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École Mines-Télécom
IMT-Université de Lille

Geopolymer applications for concrete and brick technology.

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- **Mahfoud BENZERZOUR**
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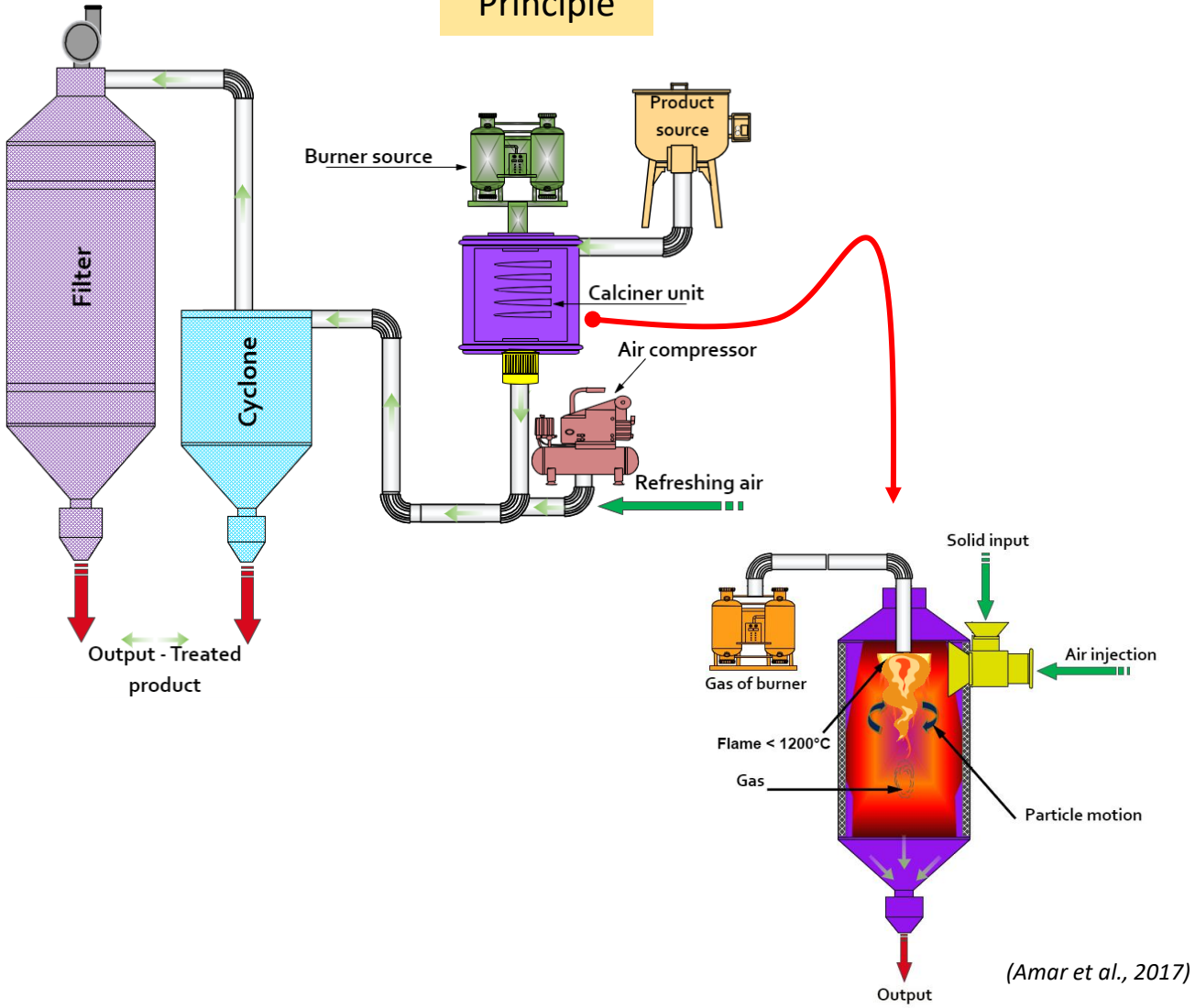
Calcination/Activation of mineral materials

Flash calcination principle

Principle



(Amar et al., 2017)

Advantages

Quickness

- The process takes about **1 s**
- Good flow rate (about a minimum **Q = 10 kg/h**)

Cost

- **Low-energy** consumption
- **No post-processing** needed

Activation

- **Pozzolanic activation** of materials
- Removal of **organic matter**
- Reduction of **pollutant** content

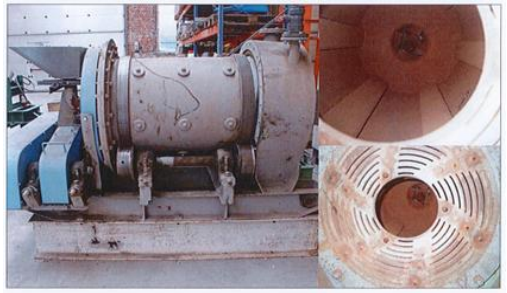
Adaptability

- **Adaptable** to different types of materials **(clay, sludge, soil, etc.)**
- Different energy sources **(gas, electricity, PV)**
- **Simple process**

Calcination/ Activation of mineral materials

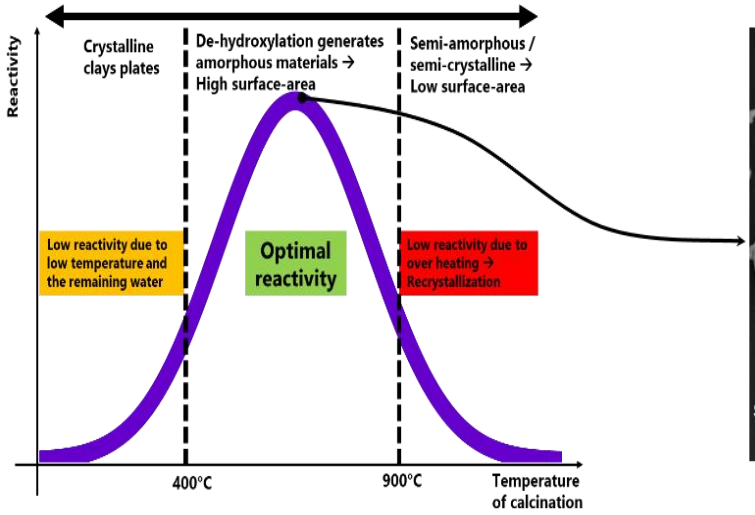
Flash calcination principle

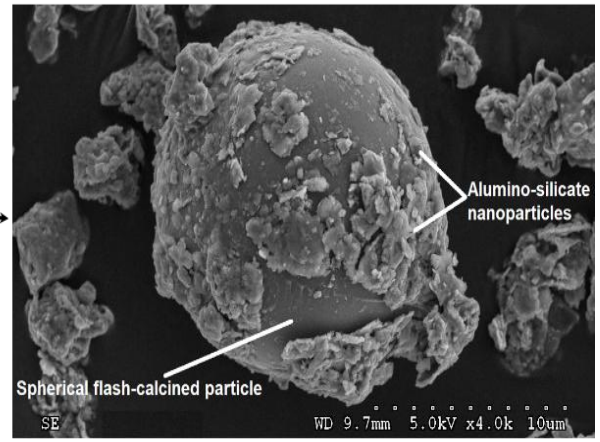
- Fine grinding after drynig (before calcination)



Broyeur à boulets

- Optimization of initial characteristics





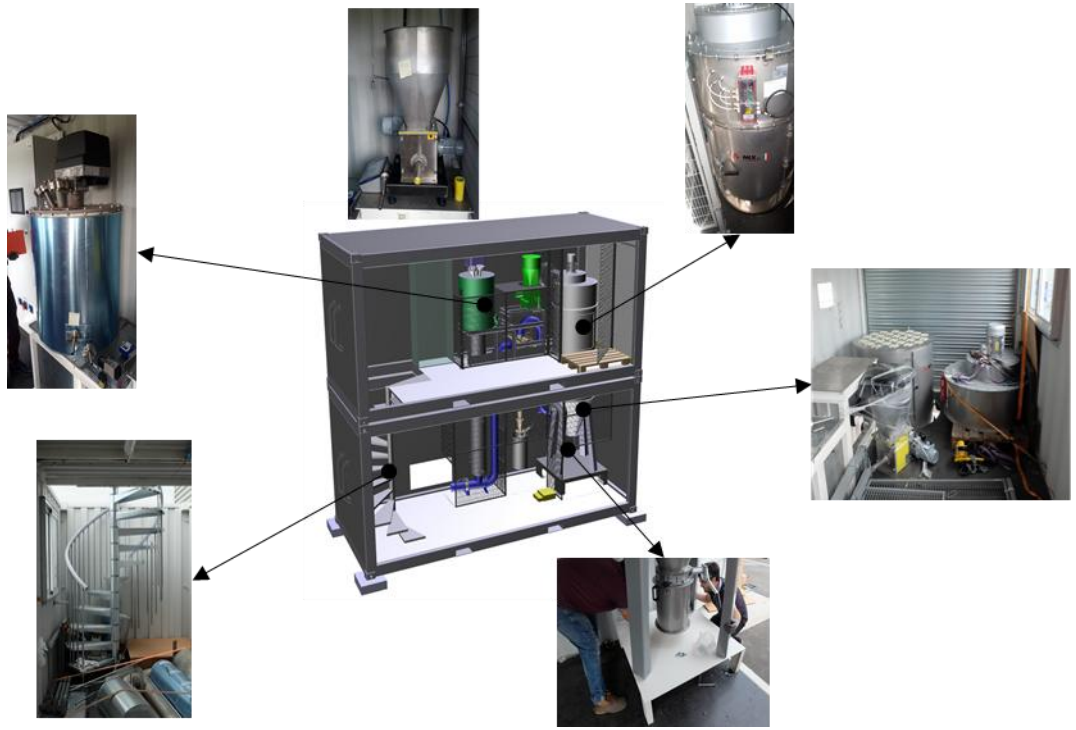
Calcination/ Activation of mineral materials

Flash calcination principle

- Pilot unit







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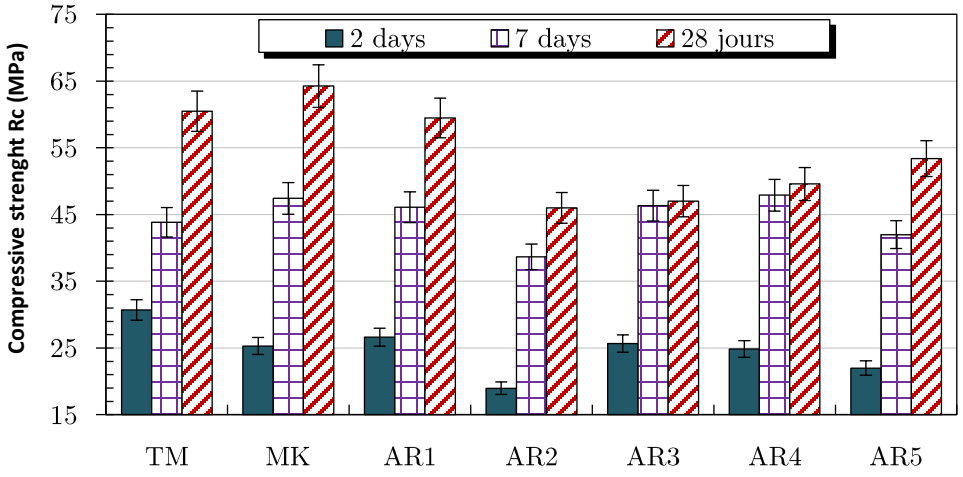
RÉPUBLIQUE FRANÇAISE
Liberté
Égalité
Fraternité

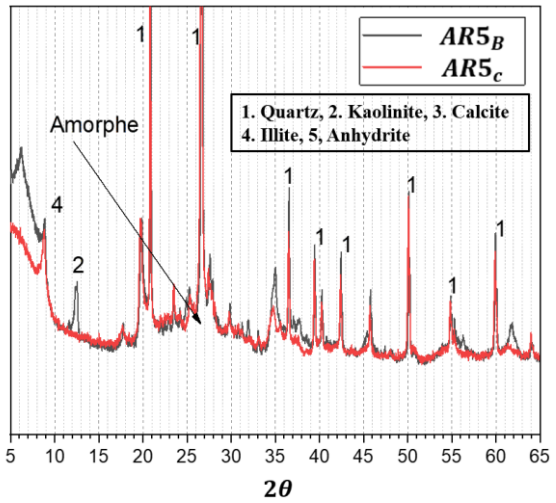
Calcination/ Activation of mineral materials

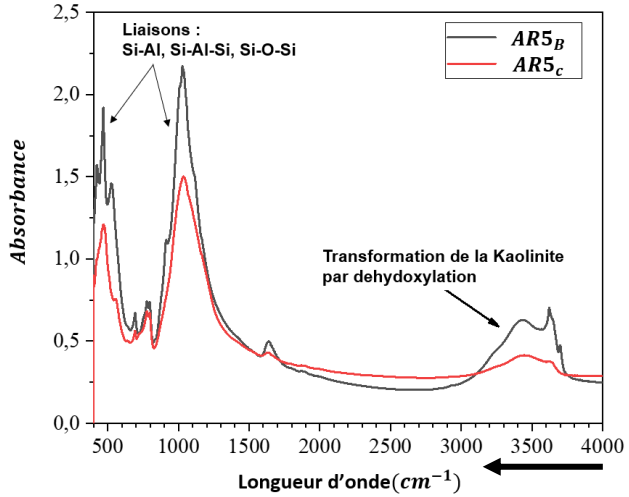
Flash calcination principle

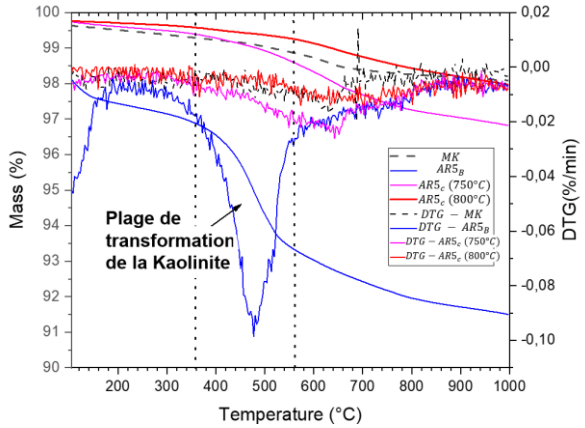
Tests and characterization results

Materials	AR1	AR2	AR3	AR4	AR5
Composition			Chemical composition		
Al_2O_3	22,41	10,99	7,29	9,98	17,64
CaO	1,42	12,23	16,27	9,1	0,91
			Mineralogical composition		
Clay content	79	68	29	48	69
Smectites	31	7	13	24	46*
Illite-Mica	2	39	4	11	17
Kaolin	46	6	-	11	6
Palygorskite	-	13	10		-
BET - Raw	47,15	16,10	15,53	21,14	44,87
BET - Calcined	45,73	57,52	20,83	25,61	22,41







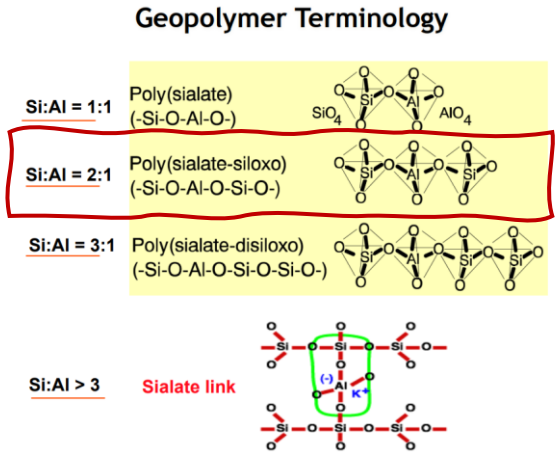


- Physico-mineralogical modifications: **density and fineness increase** if the material is rich in carbonates.
- The **reactivity** of the final product is correlated with the **fineness**, to the content in **aluminosilicates**.
- For materials rich in clays of the [T-O-T] type (**illite, smectite**, etc.), the degree of activation is **relatively limited**

Geopolymer synthesis

Formulation and optimisation

Formulation principle



$$M_2O \bullet Al_2O_3 \bullet 4SiO_2 \bullet 11H_2O$$

↓

Rigid 3D molecular structure

↓

Percentage of constituents to be defined

$$\frac{nk_2O}{nAL_2O_3} = \frac{mk_2O * MAl_2O_3}{Mk_2O * mAl_2O_3} = 1$$

1 The precursors

- Calcined clays → *Stabilisation*
- Excavated soils, sediments, fillers → *Source of aluminosilicates.*
- Slags → *Hardening*

2 Reagents

- Potassium silicate (Geosil®), sodium silicate → *Alcalination*
- Acids

Least reactive material + « Reagent » → xx min

Add the clays and other aluminosilicates → xx min

Add the slags that are the most reactive → xx min

Add the sand (and/or aggregates) → xx min

Cure at room temperature

Photo credit : Ali Allou

➢ Geopolymer Binders of High Strength and Durability cured at 20 °C

FCS

NSL river

FCC

GPE project

Waste Valorization

\+

MK

ARGICEM

Stabilization

\+

GGBFS

ECOCEM

Hardening

Percentage (%) of each precursor ?

Geosil 14157 Potassium silicate

WOELLNER

Alkaline Reagent (AR)

AR/B ratio ?

➢ Geopolymer Formulations

	Weight (%)				Ratios		
Mix	FCS	FCC	MK	GGBF	AR/B	S/B	W/B
Composition							
Reference mix							
MK0	-	-	100	-	0.8	2.64	0.45
Group with FCS							
S1	24	-	76	0	0.8	2.64	0.45
S2	20	-	70	10	0.8	2.64	0.45
S3	16	-	64	20	0.8	2.64	0.45
Group with FCC							
C1	-	27	73	0	0.8	2.64	0.45
C2	-	23	67	10	0.8	2.64	0.45
C3	-	19	61	20	0.8	2.64	0.45

- FCS: Flash Calcined Sediment
- FCC: Flash Calcined Clay
- MK: Metakaolin
- GBFS: Slag
- AR: Alkaline Reagent

Geopolymer Design

➢ Percentage (%) of each precursor

Elements	MK	GBFS	FCS	FCC
Si	27.4	14.9	20.8	28.82
Al	16.5	6.8	6.7	9.83
Si/Al	**1.66**	**2.19**	**3.10**	**2.93**

Si/Al=2

➢ Alkaline reagent – binder ratio (AR/B)

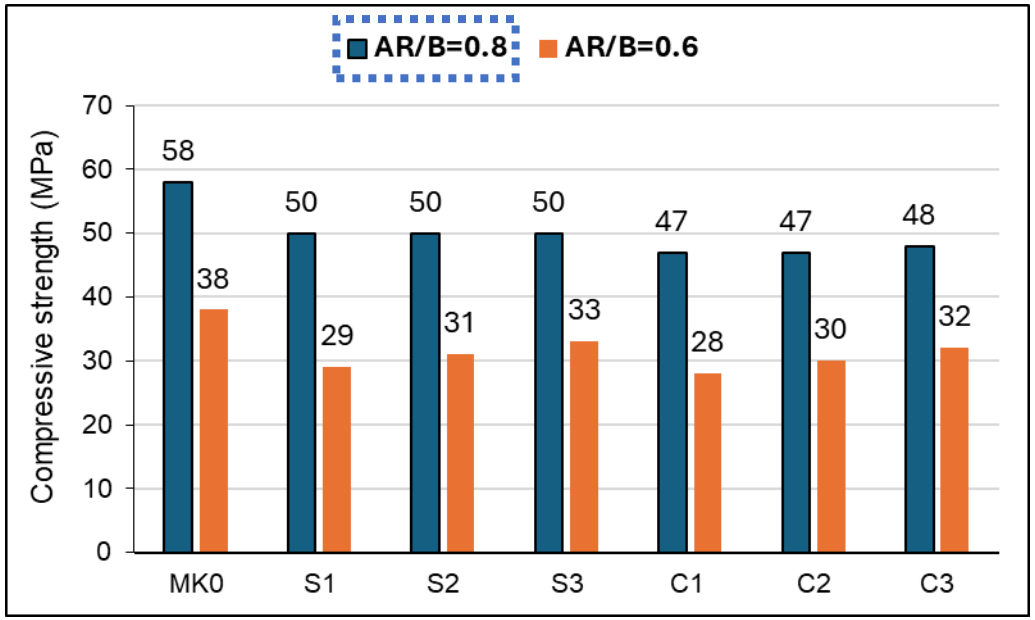
New Approach: Minimum AR needed

Workability + Compressive Strength

Ultimate Ratio
AR/B = 0.8

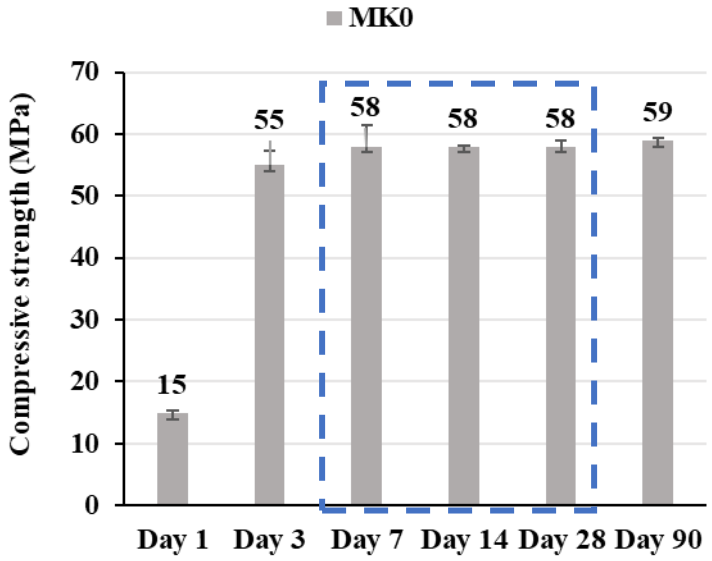
- **AR/B=0.4**

Not Workable



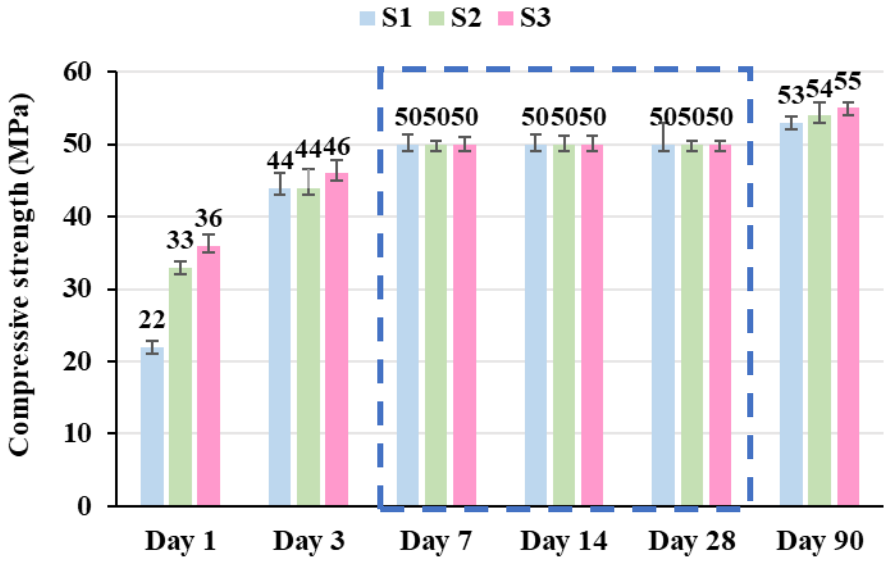
➢ Compressive strength results

MK0



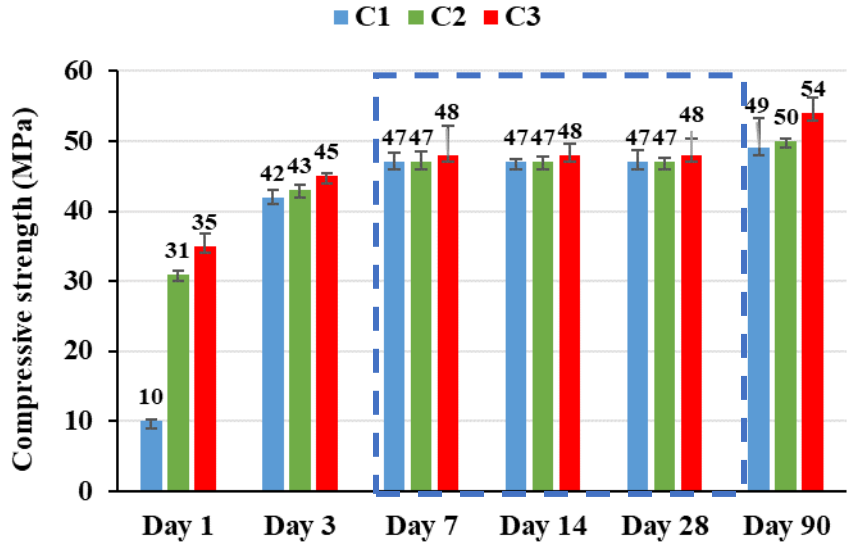
- Day 1= 15 MPa.
- Day 3= 55 MPa.
- Day 7-28 = 58 MPa.
- Day 90 = 59 MPa.

Group with FCS



- Day 1= 22-36 MPa.
- Day 3= 44-46 MPa.
- Day 7-28 = 50 MPa.
- Day 90 = 53-55 MPa.

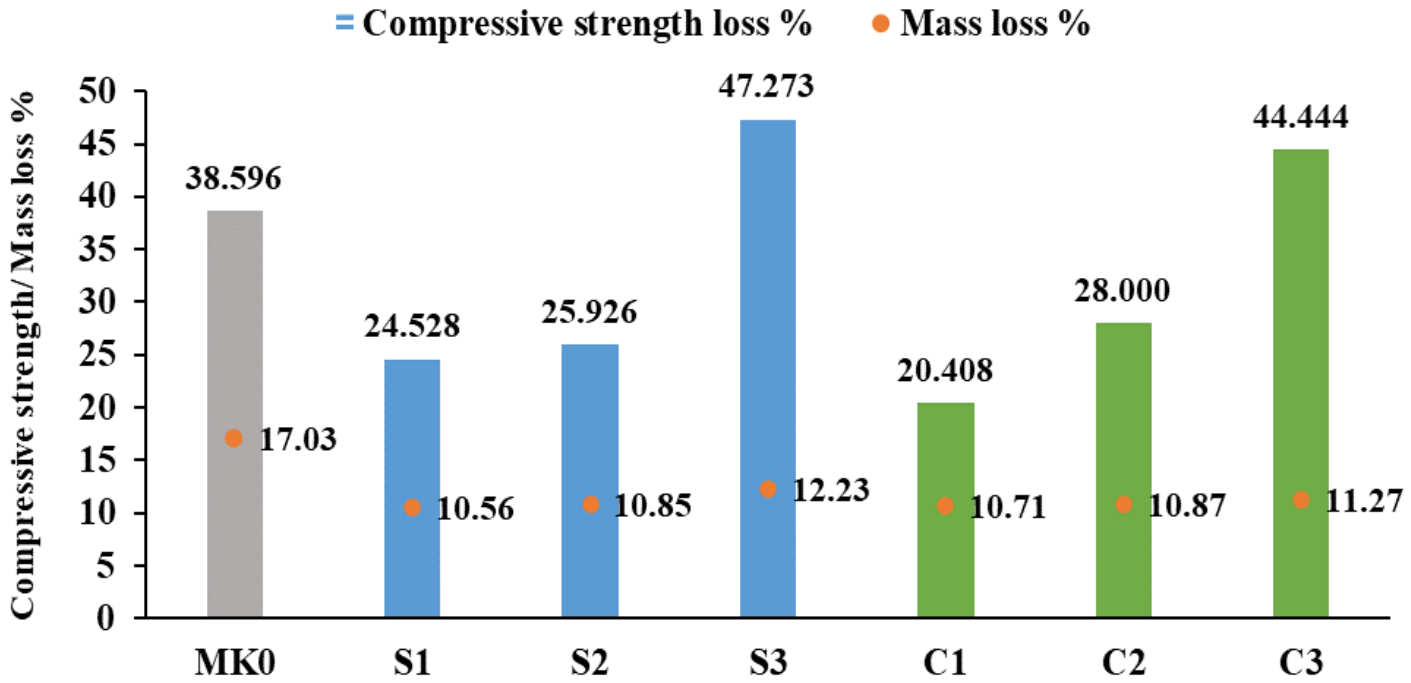
Group with FCC



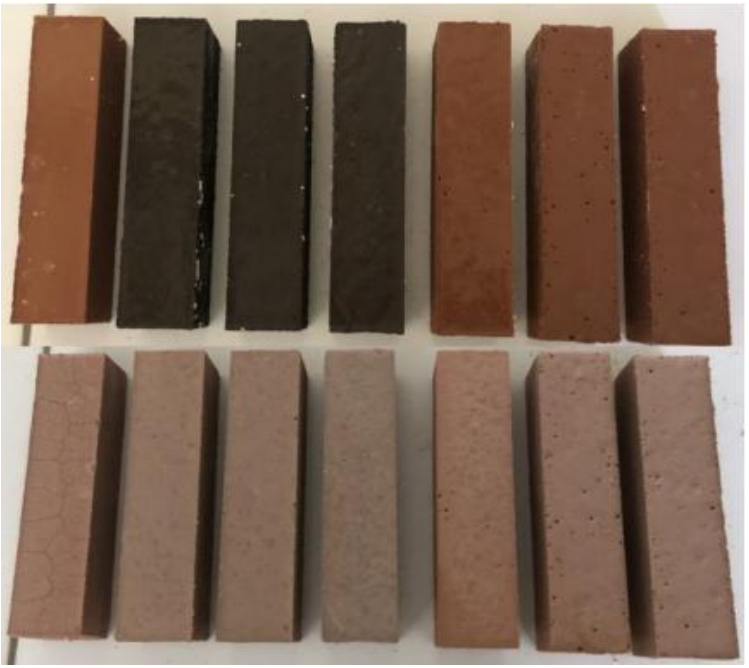
- Day 1= 10-35 MPa.
- Day 3= 42-45 MPa.
- Day 7-28 =47-48 MPa.
- Day 90 = 49-54 MPa.

➢ High-temperature resistance test

- Temperature range **200 °C to 400 °C**, range of **25 °C/min**.
- Temperature increased from **400 °C to 800 °C**, range of **5 °C/min**.



- Low loss in mass.
- S1, S2, C1, and C2 showed lower strength loss than MK0.
- S3 and C3 showed highest strength loss.
- As CaO content increased, resistance decreased.



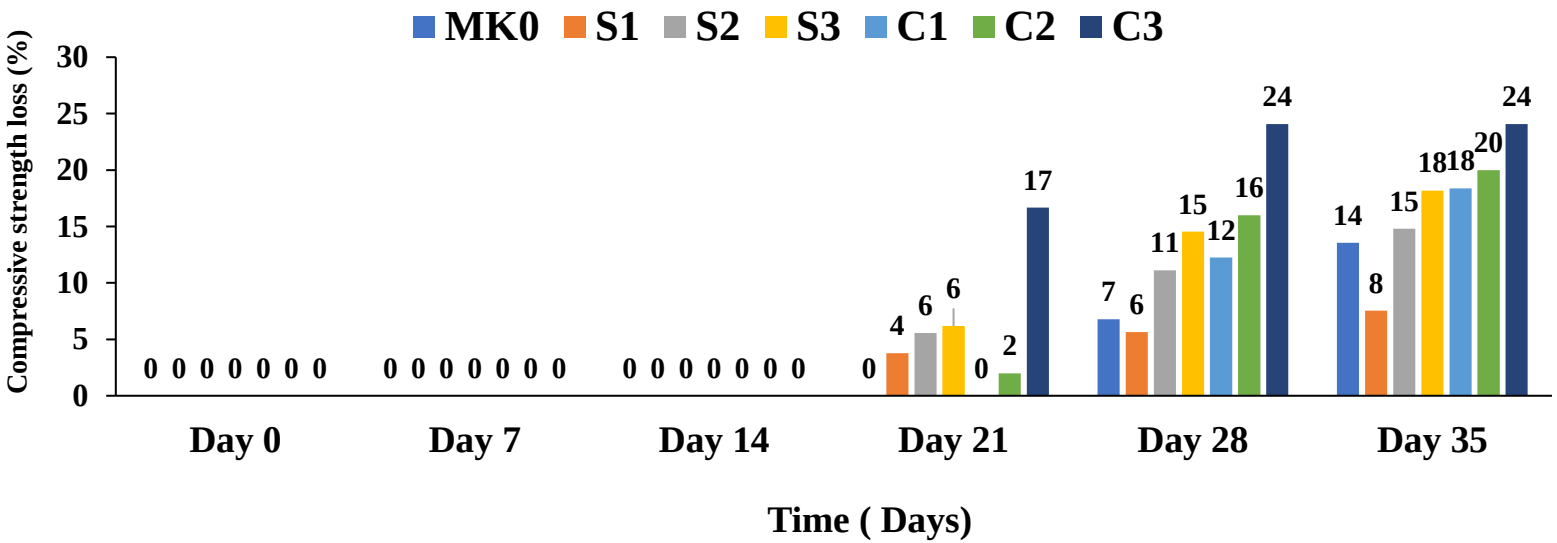
Only change in color, no cracks.

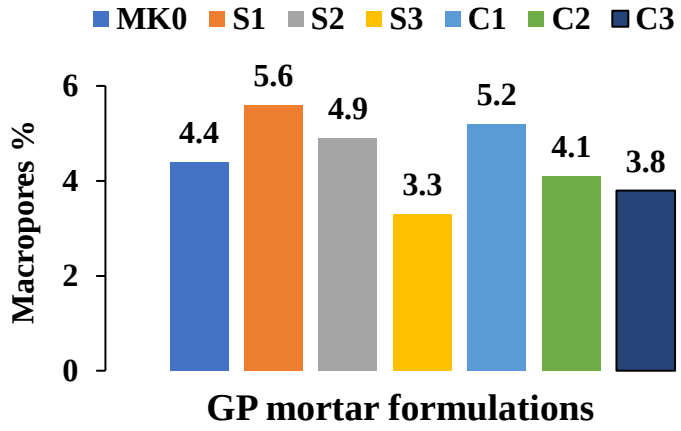
- **FCS Group: S1, S2, S3**
- **FCC Group: C1, C2, C3**

➢ Freeze and Thaw test (ASTM C666-97)

- **Cycle:**

1. **4 ± 2 °C for 6 hours.** 2. **-18 ± 2 °C for 18 hours.**





Slag % ↗

↓

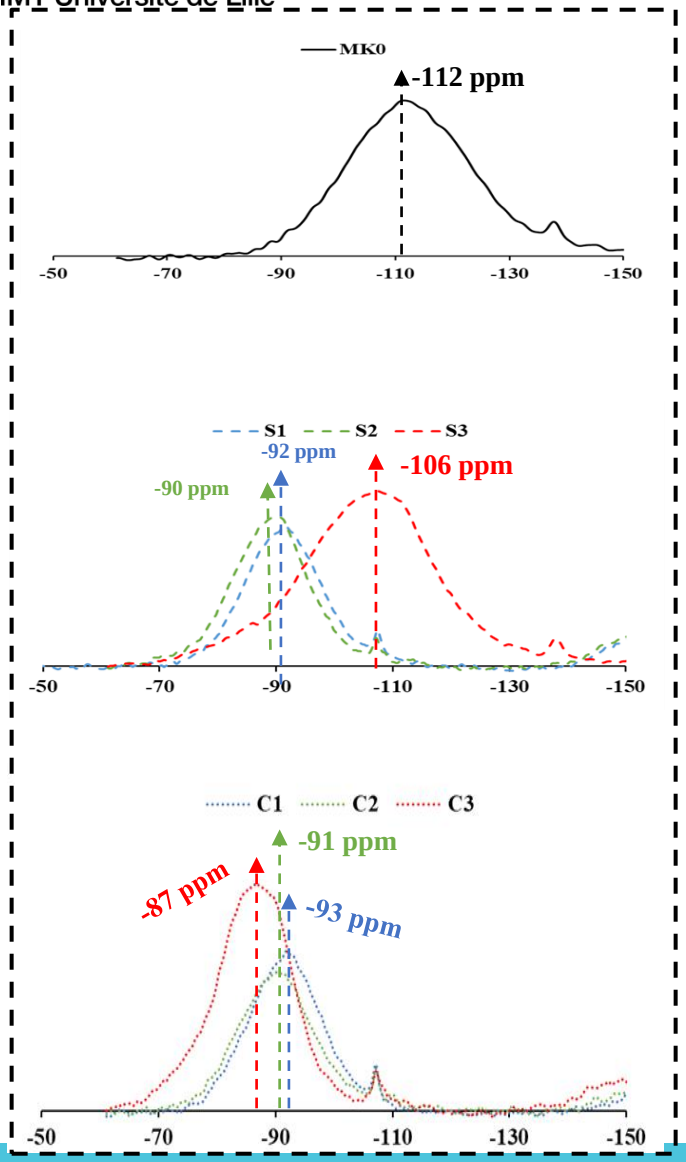
Macropores ↘

- **Day 35:** Linear increase in compressive strength loss.
- **MK0:** 14 %.
- **S1:** 8 %
- **S2:** 15 %
- **S3:** 18 %
- **C1:** 18 %
- **C2:** 20 %
- **C3:** 24 %

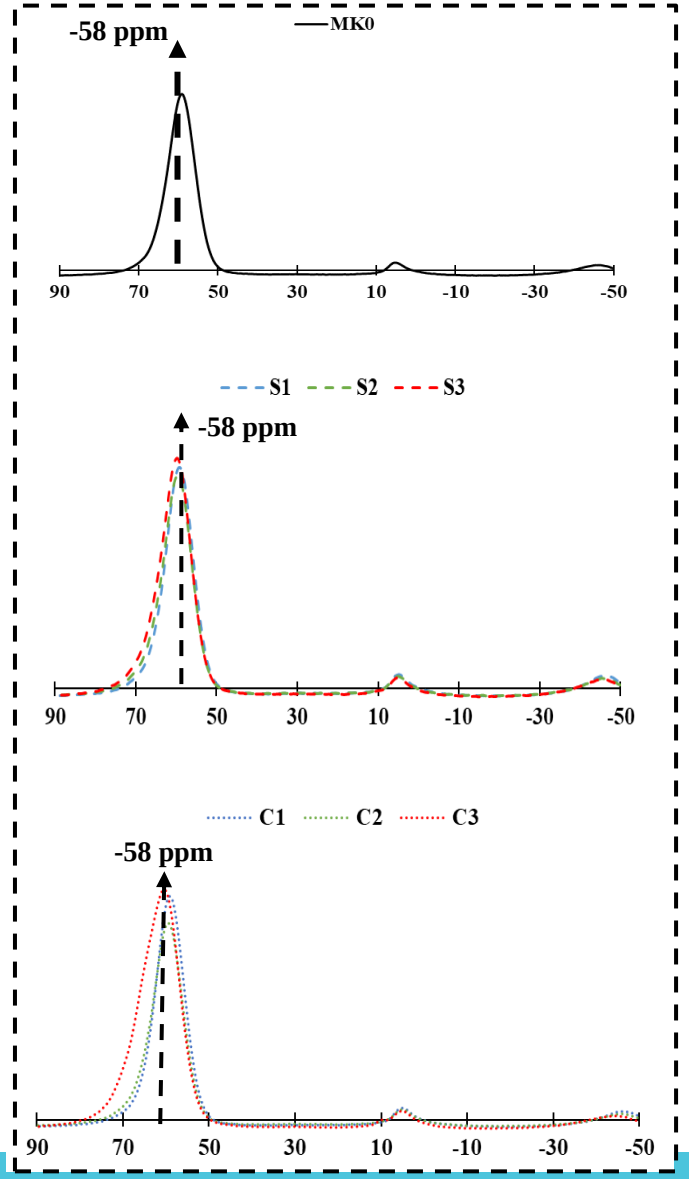
- **FCS Group: S1, S2, S3**
- **FCC Group: C1, C2, C3**

GP Classification results

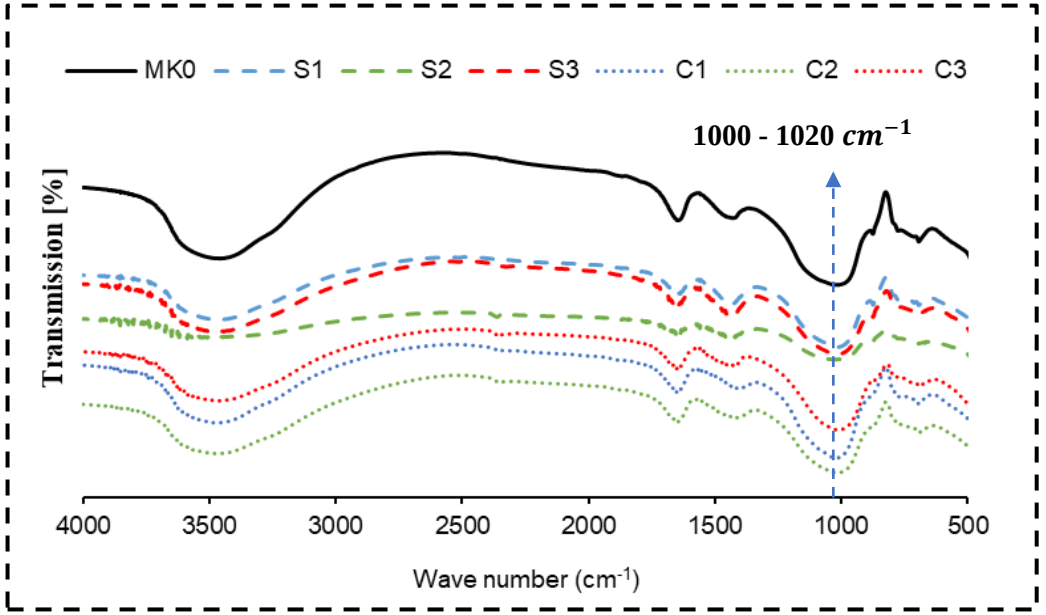
^{29}Si NMR



^{27}Al NMR



FTIR



Classification

NMR

- Tetrahedral Str.
- **MK0:** $Q^4(0Al)$
- **S1:** $Q^4(3Al)$
- **S2:** $Q^4(3Al)$
- **S3:** $Q^4(1Al)$
- **C1:** $Q^4(3Al)$
- **C2:** $Q^4(3Al)$
- **C3:** $Q^4(4Al)$

FTIR

- 1000 - 1020 cm^{-1}

Si-O-Si ⇩ Al-O-Si

⇩

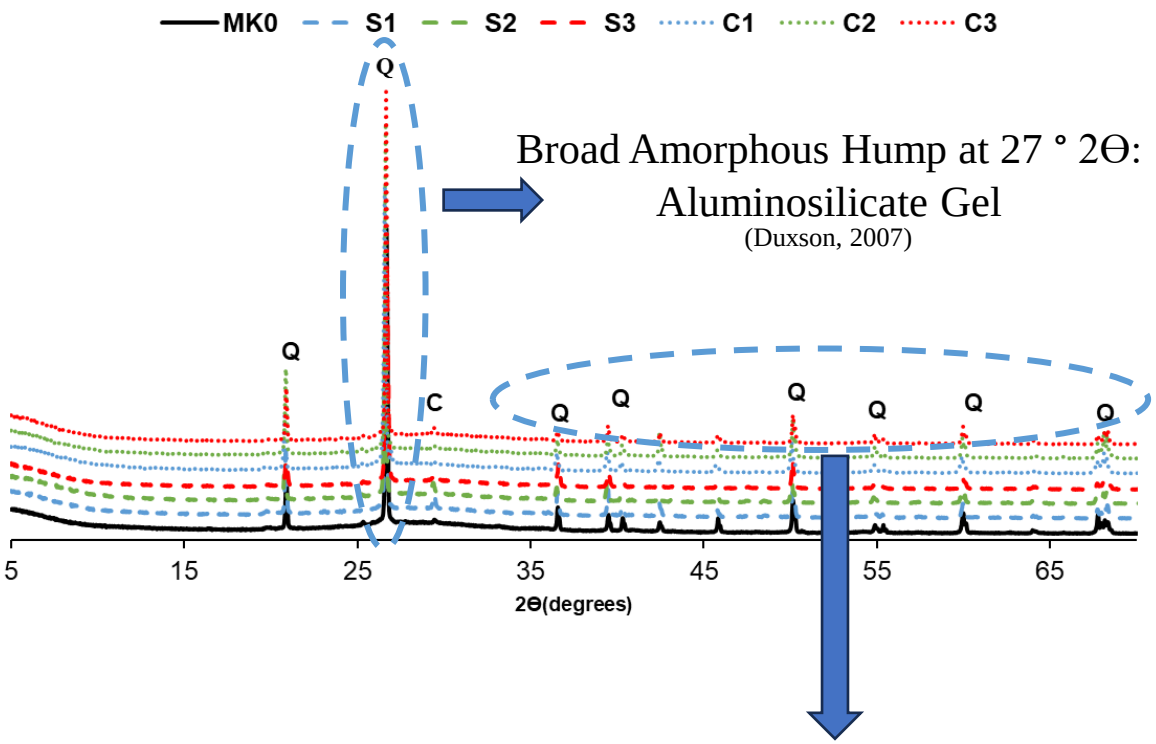
Geopolymerization “Gel”

(Salazar, 2016)

GP Characterization results

Characterization

Mineralogical characterization



Broad Amorphous Hump at 27 ° 2Θ: Aluminosilicate Gel
(Duxson, 2007)

Semi-crystalline peaks of Quartz indication a stable structure

Environmental Leaching Test (NF EN 12457-2)

Element	MK0	S1	S2	S3	C1	C2	C3	IW	NHW
As	2.615	1.926	1.689	1.542	2.012	1.77	1.508	0.5	2
Ba	< 0.008	< 0.008	< 0.008	< 0.008	< 0.008	< 0.008	< 0.008	20	100
Cd	< 0.009	< 0.009	< 0.009	< 0.009	< 0.009	< 0.009	< 0.009	0.04	1
Cr	0.131	0.027	0.03	0.035	0.026	0.025	0.024	0.5	10
Cu	0.083	0.131	0.22	0.145	0.096	0.084	0.052	2	50
Mo	0.11	0.219	0.205	0.179	< 0.09	< 0.09	< 0.09	0.5	10
Ni	< 0.05	< 0.05	0.1	0.101	0.073	0.152	0.15	0.4	10
Pb	< 0.03	< 0.03	0.041	< 0.03	0.042	0.044	< 0.03	0.5	10
Sb	< 0.06	< 0.06	< 0.06	< 0.06	< 0.06	< 0.06	< 0.06	0.06	0.7
Se	0.212	0.309	0.313	0.259	0.136	0.145	0.123	0.1	0.5
Zn	0.016	0.022	0.07292	< 0.01	0.017	0.017	0.011	4	50

Not Hazardous

Geopolymer synthesis

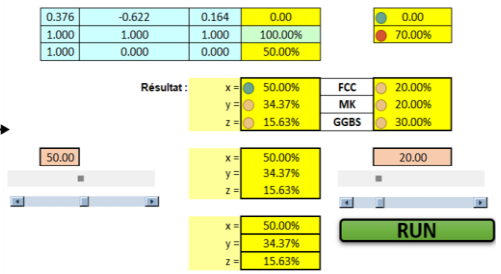
Formulation and optimisation

Formulation of sediment-based geopolymers

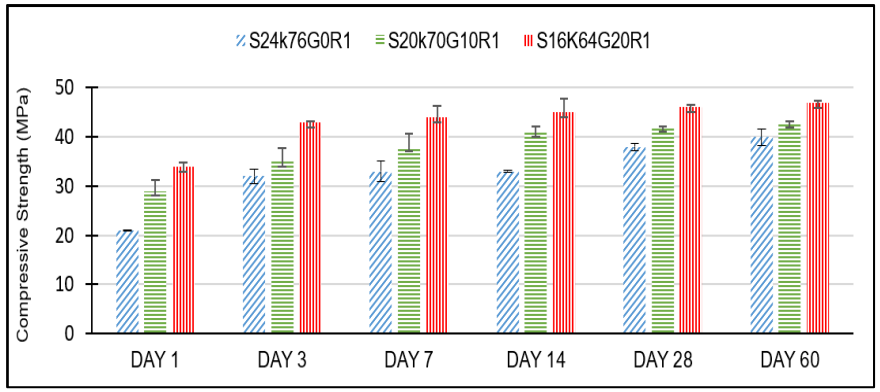
- Formulation

	MK	GGBFS	FCS
Si	27,4	14,9	20,8
Al	16,5	6,8	6,7
SiO_2	58,6	31,8	44,4
Al_2O_3	31,1	12,8	12,6

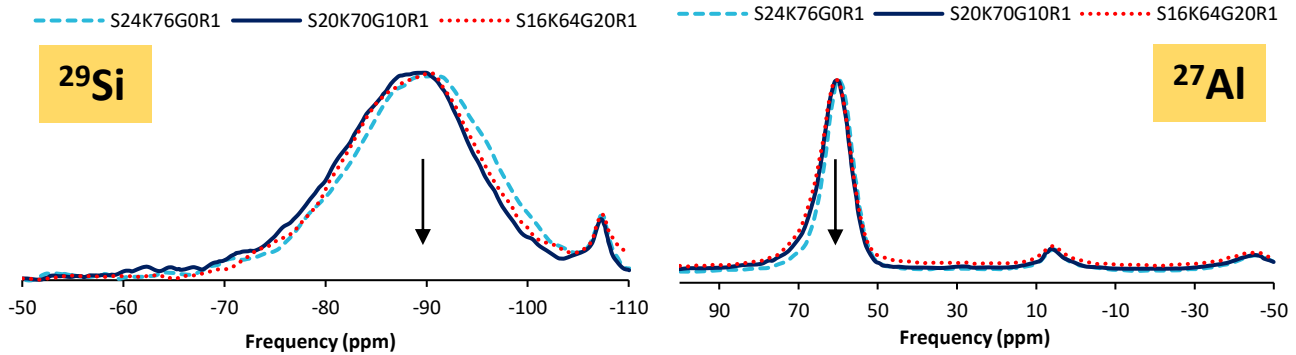
Values in %



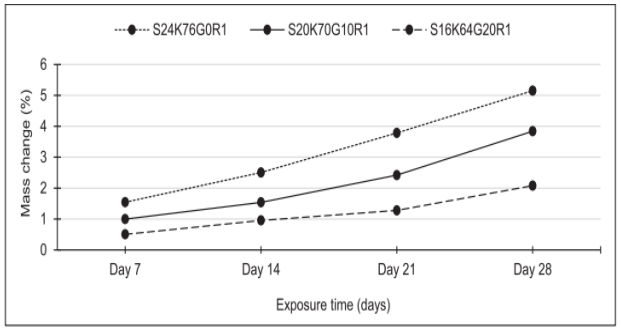
- Compressive strenht

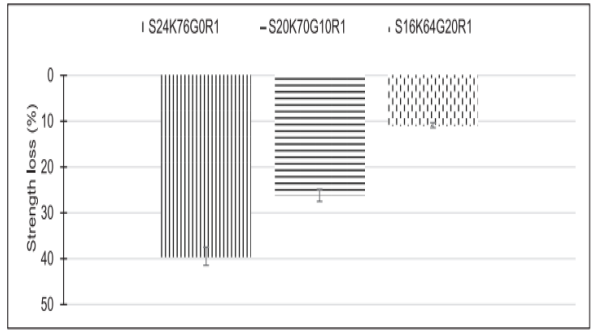


- NMR



- Acid resistance





[AP4], [AP5],[AP8]

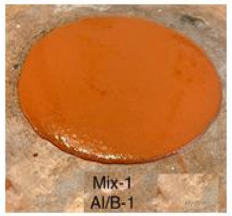
- Geopolymers with a **low ALK/B ratio** are the most resistant and durable.
- The **GGBFS** content is correlated with resistance but reduces the workability of geopolymers.
- Geopolymers based on flash-sediments have shown **structural stability (tetra-coordination)** and are **non-hazardous**.

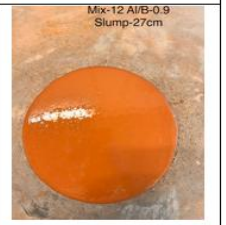
Work carried out as part of the supervision of Ali Alloul's thesis (2021-2024).

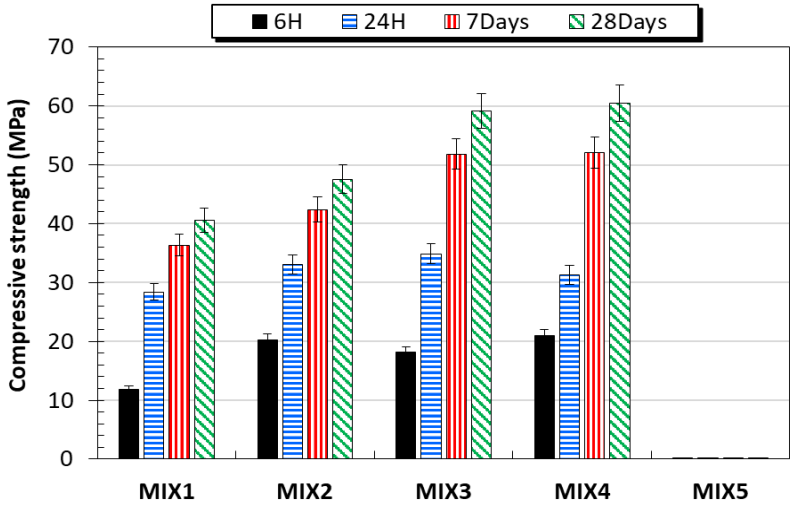
Geopolymer synthesis

Formulation and optimisation

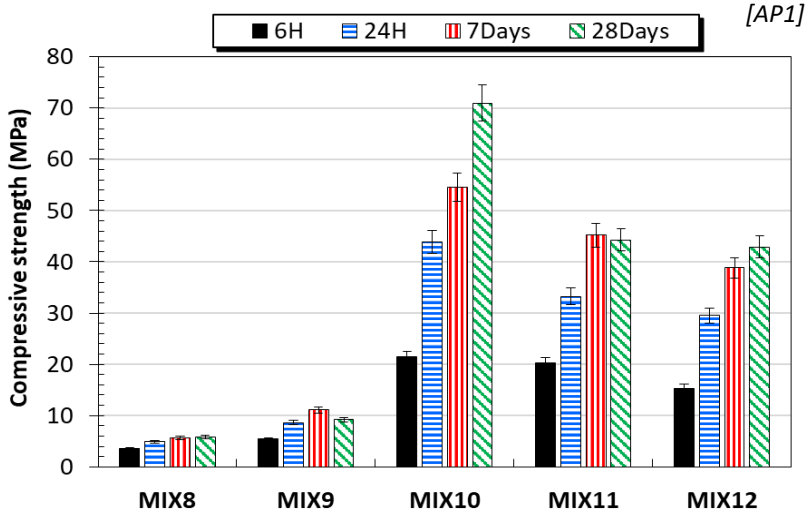
Formulation of excavated soils-based geopolymers

Formulations	Mix 1	Mix 2	Mix 3	Mix 4	Mix 5
Slump (cm)	27.6	24.1	18.3	5	no
Visual aspect					

Formulations	Mix 8	Mix 9	Mix 10	Mix 11	Mix 12
Slump (cm)	no	no	12	17	27
Visual aspect					



[AP1]

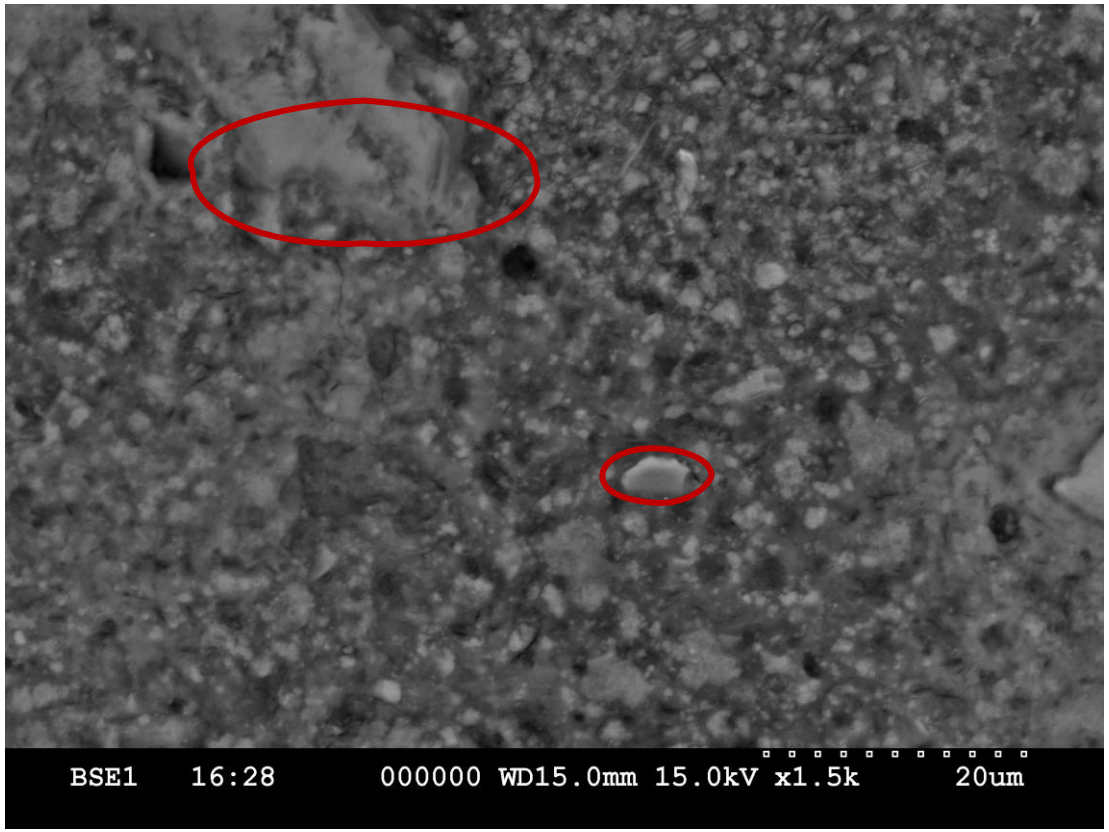


- **Microstructural analyses** such as NMR, XRD, and FTIR reveal a stable 3D polymeric structure with **strong Si–O–Al–O** bonds.
- An **ALK/B ratio** of **0.75** provides optimal performance.

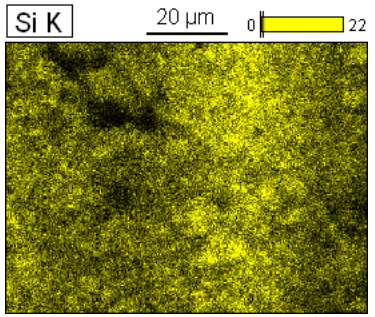
Geopolymer synthesis

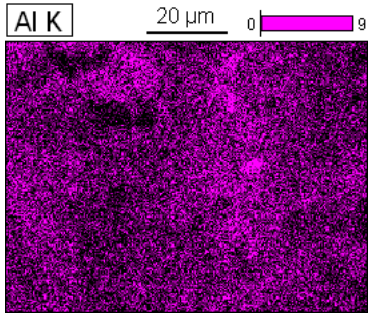
Formulation and optimisation

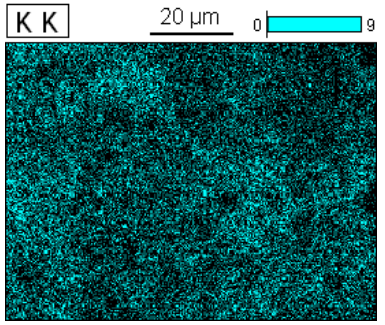
Formulation of excavated soils-based geopolymers



- SEM-EDS analyses







O	68.41 %
Mg	0.17 %
Al	**3.38 %**
Si	**20.78 %**
K	**4.90 %**
Ca	1.86 %
Fe	0.51 %

Clay	
SiO_2	79.38 %
Al_2O_3	14.72 %

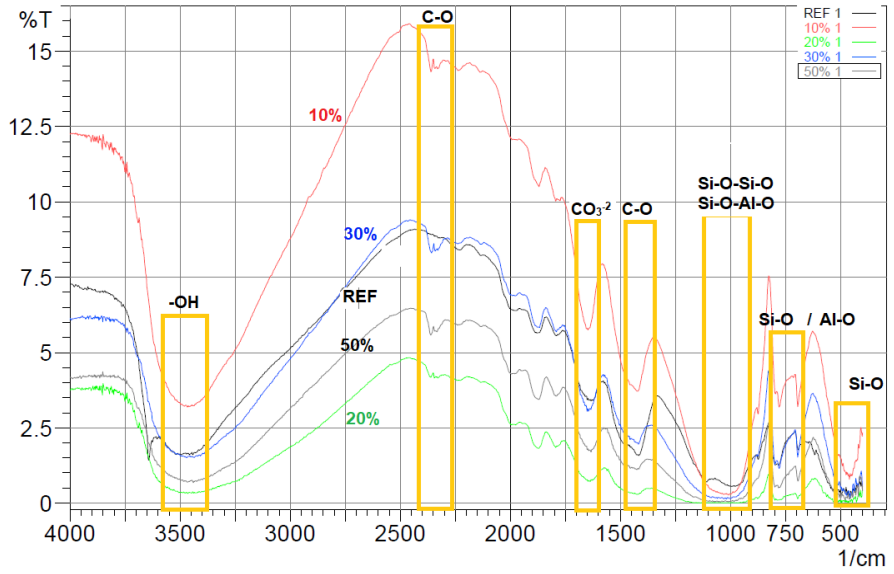
Work carried out during the supervision of the postdoctoral of Elie Mahfoud (2024)

Geopolymer synthesis

Formulation and optimisation

Formulation of excavated soils-based geopolymers

- IRFT



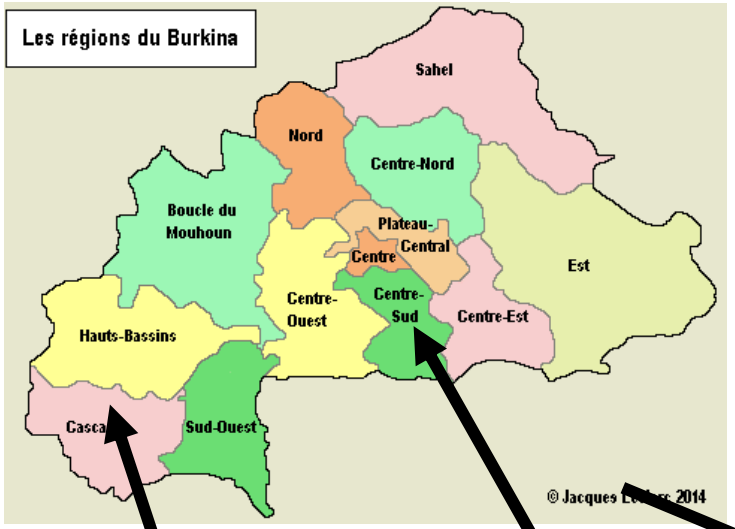
- MIP (Mercury Intrusion Porosity)

	Mean pores diameter (4V/A) (nm)	Total intrusion volume (ml/g)	Total pores surface (m^2/g)	Total porosity (%)
RMPC (Port Cem)	58.05	0.066	4.571	14.05
RMGP	34.54	0.0355	4.109	8.06
MFCC10P2	18.62	0.0296	6.358	6.92
MFCC20P2	18.15	0.0334	7.369	7.70
MFCC30P2	18.70	0.0346	7.405	7.99
MFCC50P2	18.10	0.0340	7.505	7.78

- **Mechanical strength increases** as the **ALK/B ratio decreases**.
- **Consistency decreases** as the **ALK/B ratio decreases**.
- **20–30% LHF** appears to be a **good compromise** (performance).
- **Siloxo (Si–O–Si–O) and sialate (Si–O–Al–O) bonds** have been identified.
- The **porous structure of the geopolymer** is **“better” compared to that of Portland cement–based materials**.

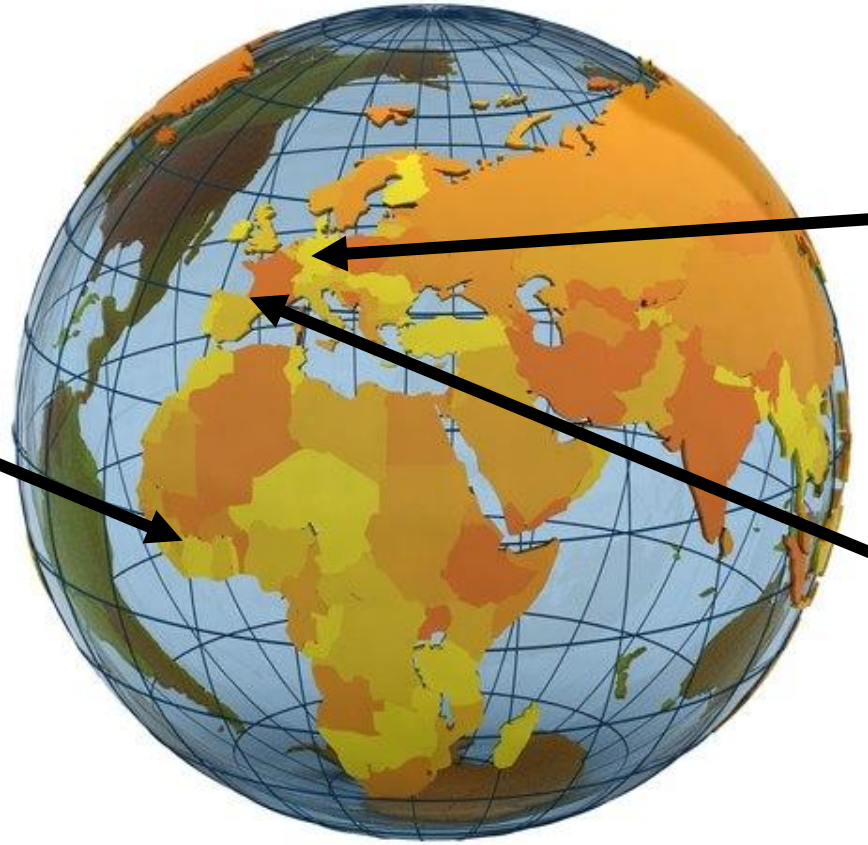
Materials

Reinventing raw earth: a geopolymer approach for high-performance, sustainable CEBs



Clay

Laterite

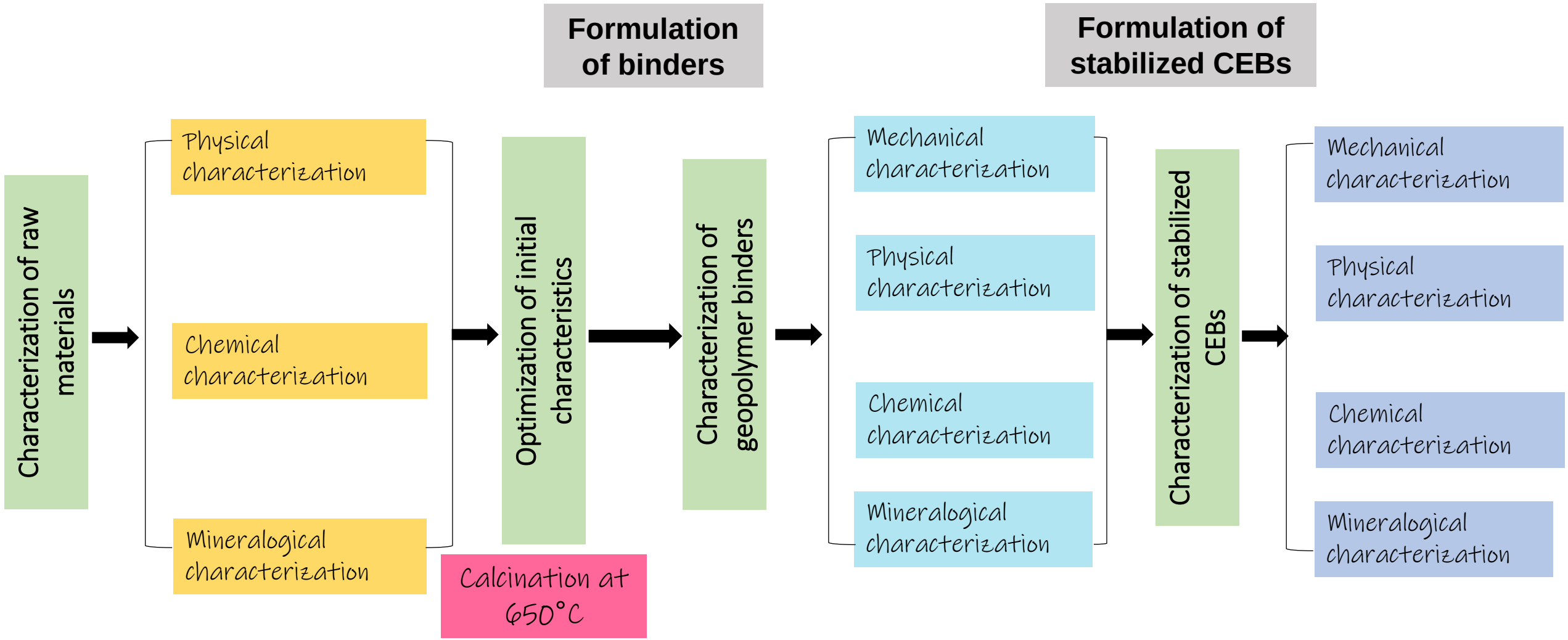


FERROSIALATE

Geosil

Silty soil

Protocol


Formulation of binders
Formulation of stabilized CEBs
Characterization of raw materials
Physical characterization
Chemical characterization
Mineralogical characterization
Optimization of initial characteristics
Calcination at 650°C
Characterization of geopolymer binders
Mechanical characterization
Physical characterization
Chemical characterization
Mineralogical characterization
Characterization of stabilized CEBs
Mechanical characterization
Physical characterization
Chemical characterization
Mineralogical characterization

Materials and Methods

Raw laterite

Raw clay

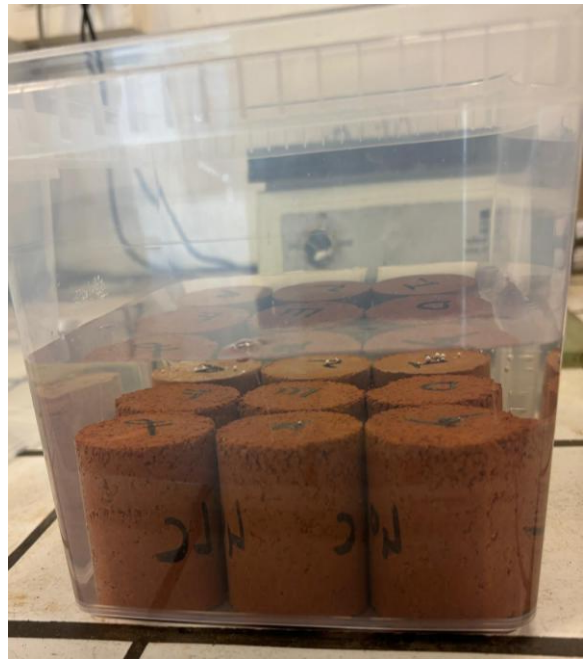
Calcined laterite

Calcined clay

Earth material for brick

Mixing of the different components before compression

Labotest press used for brick making



Total immersion for 2 hours before wet compression

Conditioning before treatment

Differents CEBs

Compression test

Characterization of CEBs

XRD and FTIR spectra of CEBs

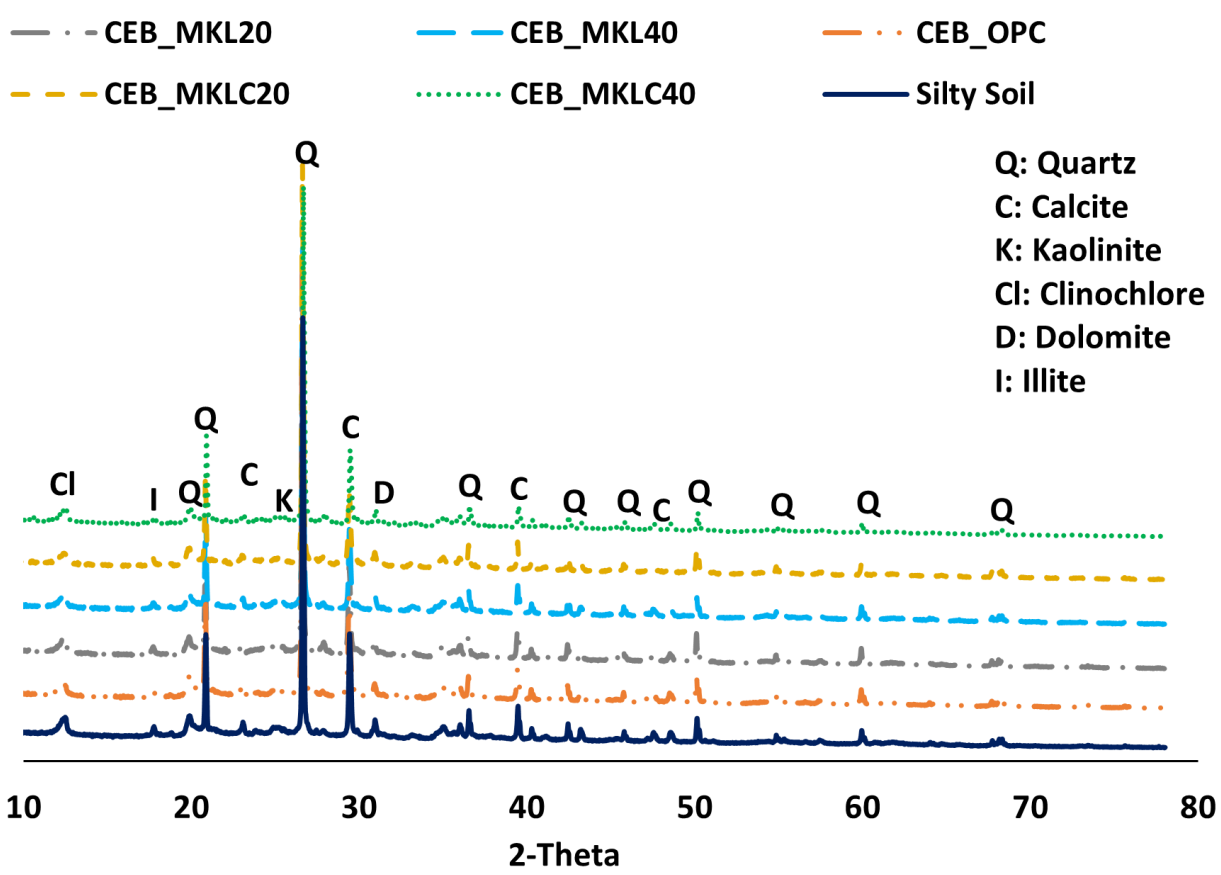


Figure : XRD of differents CEBs and silty soil

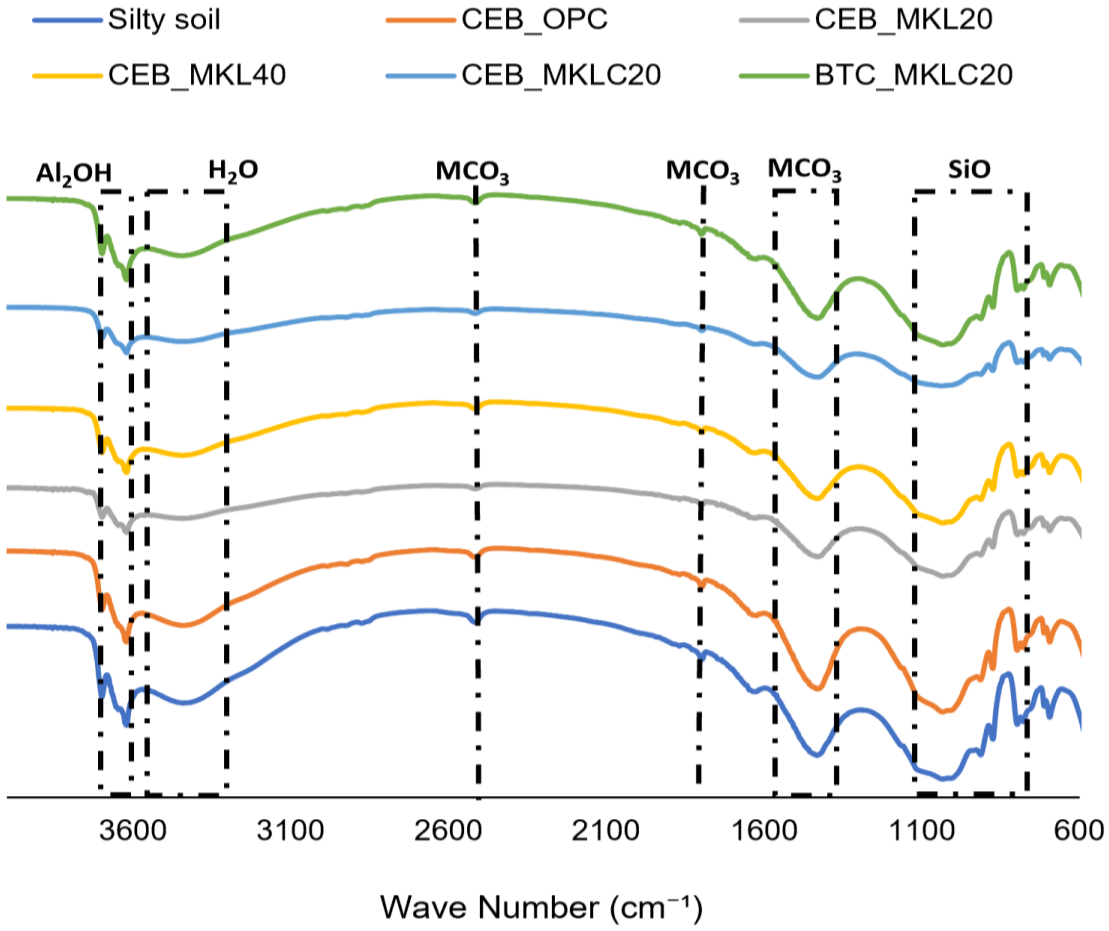


Figure : IRFT spectra of differents CEBs and Silty Soil

Characterization of CEBs

Compressive strenght and capilarry water absorption of CEBs

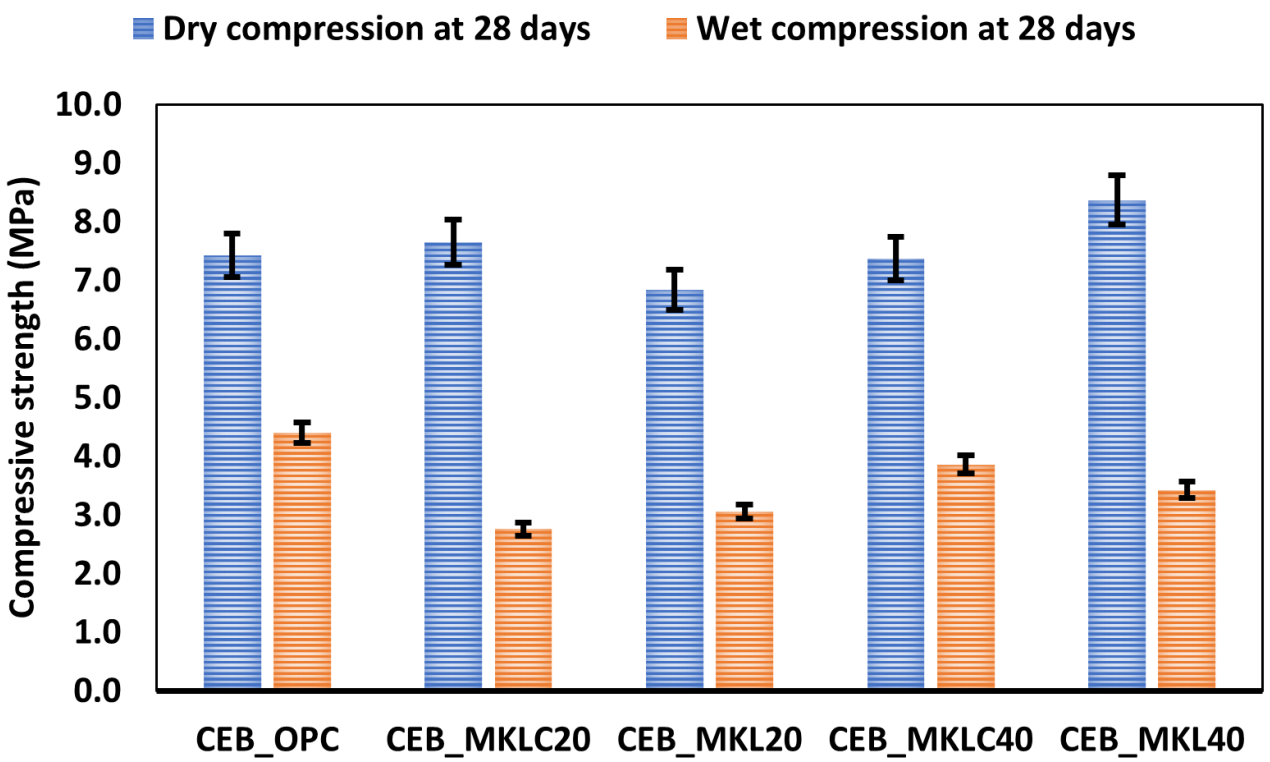


Figure : Dry and wet compressive strength of CEBs

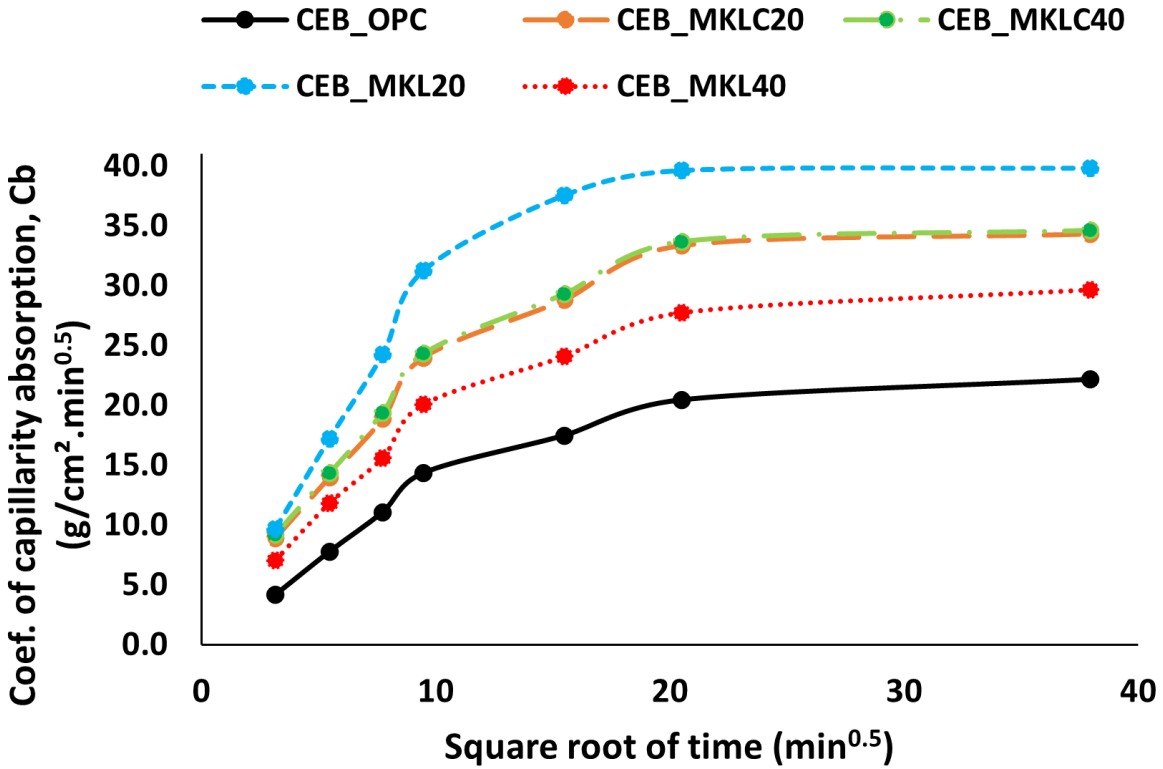


Figure : Capillary water absorption of CEBs exposed to prolonged partial immersion

Methodology

Excavated soils

Raw materia

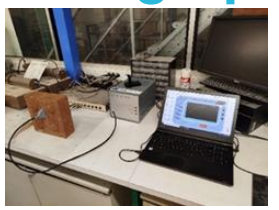
Density

cteristics

owder

Compressive strength

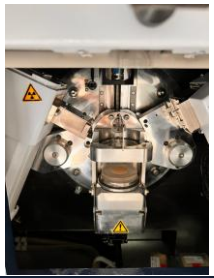
Scaling up



Thermal conductivity

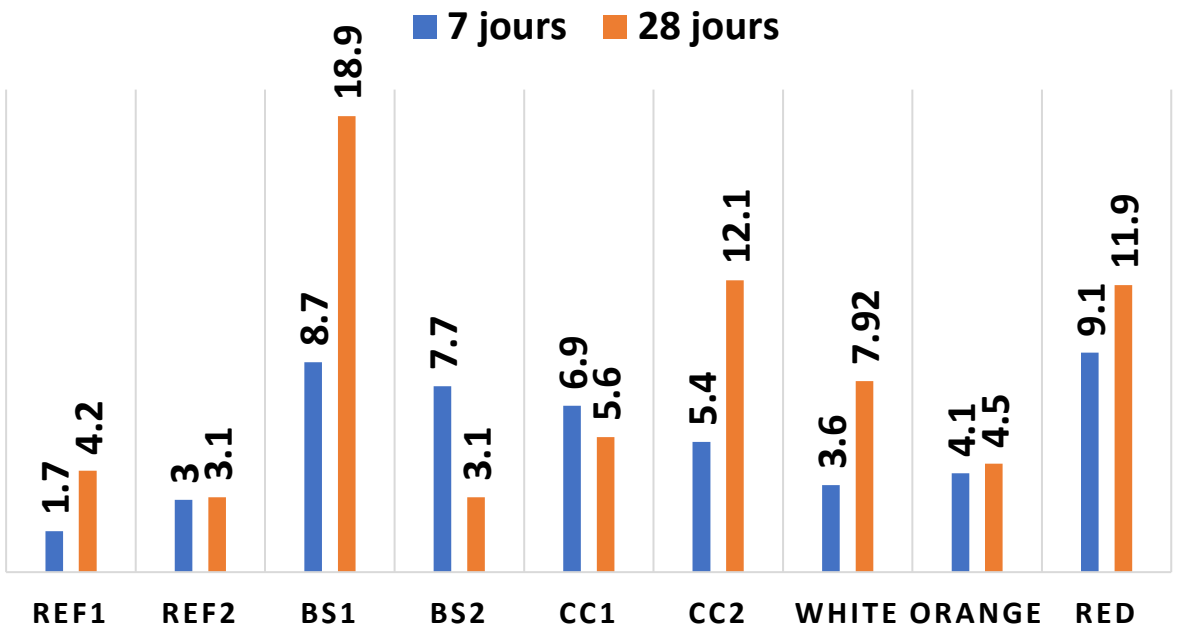
Thermogravimetric analysis
(TGA)

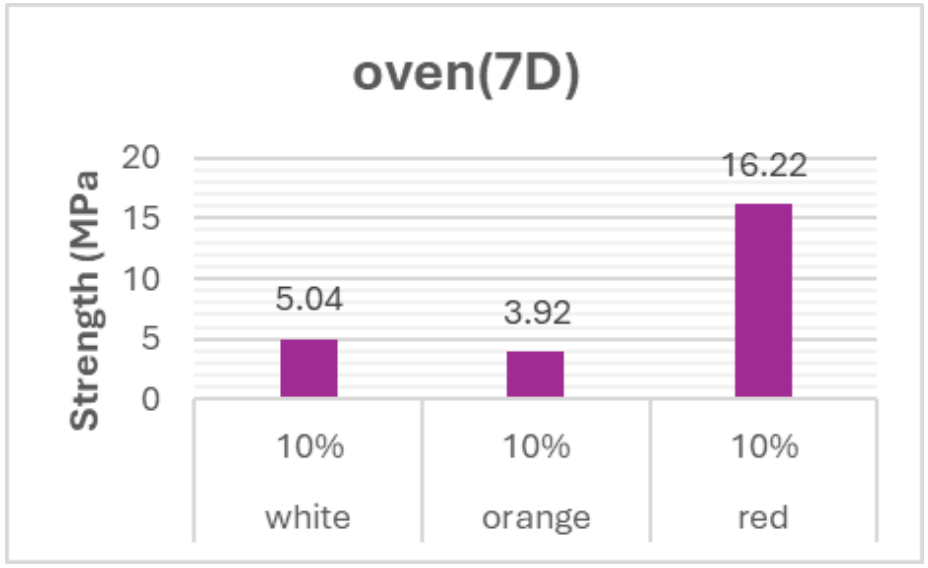
Fourier-transform infrared spectroscopy
(FTIR)



X-ray Diffraction
(XRD)

Results and discussion





Compressive strength after 7 and 28 days

Best Performing Geopolymer Bricks

BS-1 Sample: This geopolymer demonstrates the best performance among geopolymers, with a compressive strength of 12.15 MPa after 28 days. It exhibits a consistent improvement over time and provides a strong alternative to traditional materials.

Red Geopolymer Brick: Outstanding performance under oven-cured conditions with a strength of 16.22 MPa at 7 days, suggesting high early strength and faster curing, beneficial for projects with tight deadlines.

Development of bioreagent → "biopolymers"

GEL PREPARATION

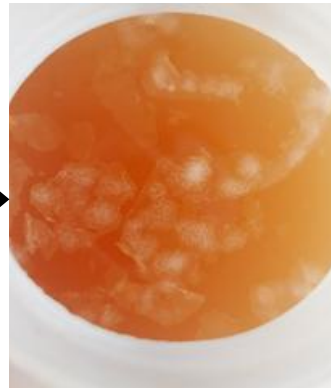
Arabica gum

After Boiling for 15mins and Filtering

Arabica gum gel in a container

The bark of Grewia bicolor (Kel)

After Boiling for 15mins and Filtering



The bark of Grewia bicolor (Kel) gel in a container

Development of bioreagent → "biopolymers"

Formulation using Kel

Formulation using Arabica gum

After 24 hours

After 7 days

Development of geopolymer pastes

FORMULATION

calcined clays

+

Potassium silicate, Geosil

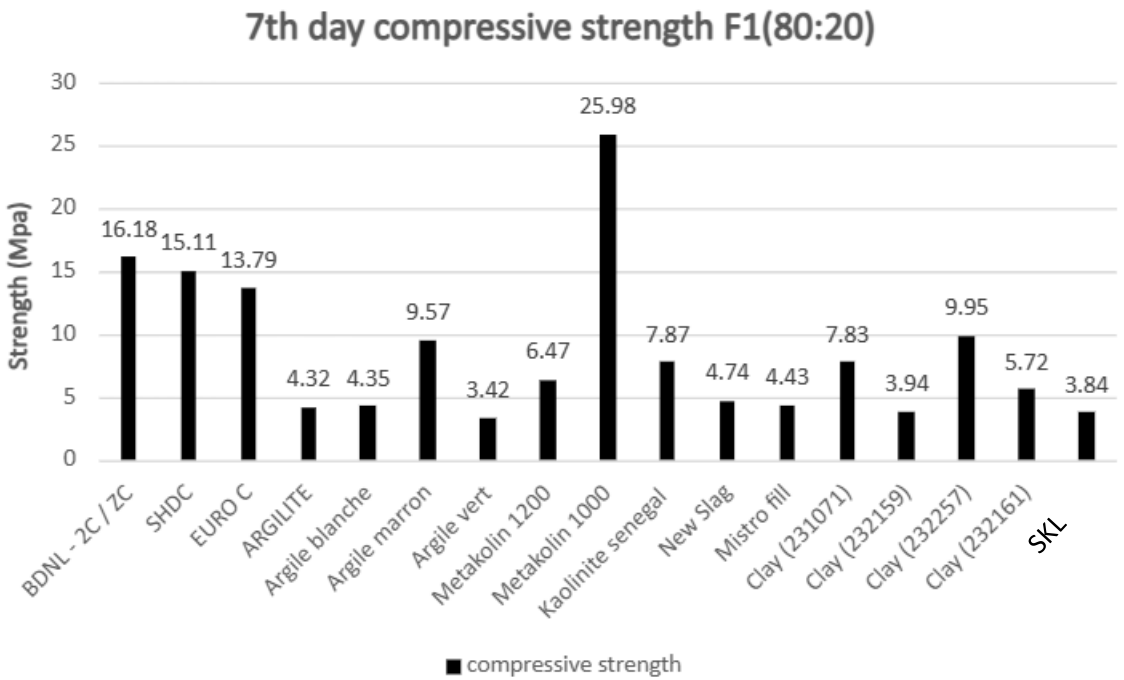
+

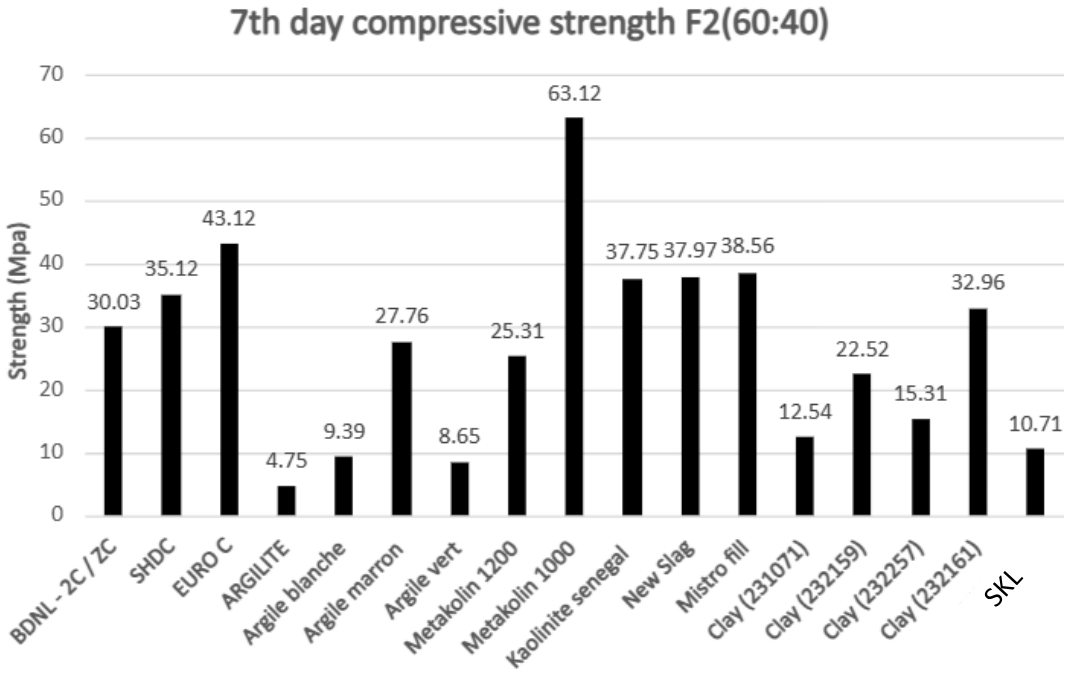
GGBFS

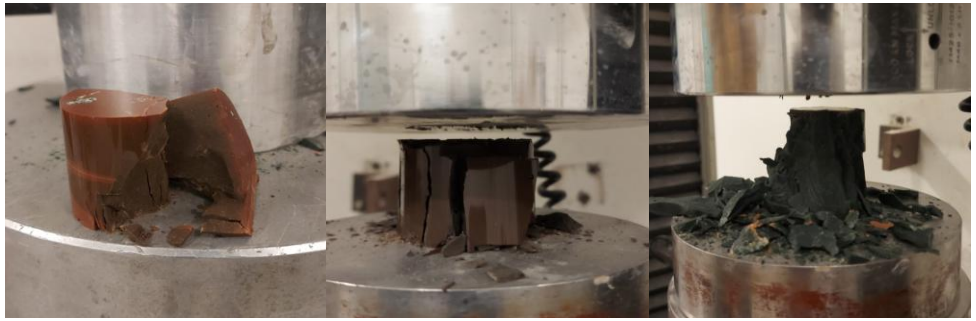
Paste mixing

Formulations F1(80:20) and F2 (60:40)

Compression Strength Formulations F1(80:20) and F2 (60:40)







F1 formulations compression testing

F2 formulations compression testing

Lessons learned

The **mixing procedure** showed to be **effective,** all GP formulations **are hardened at room** temperature

The percentage **of Al_2O_3 reacting** for **FCS** is **30%.**

Based on **compressive strength** test and **water boiling** test, the GP formulations **with the lower AR/B** are the **optimum** formulations.

NMR test showed that the designed **GP formulations have a tetrahedral 3D networking**

Lessons learned

The optimal **AR/B** for the GP formulations is around **0.7-0.8.**

In comparison with MK, both **FCS and FCC** showed **high compressive strength.**

GP formulations with **lower CaO content showed higher resistance to high temperatures** and **freeze-thaw** tests.

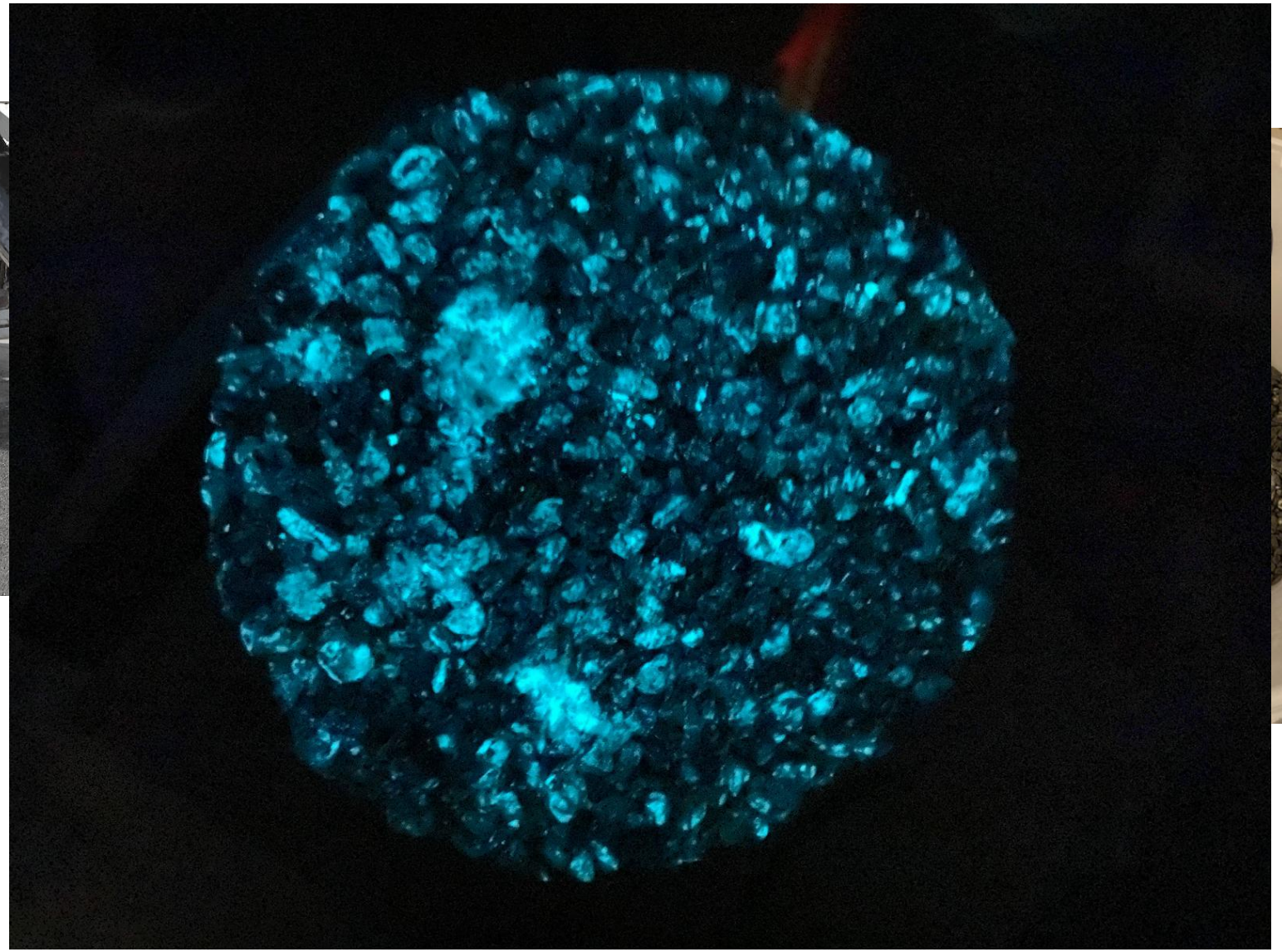
SEM/EDS, NMR, FTIR test results showed that all GP formulations **with FCS and FCC** resulted in a **geopolymerization reaction and 3D Tetrahedral network.**

The leaching test results show that GP formulations with FCS an FCC are **not hazardous.**

Different brick types can be made using geopolymers and **ranging 3-19 MPa** compressive strength

“Bioreagent” and GP making with different natural clays seems **to be promising**

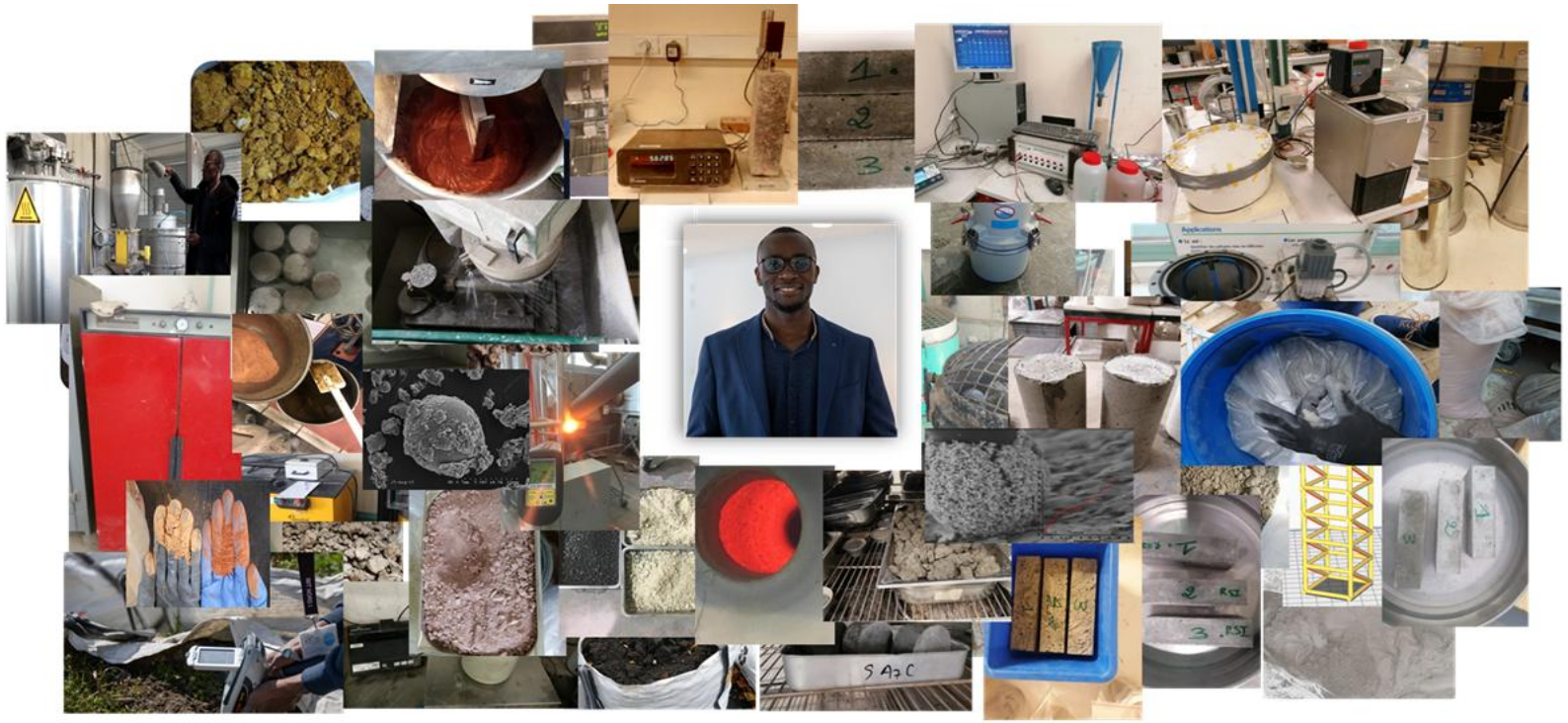
Current projects



Habilitation to Direct/Conduct/Supervise Research (HDR)

- September 2024
- A huge part of my research was dedicated to geopolymer technology
- → Moving to more company and entrepreneurship collaborations

- Email: mouhamadou.amar@imt-nord-europe.fr
- LinkedIn: Mouhamadou AMAR, HDR., PhD., Eng.
- Call: (0033)3.27.71.24.13



Thanks for your attention