One-part geopolymers from spent filtering earths using alternative activators from waste glass. An example of circular economy

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Introduction

High energy consumption

High consuption of mineral resources

High emissions of gases, mainly carbon dioxide

(5-7 % CO_2)

CEMENT PORTLAND PRODUCTION

Cement is one of the most widely used building materials

-Low Price, economical material

- Versatile

- Ability to harden under water

Cement industry more environmentally friendly

More sustainable alternative cements

Alkaline-activated cements or geopolymer cements

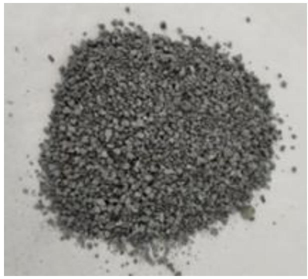
- **Low environmental impact: reduce CO_2 emissions**
- **No High temperatures in manufacturing**
- **Good mechanical properties**
- **High chemical attack resistence**

Alkaline activation binders

PRECURSOR ALUMINOSILICATES (with high or low calcium contents) + **ALKALINE ACTIVATOR** → Chemical Reaction → **ALKALINE ACTIVATED CEMENTS OR GEOPOLYMERS**

Metakaolin

Calcined clays

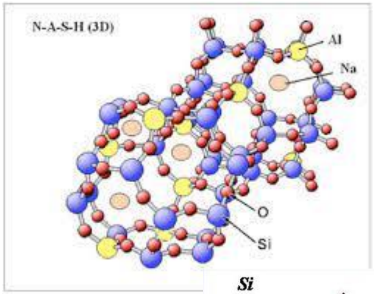


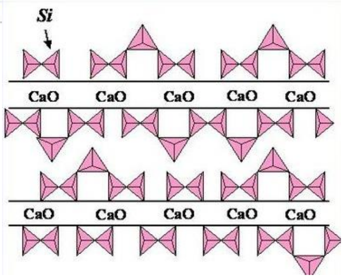
Metallurgical slag

Fly ash

Alkaline hidroxides

Alkaline silicates

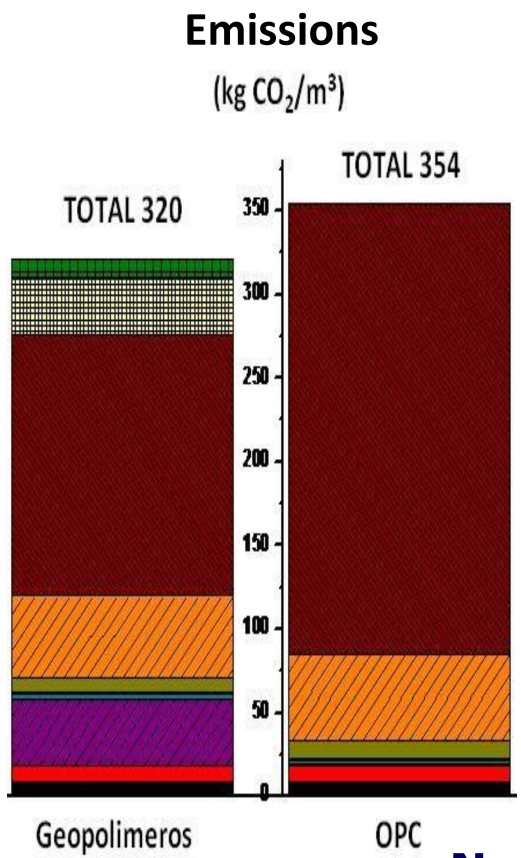




Commercial activators: Problems-Challenges

Geopolymers

- (11) Fly ash, slag
- (34) Sodium hidroxides
- (156) **SODIUM SILICATES**
- (49) Coarse aggregates
- (9) Fine aggregates
- (<1) Additions
- (3) Dosage
- (40) Curing
- (9) Transport
- (9) Commissioning



OPC concrete

- (269) CEMENT
- (51) Coarse aggregates
- (11) Fine aggregates
- (<1) Additions
- (3) Dosage
- (<1) Curing
- (9) Transport
- (9) Commissioning

Turner, L.K., Collins, F.G., 2013; Torres-Carrasco et al., 2015

Need to look for alternative activators to **commercial alkaline silicates** in order to **produce near-zero carbon footprint cements**

Alternative activators. Solutions-Proposals

Silica-based alternative activators

Alkaline hidroxide
+
Silica-rich raw material

$$2NaOH + SiO_2 \xrightarrow{t^{\circ}C} Na_2SiO_3 + H_2O$$

Alkaline silicate

Silica-rich raw materials

Contents

Objetive

The **activation** of spent filtering earth from the oil refining industry (SFE) by the **use of alternative activators** made from **waste glass (WG) with different dosages of alkali** to obtain **geopolymer cements** with near **zero carbon footprint.**

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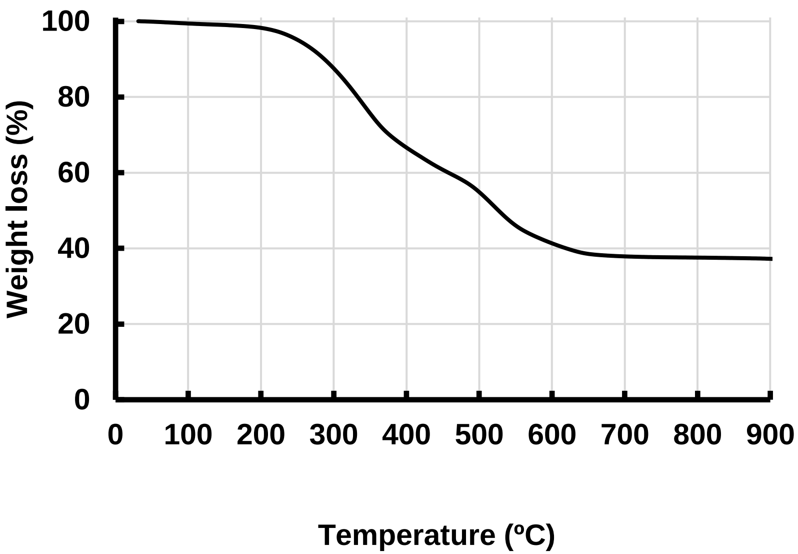
RAW MATERIAL: SPENT FILTERING EARTHS (SFE)

Filtering earths are widely used in the **agri-food industry with the** problem that the end-of-life material is a **useless waste.**



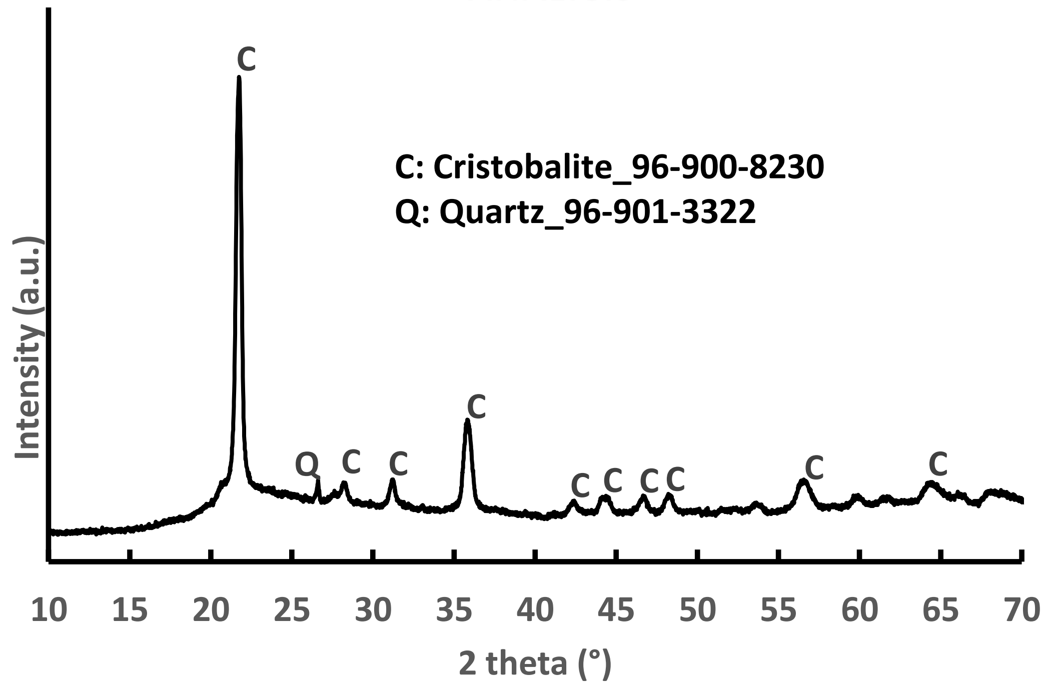
Inorganic fraction of SFE obtained by calcination at 700 ºC

RAW MATERIAL: SPENT FILTERING EARTHS (SFE)

XRF ANALYSIS

84.29 % SiO_2
5.87 % Al_2O_3
0.96 % CaO
...

XRD ANALYSIS



SYNTHESIS OF ALTERNATIVE SOLID ACTIVATOR

77.5 % Na_2O

36.4 g NaOH

99 % Purity

73.9 % SiO_2

43.6 g

Waste glass (WG)

Ground and sieved under 0.063 mm

$$Ms = \frac{mol\ SiO_2}{mol\ Na_2O} = 1$$

1. Adding water and Mixing

20.0 g

2. Heat treatment

3h - 300 ºC

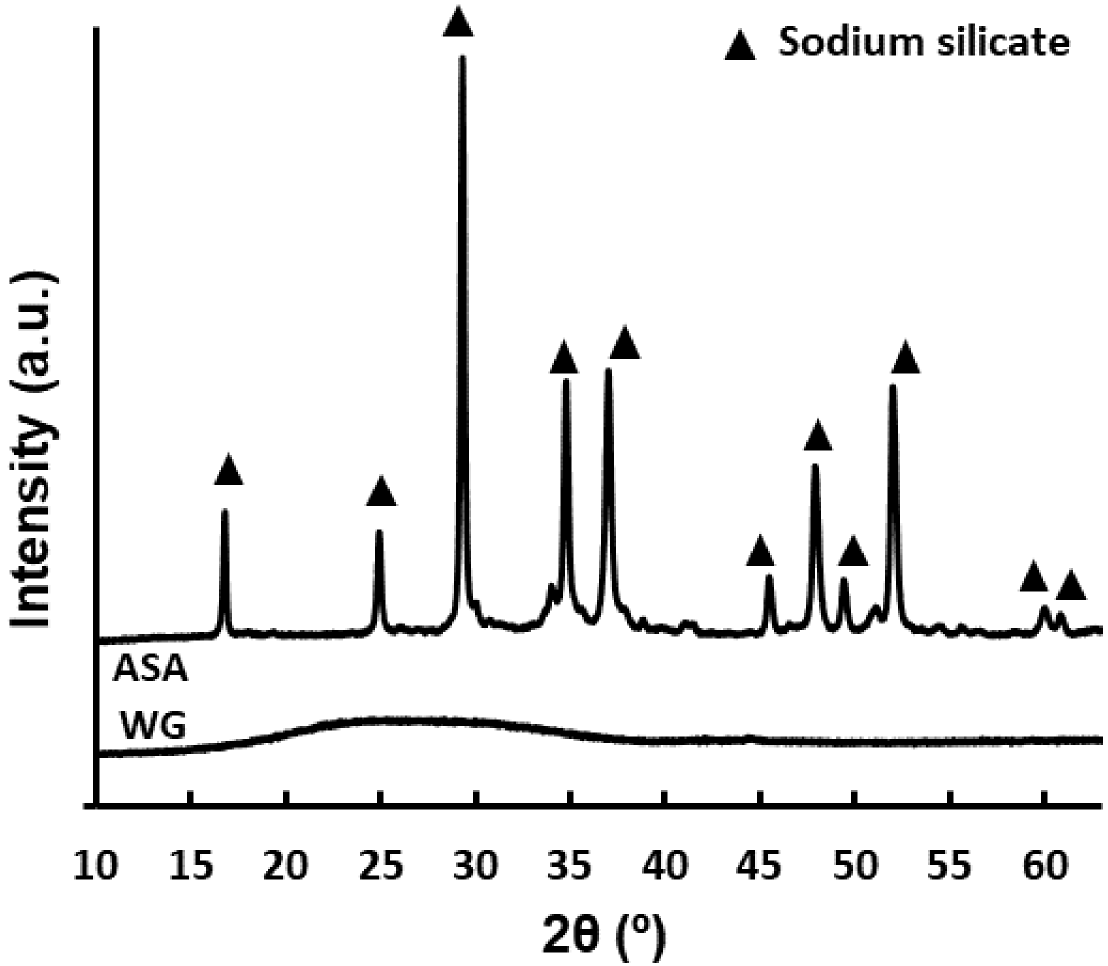
(Rate 10 ºC/min)

3. Ground in a ball mill

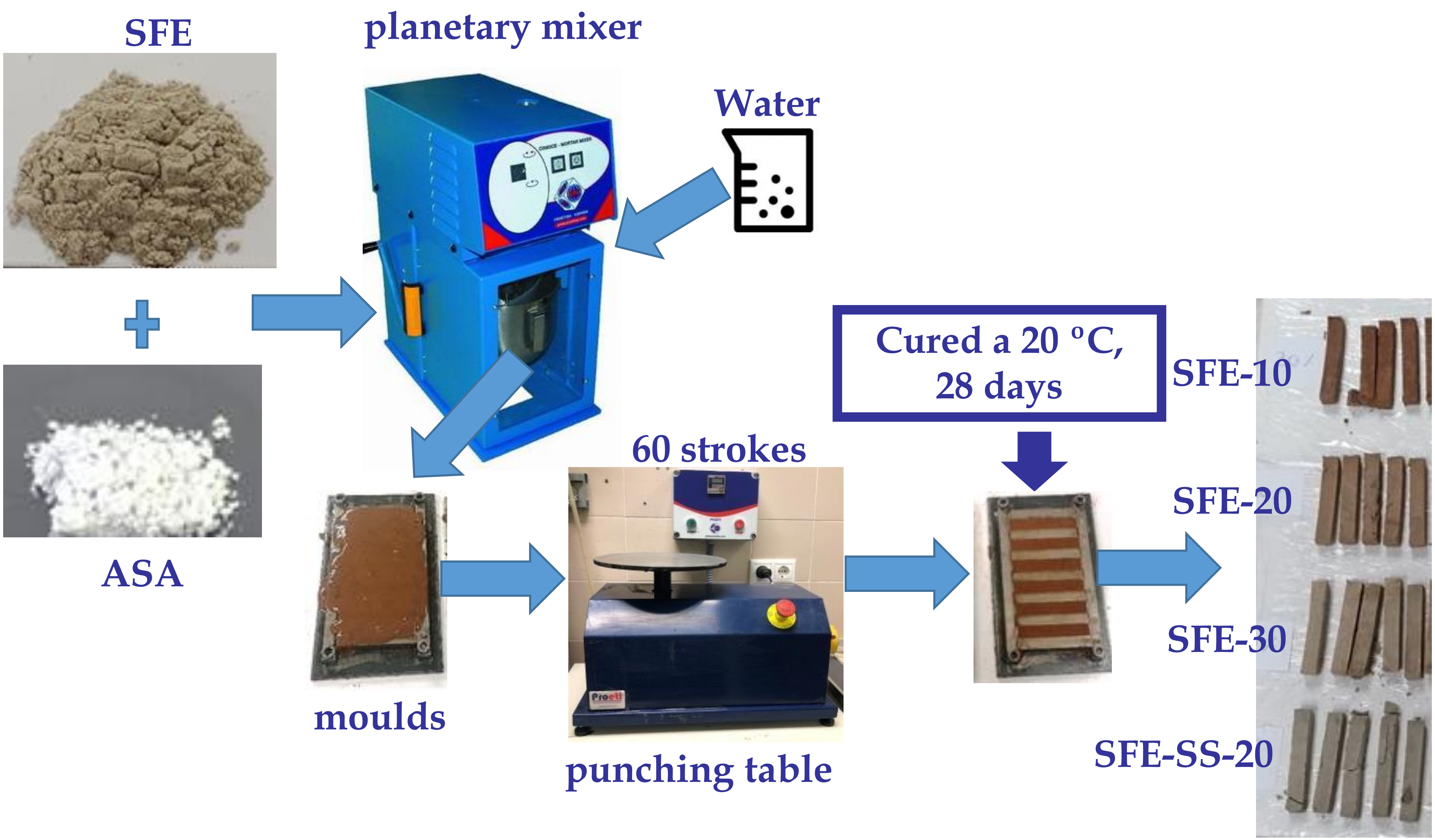
ALTERNATIVE SOLID ACTIVATOR (ASA)

100 g

XRD study


▲ Sodium silicate
Intensity (a.u.)
ASA
WG
10 15 20 25 30 35 40 45 50 55 60
2θ (º)

Manufacture of the geopolymers cements



Manufacture of the geopolymers cements

Sample	SFE (g)	Alternative solid activator (g)	Sodium hidroxide (NaOH) (g)	Sodium silicate (Na_2SiO_3) (g)	Water	Na_2O (%)
SFE-10	300	72.6	-	-	280.9	10
SFE-20	300	145.1	-	-	291.8	20
SFE-30	300	217.7	-	-	302.6	30
SFE-SS-20	300	-	55.66	199.2	176.7	20

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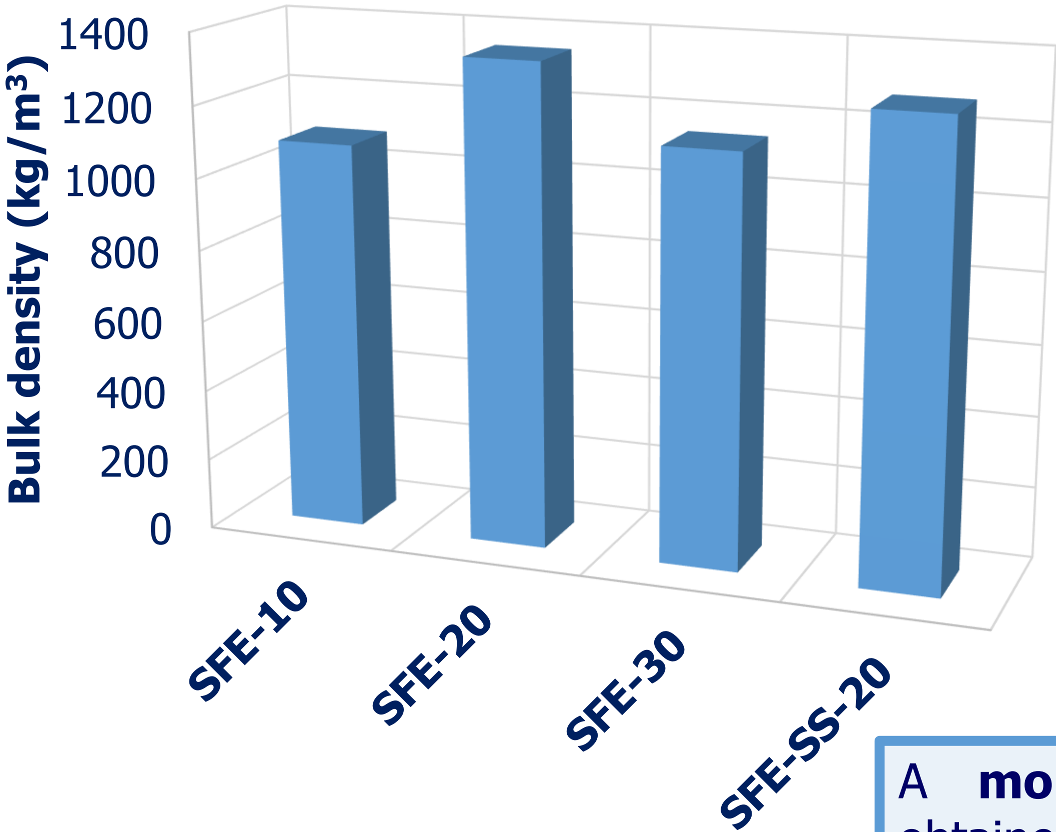
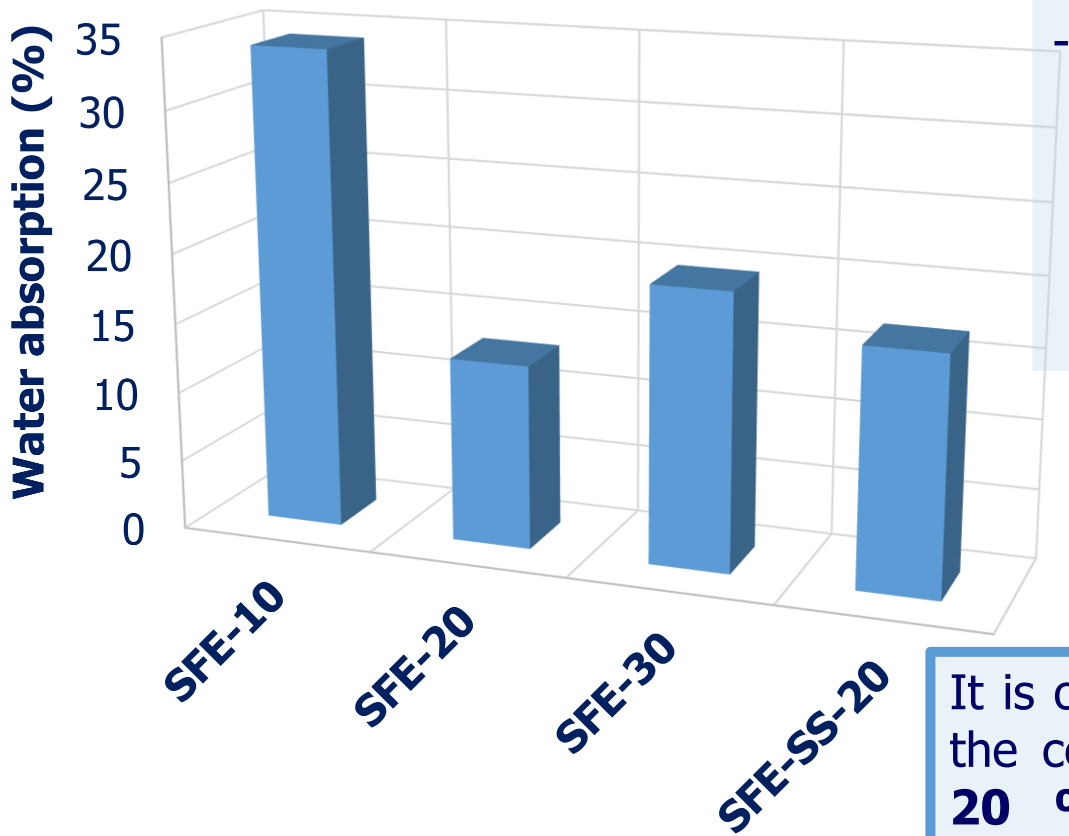
Bulk density



- Increase of BD with increasing alkali dosage from 10 to 20 % of Na_2O.
- Increase up to 30 % produced cements with a lower BD
- **Commercial silicate results in specimens with lower BD than obtained with ASA.**

A **more compact microstructure** is obtained **due to a higher advance in the geopolymerisation reaction** when **ASA is used.**

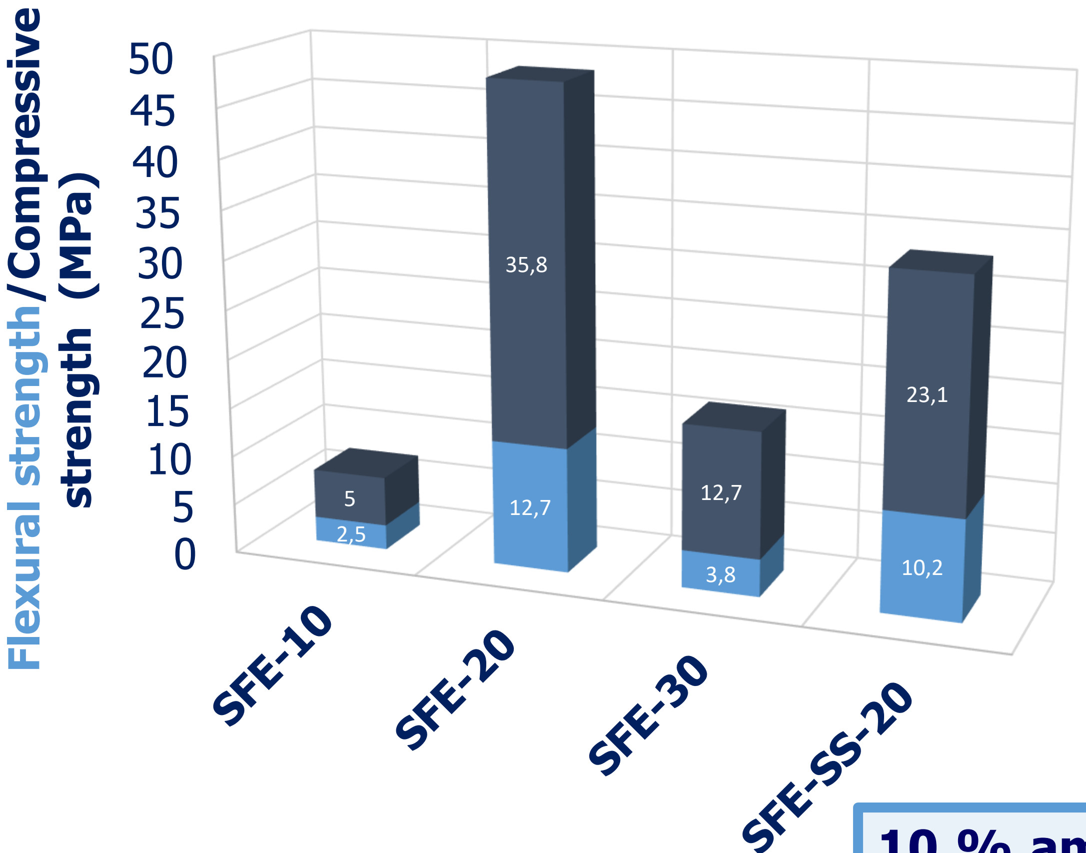
Water absorption



- WA is very high with the addition of 10 % Na_2O.
- The addition of 20 % Na_2O resulted in a significant reduction of WA up to 13 %, with a slight increase with the addition of 30 % and when commercial activator is used

It is observed that the **open porosity** of the cement **is reduced** with the use of **20 % Na_2O** because the **capillary pores** are gradually **filled** by **the formation of amorphous reaction products**.

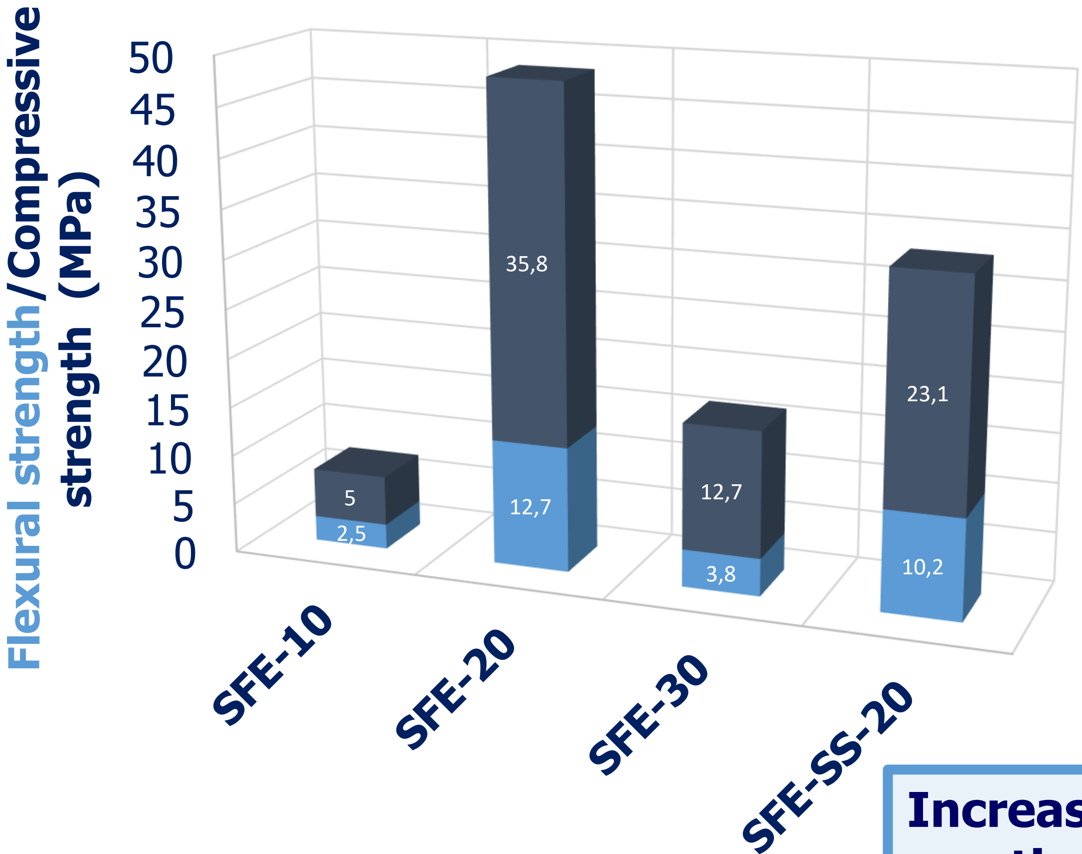
Flexural and compressive strength



- Poor mechanical properties are obtained at an alkali dosage of 10 %

10 % amount of the ASA is **insufficient to activate the pozzolanic reactivity** of the **SFE**, resulting in **many unactivated SFE particles**

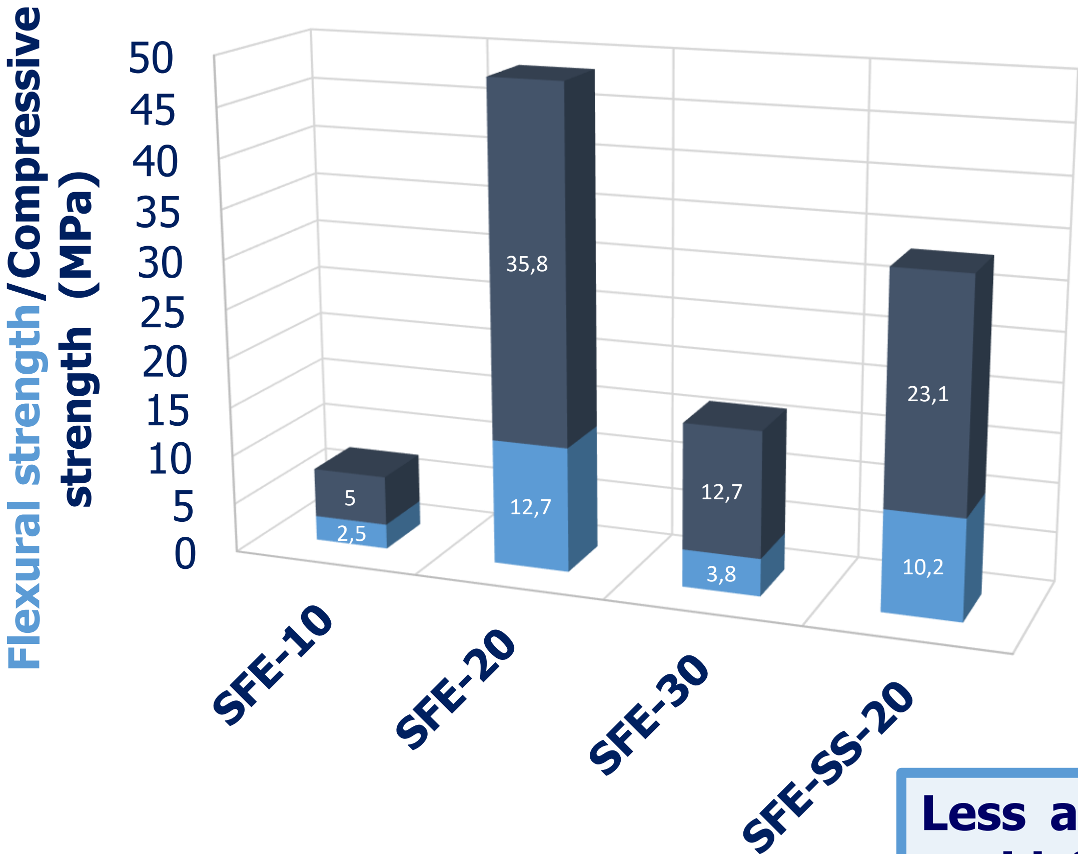
Flexural and compressive strength



- Increasing alkali dosage up to 20 %, the compressive and flexural strength shows a significant improvement

Increase in the geopolymerisation reaction, leading to the **formation** of **more geopolymer gel**, resulting in **a denser matrix**

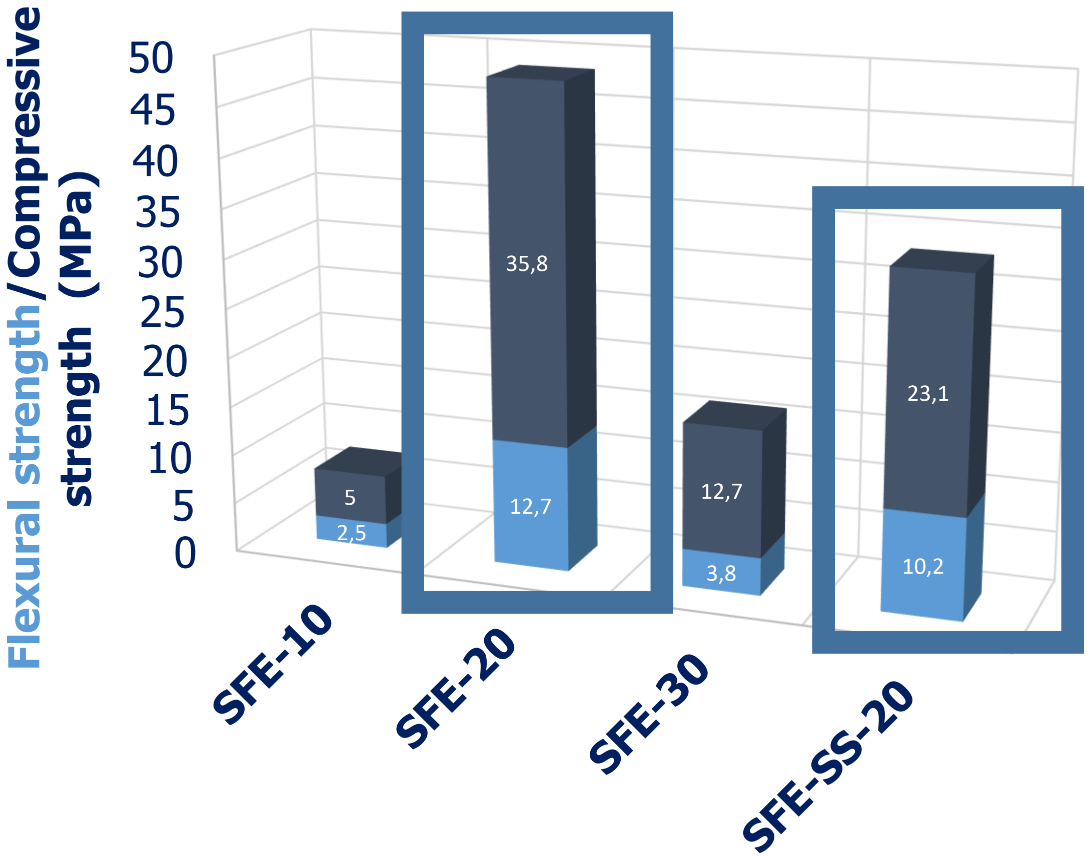
Flexural and compressive strength



- Higher alkali dosage (30% Na_2O) is unfavourable for the mechanical properties of the SFE

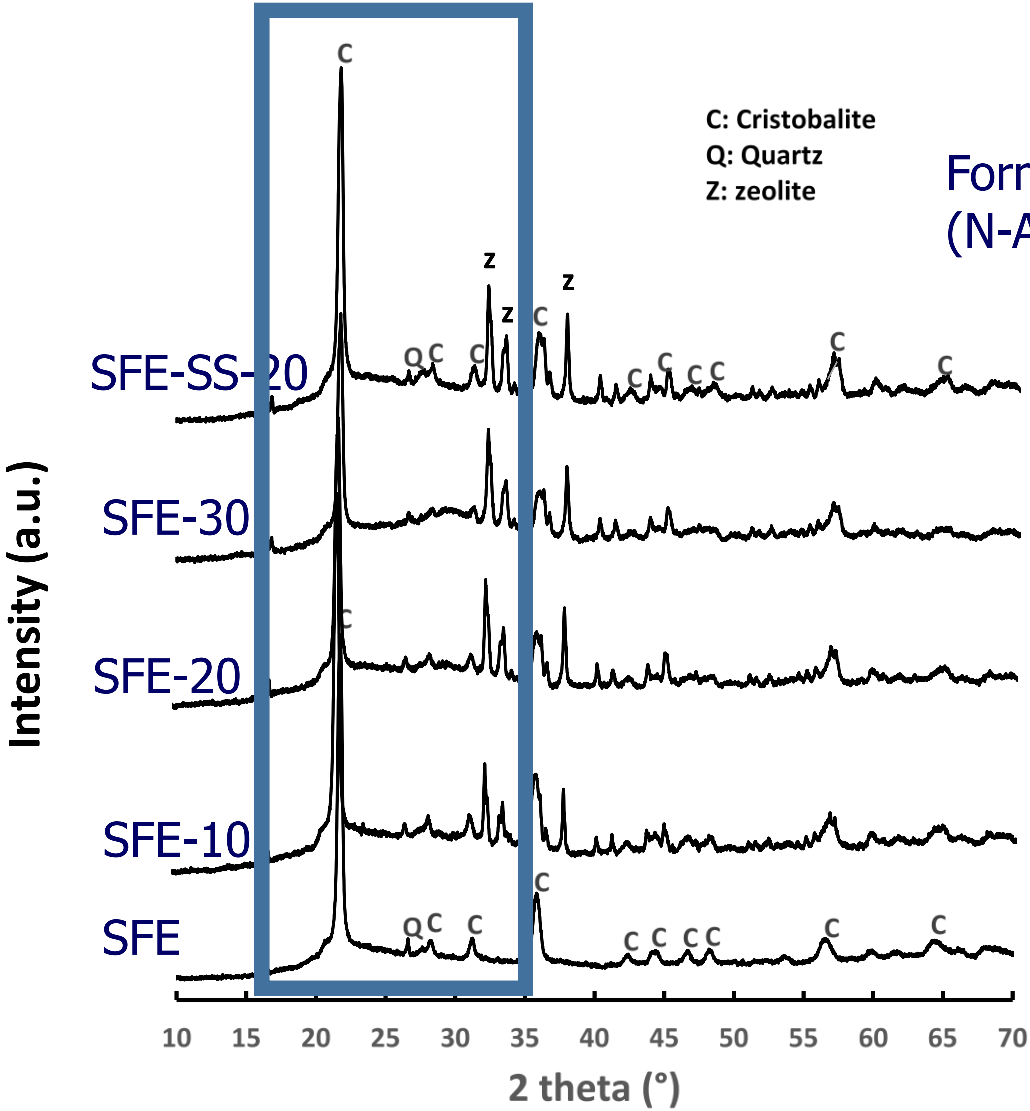
Less amorphous gel possibly due to the **rapid formation of oligomers covering the surface**, **decreasing** the **degree of reaction**

Flexural and compressive strength



- The mechanical properties of the geopolymers using the ASA are superior to those obtained for the commercial activator.

XRD

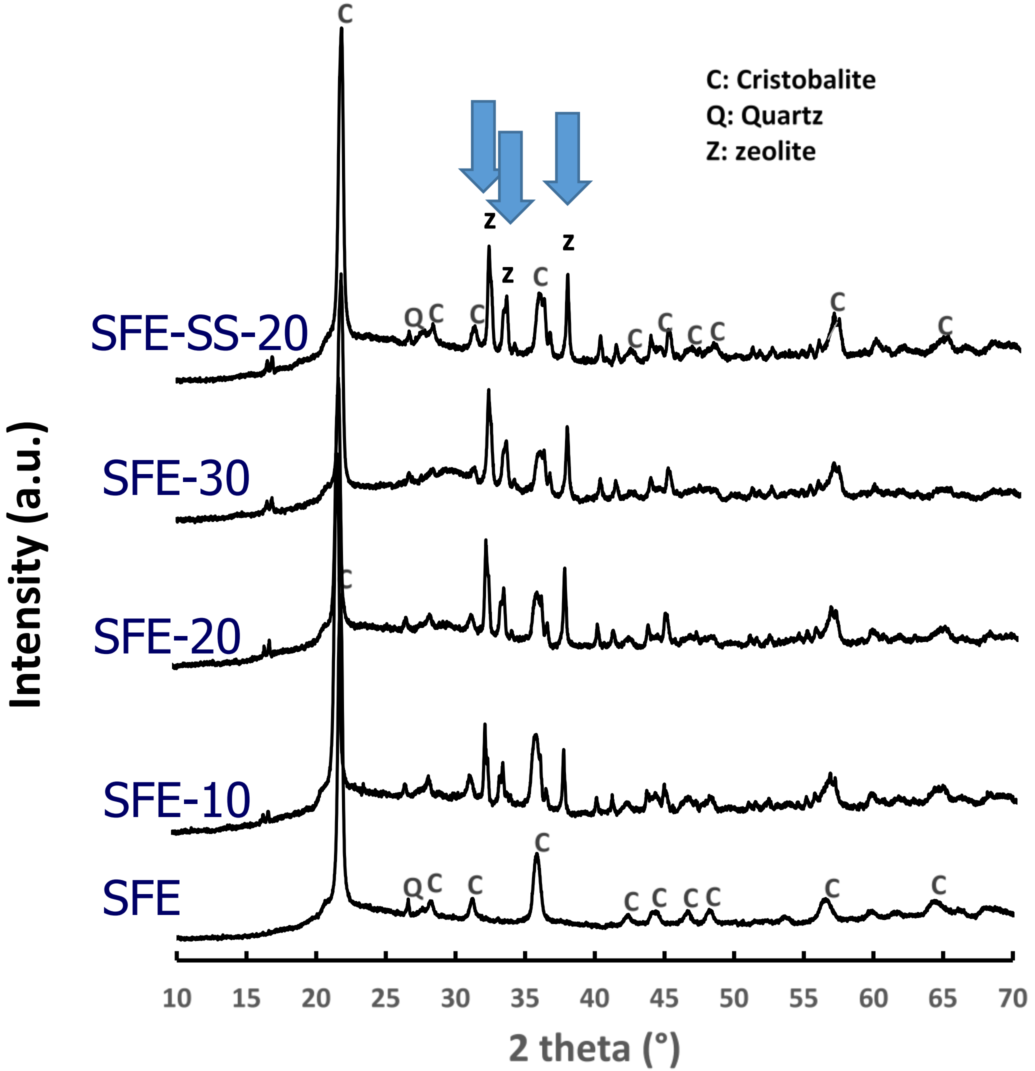


Shift of the halo in the geopolymers tower lower values of 2θ

Formation of an alkaline aluminosilicate gel (N-A-S-H gel), the main reaction product

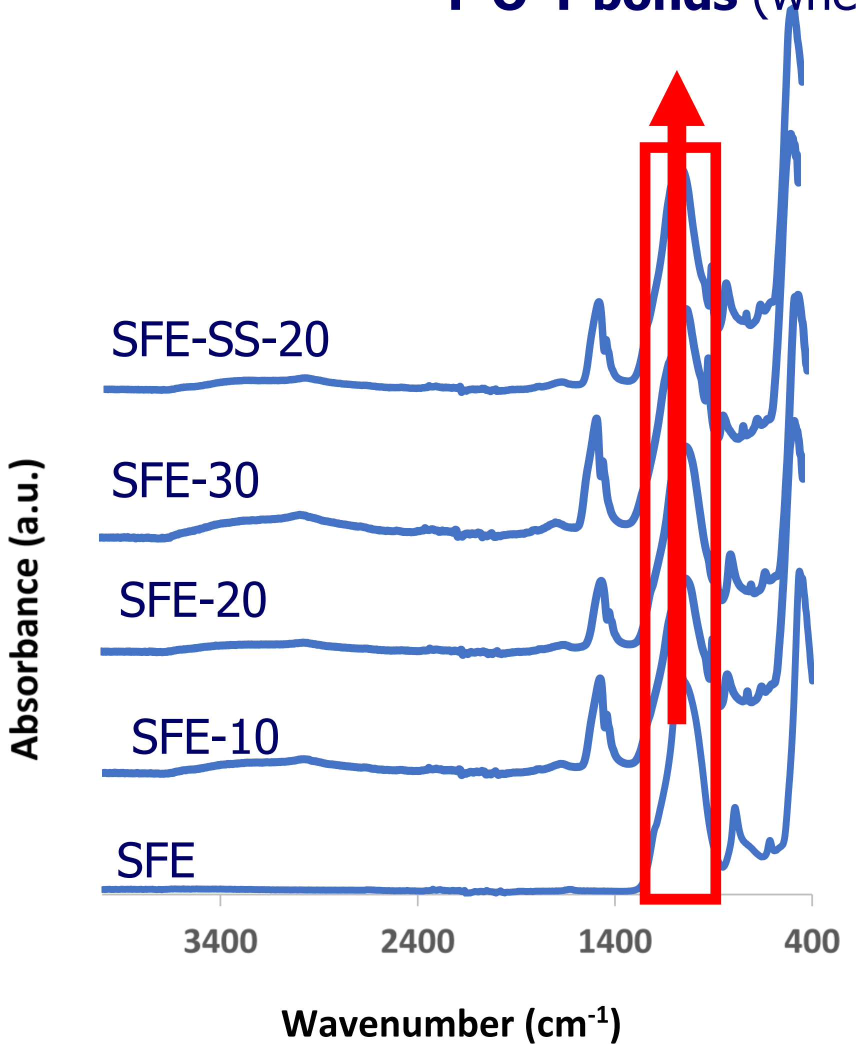
XRD

The appearance of new zeolite-type diffraction peaks which is a crystalline phase from the formation of the geopolymer gel



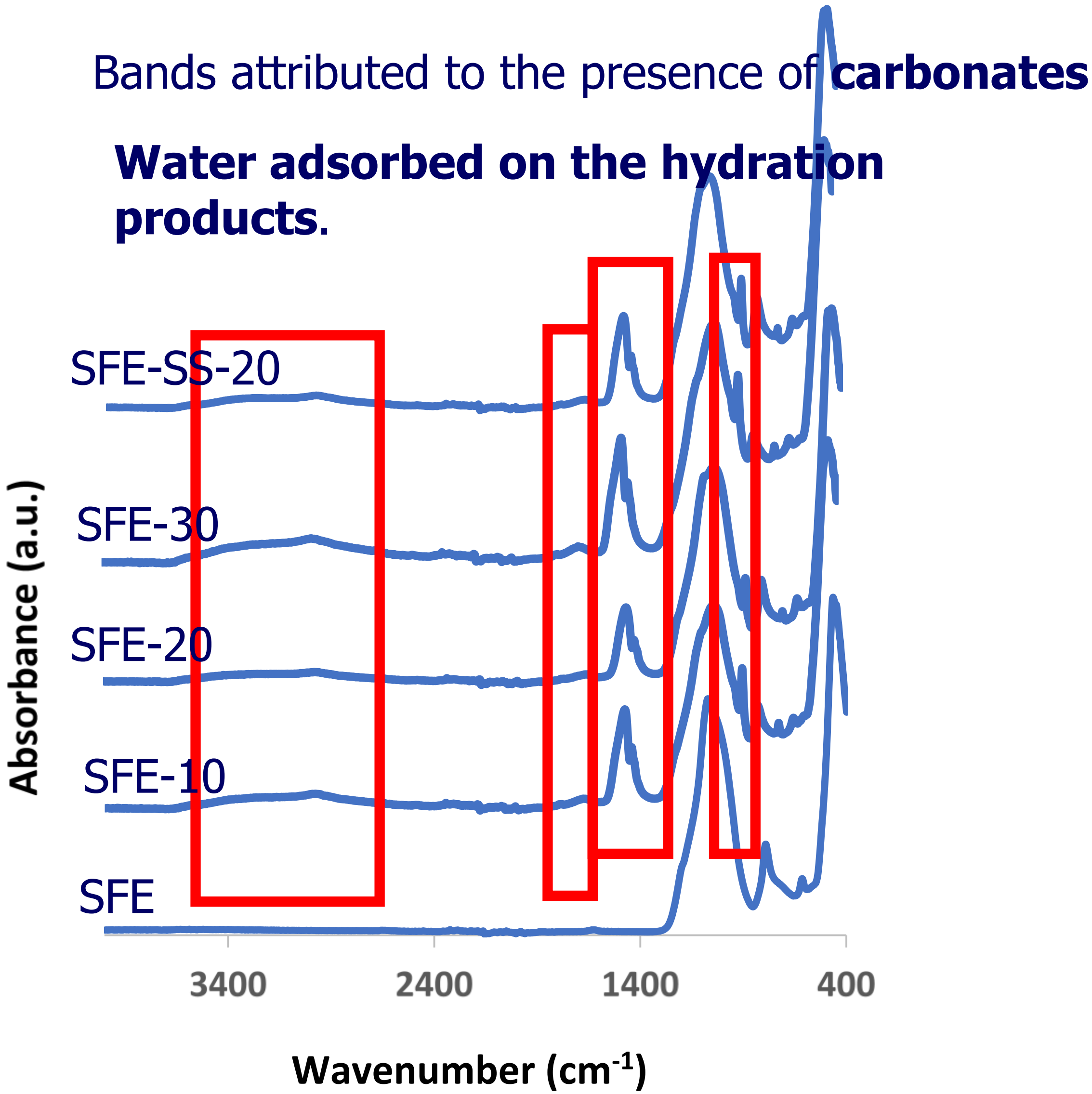
FTIR

Asymmetric vibrations generated by the **T-O-T bonds** (where T is Si or Al)



This band **shifts towards lower wavenumbers** in **geopolymers** indicating the **formation of sodium aluminosilicate gel**

FTIR


Bands attributed to the presence of **carbonates**.
Water adsorbed on the hydration products.
SFE-SS-20
SFE-30
SFE-20
SFE-10
SFE
Absorbance (a.u.)
3400
2400
1400
400
Wavenumber (cm^{-1})

Conclusions

ASA can be used in the manufacture of **SFE cements**.

The **dosage of the alternative alkaline activator** has a **significant influence on the physical and mechanical properties** of **geopolymers** using **SFE as a precursor.**

At a **low alkali dosage** of **10 % Na_2O** and a **high dosage** of **30 % Na_2O SFE geopolymers** show a **low amount of amorphous reaction products**, **high porosity** and **poor flexural and compressive strength**.

The **optimal amount of alkali (20 % Na_2O) promotes** the **geopolymerisation reaction** resulting in a **denser structure** with **lower porosity improving the mechanical properties** of **SFE geopolymers**.

One-part activation of SFE using an **solid alternative activator made** from **glass** and **sodium hydroxide results** in **geopolymers** with **higher mechanical properties** than **two-part activation** using a **sodium silicate and sodium hydroxide solution** as a **commercial activator**.

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Thank you very much for your attention!

Acknowledgments

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