Synthesis of silica particles from sugarcane bagasse ash for its application in hydrophobic coatings

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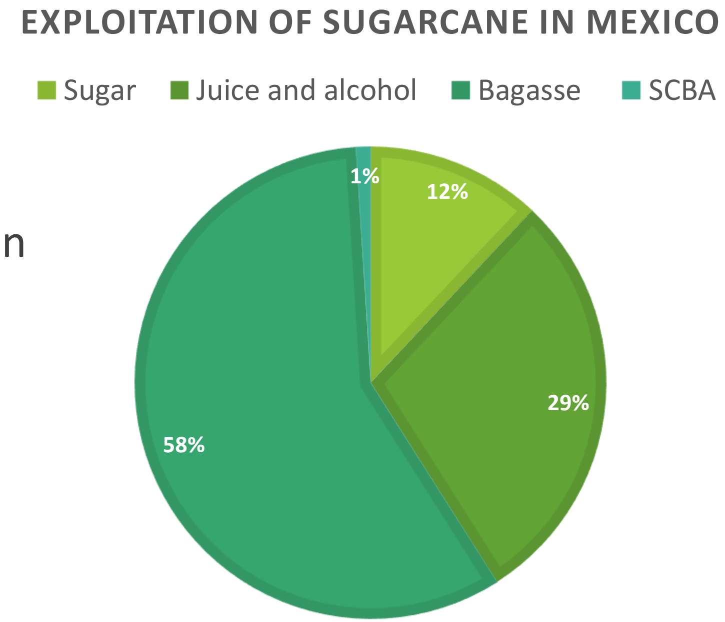
Sugarcane bagasse ash (SCBA) generation

- Mexico 2018
- Processed sugarcane: 51,218,400 ton
- Production:
- 6,009,528 ton of sugar
- 15,187,269 ton of bagasse



- [1]Informe estadístico del sector agroindustrial de la caña de azúcar Zafras 2008/09 – 2017/18. (2018)

Applications of Sugarcane bagasse ash

The high content of silicon oxide, more than 60% in the SCBA, makes possible the utilization of this waste in multiple applications.

- Glass-ceramic synthesis
- High SCBA content(82%).
- Mechanical properties equal or better than commercial materials.
- Low performance.[14]

- Light bricks elaboration
- Low SCBA content(10%).
- Higher wáter absorption.
- Lower mechanical properties.[2]

2. James, J. & Pandian, P. K., (2017).

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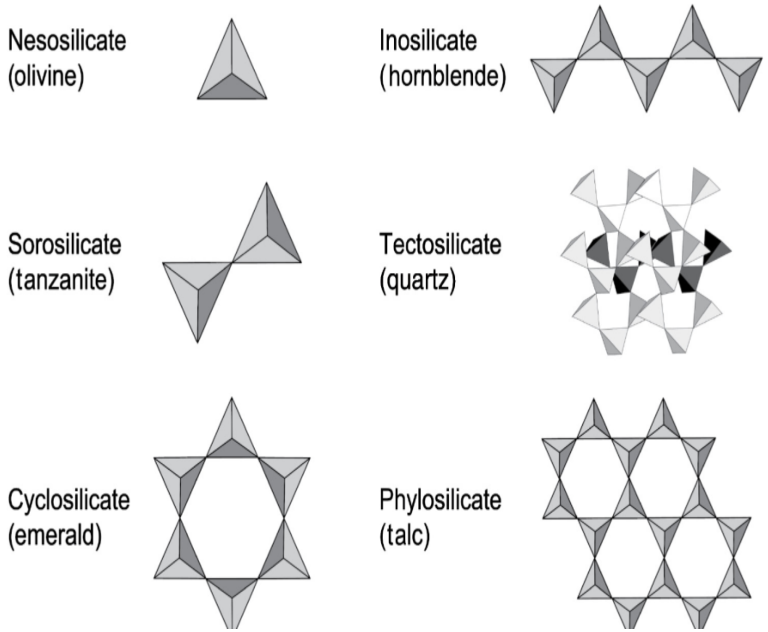
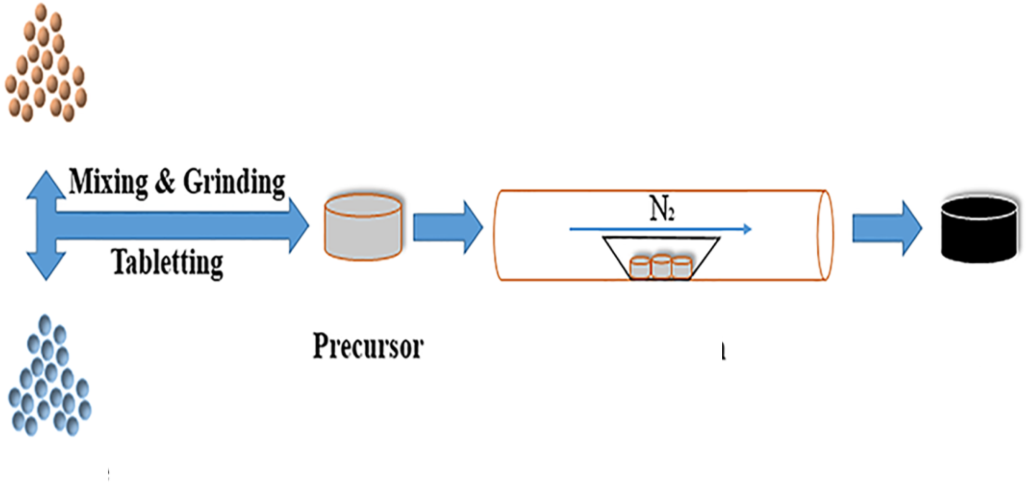
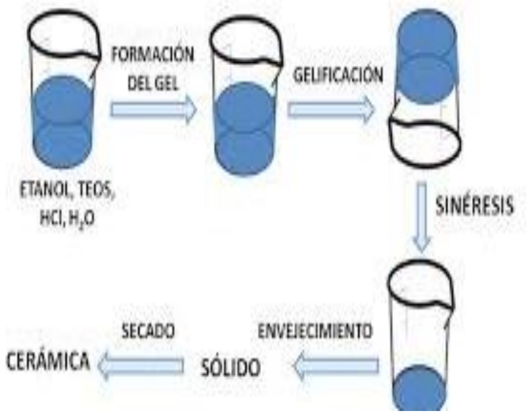


Silicates, a new usage for SCBA

More tan the 80% of earth crust are silicates and they are classified according it's structure.

For a specific use, the silicates must be purified or be synthetized from the beginning. The main methodologies are the thermochemical method and the sol-gel.





[3] Besoain, E. *Mineraalogía de Arcillas y Suelos*. 2 edición (1985)

Sodium silicate and its applications

Sodium silicate can be obtained in different structures (**$Na_{2x}SiO_{2+x}$**) depending the stoichiometry of the synthesis reaction, in addition to it, sodium silicate have multiple applications:

Concrete
binder and selfhealing concrete

Hydrophilic and hydrophobic coatings

Anticorrosive
surfaces for metals, mainly aluminum

Hydrophobic coatings

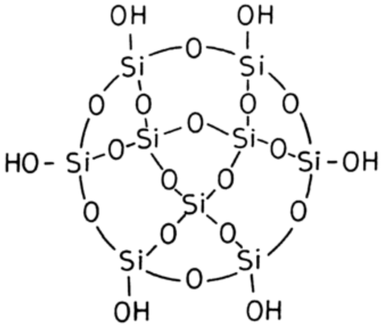
Since the rainwater can be harmful to the building and structures by the eventual dissolution of the concrete o by the corrosion of the internal structure, sodium silicate is proposed as an alternative to prevent these damages with the formation of a siloxane coat.

Sodium silicate, siloxane generation

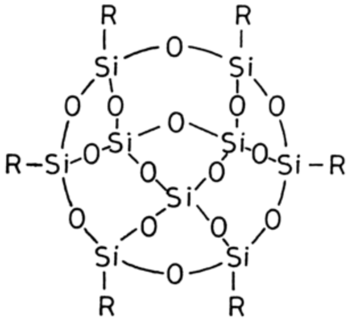
$$Na_2SiO_3 \rightarrow 2Na^+$$

$$Na_2SiO_3 \rightarrow SiO_3^{2-} \xrightarrow{CH_3OH} R - Si - OH \xrightarrow{TEOS} R - Si - O - Si - R$$

R = Alkyl or Aryl



HYDROPHILIC



HYDROPHOBIC

Venkateswara Rao, A. & Haranath, D. *Microporous Mesoporous Mater.* **30**, 267–273 (1999).

Background

Reference	Application	Observations
Venkateswara Rao, A. & Haranath, D.. *Microporous Mesoporous Mater.* **30**, 267–273 (1999).	Synthesis of a silica aerogel with hydrophobic properties	Use of methyltrimethoxysilane as a green alternative for the synthesis of silica aerogels with a siloxane structure
Krug, D. J. & Laine, R. M. *ACS Appl. Mater. Interfaces* **9**, 8378–8383 (2017).	Hydrophobic layers for metals	Use of siloxanes to generate superhydrophobic layers over aluminum
Zulfiqar, U. *et al. Mater. Lett.* **192**, 56–59 (2017).	Superhydrophobic surfaces for glass and building materials	Generation of selfhealing surfaces of silica from sodium silicate, with water contact angles higher than 150°

Proyect objective

Generate a hydrophobic coat based in silica with siloxane structure for building materials to prevent its degradation. Using sugarcane bagasse ash as main raw material for the synthesis of the precursor of sodium silicate.

Specific objectives

01

Characterize the SCBA and obtain the synthesis condition for the sodium silicate with sodium carbonate as source od sodium.

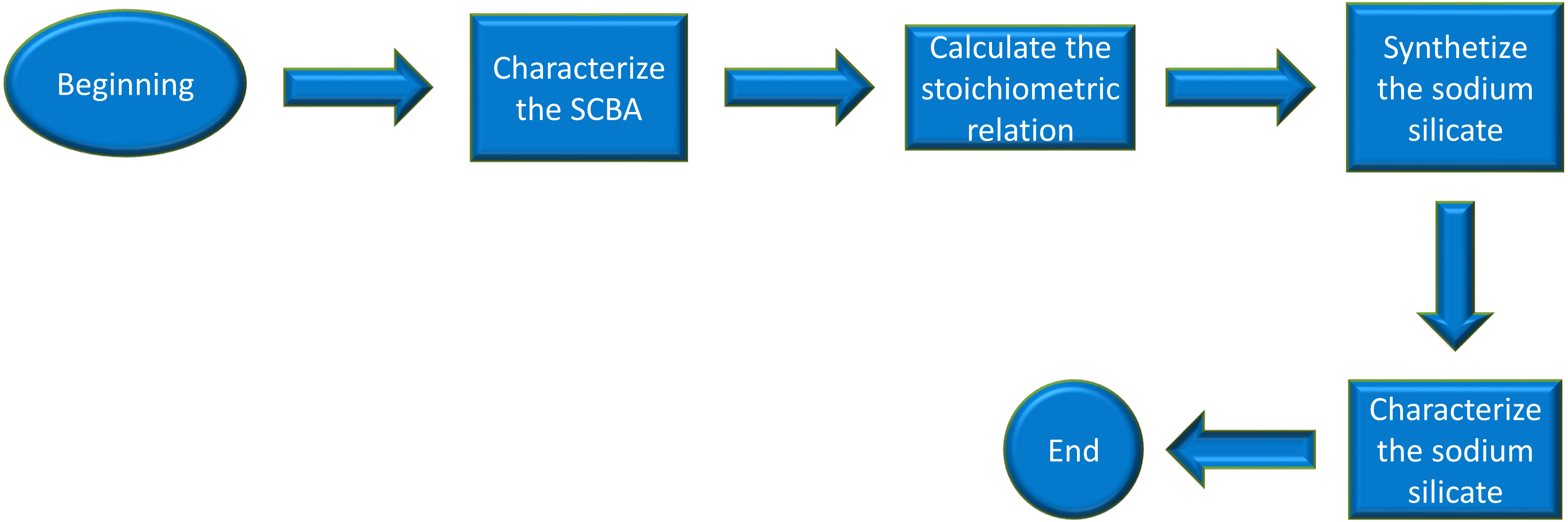
02

Synthetize the sodium silicate by the thermochemical method.

03

Modify the sodium silicate to obtain the silica particles with a characteristic coordination of siloxanes to create a hydrophobic coat for building materials.

General methodology



Methodology

$$XNa_2O + SiO_2 \rightarrow Na_{2x}SiO_{2+x} \quad \text{eq. 1}$$

Beggining

Quantify volatile material in SCBA by elemental analysis

Calcinate SCBA at 900°C and 2 h

Leach SCBA with citric acid at 2% for 2 h and 60 °C

Recover the leach to analyze by F-AAS.

Ground the ash till 150 µm

Analyze with XRF and elemental analysis.

Prepare the batch of ash and sodium carbonate.

Yahya, A. A. *et al.* Reducing Heavy Metal Element from Coal Bottom Ash by Using Citric Acid Leaching Treatment. *MATEC Web Conf.* **103**, 0–6 (2017)

Results

XRF

Compound	%mol
Na_2O	0.48
MgO	0.92
Al_2O_3	2.21
SiO_2	70.85
P_2O_5	1.15
SO_3	1.46
K_2O	4.34
CaO	12.73
TiO_2	0.44
MnO	0.13
Fe_2O_3	4.82
CuO	0.03
ZnO	0.06
SrO	0.13
ZrO_2	0.02
Ag_2O	0.18
BaO	0.04
Total	100

With the XRF analysis we confirm the silica as main component with remnants of oxides that can act as precursor in the synthesis of silicates

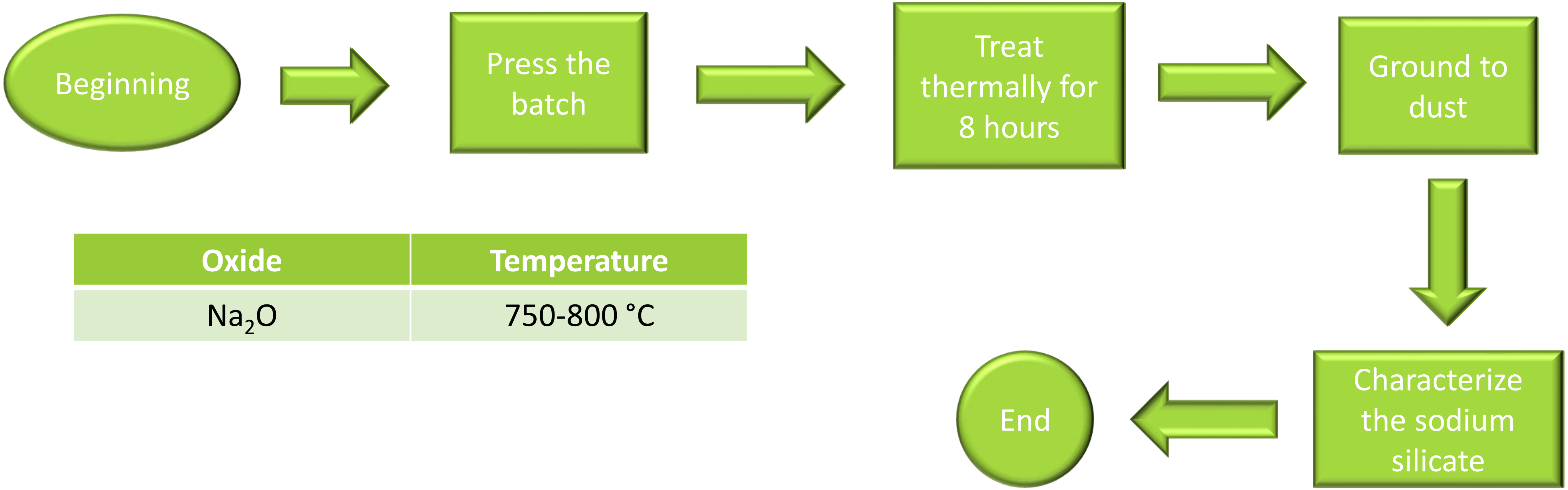
Elemental analysis

Elemento	% w/w before calcination	% w/w after calcination
C	15.86 ± 2.13	0.12 ± 0.05
H	0.15 ± 0.01	0
N	0.25 ± 0.05	0.09 ± 0.02
S	1.32 ± 0.09	0

There are a carbon elimination of almost the 100 % after the calcination, this is important to prevent the fracture of the pellets during the thermochemical synthesis.

Methodology

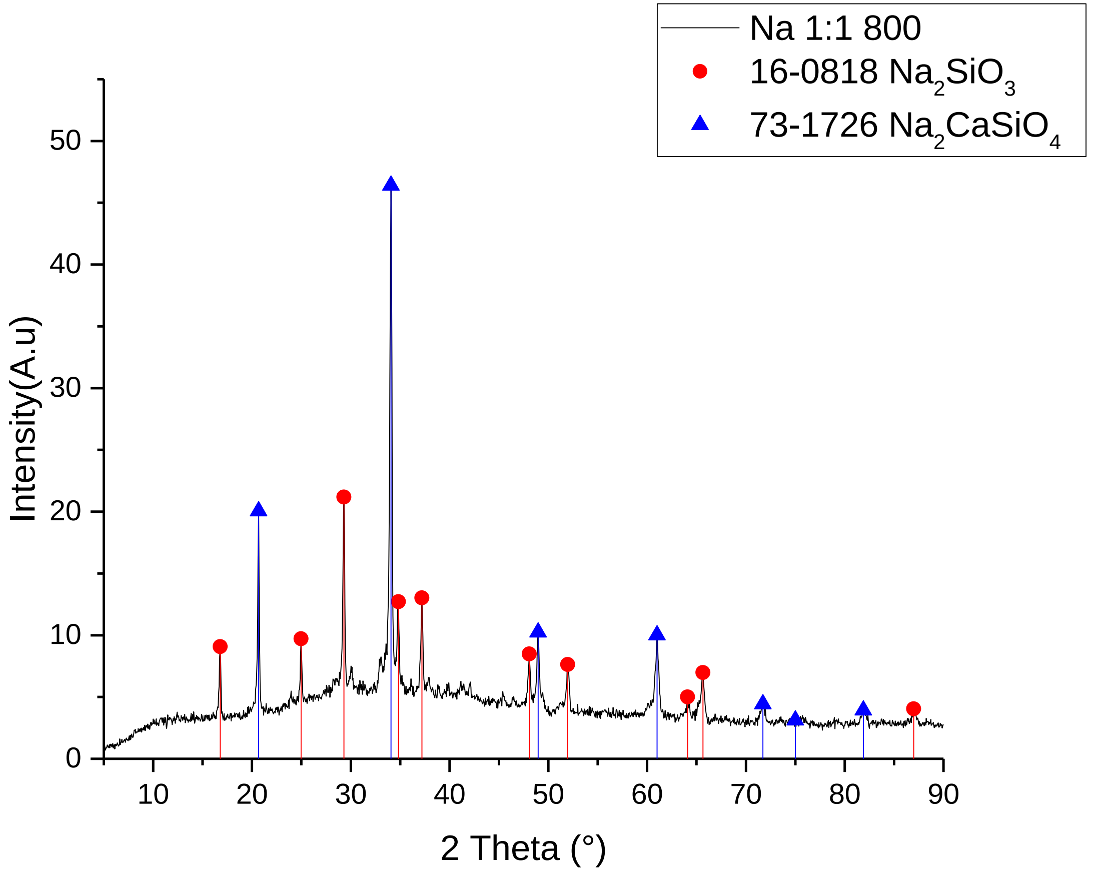
Thermochemical method



Oxide	Temperature
Na_2O	750-800 °C

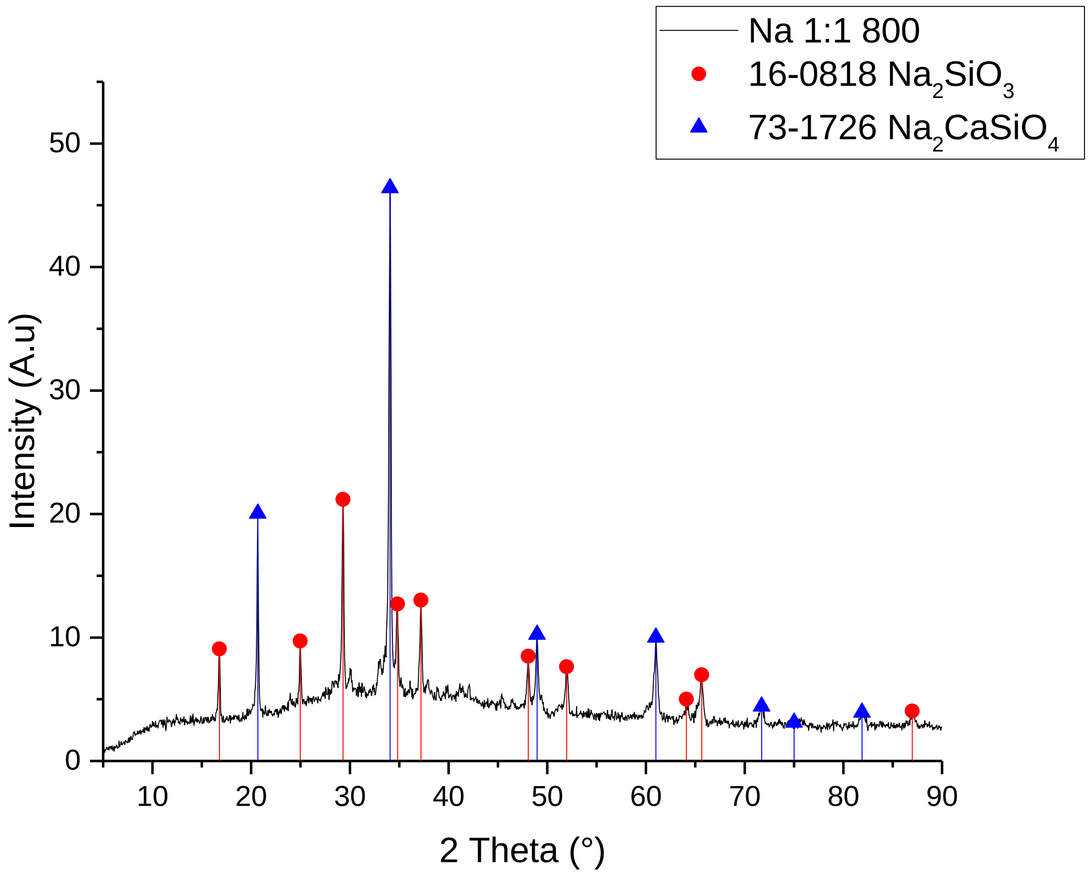
Results Na 1:1 800 °C no leached ash

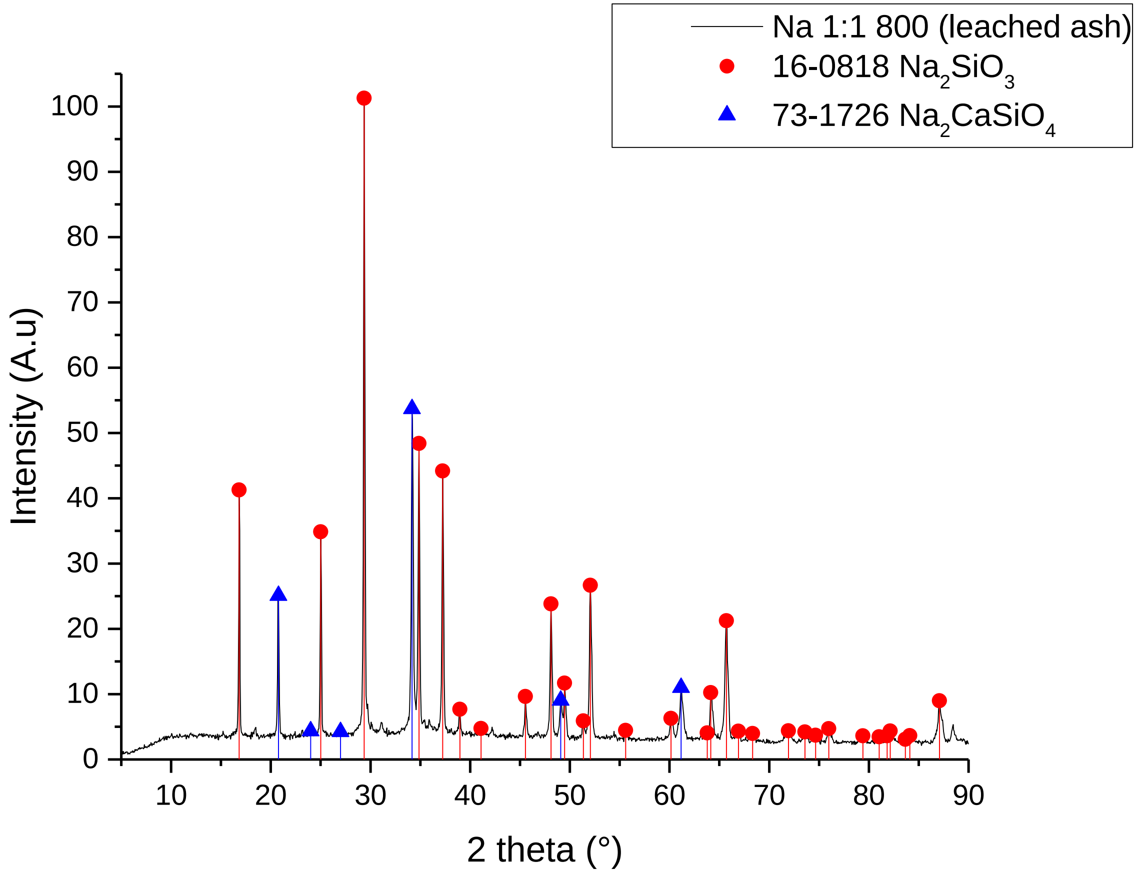
The presence of CaO in the ash in a high proportion generates the sodium calcium silicate, a no soluble silicate which in no valuable for the hydrophobic coat application, reason why the ash was leached. With the leach process the main crystalline phase was sodium silicate as shown in the next slide.



COMPOSITION FOR SODIUM SILICATE SYNTHESIZED IN AQUEOUS MEDIUM. **64**, 7–11 (2007).

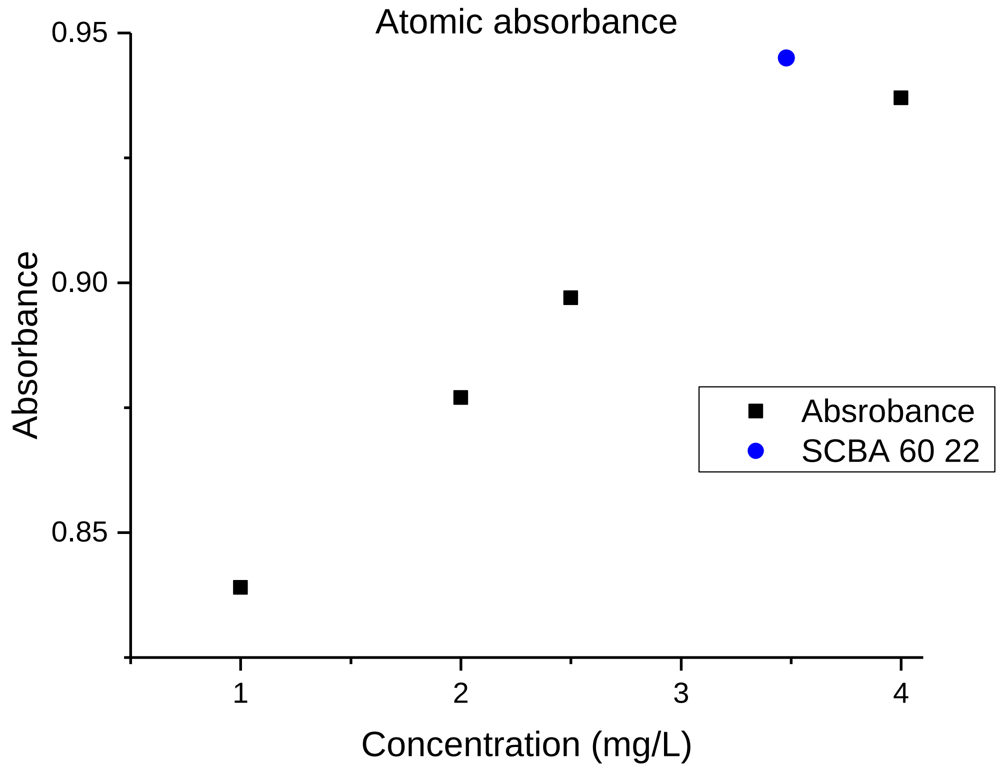
Results Na 1:1 800 °C





Results leached ash

XRF	Leached SCBA %w/w	
Compound	60° 2% 2 h	T_{amb} 2% 2 h
V_2O_5	0.01	0.01
ZrO_2	0.01	0.01
Cr_2O_3	0.01	0.01
CuO	0.02	0.02
ZnO	0.03	0.03
SrO	0.05	0.05
Ag_2O	0.06	0.05
MnO	0.11	0.12
SO_3	0.25	0.26
MgO	0.29	0.28
TiO_2	0.47	0.48
P_2O_5	1.28	1.34
Al_2O_3	1.70	1.77
Fe_2O_3	4.05	4.07
K_2O	5.61	5.66
CaO	7.45	8.14
SiO_2	78.61	77.72
Total	100.00	100.00

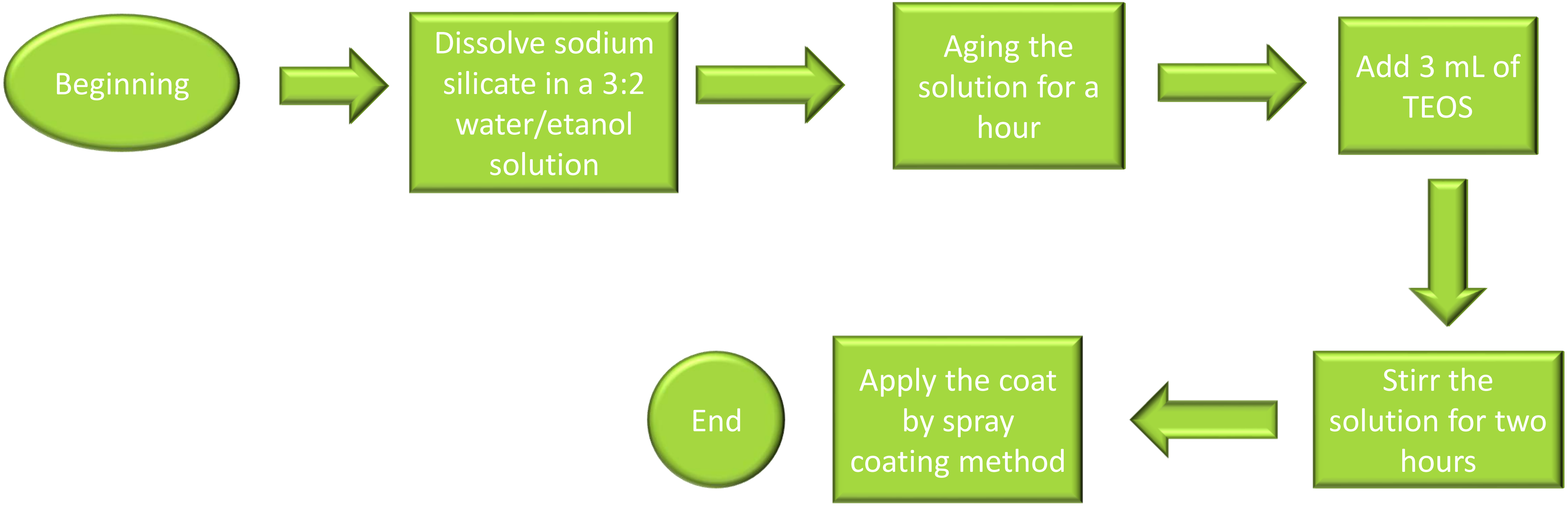


Absorbance	Conc (mg/L)
0.84	1.00
0.88	2.00
0.90	2.50
0.99	4.00
0.95	3.48

The concentration of Ca^{2+} in the diluted sample was 3.48 mg/L. In the original sample the concentration is 434.875 mg/L of Ca^{2+}

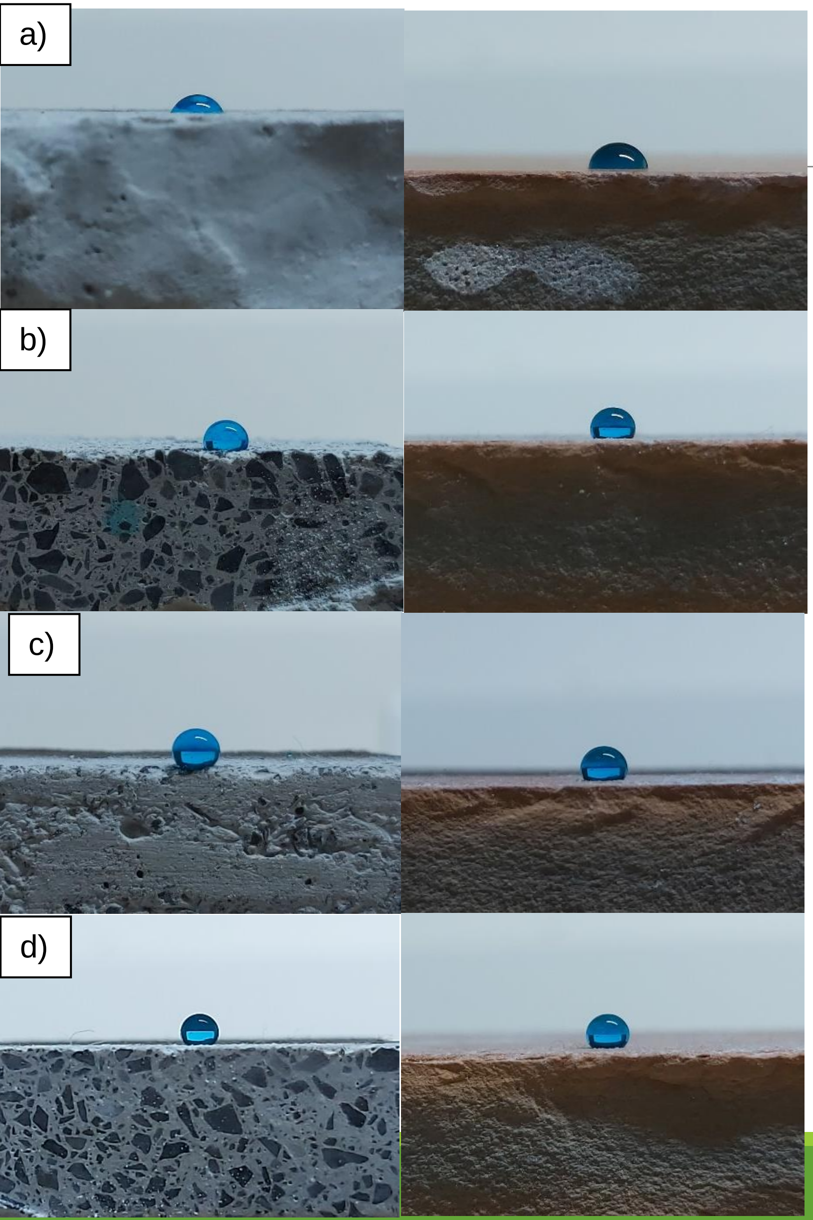
Methodology

Hydrophobic coating



Zulfiqar, U. *et al.* Durable and self-healing superhydrophobic surfaces for building materials. *Mater. Lett.* **192**, 56–59 (2017

Results SCBA no leached

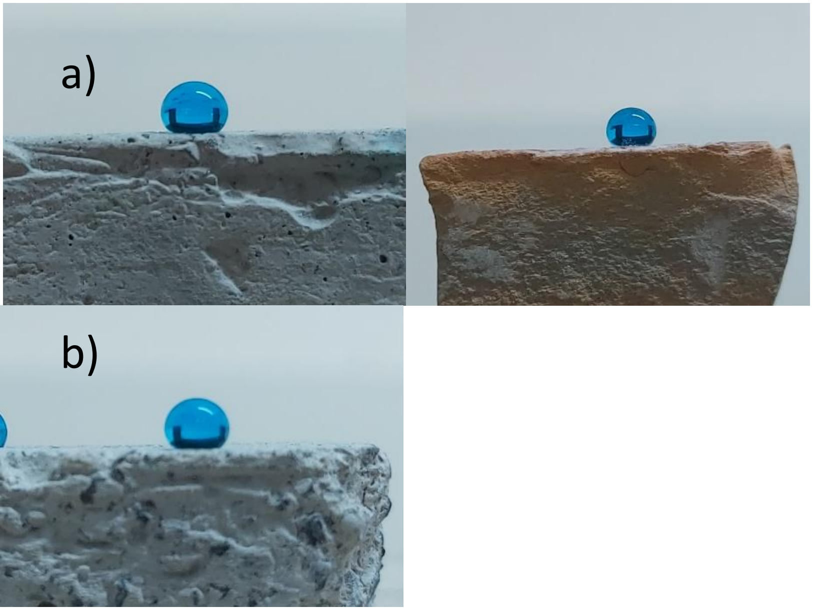


Water droplets over coated surfaces (left concrete, right clay) a) control, b) 10 layers, c)15 layers d) 20 layers

Sample	Mean angle
Concrete control	71.80
Concrete 10 layers	111.50
Concrete 15 layers	140.40
Concrete 20 layers	138.00
Clay control	88.40
Clay 10 layers	122.80
Clay 15 layers	121.40
Clay 20 layers	134.20

As there are lower water contact angle, just for hydrophobic behavior, the test was repeated with sodium silicate of leached SCBA.

Results leached ash



Water droplets over coated surfaces (left concrete, right clay) a) 10 layers, b)15 layers

Sample	*Mean angle*
60 10 concrete	142.10
60 10 clay	146.50
60 15 concrete	146.00

The no reported results correspond to samples which the water droplet bounces off the surface, characteristic of superhydrophobic behavior.

Crick, C. R. & Parkin, I. P. Water droplet bouncing-a definition for superhydrophobic surfaces. *Chem. Commun.* **47**, 12059–12061 (2011).

Conclusion

The SCBA can be used as raw materials for the synthesis of sodium silicate after being leached and calcinated to eliminate the metallic elements that can interfere in the synthesis. Although there is still a sodium-calcium silicate phase, it´s concentration is too low, This can be considered a polluting phase and does not negatively affect the coating. Sodium silicate can be used to generate coatings for ceramic materials. The samples coated showed a nearly superhydrophobic behavior and when there are 15 layers over the clay and 20 layers over the materials both showed a water repellent behavior.

References

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[7] A.A. Yahya, N. Ali, N.L. Mohd Kamal, S. Shahidan, S. Beddu, M.F. Nuruddin, N. Shafiq, Reducing Heavy Metal Element from Coal Bottom Ash by Using Citric Acid Leaching Treatment, MATEC Web Conf. 103 (2017) 0–6. doi:10.1051/matecconf/201710301004.

[8] U. Zulfiqar, M. Awais, S.Z. Hussain, I. Hussain, S.W. Husain, T. Subhani, Durable and self-healing superhydrophobic surfaces for building materials, Mater. Lett. 192 (2017) 56–59. doi:10.1016/j.matlet.2017.01.070.

[9] C.R. Crick, I.P. Parkin, Water droplet bouncing-a definition for superhydrophobic surfaces, Chem. Commun. 47 (2011) 12059–12061. doi:10.1039/c1cc14749h.

Thanks for your attention