstrated a satisfactory result.





## **Project Outcomes and Managing Uncertainty**

- Soil successfully treated to reduce PFAS leachability to below target airport reuse requirements.
- Blending uniformity and sorbent behavior uncertainties addressed.
- Flexible plant options for blending, further optimised for more reliable outcomes.
- Soil able to be processed at a high throughput rate (~1000 1500 ton per day).
- Robust QAQC method suitable for identification of treatment non-conformity employed.
- Robust multiple lines of evidence demonstrating treated PFAS impacted spoil suitable for reuse.
- Provided use cases for PLCMS and PFAS in Dust technologies









**PFAS in Dust – Occupational Monitoring** Poster Group 1 (I3) - #942



**Portable LCMS (PLCMS)** Poster Group 2 (E9) - #939





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