Use of analytical techniques for the identification of the morphology of geopolymers

Dimitra Zaharaki[1], Kostas Komnitsas[1] and Georgios Bartzas[2]

[1]School of Mineral Resources Engineering, Technical University of Crete, Chania, 73100, Greece
[2]School of Mining and Metallurgical Engineering, National Technical University of Athens, Zografos Campus, Athens, 15780, Greece

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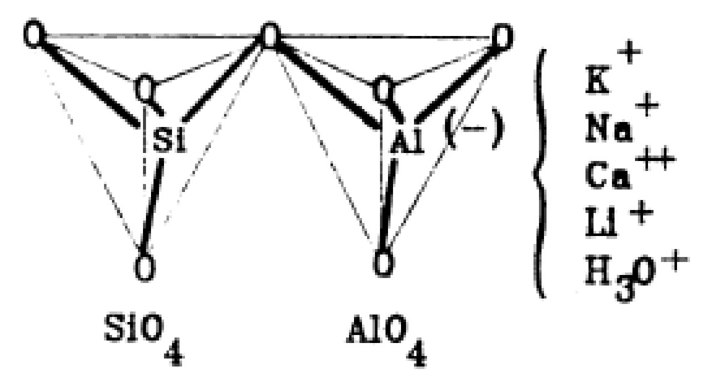
Objectives

XRD, SEM, FTIR and TG were used to provide significant insights on the geopolymerization mechanisms, the mineralogy, the microstructure and the properties of the geopolymers produced from ferronickel slag and the three main components of construction and demolition wastes (CDW), namely concrete, bricks and tiles

Geopolymers

- Geopolymers are cementitious inorganic materials formed by the alkali activation of aluminosilicates at relatively low temperatures
- Partially or fully amorphous polymeric structures consisting of Si–O–Al bonds
- The tetrahedral AlO_4 and SiO_4 units are built in three dimensional structures



Geopolymerization

- During the last two decades, various wastes such as fly ash, slag and red mud have been extensively investigated as potential raw materials for geopolymerization
- The structure and mechanical properties of geopolymers are affected by several parameters
- The complex reactions during alkaline activation of the starting materials may be explained by the values of the molar Si/Al and other ion ratios
- Also a number of simple or advanced techniques may be used to obtain maximum information and elucidate geopolymerisation mechanisms

Experimental design

Materials

- Electric arc furnace slag from the "LARCO S.A" ferronickel plant

- Bricks

- Tiles

- Recycled concrete

Table 1. Particle size (μm) of raw materials

	Slag (S)	Bricks (B)	Tiles (T)	Concrete (C)
size	<120	<140	<140	<190
d_{50}	12	7	14	10

Table 2. Chemical composition (%) of the raw materials

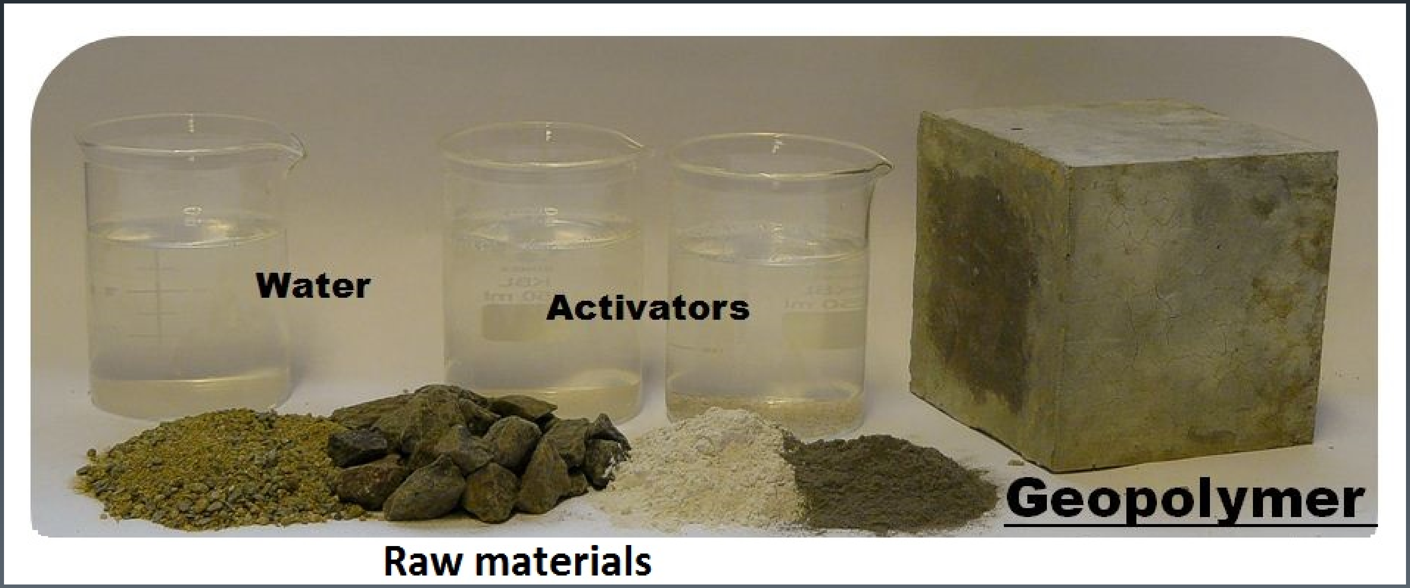
	Slag (S)	Bricks (B)	Tiles (T)	Concrete (C)
Na_2O	-	1.03	-	0.57
MgO	2.76	4.75	4.46	4.21
K_2O	-	2.80	1.37	1.26
CaO	3.73	8.79	8.78	65.42
Fe_2O_3	43.83	6.00	5.39	0.75
Al_2O_3	8.32	14.95	9.80	1.49
SiO_2	32.74	57.79	70.54	5.81
MnO	0.41	0.05	0.06	0.01
SO_3	0.45	-	-	0.82
TiO_2	-	0.85	0.77	0.03
LOI*	-	1.89	0.23	21.59
Sum	95.31**	98.89	101.41	101.95

***LOI: Loss on ignition after heating the material at 1050 ^{o}C for 4 h**

**** Cr_2O_3: 3.07%**

Experimental methodology

- The activating solution consists of NaOH anhydrous pellets, distilled water and sodium silicate solution
- Raw materials are mixed with the activating solution (8, 10 or 14 M NaOH)
- The specimens produced (5 cm edge) were heated at 80 or 90 °C for 7 days and then subjected to compressive strength testing



Analytical techniques

- X-ray diffraction (XRD) (Bruker D8 Advance diffractometer) - useful tool providing data regarding the extent to which crystalline starting materials have reacted
- Scanning electron microscopy (SEM) (JEOL 6380LV scanning electron microscope) - allows a good description of the microstructure of geopolymers
- Fourier transform infrared spectroscopy (FTIR) on KBr pellets (Perkin–Elmer Spectrum 1000 spectrometer) - provides information about the vibrational transitions and rigidity of geopolymeric bonds
- Thermogravimetric analysis (TG) (differential thermogravimetric analyzer Diamond DTA-TG of Perkin Elmer) - identification of the mechanism by which the specimens lose weight as a result of controlled heating

Triple mould for casting of geopolymeric paste

Slag geopolymer

Geopolymers from concrete, bricks and tiles (left to right)

Results and discussion

Geopolymerization potential

Table 3. Optimum conditions for the synthesis of slag (S), brick (B), tile (T) and concrete (C) based geopolymers and molar ratios of oxides of the initial paste

	Slag (S)	Bricks (B)	Tiles (T)	Concrete (C)
Max strength, MPa	52	50	58	13
NaOH, M	8	8	10	14
Temperature, ^oC	80	90	80	90
SiO_2/Al_2O_3	7.33	6.84	12.67	9.86
$SiO_2/(Al_2O_3+CaO)$	4.03	3.30	4.81	0.12
$H_2O/(Na_2O+K_2O)$	8.30	8.32	9.03	6.62
$(Na_2O+K_2O)/SiO_2$	0.10	0.14	0.12	1.53
$SiO_2/(Al_2O_3+Fe_2O_3)$	1.68	5.44	9.38	7.47

These ratios as well as the results from XRD, FTIR, SEM and TG analyses, provide very useful insights on the geopolymerization mechanisms and the microstructure of the final products

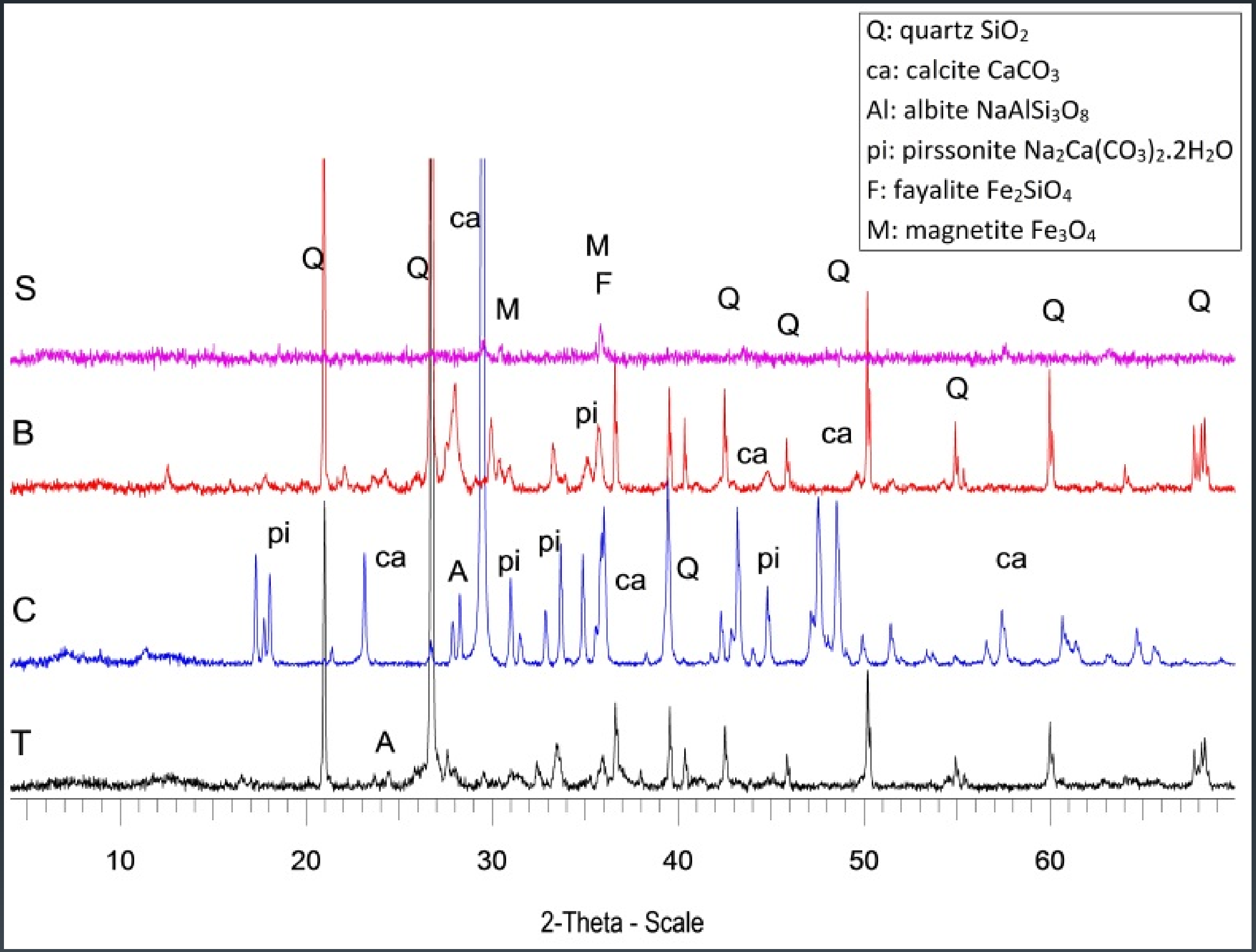


Figure 1. XRD patterns of geopolymers synthesized from slag (S), bricks (B), concrete (C) and tiles (T)

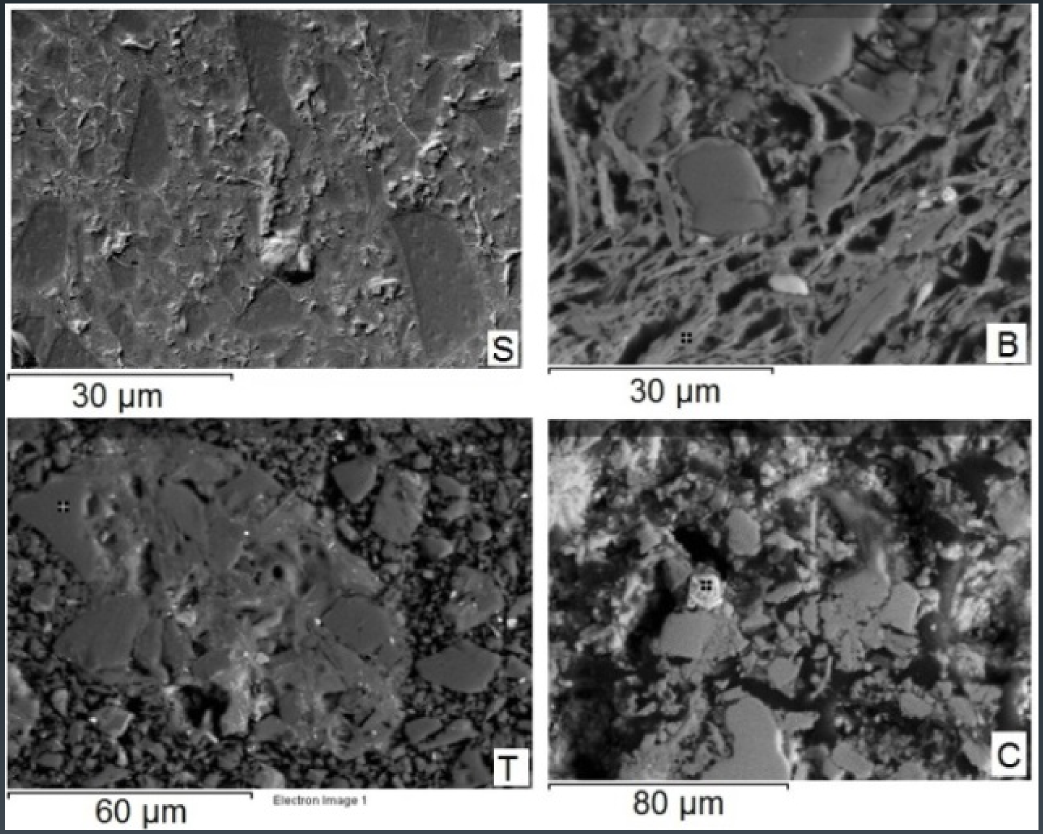


Figure 2. SEM images of geopolymers synthesized from slag (S), bricks (B), tiles (T) and concrete (C)

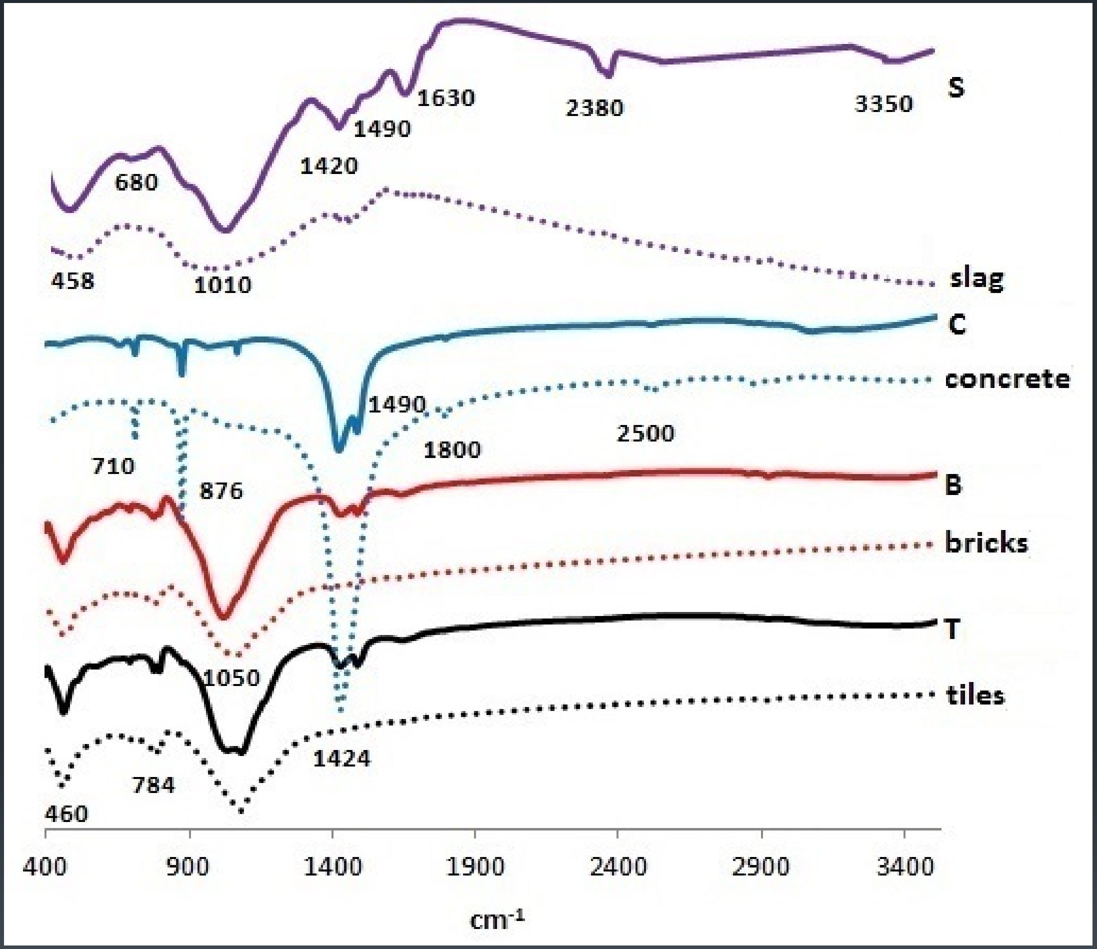


Figure 3. FTIR spectra of the initial components and the geopolymers synthesized from slag (S), bricks (B), concrete (C) and tiles (T)

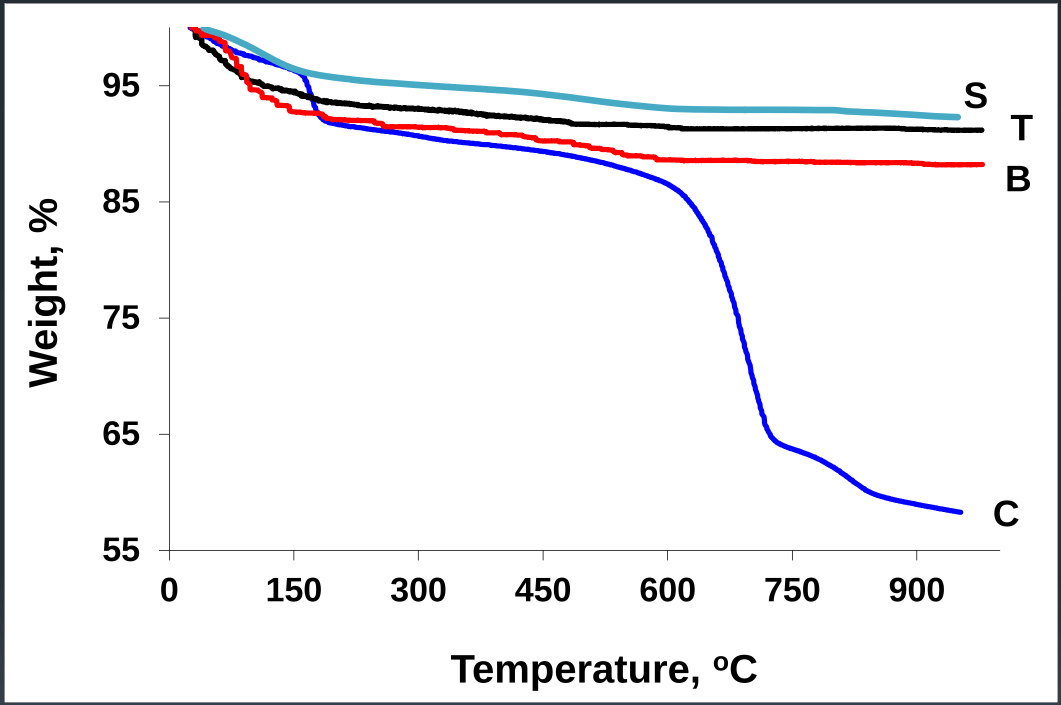


Figure 4. TG curves of geopolymers synthesized from slag (S), bricks (B), concrete (C) and tiles (T)

Conclusions

- The use of analytical techniques, XRD, SEM, FTIR and TG, contributes significantly to the complete characterisation of the microstructure of geopolymers
- An amorphous aluminosilicate gel is formed due to adequate water content and dissolution of Si and Al from the raw materials under highly alkaline conditions
- Slag, bricks and tiles which were successfully geopolymerized and acquired strength higher than 50 MPa, are characterized by a homogenous structure
- Concrete is rather poorly geopolymerized and specimens are characterized by an inhomogeneous matrix where unreacted grains are present

Thank you

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Project website: http://www.durecobel.gr/