

Innovation & Technology

Saint Quentin, 2023/07/12,  
Geopolymer Camp

Miguel Otero\*, Lorena Freire.

**Alternative applications of  
geopolymers in the environmental  
industry**



**Testing & Analysis**

**Industrial Services**

**R&D&i**

**690**  
R&D&i Projects  
In the last 10 years

**6**  
Active Patents  
As of 31/12/2020

**+750**  
Clients  
Annual average  
In the last 5 years

**280**  
Employees  
39 PhDs  
61% men  
39% women

**15,6 M€**  
Annual Income  
2019

**292.435**  
Technical Reports  
As of 31/12/2020

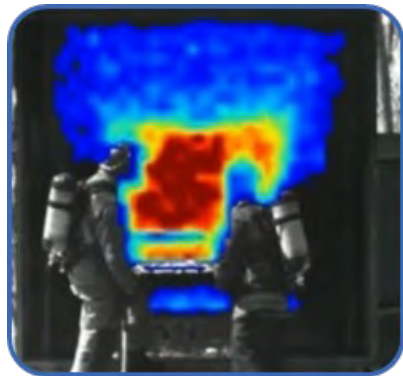
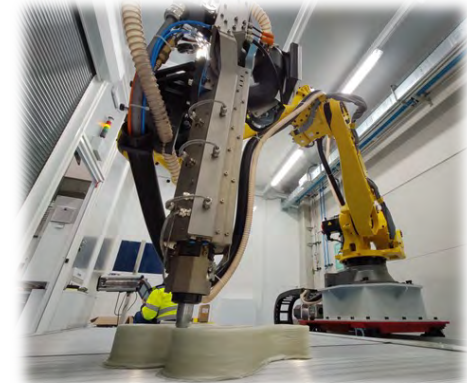
Experts in



- Laser technologies
- Industrial robotics and 3D printing
- Environmental approaches
- Advanced materials**



**Geopolymer research line**





## Environmental applications:

Management of radioactive wastes



Adsorption in wastewater treatments



## Construction applications:

### *Precast panels*



Insulation



Drainage

### *Small buildings*



### *High temperature resistant geopolymer*



### *Future: Geopolymer 3D Printing*

### *Electrically conductive geopolymer*



## Introduction:

### Organic Liquids cementation

- ❑ **OPC:** Organic liquids **affects hydration reactions** and **phase separation occurs.**
- ❑ **Geopolymers:** Their reaction **is not affected by the nature of the O.L.**

## METAKAOLIN-BASED GEOPOLYMER

### Parameters:

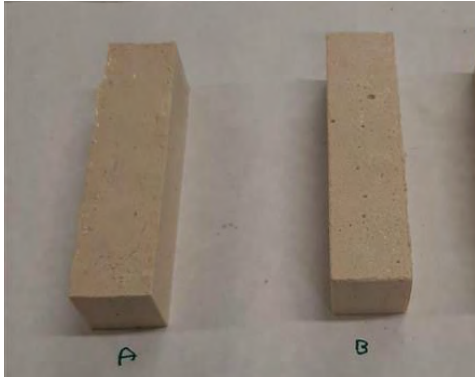
- **Incorporation methods:** Direct and pre-emulsión
- **O.L. :** engine oil and scintillation cocktail.
- **Activators:** Na and K silicates.
- **Curing treatments:** Ambient or heat.



### Goals:

- Compressive strength > 10 MPa
- O.L. Immobilization > 30% v/v without bleeding and leaching

## Results:



Bleeding or phase separation

### DIRECT vs PRE-EMULSION:

Pre-emulsion ↑ bleeding

O.L addition:  
↓ strength

O.L. wt%	Compressive strength/MPa
0	41,6
	45
10	36,8
	38,7
20	22,6
	22,9
25	17,4
	18,9
30	9,8
	8,7

Preparation	Curing	Bleeding	Mean
DIRECT	Room temperature	1,68%	1,20%
		0,85%	
	heat	2,99%	3,05%
		3,12%	
PRE-EMULSION	Room temperature	6,78%	4,46%
		2,93%	
	heat	Very high!	

### Curing treatments:

Heat: ↓ strength ↑ bleeding

Nomenclature	Curing	Flexural strength/Mpa	Compression strength/Mpa
Pre30AmbA	ambient	1,9	9,7
Pre30AmbB		1,7	10,5
Pre3080A	80 °C 2h	1,6	6,7
Pre3080B		2	6,7

## Leaching tests:

### *Control sample*

Sample	Days	HCO <sub>3</sub> <sup>-</sup> (ppm)	OC mgC/L	TC	IC
0% waste	3	605	4	123	119
	7	294	0	61	58
	14	97	0	19	19
	21	10	0	2	2
	28	11	0	2	2



### *Oil*

Samples	Days	HCO <sub>3</sub> <sup>-</sup> (ppm)	OC mgC/L	TC	IC
OIL-30%	3	737	12,4	158	145
	7	219	0	43	43
	14	49	0	10	10
	21	24	0	5	5
	28	16	0	3	3
	35	35	0	7	7
	42	23	0	5	5
	70	37	0	7	7

Oil-40%	3	416	1,2	83	82
	7	165	0	33	33
	14	5	0	1	1
	21	63	0	12	12
	28	30	0	6	6
	35	14	0	3	3
	42	6	0	1	1
	70	3	0	1	1



### *Scintillation cocktail*

Sample	Days	HCO <sub>3</sub> <sup>-</sup> (ppm)	OC mgC/L	TC	IC
Scintillation cocktail 30%	3	214	37	79	42
	7	1306	96	352	257
	14	105	29	50	21
	21	50	23	33	10
	28	29	21	27	6

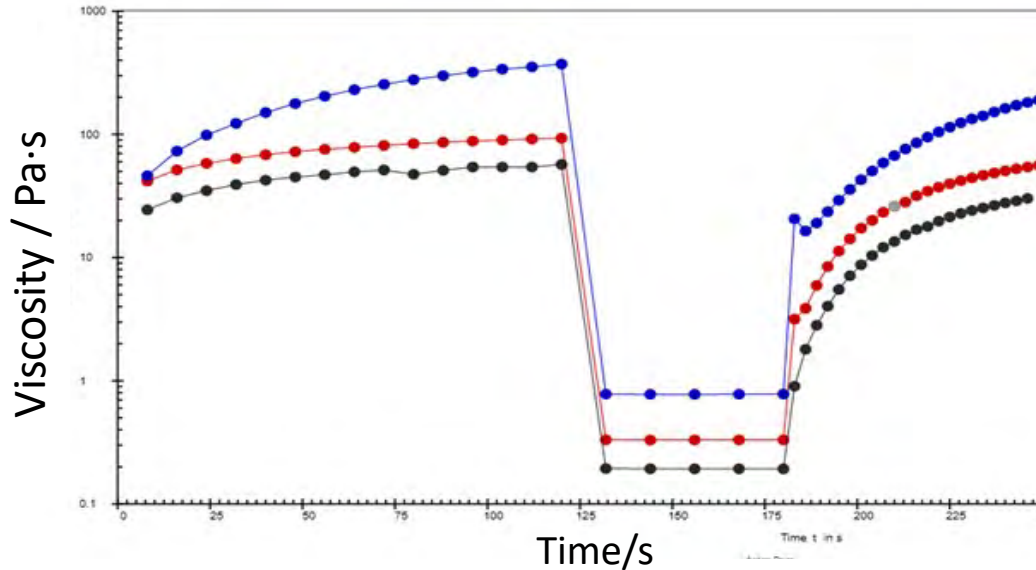
Scintillation cocktail 20%	3	147	35	64	29
	7	178	31	66	35
	14	259	29	80	51

Scintillation cocktail 10%	3	275	29	83	54
	7	158	24	55	31
	14	122	21	45	24

## Rheology:

### 3-INTERVAL TEST

Low shear rate / High shear rate / Low shear rate



Black 0%, red 20% and blue 40%

### Best results

*(without bleeding or leaching)*

- 38% v/v of oil obtaining 18MPa with Na activator.
- 40% v/v of oil obtaining 14MPa with K activator.

↑ % O.L. ↑ viscosity

Dynamic viscosity (mixer):

- 40%=0,72 Pa·s,
- 20%=0,32 Pa·s
- 0%=0,18 Pa·s

Static viscosity (after mixing):

- 40% = 431,2 Pa·s
- 20%= 93,3 Pa·s
- 0%= 57,05 Pa·s

Recovery of viscosity (after 30sec)

- 40% = 18%
- 20%= 27%
- 0%= 26%



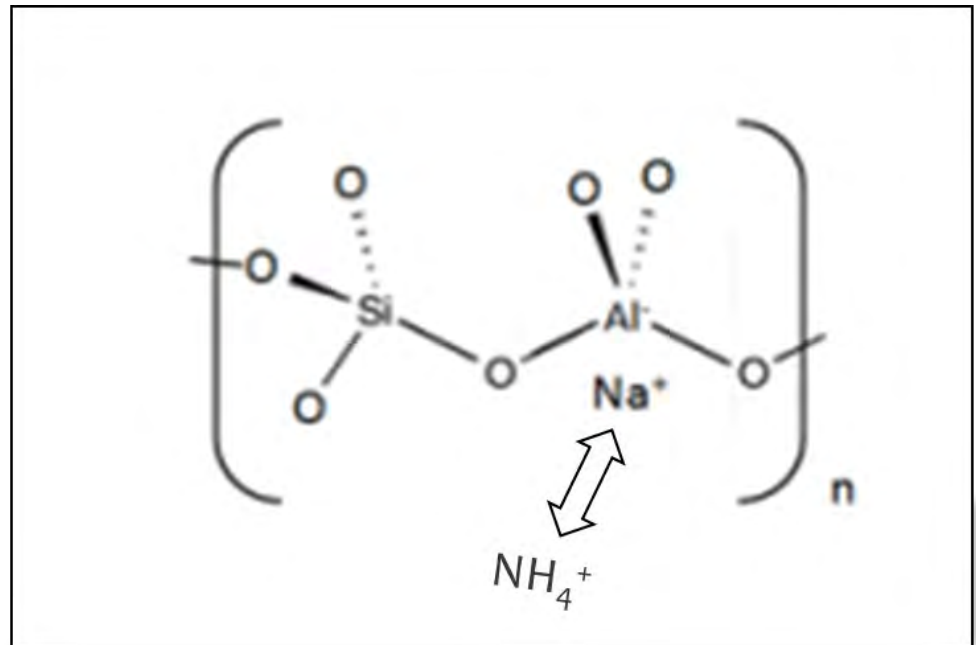
## Introduction:



Ammonium:  $\text{NH}_4^+$   
Eutrophication

### Geopolymer good adsorbent:

- Chemistry structure:** sodium cations easily exchangeable.
- Easily modified** to increase porosity.
- Sustainable and low operational cost adsorbent.**



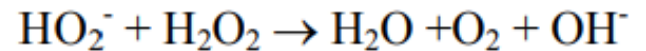
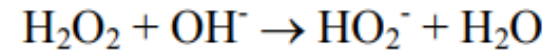
*Ionic Exchange process*

## Promising adsorbent formulation:

Parameters	Units	Values
SiO <sub>2</sub> /Al <sub>2</sub> O <sub>3</sub>	Molar ratio	1,5
SS/NaOH	Molar ratio	1,2
NaOH	Molarity	10
Granite waste	%Substitution	20
H <sub>2</sub> O <sub>2</sub>	%	1
Hardening time	Hours	24
Curing temperature	°C	25

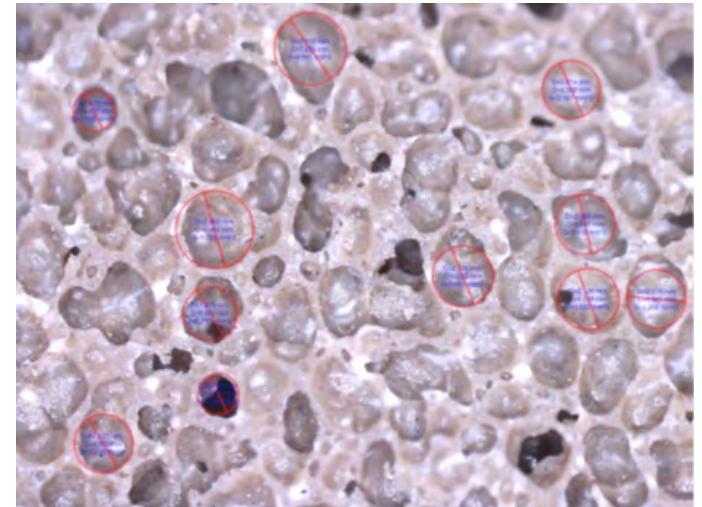
**Metakaolin-based porous geopolymer**  
**20% of granite waste (GW)**

**1% H<sub>2</sub>O<sub>2</sub> foaming agent**



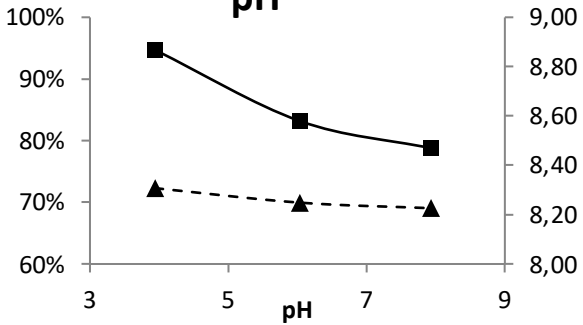
**6,1MPa compressive strength**

**58,99% porosity**

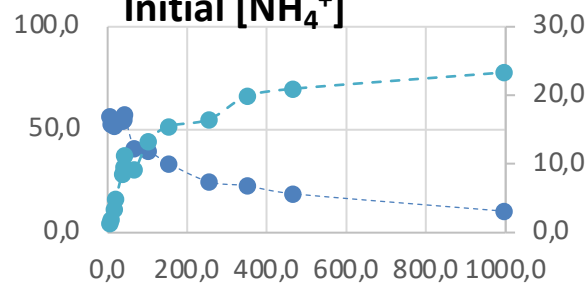


## Batch tests

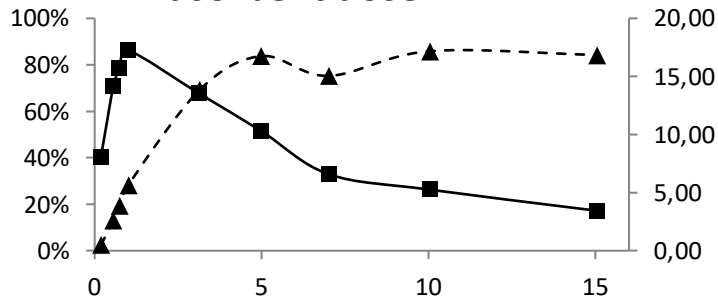
**pH**



**Initial [NH<sub>4</sub><sup>+</sup>]**



**Adsorbent dose**



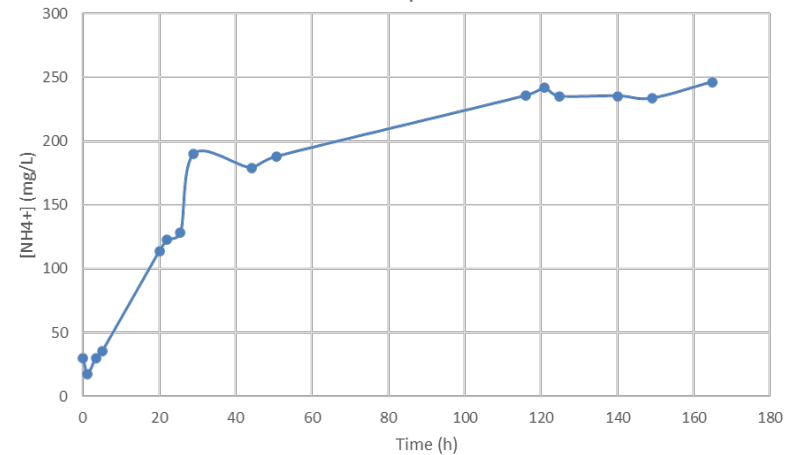
**$Q_m = 23\text{mg/g}$**

**highest removal: 5g/L and pH=4**

## Continuous tests:



Column adsorption test



**First hours: 93% ammonium removal**

**One day: 50% ammonium removal**

**120h: saturation**

## 125L cylinder geopolymer (Container)

## 125L grave geopolymer (Wetland)



## *Pilot plant (Xiloga Landfill, Galicia-Spain)*



## Results

Monitoring for 1 day every hour.

	Tank	Container (cylinder)	Wetland (grave)
pH	12,2	11,6	13,3
EC (mS/cm)	22,5	11,7	60,4
NH <sub>4</sub> <sup>+</sup> (mg/L)	91	59	17



**Total removal: 80%**  
Slightly superior to zeolites



High electrical conductivity  
High pH due to alkalis leaching



## Summary

### **Geopolymer as radioactive waste immobilizer:**

- Direct method and ambient curing best techniques
- Scintillation cocktail was leached more than oil.

Best results:

- 38% v/v of oil obtaining 18MPa with Na activator.
- 40% v/v of oil obtaining 14MPa with K activator.

### **Geopolymer as $\text{NH}_4^+$ adsorbent:**

- Sustainable and low operational cost adsorbent.
- Lab tests: 93% ammonium removal first hours and  $q_m=23\text{mg/g}$ .
- Pilot plant: 80% ammonium removal, slightly superior to zeolites.

Future works:

- ❖ Techniques/treatments to reduce the alkalis leaching.
- ❖ Treatments to reuse adsorbents in several adsorption cycles.
- ❖ 3D printing: Innovative designs to maximize adsorption

**Headquarters**

**Laser Applications Centre**

Polígono Industrial de Cataboi  
SUR-PPI-2 (Sector 2) Parcela 3  
E36418 O PORRIÑO  
Pontevedra - Spain  
Phone: +34 986 344 000

**Torneiros Facilities**

**Armando Priegue Building**

Relva 27 A – Torneiros  
E36410 O PORRIÑO  
Pontevedra – Spain  
Phone: +34 986 344 000

**A Coruña Office**

Polígono Industrial de Pocomaco  
Parcela D-22 Oficina 20  
E15190 A Coruña - Spain  
Phone.: +34 637 127 253

**Madrid Office**

C/ Rodríguez San Pedro, 2  
Planta 6, Oficina 609 Edificio Inter  
E28015 Madrid - Spain  
Phone: +34 687 448 915

[aimen@aimen.es](mailto:aimen@aimen.es)  
[www.aimen.es](http://www.aimen.es)

**Thanks for your attention**



**Miguel Otero Sáenz de Lubiano** [miguel.otero@aimen.es](mailto:miguel.otero@aimen.es)

**+34 98634400 Ext. 3210**

**Lorena Freire Piñeiro** [lorena.freire@aimen.es](mailto:lorena.freire@aimen.es)

**+34 98634400 Ext. 3210**