

# Sediment Management Working Group

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**Jacobs**

Challenging today.  
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## The Development a Rapid CQA Method to verify GAC/Sand and OC/Sand Blends for sediment caps

Dave Adams

Andy Judd, PG

Juliana Hess, PE

Alise Lampi

Nasser Hamdan, PhD

Dennis G. Grubb, PhD, PE, F. ASCE



# Motivation

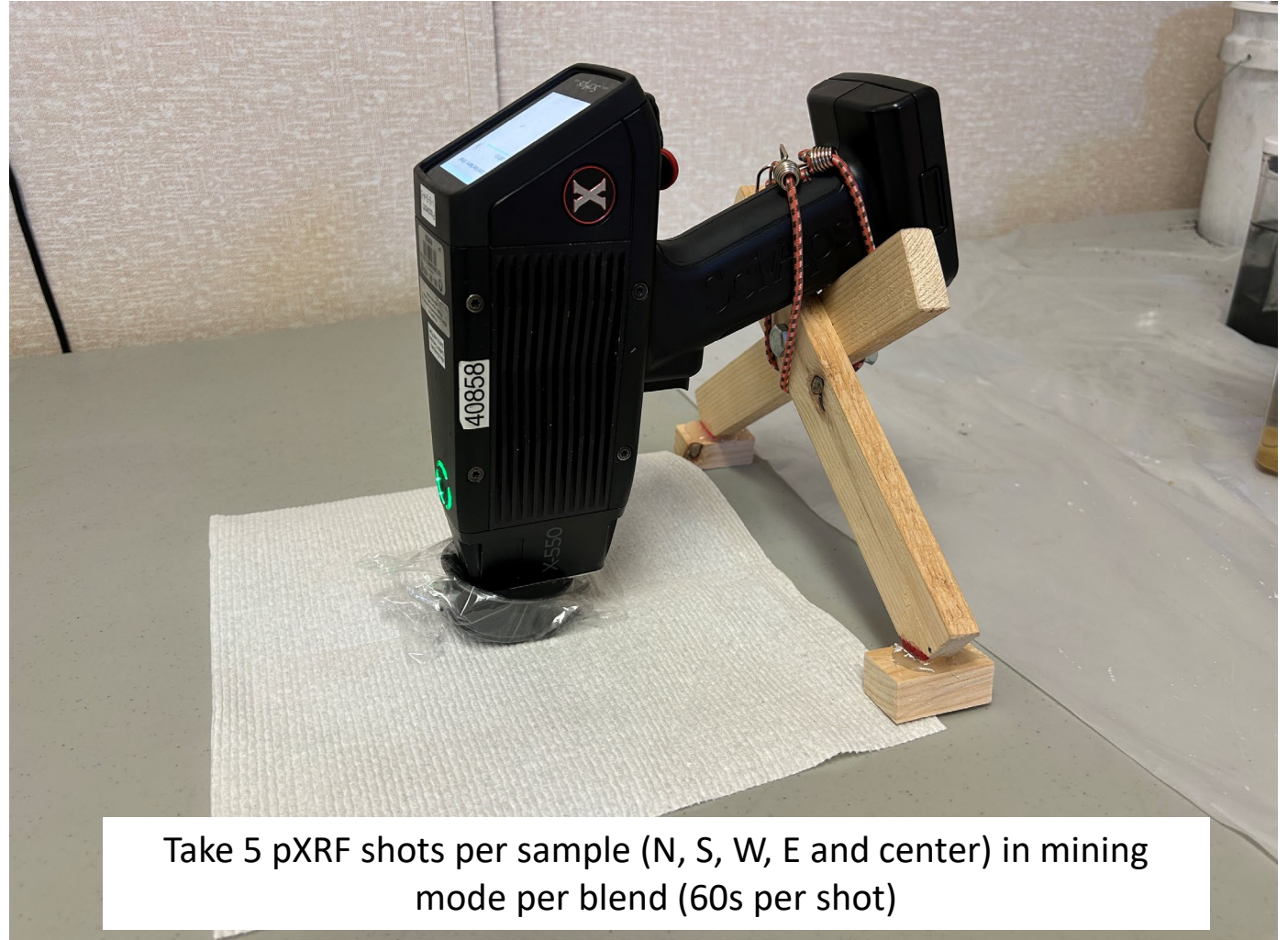
- Conventional LOI testing for OC/GAC content of their blends with sand can take up to 3 days leading to delays in construction.
- Hamdan et al. 2024 showed that pXRF could be used in a field trailer setting to rapidly confirm granular activated carbon (GAC) blended with sand in varying amounts.
  - Results in 2 to 4 hours.
  - GAC/sand blends up to 60 wt% (every 5 w%)
  - Sand from Gowanus Canal Superfund site
  - “Pathfinder” elements (Ca, Si, S)
  - Sulfur was highly correlated with GAC due to bituminous coal origin.



# GAC/Sand Blend Sample Preparation

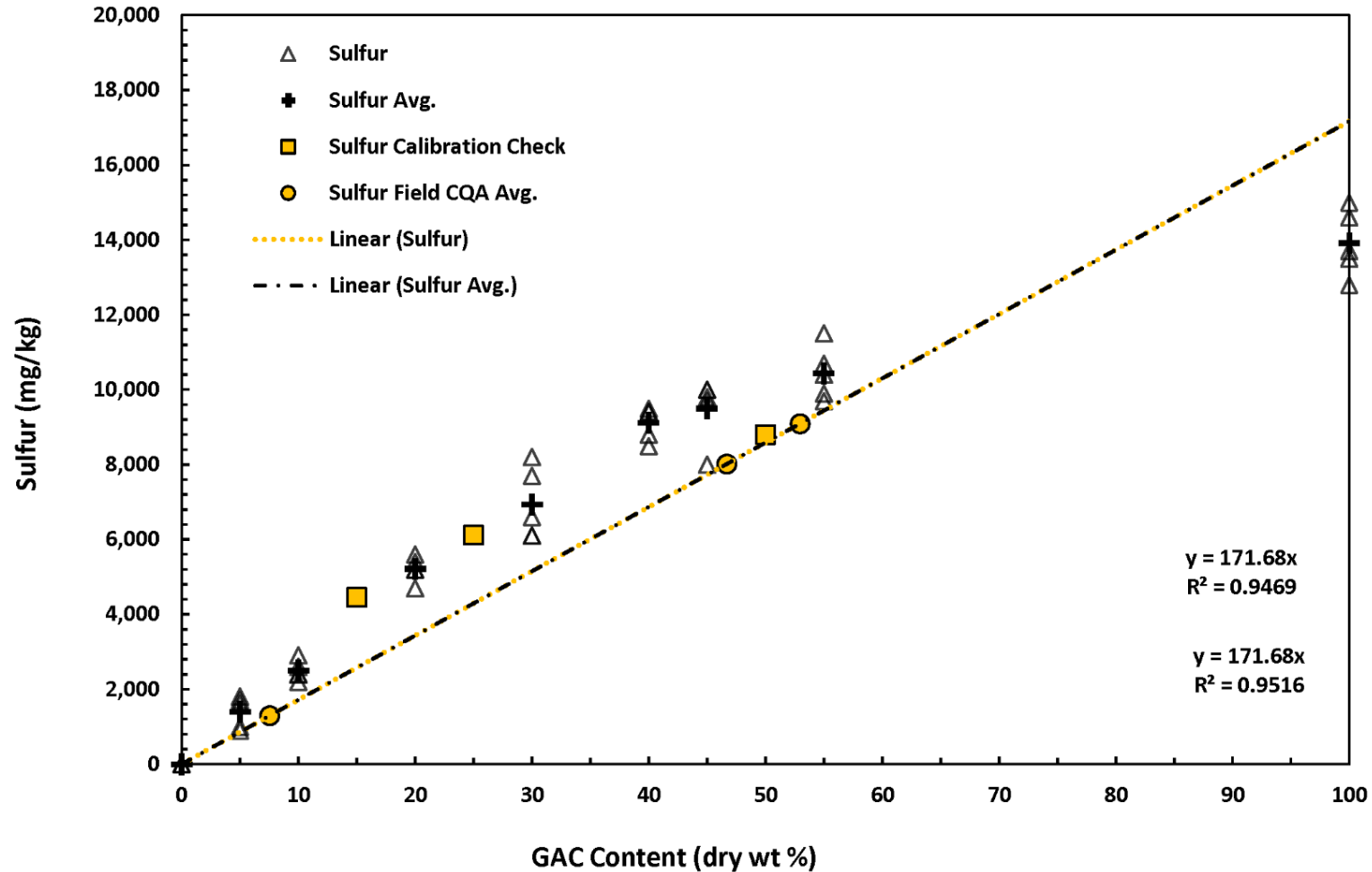


Crushed/pulverized to 100% passing  
No. 100 (150 um) sieve



Take 5 pXRF shots per sample (N, S, W, E and center) in mining  
mode per blend (60s per shot)

# XRF Results – Sulfur correlation

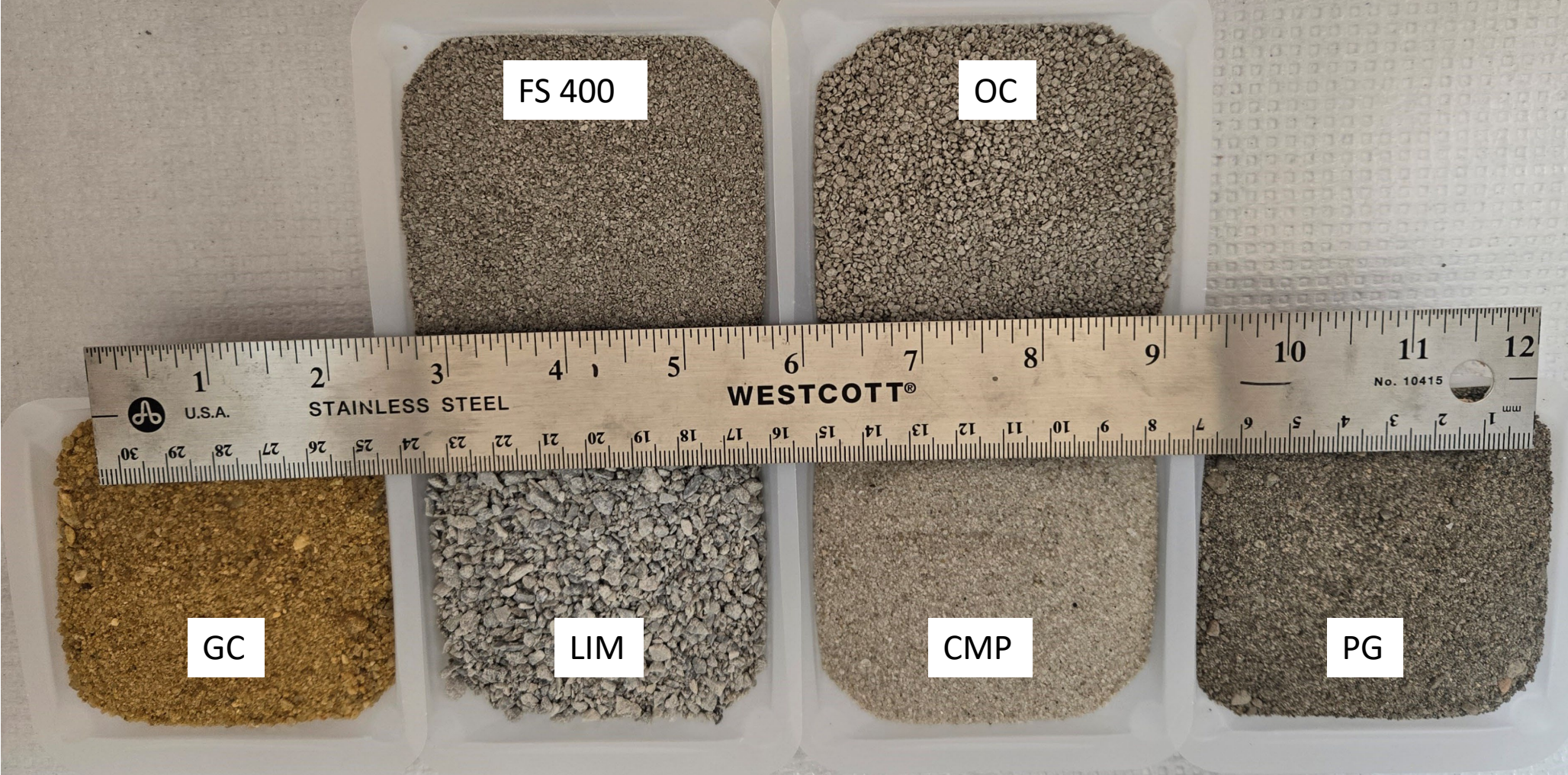


- Data appears curvilinear
  - Linear has better fit up to 60%
- What about the skewed data at 100% GAC?
  - pXRF uses multiple energy x-ray beams to characterize materials
  - X-rays are susceptible to attenuation and other matrix effects
  - Due to mesoporosity and other structural features, carbon nanotubes have been shown to attenuate X-rays up to 30 keV
  - GAC has mesoporosity and structural features similar to C-nanotubes.

## OC/Sand Testing Approach

- Expand to include 4 different “sands” including limestone “sand.”
- OC/FS only dried to 70°F based on manufacturer’s direction
- Milled with cobra crusher to pass No. 100 (150 um) sieve. Watch overheating.
- OC/sand blends from 0 to 45 wt% at 5 wt% increments plus endpoint materials
- Used plastic condiment (2 oz) containers with Saran Wrap cover as before
- pXRF shots in mining mode (4 beams) in triplicate per blending target (60s each)
- Seek out pathfinder elements having linear correlations with  $R^2$  values  $> 0.80$

# OC/FS and sand source materials



# Cobra Crusher Attachment to Angle Grinder to Pulverize Solids



# OC/Sand Testing Results -- Pure Materials

**Table 1.** Average, median and standard deviation values for Cl, Si, Ca, Mg, Fe, and Ti concentrations (mg/kg) of pure sands and sorbents via pXRF.

Analyte	Parameter	OC	LIM	PG	CMP	GC	FS
	n	24	6	6	6	6	6
Cl	Average	32,226	2,759	--	--	--	28,810
	Median	31,924	2,753	--	--	--	30,007
	S.D.	2,831	185	--	--	--	2,395
Si	Average	129,222	11,886	193,954	282,927	287,652	122,287
	Median	129,162	11,874	196,299	283,584	286,328	126,154
	S.D.	11,660	415	9,954	3,567	7,260	10,541
Ca	Average	4,044	213,431	22,112	471	17	4,299
	Median	3,832	211,996	22,618	457	0	4,362
	S.D.	1,109	5,026	1,664	90	26	384
Mg	Average	880	33,071	--	--	--	1,221
	Median	0	33,679	--	--	--	928
	S.D.	1,329	2,856	--	--	--	1,408
Fe	Average	17,358	2,078	29,461	2,453	4,726	15,492
	Median	16,959	2,102	29,780	2,454	4,795	15,974
	S.D.	2,461	71	1,490	66	293	1,165
Ti	Average	564	196	3,576	167	1,366	648
	Median	562	199	3,596	160	1,375	660
	S.D.	60	15	229	25	248	63

“n” denotes number of replicates

“S.D.” denotes standard deviation

“--” denotes concentrations below instrument LOD



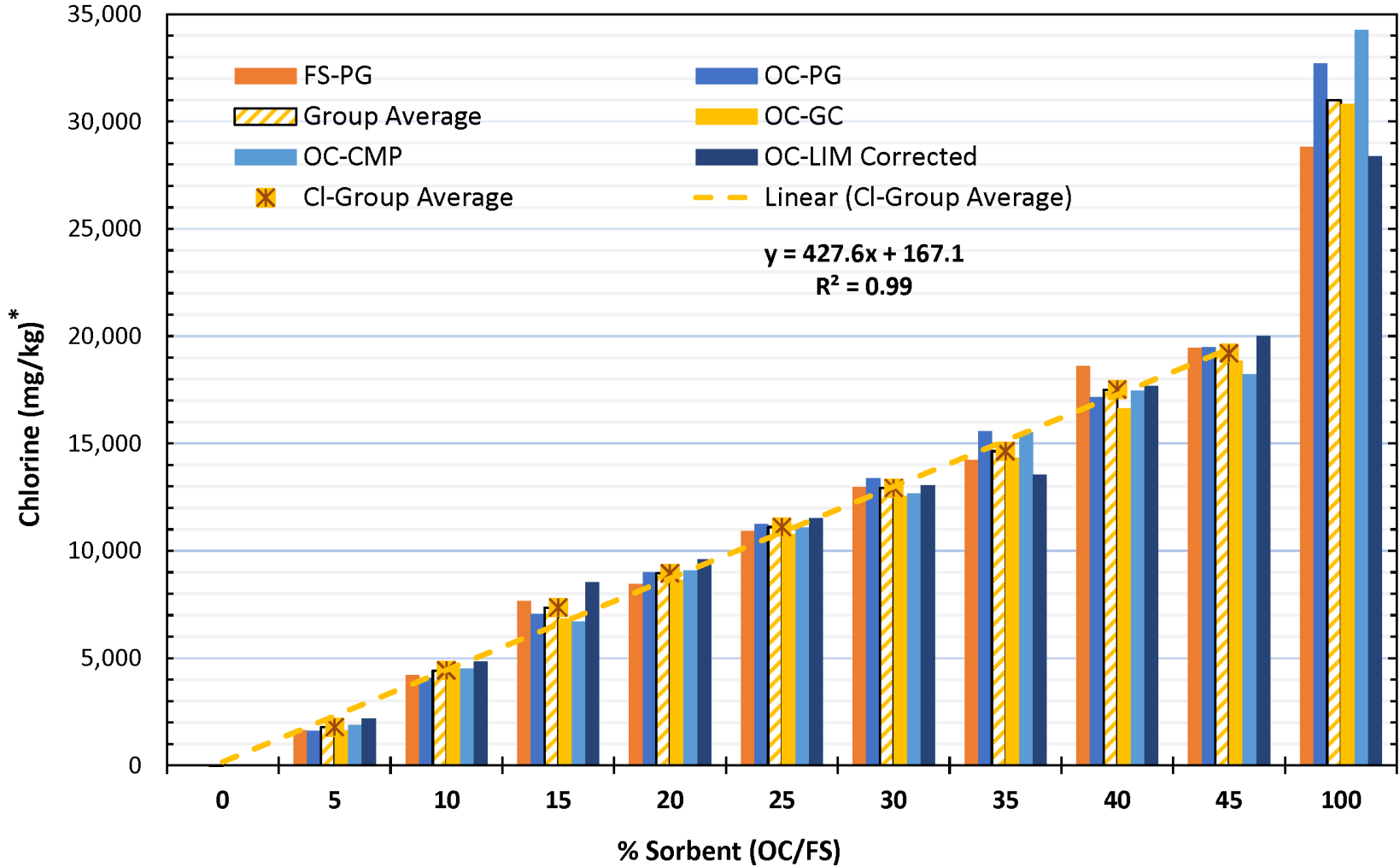
# OC/Sand Blend Pathfinder Element Summary (38 correlations)

Figure 5. Summary of 38 elemental correlations with  $R^2 \geq 0.80$  for OC-sand blends up to 45% OC/FS. Dash indicates  $R^2 < 0.80$  threshold.

Blend Type (OC/FS+Sand)	Correlation ( $R^2$ ) Values										
	Cl	Fe	Ca	Si	Ti	K	Al	Ba	Mg	Mn	Br
OC-LIM	0.98	0.97	0.97	0.98	0.89	--	0.97	0.84	0.95	--	0.94
OC-CMP	0.99	0.99	0.99	0.96	0.92	0.97	0.98	--	--	0.97	--
OC-GC	0.99	0.99	0.99	0.97	0.83	0.98	0.99	0.84	--	--	--
OC-PG	0.99	0.96	0.99	0.91	0.98	0.99	--	0.94	--	--	--
FS-PG	0.99	0.87	0.94	--	0.95	0.95	--	0.84	--	--	--
Only $R^2 \geq 0.80$ reported.					Green $R^2 \geq 0.95$		Yellow $R^2 \geq 0.90$		Orange $0.80 \leq R^2 < 0.90$		

Br shows up in LIM, not OC

# OC/Sand Testing Results -- Chlorine



**Figure 4.** Average Cl concentration for pure media and each OC/FS sand blend corrected for (\*) background Cl content in LIM sand (2,759 mg/kg-Cl). Dashed linear trendline with  $R^2=0.99$  fitted to average Cl concentration for each group (i.e., the striped bar, "Group Average") and sorbent content up to 45% OC/FS.

# OC/Sand element concentration equations for up to 45 wt% OC/FS

Element	Blend Type									
	<u>OC-LIM</u>		<u>OC-GC</u>		<u>OC-CMP</u>		<u>OC-PG</u>		<u>FS-PG</u>	
	Equation	R <sup>2</sup>	Equation	R <sup>2</sup>	Equation	R <sup>2</sup>	Equation	R <sup>2</sup>	Equation	R <sup>2</sup>
<b>Cl</b>	440.66x + 2759	0.98	419.52x	0.99	428.67x	0.99	438.63x	0.99	436.87x	0.99
<b>Fe</b>	155.8x + 2212.6	0.97	117.8x + 4575.9	0.99	170x + 2553.7	0.99	-120.19x + 29974	0.96	-162.48x + 29703	0.87
<b>Ca</b>	-2864.2x + 207395	0.97	37.449x + 106.35	0.99	53.661x + 508.71	0.99	-198.79x + 22492	0.99	-235.4x + 22411	0.94
<b>Si</b>	1626.7x + 13731	0.98	-2046.3x + 285789	0.97	-1730.4x + 278688	0.96	-694.09x + 198700	0.91	--	--
<b>Ti</b>	4.3192x + 201.2	0.89	-10.829x + 1276.2	0.83	5.3951x + 195.98	0.92	-35.209x + 3613	0.98	-37.525x + 3618.9	0.95
<b>K</b>	--	--	17.597x + 1227.5	0.98	24.173x + 677.2	0.97	-145.69x + 15297	0.99	-165.73x + 15185	0.95
<b>Al</b>	360.58x + 1568.3	0.97	428.37x + 3292.7	0.99	456.08x + 949.48	0.98	--	--	--	--
<b>Ba</b>	1.8727x + 179.8	0.84	1.971x + 120.85	0.84	--	--	-4.5082x + 630.12	0.94	-6.4744x + 695.89	0.84
<b>Mg</b>	-424.68x + 33121	0.95	--	--	--	--	--	--	--	--
<b>Mn</b>	--	--	--	--	4.8434x - 11.879	0.97	--	--	--	--
<b>Br</b>	-0.1657x + 13.866	0.94	--	--	--	--	--	--	--	--

R<sup>2</sup> values reported in Figure 5 of main text

Sand: Limestone (LIM); Gowanus Canal (GC); Cape May Point (CMP); Playground (PG)

"X" value in Table denotes OC or FS content

OC: Organoclay PM-200 (CETCO)

FS: Fluorosorb FS-400 (CETCO)

"--" Correlation excluded because R<sup>2</sup> < 0.80

# Conclusions, Observations & Lessons Learned

- “Pathfinder” elements are key to “fingerprinting” a material
  - Cobra Crusher is great time saver, can quickly mill dry solids, including malleable clays
  - For GAC/sand, sulfur is king
  - For OC/sand and FS/sand, chlorine is king
  - Sands interchangeable and scalable if pure “sand” has unique element (Cl in LIM)
  - $OC = Cl \text{ (mg/kg)} / 432$  for OC-sands (first order estimate w/o site specific correlation)
  - Correlations can have steep or shallow slopes, positive or negative depending on pairing
  - Correlations can be used to check each other. Do not extrapolate.
- 1 day to develop correlation; 2-4 hours for triplicate testing of sample including drying/milling

## References

- Lampi, A.B., Hamdan, N. and Grubb, D.G., 2025. Rapid Verification of Organoclay-Sand Capping Blends for Environmental Construction using portable X-ray Fluorescence (pXRF), *J. Hazard. Toxic Radioact. Waste.* 29(4) 04025022 (DOI 10.1061/JHTRBP.HZENG-1515)
- Hamdan, N., Grubb, D.G., Adams, D.L., Judd A.B., Tsiamis, C.D., and Hess, J., 2024. Errata for: Gowanus Canal Superfund Site VI: Rapid Field Screening Techniques to Verify Sediment Capping Blends, *J. Hazard. Toxic Radioact. Waste*, 28(4) (DOI 10.1061/JHTRBP.HZENG-1368).
- Hamdan, N., Grubb, D.G., Adams, D.L., Judd A.B., Tsiamis, C.D., and Hess, J., 2023. Gowanus Canal Superfund Site VI: Rapid Field Screening Techniques to Verify Sediment Capping Blends, *J. Hazard. Toxic Radioact. Waste.* 28(1), 04023045 (DOI: 10.1061/JHTRBP.HZENG-1282).

# Thank You

GAC/Sand

Dave Adams

Andy Judd

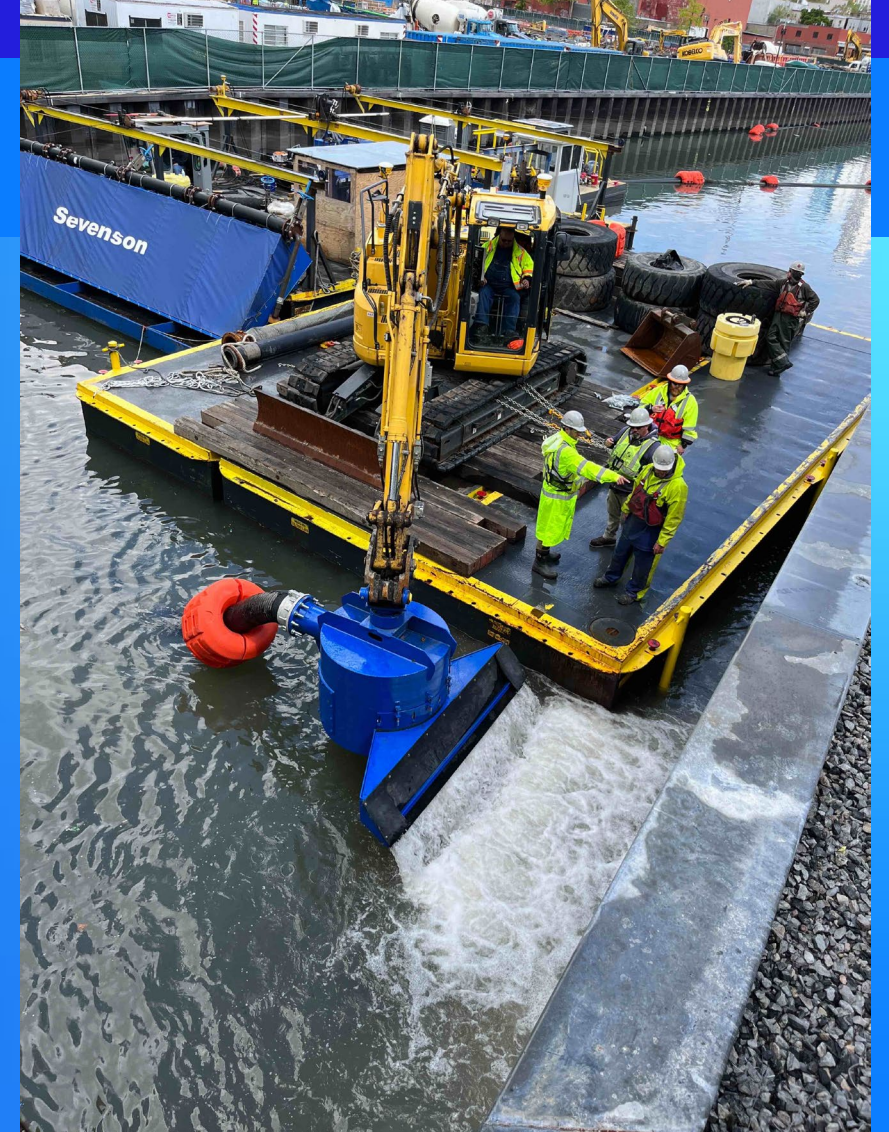
Juliana Hess

OC/Sand

Alise Lampi

Nasser Hamdan (both)

Dennis Grubb (both)



**Jacobs**

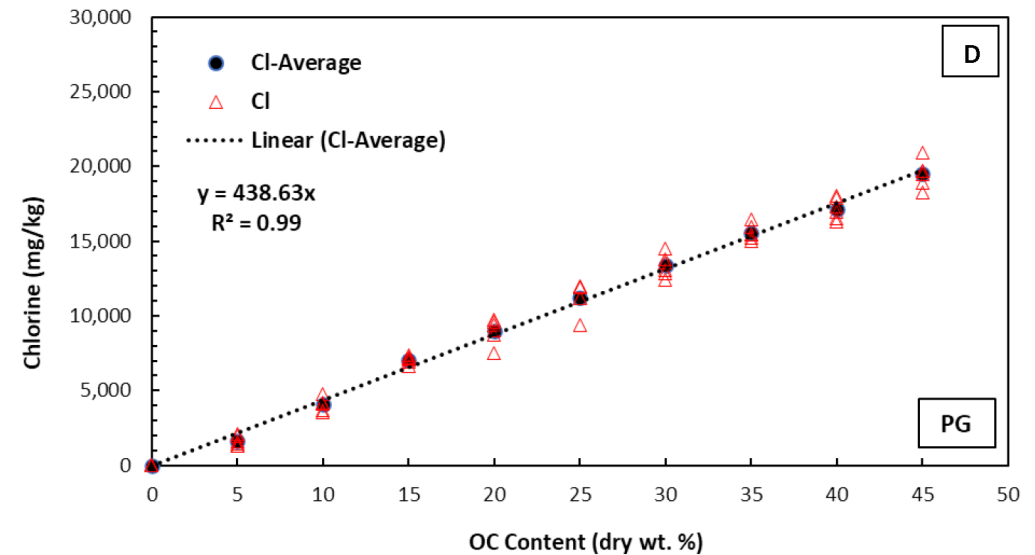
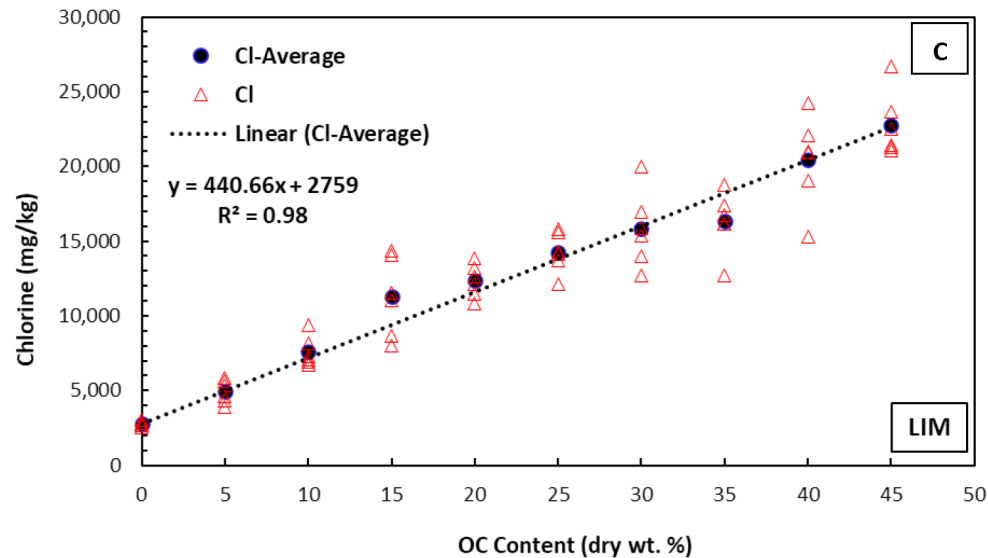
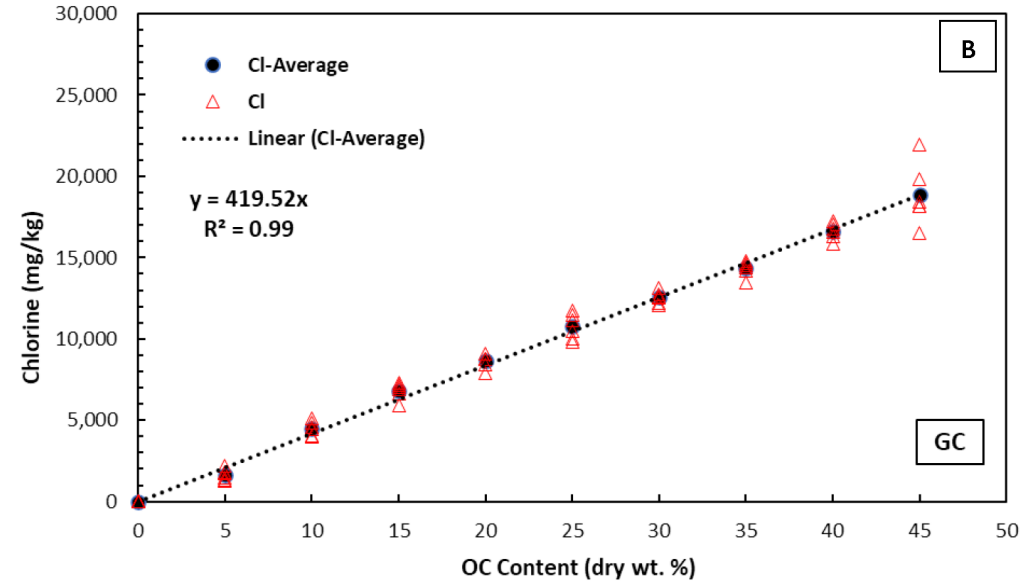
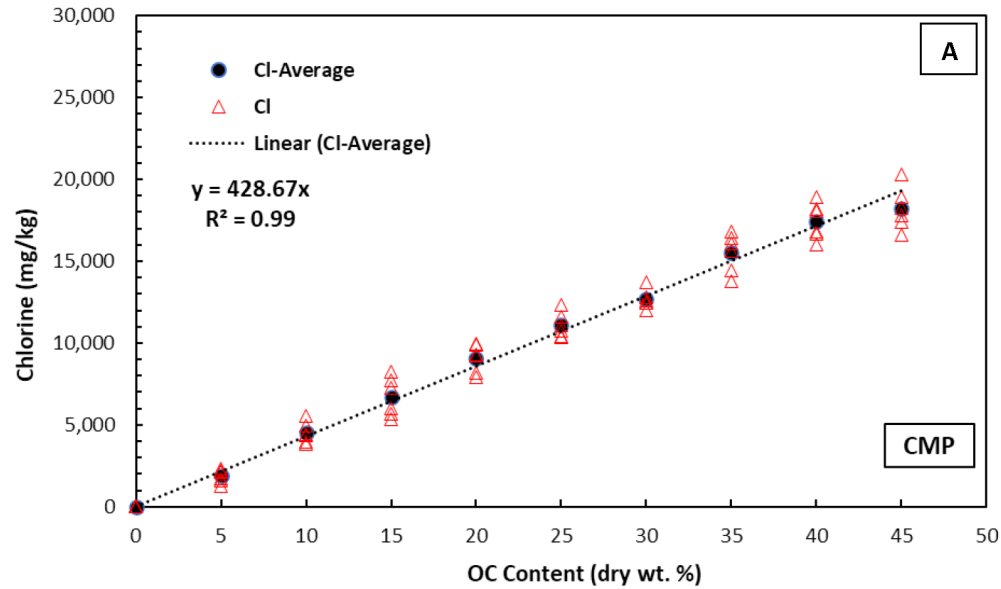
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# Organoclay (OC) Approach

- Physical crusher did not work for OC (too malleable), needed different approach
- Expand study to include 4 mineralogically different sands
  - Gowanus Canal (GC) sand (quarried)
  - Cape May Point (CMP) New Jersey beach sand (marine)
  - Playground (PG) Sand from big box store (general “sand”)
  - Limestone (LIM) sand from Chicagoland region for Great Lakes applications (Karst)
- Also tested Flourosorb 400 (FS) used for PFAS treatment since in OC family.
- Seek out pathfinder elements. Chlorine/Bromine obvious choices due to OC manufacturing processes. Common bentonite source will likely produce others.

# OC/Sand Testing Results -- Chlorine



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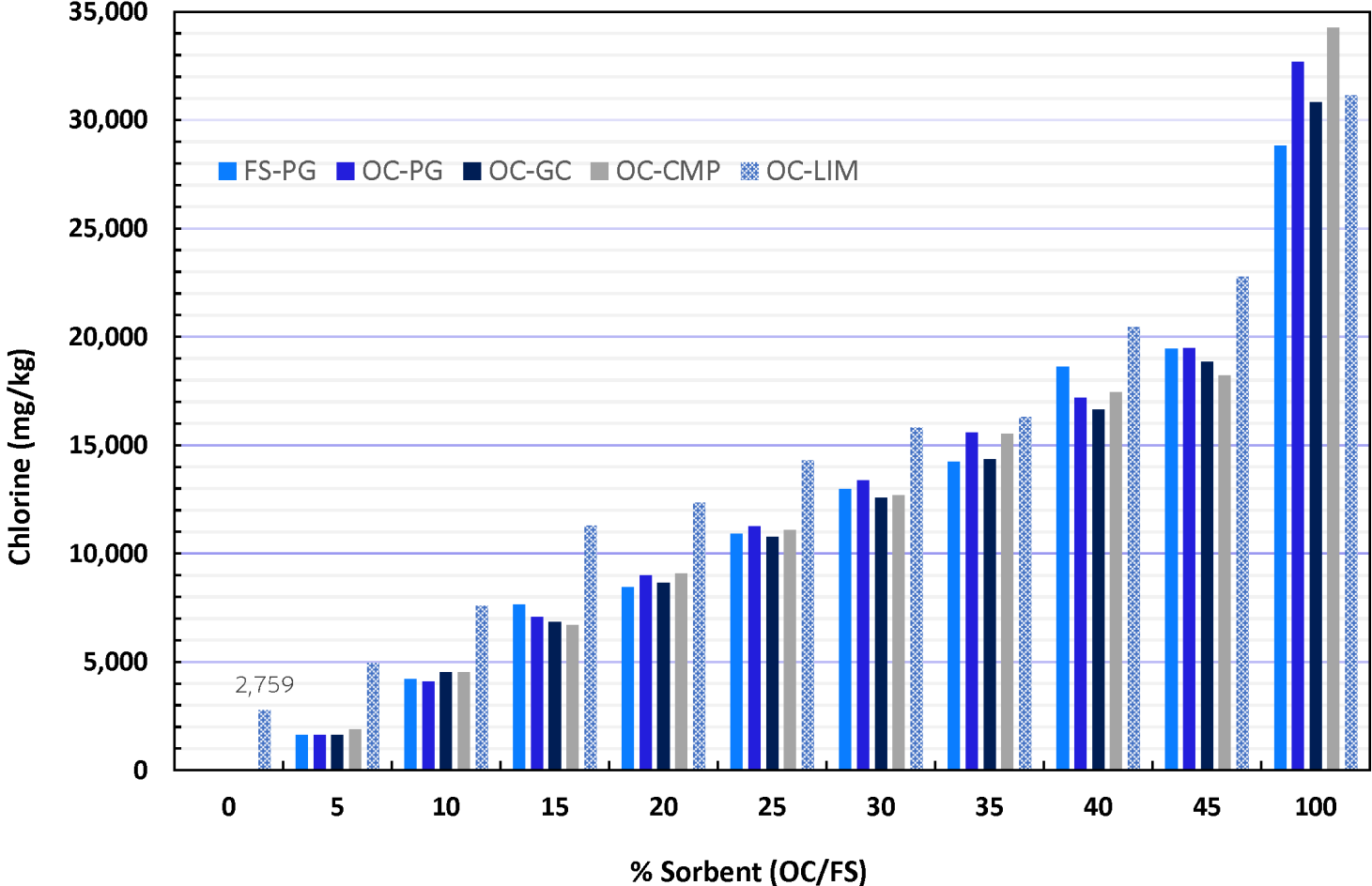
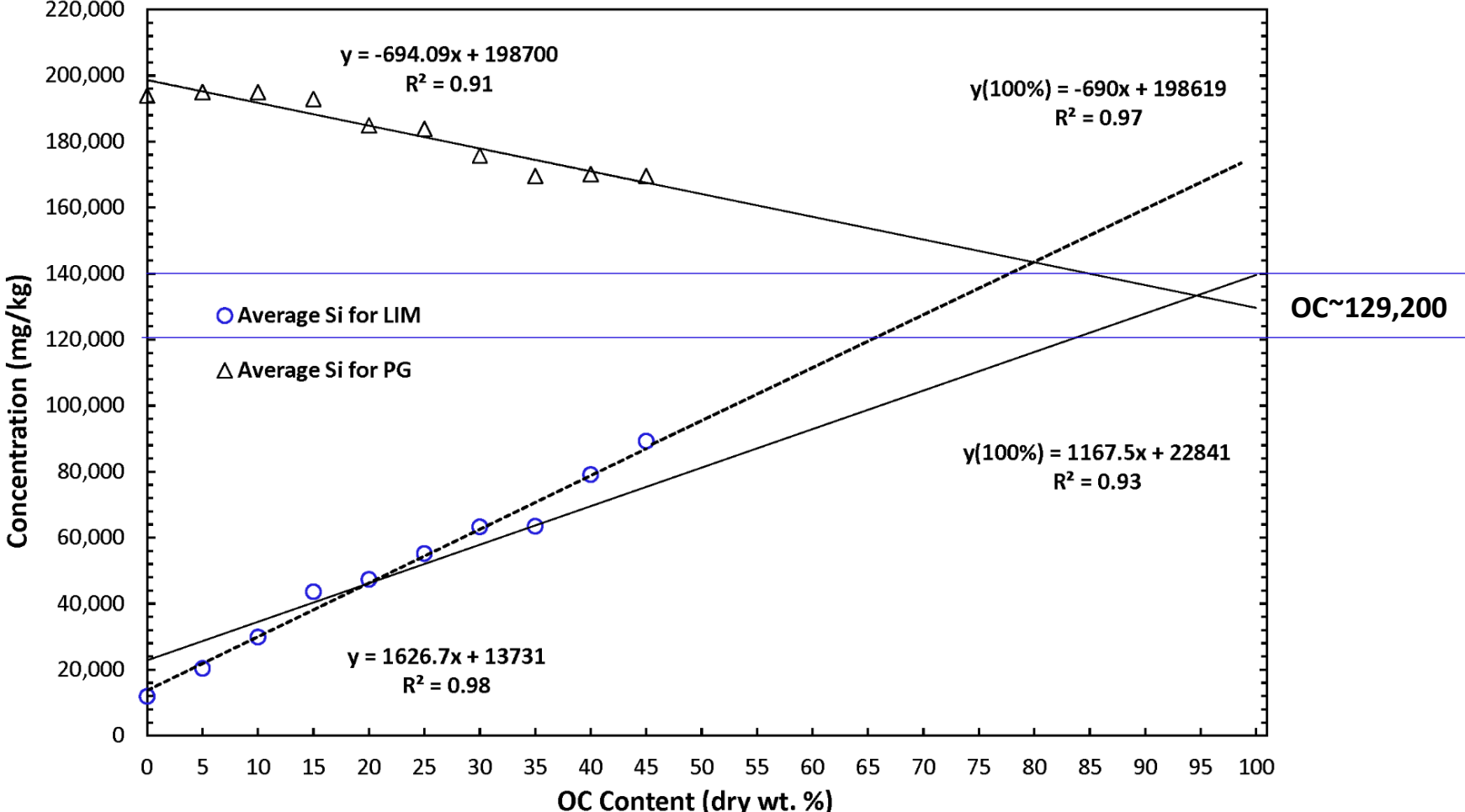


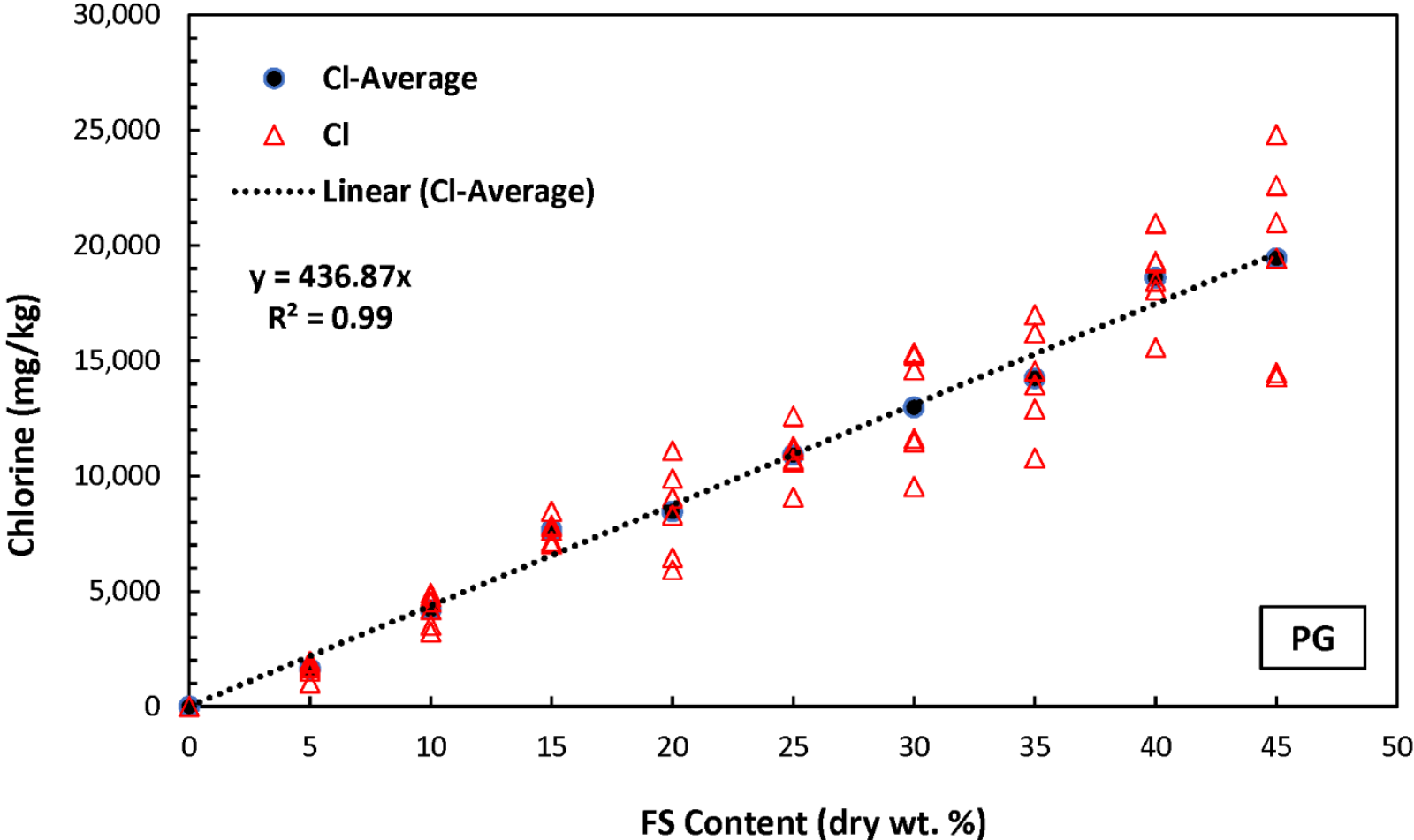
Figure 3. Average Cl concentration for each sand across all OC and FS sand blends grouped by sorbent content from 0 to 100% OC or FS and for pure test media.

# OC/Sand Testing Results -- Silicon



**Figure 7.** Relationship between average Si concentrations and OC content for LIM and PG blends. The y(100%) correlations include 100% OC.

# FS/Sand Testing Results -- Chlorine



**Figure 2.** Correlation between chlorine concentration and FS content in the blend range of 0 to 45% FS in PG sand.

# OC/Sand Testing Results – Ca and Mg

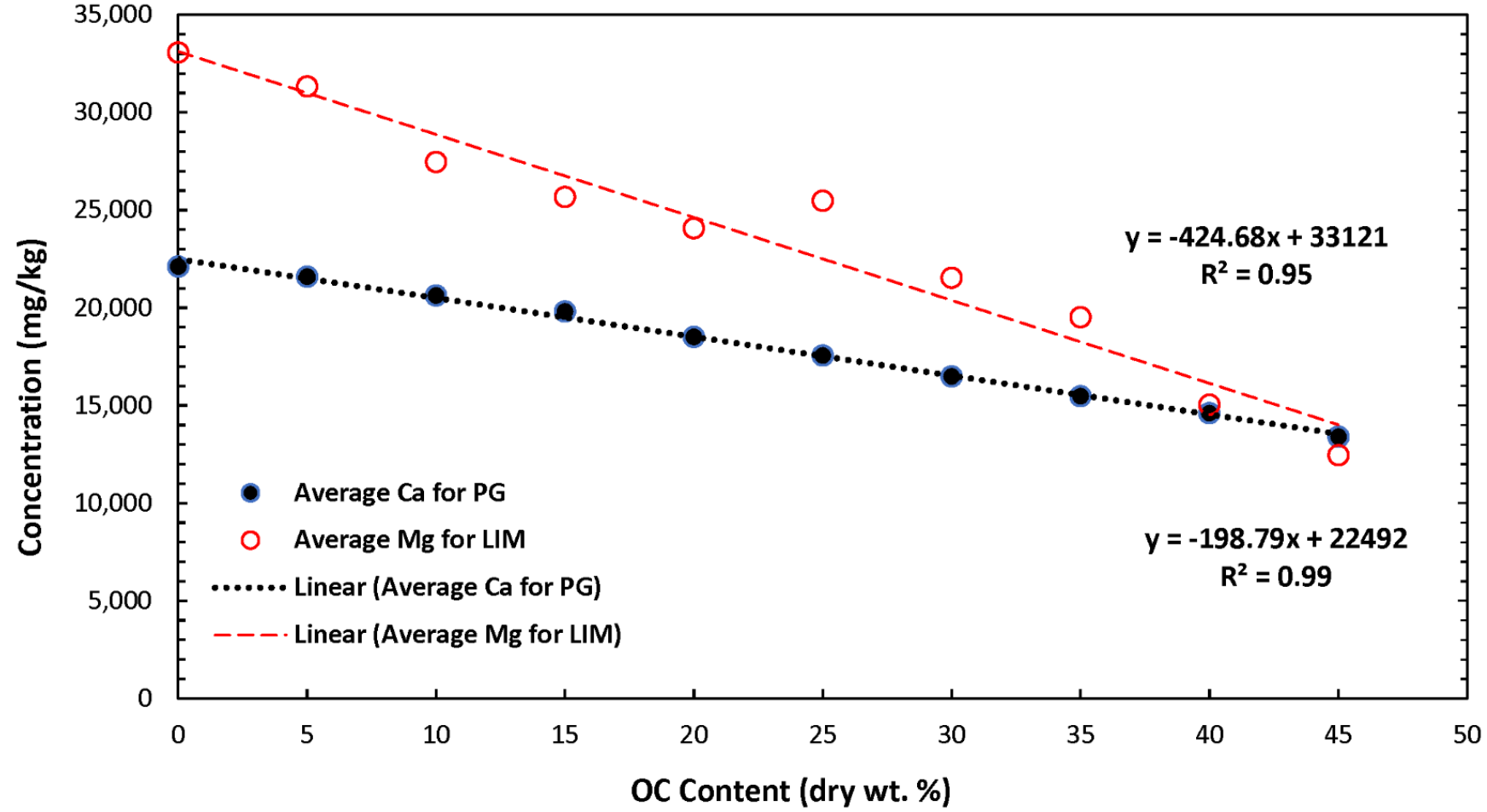


Figure 6. Relationship between average Ca and Mg concentrations and OC content for PG and LIM sands, respectively.

# OC/Sand Testing Results – Ti, Ca, Fe for PG sands

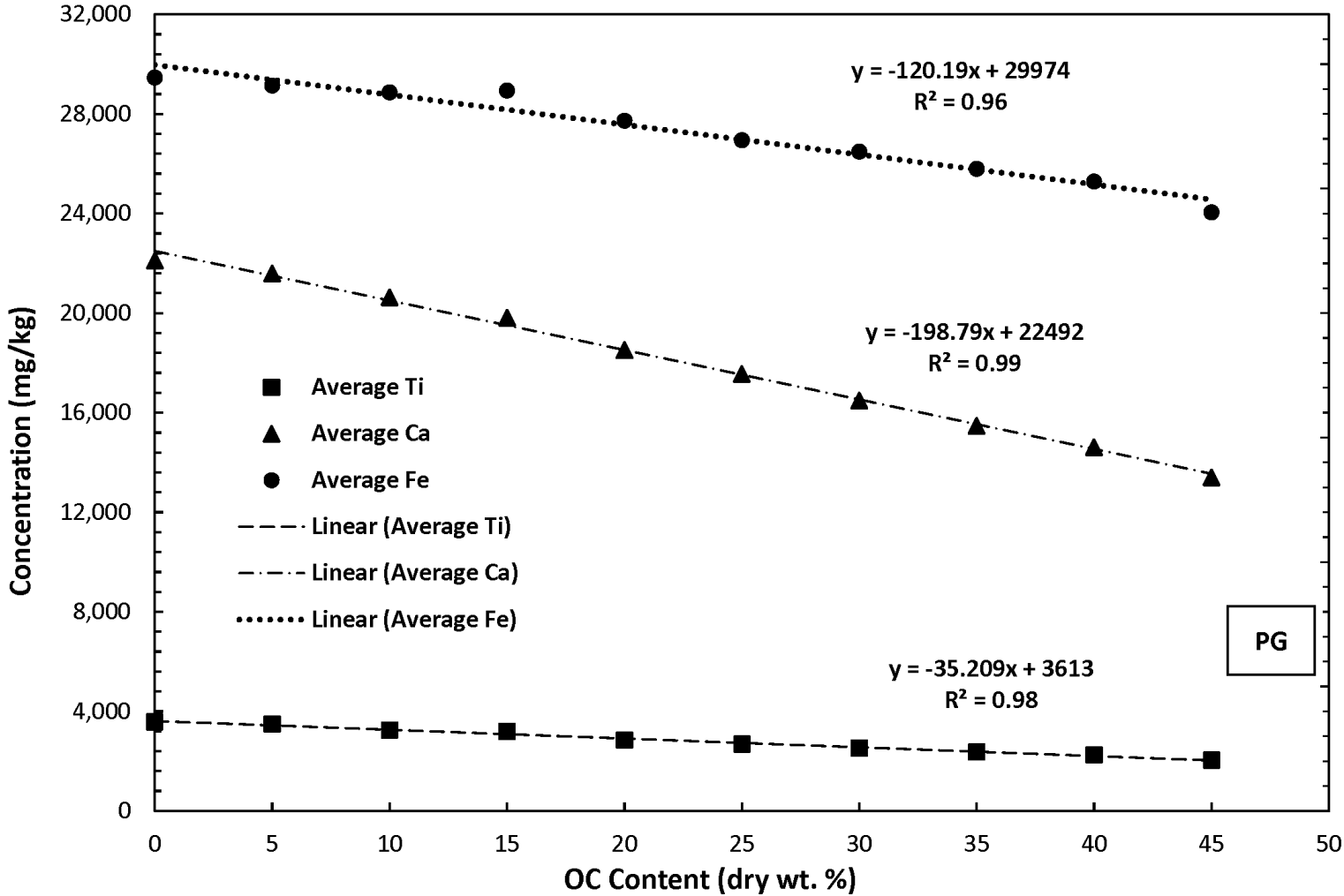


Figure 8. Relationship between average Ti, Ca and Fe concentrations and OC content for OC-PG blends.