

**NOVEMBER  
30<sup>TH</sup> 2018**

**III EUROPEAN  
GEOPOLYMER NETWORK**

**BOOK  
OF  
ABSTRACTS**

**ISTEC-CNR  
FAENZA  
ITALY**



# III European Geopolymer Network Faenza - 30<sup>th</sup> November 2018

## CHAIRS

**Dr. Valentina Medri, CNR-ISTEC**  
**Prof. Angelo Vaccari, UNIBO CNR-ISTEC**

## LOCATION

**Romagna Tech meeting room**  
**via Granarolo 62, Faenza, Italy**

## SECRETARIAT

**Elettra Papa**  
**Annalisa Natali Murri**  
**tel +39 0546 699 776**  
**egn@istec.cnr.it**  
**www.istec.cnr.it**

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*The 3<sup>rd</sup> edition of the European Geopolymer Network follows the meetings of Limoges in 2016 and Madrid in 2017. The event also belongs to the 'one-day-meeting' annually organized by the Working Group on Geopolymers of the Italian Ceramic Society. During the workshop Representatives of the scientific community and industry will meet to review progress on research, innovation and advantages of geopolymers with the aim of providing guidance on their most effective use in various industrial sectors in Europe.*

## ACKNOWLEDGEMENTS

The organization warmly thanks Prof. Sylvie Rossignol

# III EUROPEAN GEOPOLYMER NETWORK

30th November 2018  
Romagna Tech meeting room,  
via Granarolo 62, Faenza, Italy

## Daily Program

- 08.30-09.00:** Registration
- 09.00-09.30:** Opening remarks  
**Dr. Valentina Medri**, III EGN chair, CNR-ISTEC, Italy  
**Prof. Angelo Vaccari**, III EGN chair, University of Bologna, Italy  
**Dr. Anna Tampieri**, Director of CNR-ISTEC, Italy  
**Dr. Michele Dondi**, President of the Italian Ceramic Society, CNR-ISTEC, Italy  
**Prof. Cristina Leonelli**, Coordinator of the Italian Working Group on Geopolymers, University of Modena and Reggio Emilia, Italy
- Morning session I** Chair: **Dr. Valentina Medri**
- 09.30-10.00: Key note - **Prof. João Labrincha et al.**, University of Aveiro CICECO, Portugal  
*Waste-based porous geopolymers for environmental applications*
- 10.00-10.30: Key note - **Dr. Vilma Ducman**, ZAG, Slovenian National Building and Civil Engineering Institute, Slovenia  
*European and national legislation on geopolymers/ alkali activated materials*
- 10.30-10.45:** Coffee break
- Morning session II** Chair: **Prof. Angelo Vaccari**
- 10.45-11.05: Invited - **Dr. Giulia De Aloysio et al.**, CertiMaC/Netzsch, Italy  
*Thermo-physical characterization of geopolymers using the laser flash technique*
- 11.05-11.20: **Dr. Sergio Tamburini et al.**, CNR-ICMATE, Italy  
*Development of prefabricated panels for energy efficiency of buildings within the innoWEE project*
- 11.35-11.50: **Dr. Agnese Attanasio et al.**, CETMA- Centro di Ricerche Europeo di Tecnologie, Design e Materiali, Italy  
*Alkali-Activated Mortars using various Construction and Demolition Wastes*
- 11.50-12.05: **Dr. Serena Chiara Tarantino et al.**, University of Pavia, Italy  
*Reuse of Pietra Serena sandstone waste sludge by alkali activation*
- 12.05-12.20: **Dr. Maurizio Bellotto et al.**, CIRCe, University of Padova, Italy  
*Refractory binders from CAC activated GGBS*
- 12.20-12.35: **Dr. Catherine Davy et al.**, Ecole Centrale de Lille, France  
*3D structure of oil droplets in hardened geopolymer emulsions*
- 12.35-12.50: **Dr. Matteo Minelli et al.**, DICAM, University of Bologna, Italy  
*Novel geopolymer- zeolite composites for CO<sub>2</sub> capture*
- 12.50-13.00:** Poster flash presentation competing for the JECS Trust Best Poster Award
- 13.00-14.20:** Light lunch & poster session

**JECS Trust Best Oral Award Competition Chair: Dr. Michele Dondi**

- 14.20-14.35: OJ1 - **Colin Dupuy et al.**, Andra, IRCER, France  
*Control of the setting time, viscosity and pH value of geopolymer grouts*
- 14.35-14.50: OJ2 - **Elena Crotti et al.**, University of Bergamo, Italy  
*Influence of naphthalene, polycarboxylates and lignosulfonates based superplasticizers on fresh and hardened properties of alkali-activated slag mortars*
- 14.50-15.05: OJ3 - **Paolo Scanferla et al.**, University of Padova, Italy  
*3D printing of geopolymers: the path to innovative ceramic composites*
- 15.05-15.20: OJ4 - **Michiel Giels et al.**, KU Leuven, Belgium  
*Identifying the carbonation front in Fe-rich alkali activated materials*
- 15.20-15.35: OJ5 - **Denny Coffetti et al.**, University of Bergamo, Italy  
*Lightweight alkali activated slag-based plaster for seismic retrofitting and thermal insulation of existing masonry buildings*
- 15.35-15.50: OJ6 - **Enza Vitale et al.**, University of Cassino and Southern Lazio, Italy  
*Chemo-mechanical behaviour of soil improved by alkali activated binders*
- 15.50-16.05: OJ7 - **Nicolas Youssef et al.**, Ecole Centrale de Lille, France  
*A novel approach of geopolymer bricks production based on clay*
- 16.05-16.20: OJ8 - **Vittorio Vezzali et al.**, University of Modena and Reggio Emilia, Italy  
*Construction and demolition waste (CDW) valorization in alkali activated bricks*
- 16.20- 16.30: JECS TRUST voting**
- 16.30- 17.30: Round Table  
JECS Trust Awards  
Conclusive Remarks**

# POSTERS - Contributes

## Poster ID: 01

Qi Technologies s.r.l., Pomezia (RM) (Italy)  
*Planetary Centrifugal Mixer "THINKY MIXER"*

## Poster ID: 02

Garbo S.r.l., Cerano (NO) (Italy)  
*SitoH project: silicates from silicon waste*

## Poster ID: 03

L. Korat, V. Ducman  
ZAG, Slovenian Building and Civil Engineering Institute, Ljubljana (Slovenia)  
*Evaluation of fly ash based foams after exposure to elevated temperature*

## Poster ID: 04

V. Ducman  
ZAG, Slovenian National Building and Civil Engineering Institute, Ljubljana (Slovenia)  
*FLOW - Lightweight alkali activated composite foams based on secondary raw materials*

## Poster ID: 05

I. Lancellotti, A. Baraldi, M. Češnovar, K. Traven, M. Ciabocco, L. Barbieri, M. Berrettoni, V. Ducman, C. Leonelli  
Dipartimento di Ingegneria "Enzo Ferrari", Università di Modena e Reggio Emilia, Modena (Italy);  
ZAG, Slovenian National Building and Civil Engineering Institute, Ljubljana (Slovenia);  
Analisi Control S.r.l., Corridonia (MC) (Italy);  
Dipartimento di Chimica Industriale "Toso Montanari", UOS Campus Rimini, Università di Bologna, Rimini (Italy)  
*Characterization and reactivity of slags as precursors for alkali activate materials*

## Poster ID: 06

G. Barone  
Dipartimento di Scienze Biologiche, Geologiche e Ambientali, Università di Catania, Catania (Italy)  
*AGM for CuHe – Advanced Green Materials for Cultural Heritage*

## Poster ID: 07

L. Carabba, M. Santandrea, C. Carloni, S. Manzi, M. C. Bignozzi  
Department of Civil, Chemical, Environmental and Materials Engineering, University of Bologna, Bologna (Italy)  
*Fly ash-based alkali activated mortar: preliminary study of the fracture energy*

## Poster ID: 08

G. Masi, L. Carabba, M. C. Bignozzi  
Department of Civil, Chemical, Environmental and Materials Engineering, University of Bologna, Bologna (Italy)  
*Corrosion resistance of light-weight fly ash-based alkali activated mortars for fire protection of steel structures*

## Poster ID: 09

C. Ferone, I. Capasso, A. Bonati, G. Roviello, R. Cioffi  
Dipartimento di Ingegneria, Università di Napoli "Parthenope", Napoli (Italy);  
Construction Technologies Institute, National Research Council, San Giuliano Milanese (MI) (Italy)  
*Management and valorization of water potabilization sludge through use in producing geopolymers materials*

## Poster ID: 10

G. Roviello, L. Ricciotti  
Dipartimento di Ingegneria, Università di Napoli "Parthenope", Napoli (Italy)  
*Geosyl: geopolymer hybrid materials for high energy efficient systems*

## Poster ID: 11

B. Liguori, P. Aprea, G. Roviello, C. Ferone  
Applied Chemistry Labs-Department of Chemical, Materials and Industrial Engineering, University of Naples Federico II, Naples (Italy);  
Materials Science and Engineering Research Group MASERG, Department of Engineering, University of Naples Parthenope, Naples (Italy)  
*Self-supporting zeolite by geopolymer gel conversion (GGC)*

**Poster ID: 12**

C. Bordignon, E. Apollo

Aeronautical Service s.r.l., Fiumicino (RM) (Italy)

*Nano-structured carbon fiber material for high temperature applications*

**Poster ID: R1**

C. Ferone, M. Vukcevic, G. Roviello, I. Boškovic, V. Grudic, S. Radusinovic

Dipartimento di Ingegneria, Università di Napoli «Parthenope», Napoli (Italy);

Faculty of Metallurgy and Technology, University of Montenegro, Podgorica (Montenegro)

*Project: valorization of red mud waste in the sustainable production of advanced geopolymer components*

## JECS TRUST Best Poster Award Competition

**Poster ID: PJ1**

E. Kamseu, V. Alzari, D. Nuvoli, D. Sanna, A. Mariani, C. Leonelli

Department of Chemistry and Pharmacy, INSTM Unit, Sassari (Italy);

Department of Engineering Enzo Ferrari, University of Modena and Reggio Emilia, Modena (Italy)

*Dependence of the geopolymerization and end products from the nature of the solid precursors*

**Poster ID: PJ2**

K. Traven, M. Češnovar, V. Ducman

ZAG, Slovenian National Building and Civil Engineering Institute, Ljubljana (Slovenia);

Jozef Stefan International Postgraduate School, Ljubljana (Slovenia)

*Influence of particle size on mechanical properties of slag based alkali activated materials*

**Poster ID: PJ3**

M. Češnovar, K. Traven, V. Ducman

ZAG, Slovenian National Building and Civil Engineering Institute, Ljubljana (Slovenia);

Jozef Stefan International Postgraduate School, Ljubljana (Slovenia)

*Ageing and curing of alkali activated slag based materials*

**Poster ID: PJ4**

L. Valeriani, M. C. Bignozzi, C. Beneventi

Dipartimento di Ingegneria Civile, Ambientale e dei Materiali, Università di Bologna (Italy);

Marazzi Group S.r.l. (Italy)

*New geopolymers based on wastes of ceramic industry as innovative products for tile industry*

**Poster ID: PJ5**

G. Barone, C. Finocchiaro, P. Mazzoleni, A. Stroschio, C. Sgarlata, I. Lancellotti, C. Leonelli

Dipartimento di Scienze Biologiche, Geologiche e Ambientali, Università di Catania, Catania (Italy);

Dipartimento di Ingegneria "Enzo Ferrari", Università di Modena e Reggio Emilia, Modena (Italy)

*Effect of metakaolin addition on AAM based on volcanic ash and ghiara (Etna-Sicily)*

**Poster ID: PJ6**

A. Alfocea-Roig, A. Maldonado-Alameda, J. Mañosa, J. Giro-Paloma, J. Formosa, J.M. Chimenos

Departament de Ciència de Materials i Química Física, Universitat de Barcelona, Barcelona (Spain)

*Development of alkali activated cements by using municipal solid waste incineration (MSWI) weathered bottom ash*

**Poster ID: PJ7**

K. Goulart de Oliveira, L. Martone, M.C. Bruzzoniti, P. Colombo

Department of Industrial Engineering, Università di Padova, Padova, (Italy);

Department of Chemistry, Università di Torino, Torino (Italy)

*Geopolymeric structures for water purification*

# KEYNOTE LECTURE

## WASTE-BASED POROUS GEOPOLYMERS FOR ENVIRONMENTAL APPLICATIONS

Rui M. Novais, João A. Labrincha\*

\*jal@ua.pt

Department of Materials and Ceramic Engineering / CICECO- Aveiro Institute of Materials, University of Aveiro, Campus Universitário de Santiago, 3810-193 Aveiro, Portugal

**Key Words:** waste; inorganic polymers; pH buffering; pollutants adsorption.

Industrial wastes perception has dramatically changed over the past decades. Nowadays, wastes are widely considered as a vital, abundant and low-cost resource to cope with the increasing demand and distressing scarcity of virgin raw materials. Despite the paradigm change, from waste to resource, several challenges (e.g. public acceptance, wastes variability, hazardous elements leaching) still remain hindering wastes recycling into common applications (e.g. building materials). In this context, innovative waste management methodologies are eagerly pursued. Geopolymerization is an exciting technology that may incorporate significant amounts of wastes in their composition, either as raw materials [1-3] or fillers [4]. However, it is a rather complex process, in particular when using waste materials. Nevertheless, waste-based geopolymers can be optimised and tailored to desired applications. Geopolymers have been mainly considered as environmental friendlier alternative to Portland cement. However, these innovative materials present other very interesting properties that may allow their use in high added-value applications. In this work, biomass fly ash, produced by a Portuguese pulp and paper industry, as used as aluminosilicate source in inorganic polymer production. Then the possibility of using them in distinct applications was evaluated: i) as pH regulators in the anaerobic digestion [5] and ii) as adsorbents for wastewater treatment (e.g. methylene blue [6] and lead extraction [7]). Results show remarkable pH buffer ability for the produced waste based geopolymer. The inorganic polymers can also efficiently remove heavy metals or dyes from industrial wastewaters.

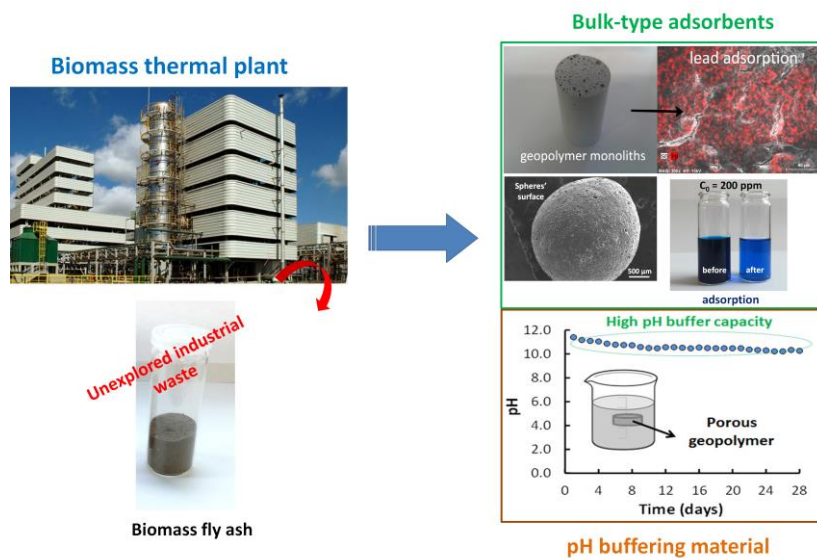


Figure 1 – From waste to resource: biomass fly ash as an aluminosilicate source in the synthesis of inorganic polymers for environmental remediation applications.

### ACKNOWLEDGMENTS

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- [2] Novais et al. (2017) Journal of Cleaner Production 143, 1114-1122.
- [3] Novais et al. (2016) Journal of Cleaner Production 119, 99-107.
- [4] Novais et al. (2018) Construction and Building Materials 184, 464-472.
- [5] Novais et al. (2018) Journal of Cleaner Production 178, 258-267.
- [6] Novais et al. (2018) Journal of Cleaner Production 171, 783-794
- [7] Novais et al. (2016) Journal of Hazardous Materials 318, 631-640.



# KEYNOTE LECTURE

## EUROPEAN AND NATIONAL LEGISLATION ON GEOPOLYMERS/ ALKALI ACTIVATED MATERIALS

Vilma Ducman  
vilma.ducman@zag.si

Slovenian National Building and Civil Engineering Institute, Dimičeva 12, 1000 Ljubljana, Slovenia

**Key Words:** alkali activation technology, construction product regulation (CPR), technical assessment

Technical requirements and marketing of construction products is regulated by Regulation (EU) No 305/2011 (Construction Product Regulations or CPR) [1] which defines procedures for placing construction products legally on the EU market. For a majority of building products these procedures are described in detail in harmonized European Standards (hEN). But innovative products are often not covered by the scope of hENs. For such products there are other ways of legally entering the EU market. Alkali activated materials (AAM) belong to a group of innovative building products where it is very likely that products are not covered by the scope of hENs. Apart from the Construction Sector, AAM can also be used in other fields, like refractory sector or automotive industry. For such sectors there are many EN standards available but there is no legal requirement to follow those standards. Placing such products on the EU market is regulated through the General Product Safety Directive 2001/95/EC [2].

Innovative construction products not covered by harmonized standards can be CE marked based on European Assessment Documents (EAD). In case of such products the producer can apply for an European Technical Assessment (ETA) at a notified Technical Assessment Body (TAB). The basis for the ETA is a corresponding EAD. If no such EAD exists the TAB develops an EAD in collaboration with other members of the European Organization for Technical Assessment (EOTA). ETA includes information on the intended use and performance of a product. The procedure for market entrance for building products is very shortly and very clearly presented on EC or different national sites related to the CPR [3].

Innovative products can also enter the market through a national assessment procedure (National Assessment Regulations), and subsequently enter the European Market [4]. Usually national procedure is a bit faster (at least there is no need to circulate the assessment among TABs) but if such assessment is used then in another member state there might be a need for specific additional testing (like seismic resistance on seismic territory, or freezing resistance...) about what manufactures can be informed through National Contact Points.

Examples of selected case studies, like AA binders, AA aggregate, AA concrete, foamed insulating AA boards and façade panels based on AAM [5] developed within various research projects have been analyzed and procedures for technical assessment and market entrance have been proposed.

### ACKNOWLEDGMENTS

The present work has been partially supported by **H2020 InnoWEE** project (2016-2020), funded from the European Union's Horizon 2020 research and innovation programme under grant agreement No. 723916, **ERA-MIN FLOW** project funded from the Ministry of education, science and sport (acronym: MIZS) under grant agreement No. C 3330-18-252010, and Slovenian National project **ARRS J2-9197** (2018-2021) funded by Slovenian Research Agency (ARRS).

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[2] [https://ec.europa.eu/info/business-economy-euro/product-safety-and-requirements/consumer-product-safety/product-safety-rules\\_en](https://ec.europa.eu/info/business-economy-euro/product-safety-and-requirements/consumer-product-safety/product-safety-rules_en)

[3] <https://www.gov.uk/guidance/eu-construction-products-regulation-and-ce-marking-including-uk-product-contact-point-for-construction-products>,

[4] REGULATION (EC) No 764/2008 laying down procedures relating to the application of certain national technical rules to products lawfully marketed in another Member State

[5] PAS 8820:2016 Construction materials. Alkali-activated cementitious material and concrete. Specification.



## THERMO-PHYSICAL CHARACTERIZATION OF GEOPOLYMERS USING THE LASER FLASH TECHNIQUE

Luca Laghi<sup>1</sup>, Giulia De Aloysio\*<sup>1</sup>, Annalisa Natali Murri<sup>2</sup>

\*g.dealoyisio@certimac.it

<sup>1</sup> CertiMaC ScarL, Faenza, Italy

<sup>2</sup> ISTECCNR, Institute of Science and Technology for Ceramics, Faenza, Italy

**Key Words:** Metakaolin-based geopolymer, composite, Alkali bonded lightweight material, Laser flash technique, Penetration of the laser flash

The thermo-physical characterization of two different kinds of porous geopolymeric materials carried out by means of the laser flash (LFA) technique is presented in this work in order to investigate high-temperature applications and to confirm the LFA method for porous and lightweight materials.

The first material under consideration is a metakaolin-based geopolymer composite for which ashes derived from combustion of vegetal and animal biomass are employed. This material is obtained through the activation of a blend of metakaolin and biomass ash in a weight ratio of 1:1 and foamed with the addition of H<sub>2</sub>O<sub>2</sub> in measure of 5 wt %, in order to maximize the volume of disposed ash and to guarantee appropriate characteristics to the material. The metakaolin-based geopolymer composite has an average bulk density of about 488 kg/m<sup>3</sup>. The fully inorganic nature of the material allows a significant high temperature stability up to 800°C to be ensured and the employment of a direct foaming technique enables to build up a tailored pore network to enhance its insulation ability without affecting its mechanical properties. Therefore, this composite under consideration is able to satisfy both the environmental and performance needs and it represents a promising solution for low-cost self-bearing thermal insulating partitions or lightweight cores for thermos-structural sandwich panels to be used in the construction and in the transportation industries.

The other material is a “non-conventional” geopolymer. It deals with an alkali bonded lightweight material, with a density of about 467 kg/m<sup>3</sup> and a temperature stability ensured up to about 550°C. It is produced by means of fine granules of expanded perlite as raw powder and potassium di-silicate, that was chosen due to its slightly higher softening points in comparison with sodium silicate. The potassium di-silicate plays the role of a binder for expanded perlite granules but also of an alkali activator because a partial geopolymerization was promoted through shear mixing. Due to their role of binder for fillers, aggregates and eventually not reacted materials, the geopolymer nanoprecipitates enable to produce composites which may have the ideal features of lightweight materials for building and industrial sectors, like non flammability, durability, chemical resistance to organic solvents and safety for human health.

The thermo-physical characterization of both the materials is performed in the temperature range between 25°C and 550°C, where they are both thermally stable. The results of the measurements have been then evaluated by means of different models available and implemented in the software Proteus® associated to LFA 467 HT Hyperflash experimental apparatus, by NETZSCH.

In particular, the thermal model based on Mc Master et al. [1] has been considered, when the first material is taken into consideration. It is the so called “Penetration Model” and it is particularly suitable for porous products, because it considers the absorption of the pulse energy into the porosities. Moreover, the model allows for a thorough evaluation of the heat transfer mechanism inside the material itself.

With regard to the second material, the thermal diffusivity was modelled by means of the improved Cape Lehmann Model [2] which allows us for the consideration of two dimensional heat flow and for heat losses, but also for the radiation effect above 500°C.

After determining the thermal diffusivity, the specific heat was measured with the same apparatus for both the materials. From the data regarding the thermal diffusivity, the specific heat and the density, it has been possible to determine the thermal conductivity of both the materials.

In particular, when focusing on the metakaolin-based geopolymer composite, the thermal conductivity ranges between 0.072 W/mK at 25°C and 0.128 at 550°C, showing interesting thermal insulating ability.

With regard to the alkali bonded lightweight material, the thermal conductivity varies between 0.084 W/mK at 25°C and 0.121 W/mK at 550°C. These values give evidence that lightweight alkali bonded expanded perlite materials may be produced with enhanced thermal conductivity.

### ACKNOWLEDGMENTS

The authors thank Dr. Daniele Rebecchi and Dr. Chiara Baldini from NETZSCH and Mrs. Andrea Kesselboth and Mr Alexander Spengler from NETZSCH for the precious suggestions in the samples analysis and in the post-analysis phase.

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**DEVELOPMENT OF PREFABRICATED PANELS FOR ENERGY EFFICIENCY OF BUILDINGS WITHIN THE INNOWEE PROJECT**

S. Tamburini<sup>1</sup>, M. Natali<sup>1</sup>, M. Panizza<sup>1</sup>, C. Tsoutis<sup>2</sup>, V. Ducman<sup>3</sup>, F. Sonzogni<sup>4</sup>, A. Bernardi<sup>5</sup>

\* sergio.tamburini@cnr.it

<sup>1</sup> ICMATE-CNR, Institute of Condensed Matter Chemistry and Energy Technologies, Padua, Italy

<sup>2</sup> Advanced Management Solutions 15, I. Nika, Acharnai, Athens, GR 14122, Greece,

<sup>3</sup> Slovenian National Building and Civil Engineering Institute ZAG, Dimičeva 12, 1000 Ljubljana, Slovenia

<sup>4</sup> Magnetti Building SPA, Bergamo, Italy

<sup>5</sup> CNR, ISAC, C.so Stati Uniti 4, 35127, Padova, Italy

**Key Words:** Geopolymers, Construction & Demolition Waste, Sustainability, Energy-efficiency

Construction and Demolition waste (CDW) is one of the main sources of waste in Europe, with approximately one billion tons generated in the EU 27 every year. The Waste Framework Directive 2008/98/EC set the target for its reuse, recycle or recover at 70% in weight by 2020.

In this context, the H2020 project InnoWEE – Innovative pre-fabricated components including different Waste construction materials reducing building Energy and minimising Environmental impacts, started in 2016. The project is focussed on the development of prefabricated panels, made of geopolymer materials containing either inorganic or wood CDW, for the improvement of the energy efficiency of new and renovated buildings.

The geopolymer mixtures include 50% by weight of inorganic CDW, or either 40% or 50% of wood CDW according to their application (exterior or interior). Waste aggregates were extensively characterized from a chemical, physical and health/safety standpoint, and their production was assessed in terms of input materials and processing to better exploit the properties of geopolymer binders. The binders were optimized to meet the requirements of real-scale panels, such as robustness, absence of flaws, impact behaviour, etc., and were tuned to suit the needs of their pilot plant, especially regarding open time and fluidity.

Four types of products are under development, namely an ETICS-like panel for the exterior insulation of building walls, a ventilated façade panel, and two modular hydronic radiating panels for either ceiling or wall heating/cooling systems. The prototyping phase of the panels was concluded in March 2018, and the pilot production for the four demo sites located in Italy, Belgium, Greece and Romania, is ongoing.

The installation on real buildings was preceded by a one-year environmental monitoring that will allow a direct comparison of the energetic performance before and after the installation. Models calibrated on real sites will be applied to four virtual demo cases, in order to demonstrate the possible advantages of the application of InnoWEE panels.



*Figure 1 – Installation test of ETICS-like and ventilated façade panels on a free-standing wall*

**ACKNOWLEDGMENTS**



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ALKALI-ACTIVATED MORTARS USING VARIOUS CONSTRUCTION AND DEMOLITION WASTES

Agnese Attanasio\*<sup>1</sup>, Alessandro Largo<sup>1</sup>

\*agnese.attanasio@cetma.it

<sup>1</sup> CETMA - Centro di Ricerche Europeo di Tecnologie, Design e Materiali / Brindisi, Italia

**Key Words:** Alkali-Activated Binders (AAB), Construction and Demolition Wastes (CDW), Sustainable building materials, RE<sup>4</sup> project

Alkali-Activated Binders (AAB) have recently gained considerable interest as an eco-sustainable alternative to Ordinary Portland Cement (OPC), commonly used in the construction sector although its production has high environmental impacts (energy consumption, CO<sub>2</sub> emissions). Industrial by-products have been considered as precursors for the development of AAB suitable for mortars/concretes, these includes pulverized fuel ash (PFA) and ground-granulated blast-furnace slag (GGBS or GGBFS) - respectively, produced and disposed by thermal-power and metallurgical plants. In addition, ceramic powders recycled from Construction and Demolition Wastes (CDW) – can be investigated as precursors for AAB usable for mortars/concretes.

CDW represent a significant waste stream in Europe and, therefore, their management has been identified as a priority target. This waste is regulated by Directive 2008/98/EC which establishes that re-use, recycling and recovery of non-hazardous CDW shall be increased, by 2020, to a minimum of 70% by weight. Attention concerning CDW has recently increased, mainly related to their environmental impacts but also to the recycling potential of such valuable resources for the building sector. CDW contain various recyclable materials such as concrete, mortar, ceramics, wood, various plastics, metals, glass, paper/cardboard, paints/glues, bitumens and soils. Their integration in the construction sector is an effective way to reduce CDW pressure on the environment and, at the same time, to limit the consumption of non-renewable resources for building materials production. RE<sup>4</sup> project - founded by the European Commission and currently in progress (duration 2016-2020) - specifically focus on the integration of CDW in the production cycle of building materials. RE<sup>4</sup> project promotes new technological solutions for the development of sustainable materials, pre-fabricated and cost-effective components and elements incorporating CDW derived materials and intended for both new and refurbished buildings [1].

This study deals with innovative alkali-activated mortars, developed in RE<sup>4</sup> project, based on both industrial wastes (PFA, GGBS) and construction and demolition rubbles (mineral fractions, ceramics, plastics, wood). Two binders have been produced through alkaline activation and consist, respectively, in PFA-GGBS and ceramics-GGBS blends. The binders have been combined with various CDW derived aggregates, which include mineral fractions but also heterogeneous rigid plastics and mixed wood/plastic usually not recycled. The compatibility of CDW aggregates with the binders has been evaluated and technical performance (consistency, density, mechanical strength, thermal conductivity) of the produced mortars assessed and compared. Upon 7 days of curing in normal lab conditions, independently from the specific binder, on average mortars with 100% mineral aggregates reach 20 MPa, mortars with 30% rigid plastics aggregates reach 10 MPa while those with 10% wood/plastics aggregates result in 5 MPa. On the other hand, the combined use of mineral and lightweight aggregates contribute to lower the density (15% reduction of fresh density with rigid plastics, 30% using wood/plastic) and, in turn, in a thermal conductivity



reduction. Overall the developed alkali-activated mortars, integrating recycled binders and aggregates, represent sustainable and low-cost solutions and also encourage the use of secondary raw materials as valuable resources for the building sector.

Figure 1 – RE<sup>4</sup> mortars: alkali-activated binders (PFA/GGBS and ceramics/GGBS blends) combined with CDW-derived aggregates (mineral fraction, rigid plastic and wood/plastic scraps).

**ACKNOWLEDGMENTS**

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REUSE OF PIETRA SERENA SANDSTONE WASTE SLUDGE BY ALKALI ACTIVATION

Serena C. Tarantino<sup>\*1</sup>, Marina Clausi<sup>1</sup>, Ana M. Fernández-Jiménez<sup>2</sup>, Angel Palomo<sup>2</sup>, Michele Zema<sup>1</sup>

\*serenachiara.tarantino@unipv.it

<sup>1</sup> Department of Earth and Environmental Sciences, University of Pavia, Italy

<sup>2</sup> Instituto Eduardo Torroja (IETcc), CSIC, Madrid, Spain

**Key Words:** alkali activated materials; sewage sludge; reuse; circular economy

Approximately 700 million tons of mining and quarrying wastes are produced worldwide each year. They are usually collected, transported and placed in large heaps or dams, whose collapse may have serious impacts on environment and human health and safety. A novel and environmentally sound alternative to current waste sludge management options is badly needed.

Wastes from mineral industry offer potential for upcycling through alkali activation, allowing to make a step towards the implementation of closed-loop material flows.

In this work, waste sludge deriving from the cultivation of an Italian sandstone known as Pietra Serena as precursors has been used as precursor in the alkaline activation process.

Pietra Serena waste sludge (sPS) were used after a thermal treatment at 800 °C for two hours. Mixtures with a high-quality kaolin, heated at 800 °C for two hours to obtain the relative metakaolin, were also used with the aim to produce valid construction materials, being metakaolin one of the most studied precursors, due to its high reactivity and the good properties in terms of resistance and durability of the final products [1].

AAMs obtained by different proportions of binary mixtures of sPS and metakaolin were synthesized using 8M and 12M NaOH solutions and curing samples at 85 °C and 100% R.H. for 5 or 20 hours. Materials were characterized by X-ray power diffraction (XPRD), Fourier transform infrared (FTIR) spectroscopy, scanning electron microscope (SEM), colorimetry and mechanical tests.

The alkali activation of untreated sPS gives a material with limited amount of amorphous gel, in which unreacted particles can be still recognized, and showing a compressive strength of 3.6(2) MPa. The use of fly ashes and metakaolin in different proportions enhances mechanical strength up to 36(2) MPa. In these blends, the sludge acts basically like a filler [2].

The heat treatment aids the valorisation of this type of waste. The induced decarbonation process of the sludge increases the availability of Ca in solution for the alkali activation of the sPS itself and in blends. The produced calcium oxide is hydrated and reacts with the silica present in the material to generate a C-S-H gel with Al incorporated in its structure. In the mix produced by using the calcinated sPS, an interaction occurs, giving rise to a mixture of N,C-A-S-H / C-A-S-H gels, similar to those produced in the so-called mixed alkali cements or hybrid cements.

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REFRACTORY BINDERS FROM CAC ACTIVATED GGBS

Maurizio Bellotto\*<sup>1</sup>, Silvia Contessi<sup>2</sup>, Maria Chiara Dalconi<sup>2</sup>, Enrico Garbin<sup>1</sup>, Gilberto Artioli<sup>2</sup>  
 \*mauriziopietro.bellotto@polimi.it

<sup>1</sup> Centro Interdipartimentale di Ricerca per lo studio dei materiali Cementizi e dei leganti idraulici (CIRCe),  
 c/o Dipartimento di Geoscienze, Università di Padova, Padova, Italy  
<sup>2</sup> Dipartimento di Geoscienze, Università di Padova, Padova, Italy

**Key Words:** alkali activated slag cement, calcium aluminate cement, refractory cement, strätlingite

In the framework of our investigation of all possible approaches to produce one-part, dry powder binders based on ground granulated blastfurnace slag (GGBS), we have investigated the use of Calcium Aluminate Cements (CACs). This activation system is quite interesting thanks to the good reactivity it delivers, the nature of the hydration phases that form upon setting and the properties of the final hardened material. The basic activation of GGBS by Na<sub>2</sub>CO<sub>3</sub> in combination with hydrated lime to accelerate setting and hardening delivers a final hardened product with good fire-resistance properties (compressive strength of 29.5 MPa and 25.1 MPa at 7 days and after calcination to 500°C for 4 hours, respectively). Nevertheless the large amount of calcite forming upon hydration prevents the attainment of a material withstanding high temperatures.

The hydration of mixtures of CAC with GGBS occurs with no, or minimal, precipitation of calcium aluminate hydrates, making these systems free from the physico-mechanical issues caused by the conversion reaction. The activation with CAC40 (calcium aluminate cement with 40% Al<sub>2</sub>O<sub>3</sub> by elemental composition) or CAC80 yields reactive systems already with the presence of 10% CAC. The reactivity, measured by isoperibolic calorimetry, appears to be a function of the Al<sub>2</sub>O<sub>3</sub> content of the CAC, with 10% CAC80 yielding similar hydration kinetics than 20% CAC40. After 1 day hydration all anhydrous CAC phases have disappeared, and the main hydration products are strätlingite and hydrotalcite. Specimens hydrated for 7 days were calcined up to 1400°C. They did not show sign of softening, macroscopic cracking or even surface crackling.

The addition of MgO to these formulations increases the hydration rate by incrementing the quantity of hydrotalcite precipitated, but at the cost of reducing the high temperature properties. The addition of MgO moves the system towards akermanite in the gehlenite/akermanite solid solution, thus decreasing the melting temperature. Consequently we prepared a set of samples based on CAC40, CAC50 and CAC80 with no MgO addition and with Ca:Al:Si ratio approaching as far as possible the one of gehlenite. Where necessary this has been achieved by the addition of fly ash (class C) or metakaolin. The formulations were always designed with the goal of maximising hydraulic reactivity.

The mechanical strength of the specimens at 7 days of curing increases upon calcination at 1100°C for 4 hours, reaching values ranging from 14.5 MPa to 32.4 MPa according to the specific formulation. All materials showed no melting after calcining overnight at 1450°C, as shown in Figure 1 for the formulations based on CAC50 and CAC80.



Figure 1. Prismatic specimens based on CAC50 (a) and CAC80 (b) after calcination at 1450°C overnight.

### 3D STRUCTURE OF OIL DROPLETS IN HARDENED GEOPOLYMER EMULSIONS

C. A. Davy<sup>\*1</sup>, G. Haus<sup>2</sup>, B. Planel<sup>3</sup>, D. Lambertin<sup>4</sup>

\*catherine.davy@centraledille.fr

<sup>1</sup> Centrale Lille and UCCS UMR CNRS 8181, Villeneuve d'Ascq, France

<sup>2</sup> Plateforme d'imagerie par rayons X ISIS4D, 59655 Villeneuve d'Ascq Cedex, France

<sup>3</sup> CEA, DAM, F-21120 Is-Sur-Tille France

<sup>4</sup> CEA, DEN, DE2D, SEAD, LCBC, F-30207 Bagnols sur Cèze, France

**Key Words:** ceramic matrix composite, geopolymer, porous material, percolation, three-dimensional X Ray tomography

Composite materials made of geopolymer (GP) cement [1-4] and organic liquids (oils) have been proposed recently for the synthesis of porosity-controlled media, e.g. as thermal insulating foams or filtration supports [5-10], for the management of radioactive organic liquid waste [11-13], and as phase change materials (PCM) [14]. Indeed, GP cements have the ability to integrate huge amounts of organic oils by direct emulsion in the fresh paste. Moreover, the oil emulsion remains stable during GP hardening. This allows to design tailored GP/oil (GEOIL) composites for an array of industrial applications.

By using 3D X-Ray micro-Computed Tomography (micro-CT) with a voxel size of 1 micron<sup>3</sup>, this research determines the spatial distribution of an industrial oil emulsion inside a GP cement (emulsification in the fresh state, imaging in the hardened state), depending on the oil volume fraction (from 5 to 60% total volume). The oil droplet size distribution, mean distance between droplets, and connectivity of the oil system are determined quantitatively.

In particular, Fig. 1 shows a 3D representation of the oil structure inside the geopolymer for the whole range of oil proportions investigated. One is for 5% vol oil (left) and the other for 60% vol oil (right). From these structures, the size distribution of the oil droplets is identified. The droplet diameter  $d_{50}$  at 50% of the size distribution is similar, without any statistically significant difference, from 5 to 30% oil, with an average value of 21-25 microns depending on the oil percentage. On the opposite,  $d_{50}$  goes up to 34 microns at 40 %vol, 76 microns at 50 %vol and finally 716 microns at 60 %vol. The most significant increase in  $d_{50}$  is therefore obtained above 50 %vol. This indicates a transition in the morphology of the oil network between 30 and 40%vol oil, where the droplets possibly coalesce and become significantly closer to one another.

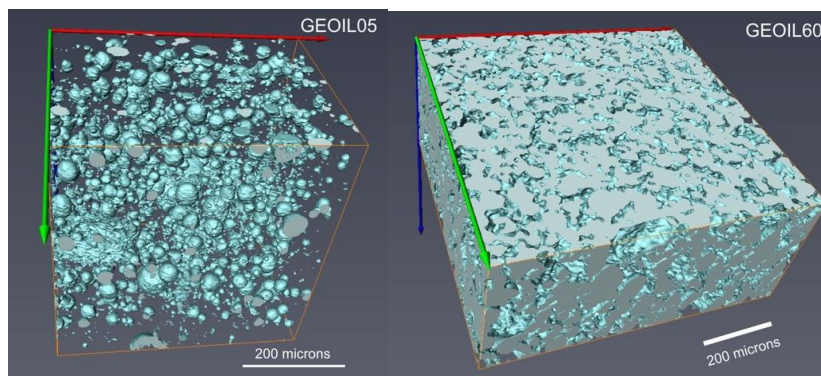


Figure 1: Spatial representation of the oil volume (in light blue) present in the geopolymer composites GEOIL05 and GEOIL60. Axis X is in red, axis Y in green and axis Z is in blue.

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NOVEL GEOPOLYMER- ZEOLITE COMPOSITES FOR CO<sub>2</sub> CAPTUREM. Minelli<sup>1</sup>, E. Papa<sup>2</sup>, P. Benito M.<sup>3</sup>, V. Medri<sup>2</sup>, E. Landi<sup>2</sup>, F. Miccio<sup>2</sup>, F. Doghieri<sup>1</sup>

\*matteo.minelli@unibo.it

<sup>1</sup> Department of Civil, Chemical Environmental and Materials Engineering, University of Bologna, Bologna, Italy<sup>2</sup> ISTECCNR, Institute of Science and Technology for Ceramics, Faenza, Italy<sup>3</sup> "Toso Montanari" Department of Industrial Chemistry, University of Bologna, Bologna, Italy**Key Words:** CO<sub>2</sub> capture, gas adsorption, geopolymer, zeolite, solid adsorbents

Novel porous ceramic materials have been prepared by combining zeolite Na13X with amorphous alkali aluminosilicates (geopolymers) [1], purposely developed for CO<sub>2</sub> capture applications by means of low temperature adsorption processes. The main aim is the combination of the intrinsic microporosity of the zeolite, a benchmark system for CO<sub>2</sub> separation, with the mesoporosity of geopolymers, in order to improve the performance of the sorbent both in terms of sorption capacity and effective diffusional kinetics [2]. The zeolite, indeed, is embedded in the amorphous geopolymer matrix, whose formulation has been tuned (K<sup>+</sup> and Na<sup>+</sup> silicates), to explore different chemical affinity between the two phases and with the gaseous sorbates [3]. The innovative composite systems have been produced in the form of porous monoliths, suitable for application in real separation processes.

The composite ceramic materials are prepared with a Si/Al molar ratio equal to 2, typical of a poly(sialate-siloxo) geopolymer with a K<sup>+</sup> counterion, while a Si/Al ratio equal to 1.2 is used for Na<sup>+</sup> based systems, resembling that of the zeolite Na13X. The fully hydrated zeolite is incorporated into the geopolymer slurries and then casted in plastic molds and cured at 80°C for 48h [3].

The resulting composites have been characterized by SEM analysis that revealed a good compatibility among the two moieties with the formation of geopolymer nanoprecipitates covering zeolite particles in the K<sup>+</sup>-based composite, while a the formation of a new NaA zeolite phase has been observed in Na<sup>+</sup>-based composites, with a partial modification of the Na13X grains.

The BET surface area analysis, carried out by N<sub>2</sub> adsorption at 77 K, showed the characteristic Type I isotherm, typical of microporous solids. The assessment of the gas adsorption properties has been obtained by a dedicated pressure-decay apparatus and a thermogravimetric analysis, both at 308 K.

The Na<sup>+</sup>-based composites present a significantly larger (2–3 times) CO<sub>2</sub> capacity than those based on K<sup>+</sup>, approaching the values obtained for pure Na13X zeolite, as illustrated in Figure 1. A synergistic interplay among the two phases is also obtained, as the CO<sub>2</sub> capacity resulted about 20% larger than that expected; such an effect is mainly related to the large interaction obtained by an efficient chemical mixing provided by the geopolymerization reaction [4]. Furthermore, the formation of a NaA phase, well know to be a good CO<sub>2</sub> adsorbent material, in the geopolymer phase allows to an enhancement of the composite performances.

Much lower capacity have been observed for lighter gases such as N<sub>2</sub> and CH<sub>4</sub>, with linear behaviors with penetrant pressure; that leads to large ideal selectivity values around 120 for a 10/90 mixture of CO<sub>2</sub>/N<sub>2</sub> and 20 for a 50/50 mixture, calculated at 1 atm.

Therefore, the composite systems prepared are interesting and suitable for a possible carbon capture applications, as the geopolymer system provides good mechanical properties and a remarkable formability, which can be combined with highly performing materials such as a zeolite with high CO<sub>2</sub> capacity, eventually allowing to the development of advanced solid adsorbents with improved properties.

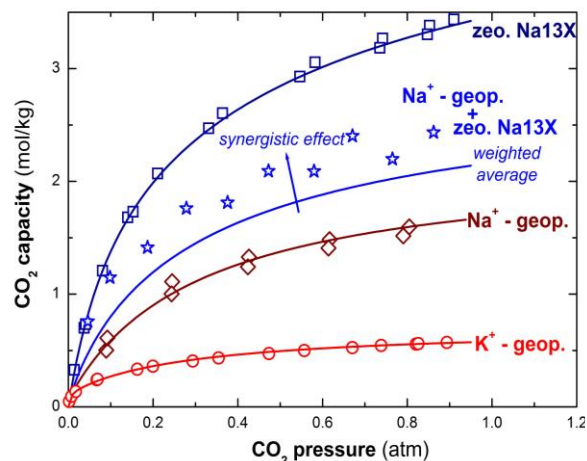


Figure 2 – CO<sub>2</sub> adsorption capacity at 35°C

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## JECS TRUST Best ORAL Award Competition

### CONTROL OF THE SETTING TIME, VISCOSITY AND PH VALUE OF GEOPOLYMER GROUTS

Dupuy Colin<sup>1,2</sup>, Nathalie Texier-Mandoki<sup>1</sup>, Xavier Bourbon<sup>1</sup>, Sylvie Rossignol<sup>\*2</sup>  
\*sylvie.rossignol@unilim.fr

<sup>1</sup> Andra (french national institute for the managing of the radioactive waste, Châtenay-Malabry, France)

<sup>2</sup> IRCER (Institut de Recherche sur les Céramiques, Limoges, France)

**Key Words:** geopolymer grout, setting time, viscosity, pH value, additives

Geopolymer binders are obtained from the dissolution of a mineral source in an alkaline silicate solution. Then the dissolved species in the mixture form oligomers and polycondensation reactions occur, resulting in a solid geopolymer. Geopolymers, in a fresh state, remain liquid and then are easy to shape. Therefore, they are good candidates for the elaboration of an injectable grout. In the aim of the development of a geological radioactive waste disposal, research is being conducted to design a grout with specific characteristics in order to be injected between the geological medium and the disposal structures. So, this study focuses on the elaboration of a geopolymer appropriate for a use as a injection grout in the disposal facilities. This grout must present a setting time over 24 hours, a moderate viscosity (0.5-5 Pa.s) and a controlled pH value (10-11). In the aim to develop such a grout, two steps are required. This first one is the development of the reactive mixture with the optimization of the alkaline silicate solution and of the mineral source amount. The second one is the study of the addition of external compounds to enhance the grout physical characteristics.

In order to adapt the mixture composition, several parameters need to be adjusted. A potassium alkaline silicate solution is used. Indeed, the setting time is higher and the viscosity lower with a potassium-based solution compared to a sodium-based solution. Then the alkalinity is moderated in order to favor polycondensation with the smallest amount of alkaline species. A Si/K *ratio* close to 0.7 seems to be a good compromise. More, the nature of the used mineral source has a major influence. The development of a grout requires to favor moderately reactive metakaolin or aluminosilicate mineral (without calcium). Last, the main parameter to control setting time and viscosity is the solid to liquid *ratio* (S/L) of the mixture. This *ratio* requires to be as small as possible. Using the Imerys M1000 metakaolin, a S/L < 0.8 seems ideal. However, these adjustments are not enough to reach the grout specificities concerning the setting time and pH value. Thus, the use of additive is required to increase the setting time from 6 to 24h and to decrease the pH value from 12.3 to ~10.5.

According to the application specificity, only mineral additives can be used. In this aim, the addition of phosphate and borate compounds is studied. It results that the setting time can be easily controlled and extended above 24 hours with the addition of acids (2.6 %<sub>weight</sub> of boric or orthophosphoric acids) or 10 %<sub>weight</sub> of decahydrated borax. The different acids decrease the reactivity of the alkaline silicate solution, which result in slower dissolution/polycondensation reactions. The borax, decahydrated sodium borate, provides a large amount of water which delays the consolidation. This increase of setting time is also related to kinetic variation due to the presence of boron in the mixture. More, the insertion of both acids and borax results to geopolymer mixtures with the targeted viscosity. Concerning the decrease of the pH value, the target of 10-11, can only be reached with the use of lithium or sodium tetraborates. This decrease seems to be related to the presence of B<sup>IV</sup> in the mixtures. This boron can be inserted in the geopolymer network, which enhanced the polycondensation and the alkali-consumption.



### INFLUENCE OF NAPHTHALENE, POLYCARBOXYLATES AND LIGNOSULFONATES BASED SUPERPLASTICIZERS ON FRESH AND HARDENED PROPERTIES OF ALKALI-ACTIVATED SLAG MORTARS

Coppola Luigi<sup>1,2</sup>, Coffetti Denny<sup>1,2</sup>, Crotti Elena<sup>\*1,2</sup>

\*elena.crotti@unibg.it

<sup>1</sup> Department of Engineering and Applied Sciences, University of Bergamo, 24044 Dalmine (BG)

<sup>2</sup> INSTM, UdR Materials and Corrosion, 50121 Florence

**Key Words:** Ground Granulated Blast Furnace Slag (GGBS); Alkali-Activated Materials; Alternative Binders; Mortars; Superplasticizers.

This paper presents an experimental study carried out to investigate the influence of different types of superplasticizers on the fresh and hardened properties of alkali-activated slag mortars. Three different admixtures were added to the mixtures (1.3% of dry polymer by binder mass). In particular, a naphthalene polymer-based (BNS), a polycarboxylates-based (SPC) and a lignosulfonates-based (LGS) high-range water reducers were used. In addition, a hardening accelerator was added to the mortars (1.0% respect to binder mass). Ground granulated blast furnace slag (according to EN 15167-1) as precursor and sodium metasilicate pentahydrate: potassium hydroxide : sodium carbonate = 7:3:1 in powder form as activator were used to produce different mortars with the dosage of activator between 2% and 16 % vs binder mass (1). The amount of water was adjusted in order to attain the same workability at the end of the mixing procedure, equal to 160 mm ± 10 mm (measured by means of a flow table). The specimens were cured in climatic chamber at 20°C and R.H. 60%. The effectiveness of the admixtures has been investigated in terms of percentage of water reduction, workability loss over time and compressive strength.

The experimental data show that all superplasticizers provides a reduction in mixing water. However, the efficiency of the admixtures in water reduction is influenced by the presence of the activator. In fact, the water reduction, at the same initial workability, is minimum in mortars manufactured with high activator/precursor ratio. Moreover, results indicated that as a consequence of superplasticizer addition, the pot-life of reference mortars manufactured without superplasticizer (60 minutes) is extended up to 160 minutes. The mechanical properties are influenced by the type of water reducer. In fact, the addition of a polycarboxylates based (SPC) superplasticizer decreases the early compressive strength, but positively influences the compressive strength at 7 days in mortars manufactured with activator/precursor ratio equal to 8 and 16. On the other hand, only the mortar manufactured with the dosage of activator equal to 16% has a compressive strength at 28 days higher than that of the reference mortars (without the superplasticizer). On the contrary, the addition of a naphthalene based (BNS) superplasticizer increases the compressive strength at early and long ages. The addition of a lignosulfonates-based (LGS) superplasticizer decreases the early compressive strength. In the case of mortars with 2% or 4% of activator, in particular, it was not possible to determine the mechanical properties up to 3 days because mortars were not hardened. Moreover, only mortars with percentage of alkaline activators higher than 4% had a 7 and 28-day compressive strength equal to of the reference mortars (without the superplasticizer). Finally, use of hardening accelerator admixture does not determine a reduction in workability loss over time while no improvement was detected on the mechanical strength at early and long ages. In addition, the hardening accelerator only improves behavior of mortars with SPC and a percentage of alkaline activators less than 4%. The AAMs are characterized by very high shrinkage compared to mixtures manufactured with traditional binders, as widely reported in the scientific literature (2). The addition of superplasticizers does not change the free shrinkage values.

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## JECS TRUST Best ORAL Award Competition

### 3D PRINTING OF GEOPOLYMERS: THE PATH TO INNOVATIVE CERAMIC COMPOSITES

Paolo Scanferla<sup>\*1</sup>, Giorgia Franchin<sup>1</sup>, Paolo Colombo<sup>1</sup>

paolo.scanferla@phd.unipd.it

<sup>1</sup> University of Padova, via Marzolo, 9, 31151, Padova, Italy

**Key Words:** Geopolymers, Direct ink writing, Geopolymeric Composites, Porous ceramics, Robocasting.

Geopolymers are inorganic materials generally obtained by the reaction of aluminosilicate powder in an alkaline solution. They can consolidate at low or even room temperatures and possess good mechanical properties, weather and heat resistance, which make them suitable for a wide range of applications, such as structural materials, thermal insulation, and so on.

Our group developed mixtures based on geopolymer for additive manufacturing of porous components via direct ink writing (DIW). We optimized the rheological properties in order to obtain suitable inks for the production of highly porous lattices. It should be noted that, as geopolymer mixtures are subjected to ongoing polycondensation reactions, their viscosity changes with time in what can be seen as a 4D printing process. Different materials were added to the mixture, such as fibers and micro particles, to produce innovative 3D printed geopolymeric composites.

## IDENTIFYING THE CARBONATION FRONT IN Fe-RICH ALKALI ACTIVATED MATERIALS

Michiel Giels<sup>\*1</sup>, Sam Eggermont<sup>1</sup>, Yiannis Pontikes<sup>1</sup>

\*michiel.giels@kuleuven.be

<sup>1</sup> KU Leuven, Department of Materials Engineering, 3001 Leuven, Belgium

**Key Words:** AAM, Durability, Carbonation, front, Phenolphthalein

Phenolphthalein is a common pH indicator to investigate the carbonation extent for OPC-based binders. The pH of concrete decreases by reaction with carbon dioxide. At lower alkaline pH values (below 8), phenolphthalein is colorless, at higher pH (above 10) it is bright pink. The method is quite simple and leads to reproducible data: after applying the indicator on a cut surface, one measures the distance of the decolored zone. The extent of carbonation can be used to estimate the life span of the reinforcements before corrosion initiates and it is obviously of interest for alkali-activated materials (AAM) as well. However, this method does not work well for AAM containing a high fly ash content. Several authors noticed a weak coloring of this indicator in certain zones or even no distinct carbonation front at all [1][2][3][4]. This carbonation front corresponds with an approximate pH of 10.5 [1]. The same challenge was observed also for Fe-rich AAM, being dark grey/black in color. In this study, a new method is developed to improve the contrast of indicators for the estimation of the carbonation rate for different alkali activated materials (AAM) with a characteristic darker color. Three pH indicators phenolphthalein, thymolphthalein and curcumin were tested for their stability in an alkaline environment. In addition, the traditional method with phenolphthalein is compared to the newly developed method. With the traditional method, phenolphthalein solution is sprayed on the sample and the pink contrast is only visible for 10 – 30 min. After rehumidifying the surface, the pink contrast returned shortly for 1 – 2 min. The new method pre-treats the sample surface by pouring a homogeneous solution of demineralized water with rutile powder (TiO<sub>2</sub>). After 5 seconds the solution is carefully tapped with absorbing paper to leave a white surface. After spraying the phenolphthalein on the surface, the carbonation front was clearly visible (Figure 1) and the contrast remained for up to 2 – 4 h. This study demonstrates that the high amount of alkalis and samples with dark surface are problematic for the determination of a clear carbonation front. By applying a surface treatment, the contrast of the carbonation front could be enhanced up to 2 – 4 h using a phenolphthalein solution.

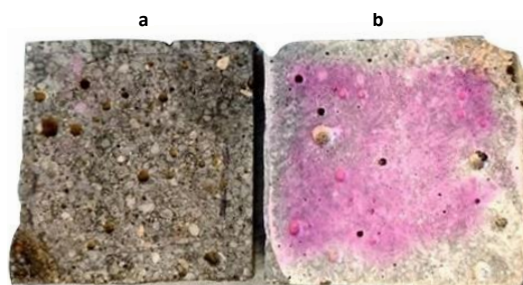


Figure 3: a) Phenolphthalein sprayed on cut AAM specimen. b) Phenolphthalein sprayed on rutile-treated AAM specimen.

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### LIGHTWEIGHT ALKALI ACTIVATED SLAG-BASED PLASTER FOR SEISMIC RETROFITTING AND THERMAL INSULATION OF EXISTING MASONRY BUILDINGS

Coppola Luigi<sup>1,2</sup>, Coffetti Denny<sup>\*1,2</sup>, Crotti Elena<sup>1,2</sup>  
\*denny.coffetti@unibg.it

<sup>1</sup> Department of Engineering and Applied Sciences, University of Bergamo, 24044 Dalmine (BG)  
<sup>2</sup> INSTM, UdR Materials and Corrosion, 50121 Florence

**Key Words:** slag cement; lightweight plaster; one-part alkali activated binder; seismic retrofitting; thermal insulation.

The issue of retrofitting and seismic upgrade of existing masonry buildings has become of primary interest, due to the huge architectural heritage widespread all over the world. This topic is extremely complex, especially because of many compatibility issues between existing structures and Portland cement repair mortars. The use of cement-free alkali-activated materials (AAM), such as ground granulated blast furnace slag (GGBFS), could be a suitable alternative to Portland cement mixtures. The aim of this research was to develop a lightweight alkali-activated slag-based plaster for seismic retrofitting and thermal insulation of stone or brick masonry buildings. In particular, the following target performances have been identified: a) 28-day compressive strength not lower than 5 MPa, b) specific mass at hardened state not higher than 1000 kg/m<sup>3</sup> and c) adhesive bond after 28 days at 20°C and R.H. 60% at least 0.2 MPa.

Three different series of lightweight plasters were produced: a traditional Portland-free mortar (TP) manufactured with hydrated lime (CH) CL90-S and ground granulated blast furnace slag with 28-day pozzolanic activity index equal to 0.76 (GGBFS) and two innovative alkali-activated slag-based mixtures (IP) with different activator/precursor ratio [1]. A blend of activator in powder form (sodium metasilicate pentahydrate : potassium hydroxide : sodium carbonate = 7 : 3 : 1) was used to produce the innovative plasters with the dosage of activator equal to 20% and 24% by binder mass. An air-entraining agent (AEA) based on cocamide diethanolamine was added (2 kg/m<sup>3</sup>) to lighten the mortars, reducing, at the same time, the tendency to bleeding and segregation. In addition, the natural siliceous aggregates (NA) were replaced by expanded recycled glass aggregates (EGA), properly combined to meet the Bolomey curve. Furthermore, due to the high shrinkage of alkali-activated slag-based materials [2], methylcellulose (MC), modified starch (MS), polypropylene fibers (length 6.5 mm, aspect ratio 200) and a ethylene glycole based shrinkage reducing admixture (SRA) were added to the mortar in order to minimize the risk of cracking and detachments. Compressive strength test results show that the mechanical strength of GGBFS/lime-based mortars (TP) are not influenced by the EGA/NA ratio. In fact, regardless of the aggregate used, the compressive strength is poor (about 2.5 MPa at 28 days) and not compatible for use in seismic improvement of existing masonry buildings. On the contrary, the innovative mortars (IP) based on alkali-activated slag show much greater compressive strength than those measured in traditional plasters (TP). Moreover, the compressive strength decreases with the increase of EGA/NA ratio similarly to the specific mass. The secant modulus of elasticity is very low, close to 1.5 GPa, with a specific mass at hardened state of about 750 kg/m<sup>3</sup>.

The AAMs are characterized by very high shrinkage compared to mixtures manufactured with traditional binders, as widely reported in the scientific literature [3]. However, the addition of MC, MS, SRA and fibers reduced shrinkage in alkali-activated slag mortars up to 50%. Furthermore, thanks to the lower elastic modulus of slag mortars, tensile stress induced by restrained shrinkage is very low, preventing the mortars from cracks and detachments. This thought is also supported by the absence of cracks on the panels made for thermal conductivity and adhesion tests. Moreover, adhesion tests were carried out on masonry panels by varying the thickness of plaster from 20 to 45 mm. The bond strength values are higher when the mortar has been applied with a lower thickness. For plasters 45 mm thick a 100% cohesive failure was observed in the alkali activated mortar layer (average adhesive bond 0.33 MPa), indicating that the repair material is the weakest part of the system. On the other hand, the average adhesive bond was higher (0.89 MPa) and the failure mode was more variable in the case of plaster 20 mm thick, where the observed failures were both cohesive (in the mortar layer) and cohesive (along the interface). Moreover, it can be noted that the adhesive bond is always higher than that required (0.2 MPa). Finally, the value of the thermal conductivity of the lightweight plaster (0.35 W/mK) is lower by about 75% compared to that of a traditional mortar based on cement or lime (~1.30 W/mK).

In conclusion, experimental data show that the Portland-free plaster based on alkali activated slag is able to provide a structural upgrade (28-day compressive strength equal to 8 MPa) with an improvement of the thermal insulation of the masonry (thermal conductivity 0.35 W/mK due to a density close to 800 kg/m<sup>3</sup>). The reduced shrinkage and the low elastic modulus ensure an excellent adhesion to the substrate and the absence of cracks and detachments.

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## CHEMO-MECHANICAL BEHAVIOUR OF SOIL IMPROVED BY ALKALI ACTIVATED BINDERS

Enza Vitale\*<sup>1</sup>, Giacomo Russo<sup>1</sup>, Dimitri Deneele<sup>2</sup>  
\*e.vitale@unicas.it

<sup>1</sup> University of Cassino and Southern Lazio, Italy

<sup>2</sup> Institut des Matériaux Jean Rouxel (IMN), Université de Nantes, CNRS; IFSTTAR

**Key Words:** alkaline activation; fly ash; soil treatment, chemo-mechanical behaviour

The use of alkali-activated binders to improve engineering properties of clayey soils is a novel solution, alternative to the widely diffused improvement based on the use of traditional binders such as lime and cement. The development of these binders for geotechnical applications is a promising issue in terms of sustainability since it reduces the carbon footprint and allows reusing secondary by-products such as artificial pozzolans. By-products can be involved in soil improvement as cementing agents if properly activated, inducing a mechanical improvement of natural soils, not suitable for construction purposes.

Alkali activation of by-products is the consequence of a chemical reaction between of an aluminosilicate source with an alkaline solution (i.e. sodium hydroxide, sodium silicate). The aluminosilicate source is formed by precursor materials like natural pozzolan (e.g., pyroclastic soils), or artificial pozzolans (e.g., fly ash, silica fume, steel sludge). The chemo-physical evolution occurring after the activation promotes the formation of cementitious compounds [1; 2]. The reaction mechanism is promoted by the alkaline solution, which enables the dissolution of the aluminosilicate source (precursor) and the subsequent precipitation of gel phases which condense in a three-dimensional aluminosilicate network [3; 4; 5; 6].

In the present work, alkaline activation of two Ca-rich fly ashes, by-products of coal combustion thermoelectric power plants working at low and high temperature, has been investigated. These alkali activated binders have been mixed with a clayey soil for evaluating the improvement of its mechanical behaviour. One-dimensional compression tests on raw and treated samples have been performed with reference to the effects induced by type of binder, binder contents and curing time. The experimental evidences at volume scale of the treated samples have been directly linked to the chemo-physical evolution of the system, investigated over curing time by means of X Ray Diffraction and Nuclear Magnetic Resonance (NMR).

One-dimensional compression tests performed on treated sample highlighted the effectiveness of alkali activated binders to promote an improvement of the mechanical behaviour of treated soil. A reduction of compressibility and an increase of the yield stress soil were observed since the very short term. The macroscopic behaviour of treated soil has been linked to the experimental evidences at microscale. Microstructural analyses show a high reactivity of the alkali activated fly ashes as alumino-silicate source promoting the precipitation of new mineralogical phases forming chains and networks with cementitious properties. The reactions kinetic controlling the chemo-physical evolution of alkali activated binders and therefore the macroscopic evolution of soil properties depends on fly ashes mineralogy, as evidenced by the compressive behaviour of treated samples as function of curing times and binder contents. The efficiency of treatment has been highlighted by comparing the mechanical performance induced by alkali-activated binder and ordinary Portland cement.

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## A NOVEL APPROACH OF GEOPOLYMER BRICKS PRODUCTION BASED ON CLAY

Nicolas Youssef<sup>1,2</sup>, Zakaria Dakhli<sup>1</sup>, François Waendendries<sup>3</sup>, Fadi Hage Chehade<sup>3</sup>,  
Zoubeir Lafhaj<sup>1</sup>

nicolas.youssef@phd.centralelille.fr

<sup>1</sup> Ecole Centrale de Lille, Civil Engineering, Cité Scientifique, CS 20048, 59651 Lille, France

<sup>2</sup> Doctoral School of Sciences and Technology, Lebanese University, Lebanon

<sup>3</sup> Minealithe, 02240 SISSY, France

**Key Words:** Clay, Alkaline Activation, Geopolymer, Brick, Building materials

This paper presents a new geopolymer formulation based on clay. The traditional method of fired bricks production is energy intensive because of the high temperature and curing requirements. The large volume of CO<sub>2</sub> emitted by this production of building materials has motivated the scientific community to look for new ways to reduce the environmental impact. One of the potential areas of work is to use the geopolymerization process. The goal of this work is to study the geopolymerization process to produce a brick substitute material by alkaline activation of clay. The manufacturing of this geopolymer bricks was carried out by mixing clay, ground granulate blast furnace slag (GGBFS), sand with a solution of hydroxide (NaOH) and sodium silicate (Na<sub>2</sub>SiO<sub>3</sub>). Prismatic samples were cured at low temperatures for 16 days. The samples were prepared according to different formulations. The optimal compressive strength obtained is 92 MPa, for a GGBFS / clay ratio, a molarity of NaOH and a silicate / hydroxide ratio, very well defined. This study gives an overview of the potential of geopolymer materials as a substitute of fired bricks for the future construction.

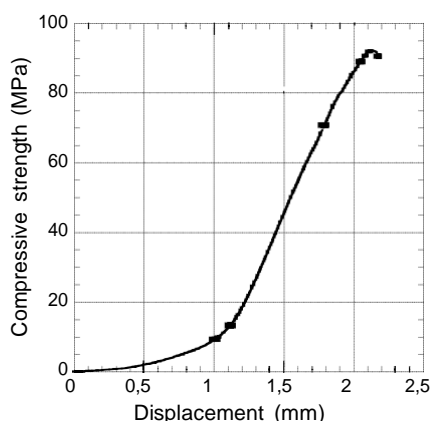


Figure 1: The maximum compressive strength obtained

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## JECS TRUST Best ORAL Award Competition

### CONSTRUCTION AND DEMOLITION WASTE (CDW) VALORIZATION IN ALKALI ACTIVATED BRICKS

V. Vezzali<sup>\*1</sup>, A. Grillenzoni<sup>2</sup>, I. Lancellotti<sup>1</sup>, L. Barbieri<sup>1</sup>, C. Leonelli<sup>1</sup>

\*vittorio.vezzali@unimore.it

<sup>1</sup> Department of Engineering "Enzo Ferrari", Modena, Italy

<sup>2</sup> GARC spa, Via dei Trasporti, 14, 41012, Carpi, Italy

**Key Words:** CDW (Construction and Demolition Waste), earthquake, bricks, alkali activation, circular economy

The present study investigates the synthesis of alkali activated (AA) bricks based on the activation through different alkaline solutions of construction and demolition waste (CDW) coming from 2016 central Italy earthquake rubble. These wastes are provided by GARC spa, in the context of Emilia Romagna "PO FSE 2014/2020" regional project, and consists especially in inert debris, as local rocks, red clay waste bricks and concrete, which can be usefully exploited as precursors or as fine and coarse aggregates in the production of AA bricks and cements. This practice leads to several advantages, including: (i) savings in the use of non-renewable raw materials, (ii) given the cold forming process, a reduction in CO<sub>2</sub> emissions and energy consumption with consequent positive climate action; (iii) improving of waste management and increasing recycling in the context of a circular economy, in accordance with the European and Italian legislative framework aimed at reducing waste production<sup>1</sup>. Several parameters are currently being investigated during the different phases of the work. First of all the composition, mineralogy and particle size distribution of the raw materials, aiming to a correct percentage of precursors in the final product. The material it's grinded and homogeneously mixed in order to work with a representative portion of the amount of CDW. The forming procedure chosen for this work it's uniaxial pressing (0.1-0.5 MPa). Typology, correct molarity of alkaline activators and curing conditions were also investigated. The created bricks were firstly subjected to integrity tests and water absorption tests to verify or not the success of activation reaction. Compressive strength (28 days) testing and adhesion testing with concrete and AA concrete were then evaluated to choose the best samples. To characterize the structure and the morphology of all the AA products, several techniques such as Scanning Electron Microscopy (SEM) and X-ray Diffraction (XRD) are used. A moisture absorb-and-release experiment it's also in testing phase, conducted on the most significant specimens. During this work were also added bottom ash derived secondary raw material, in order to evaluate the contribution of another allumino-silicate source richer in amorphous phase with respect to CDW powders<sup>2,3,4</sup>. Same characterization as before are currently being performed on these specimens. Future activities will be addition of oxides, foaming agents, reinforcement in the form of fibers or particles and other products; in this way the materials can be funzionalized in order to engineer properties. Other testing steps will be for example freeze-thaw cycles, necessary to verify the on-site applicability of these materials.



Figure 1: AA bricks made from CDW with uniaxial pressing



Figure 2: Adhesion test on commercial bricks with concrete and with AA concrete

### ACKNOWLEDGMENTS

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## POSTER

### PLANETARY CENTRIFUGAL MIXER "THINKY MIXER"

Riccardo Cossi  
[r.cossi@qitech.it](mailto:r.cossi@qitech.it)

Qi Technologies s.r.l., Pomezia (RM) (Italy)

**Key Words:** planetary centrifugal mixer, deaeration, defoaming

The mixing of raw materials is very important in any chemical synthesis. Geopolymers are produced by mixing aluminosilicate powders with liquid alkaline activators and the extent of dissolution of raw materials is fundamental as the properties of the geopolymer depend on the Si/Al ratio. For this reason, since 2008 Thinky mixers (Thinky, Japan) were used to synthesis geopolymer materials and composites [1-17]. The THINKY Mixer [18] is a no-blade planetary centrifugal mixer that mixes materials employing "rotation" and "revolution". In much the same way the Earth revolves around the Sun, the mixing container simultaneously "revolves" around the center of the mixer while "rotating". These two contradictory forces simultaneously and thoroughly mix, disperse and deareate materials in the container. Air bubbles can be also removed efficiently by mixing and defoaming under constant reduced pressure with Thinky's planetary centrifugal vacuum mixer.

The centrifugal force (400G) produced by the high-speed rotation and revolution depresses the material in the container, and generates vertical spiral convection as shown in Figure 1. This vertical spiral convection is continuously generated to blend and disperse the materials evenly.



*Figure 1 Oil clay mixing experiment*

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## POSTER

### SitoH PROJECT: SILICATES FROM SILICON WASTE

Guido Fragiaco, Gianluigi Noja  
[guido.fragiacomo@garbosrl.net](mailto:guido.fragiacomo@garbosrl.net); [gianluigi.noja@garbosrl.net](mailto:gianluigi.noja@garbosrl.net)

Garbo Srl Via Prati Nuovi 9 28065 Cerano (Novara) Italy

**Key Words:** alkaline activator, sustainability, environmental impact

Geopolymer is known to be an environment friendly material, as, compared to the standard OPC (Ordinary Portland Cement), shows much lower CO<sub>2</sub> emission per ton of final product [1]. This is true when waste or byproducts are used to produce the geopolymer, as the environmental impact of metakaolin, sodium silicate and other raw materials used for the production of the Geopolymer, is not small.

Ashes, slags and spent catalyst are frequently used as Alkali Soluble Aluminosilicate Sources and are easily available in quantities. But it's not the same for the activator/alkaline-reagents.

Aluminium hydroxide waste (Alwaste) was firstly used as a source material for producing geopolymer [2], but the availability of this waste is (only) 3000 ton/year over Italy and is usually used for water treatment application, causing this a cost increase in its procurement. Therefore it is mandatory to look for activator/alkaline-reagents produced with innovative processes to reduce their environmental impact.

***The SitoH (Silicon to Hydrogen) project has the aim to produce sodium silicate in such a way, starting from metallic silicon waste from the PV (Photovoltaic) production chain or from PV modules dismantling and being, by the way, cheaper than the standard process.***



Figure 1 – Garbo plant

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## POSTER

### EVALUATION OF FLY ASH BASED FOAMS AFTER EXPOSURE TO ELEVATED TEMPERATURE

Lidija Korat<sup>1</sup>, Vilma Ducman<sup>1\*</sup>

\*[vilma.ducman@zag.si](mailto:vilma.ducman@zag.si)

<sup>1</sup> ZAG, Slovenian Building and Civil Engineering Institute, Ljubljana, Slovenia

**Key Words:** alkali activation, foams, fly ash, high temperature evaluation

Alkali activated (AA) foams are one of the most promising type of alkali-activated materials due to potentially higher added value. They are produced at low temperatures (below 100 °C) what is competitive advantage to ceramic or glass foams. AA foams are widely researched [1,2], because they are dimensionally stable and non-combustible, and they possess high strength and good insulation properties at room temperature. Investigations have been recently focused on their behaviour at elevated temperatures. It has already been confirmed that elevated temperature of alkali activated materials provide improved compressive strengths, which in the investigated system of fly ash increased for about 53% after temperature exposure [3]. Rivera et al. [4] have also investigated the influence of mineralogy of fly ash/metakaolin geopolymers exposed to elevated temperatures, firstly on strength properties and secondly, to the air voids distribution and overall porosity.

The primary goal of present research was investigation of the thermal stability of foamed alkali-activation materials based on different fly ashes. Mineralogy and chemical composition of fly ashes were determined by XRD and XRF, respectively. After foaming and curing at 70 °C, samples were first characterized on density and compressive strength, and after exposure to the elevated temperature (1000 °C) both tests were repeated. Dilatometry was used to follow the dimensional changes throughout the firing process. Additionally, X-ray computed microtomography and SEM were used for determination of porous inner structure before and after exposure to elevated temperature.

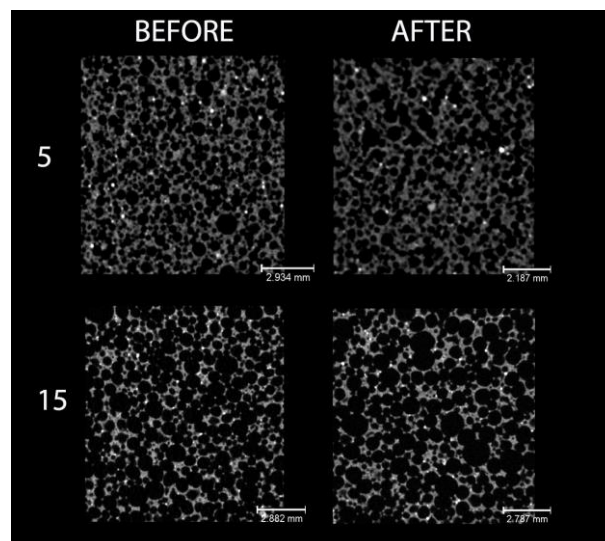


Figure 2 – Alkali activated foams before and after exposed to elevated temperatures, determined by X-ray computed microtomography

#### ACKNOWLEDGMENTS

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## POSTER

### LIGHTWEIGHT ALKALI ACTIVATED COMPOSITE FOAMS BASED ON SECONDARY RAW MATERIALS (FLOW)

Vilma Ducman<sup>1</sup>, Lidija Korat<sup>1</sup>, Christina Leonelli<sup>2</sup>, Paivo Kinnunen<sup>3</sup>, Mirja Illikainen<sup>3</sup>

1 Slovenian National Building and Civil Engineering Institute, Dimičeva 12, 1000 Ljubljana, Slovenia  
[vilma.ducman@zag.si](mailto:vilma.ducman@zag.si)

2 Dipartimento di Ingegneria "Enzo Ferrari", Università di Modena e Reggio Emilia, Modena, Italy

3 Fibre and Particle Engineering, Faculty of Technology, Univ. of Oulu, P.O. Box 4300, 90014 Oulu, Finland

**Key Words:** slag, geopolymers, alkali activation, lightweight materials

New possibilities for the recycling of inorganic wastes or industrial residues are investigated in order to avoid the disposal of waste materials in landfills. Especially, aluminate- and silicate-containing materials can be utilized in alkali activation technology; when treated with an alkaline activator solution these precursors form a solid material at room temperature which could be used to replace concrete, ceramic and some other industrial materials. Additionally, significant environmental advantages are achievable by replacing the production of these energy-intensive materials by more sustainable processes.

The main objective of this project is to develop new lightweight alkali activated foams based on secondary raw materials (e.g. fly ash, slags). To obtain highly porous structures, properly selected foaming agents and foam stabilizing agents need to be included in the basic compositions. Currently, the main disadvantage of such lightweight materials is their high fragility. Addition of fibers will be used to overcome this drawback and help to produce materials with more elastic nature. By incorporating organic fibers from a bio-based renewable source, and simultaneously using inorganic secondary resources as raw materials for alkali activated foams, a high performance in terms of energy efficiency and environmental impact will be reached. The developed materials will have applications in wide range of thermal and acoustic insulating products.

The project is divided in 5 working packages, namely: WP1-Alkali-activation development; WP2-Performance testing and validation; WP3-Piloting and LCA assessment; WP4-Industrial stakeholder co-operation; WP5-Dissemination and management.



The consortium partners are: Slovenian national Building and Civil Engineering Institute, Fiber and Particle Engineering Unit at University of Oulu, University of Modena and Reggio Emilia. In addition to research partners, several industrial companies participate in the project as potential exploitation partners.

#### ACKNOWLEDGMENTS

Project ERAMIN 2, Lightweight alkali activated composite foams based on secondary raw materials (FLOW), Project ID-94, 2017 ERA-MIN Joint Call. (<http://flow.zag.si>)

## POSTER

### CHARACTERIZATION AND REACTIVITY OF SLAGS AS PRECURSORS FOR ALKALI ACTIVATED MATERIALS

Isabella Lancellotti<sup>1\*</sup>, Andrea Baraldi<sup>1</sup>, Mark Češnovar<sup>2</sup>, Katja Traven<sup>2</sup>, Michela Ciabocco<sup>3</sup>, Luisa Barbieri<sup>1</sup>, Mario Berrettoni<sup>4</sup>, Vilma Ducman<sup>2</sup>, Cristina Leonelli<sup>1</sup>,

\*[isabella.lancellotti@unimore.it](mailto:isabella.lancellotti@unimore.it)

<sup>1</sup> Dipartimento di Ingegneria "Enzo Ferrari", Università di Modena e Reggio Emilia, MODENA (Italy)

<sup>2</sup> Slovenian National Building and Civil Engineering Institute, Dimičeva 12, 1000 Ljubljana (Slovenia)

<sup>3</sup> Analisi Control S.r.l., Via San Claudio, 5, 62014 Corridonia (MC) (Italy)

<sup>4</sup> Dipartimento di Chimica Industriale "Toso Montanari", UOS Campus Rimini, Università di Bologna, Via dei Mille 39, 47921 RIMINI (RN), (Italy)

**Key Words:** slag, geopolymers, alkaline reactivity, leaching test

Slags coming from different metallurgical industries in Slovenia, e.i. electric arc furnace steel slag (EAF) and the ladle furnace basic slag (LF) from have been characterized in order to evaluate their suitability to obtain alkali activated materials.

In order to evaluate the reactive fraction of slags, a basic attack was performed according to the test reported in literature [1]. Slag was placed in 8M NaOH solution with a solid/liquid ratio = 1 and stirred constantly for five hours in a flask bathed at  $80 \pm 2$  °C. The resulting solution was then filtered, the residue was rinsed with distilled water until a neutral pH was attained and the amount of insoluble material was quantified. Eluates were acidified at pH =2 with HNO<sub>3</sub>. Al and Si contents in the leachate were quantified with ICP/AES.

XRD diffraction on slag before and after alkaline attack was performed in order to evaluate both amorphous/crystalline nature of slag and the modifications induced by the alkaline environment. The nature of the slags is mainly crystalline and the main crystalline phases identified are Q-quartz (SiO<sub>2</sub>), C-calcite (CaCO<sub>3</sub>) and P-portlandite (Ca(OH)<sub>2</sub>), with a significant decrease of quartz and calcite and an increase of portlandite after the basic attack.

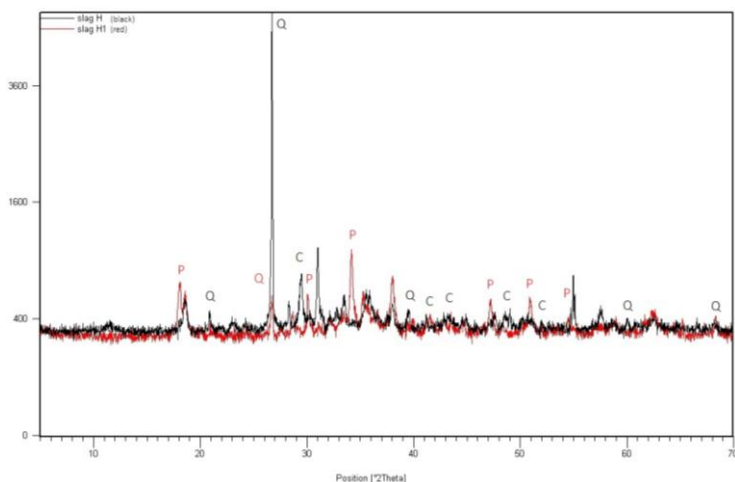


Figure 3 – XRD patterns of slags before and after alkaline attack

To evaluate the hazardousness of the bottom ash, leaching test in distilled water for 24 hrs was performed according to European norm EN 12457. Solid residue was separated by filtration and each eluate solution, after acidification (with HNO<sub>3</sub> to pH=2), was analysed by ICP/AES to determine the amount of heavy metals. pH of slags was also measured at T=0 and after 24h resulting that one slag presents pH 10,2 and 10,3 and the other 11.6 and 11.3 at T =0 and 24h, respectively.

#### ACKNOWLEDGMENTS

Project ERAMIN 2, Lightweight alkali activated composite foams based on secondary raw materials (FLOW), Project ID-94, 2017 ERA-MIN Joint Call; Coordinator: Dr Vilma Ducman, ZAG\_Slovenia.

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## POSTER

### AGM for CuHe Advanced Green Materials for Cultural Heritage

Germana Barone

[gbarone@unict.it](mailto:gbarone@unict.it)

Dipartimento di Scienze Biologiche Geologiche e Ambientali  
Università di Catania (Italy)

**Key Words:** conservation, restoration, cultural heritage, eco-friendly product, wastes

AGM for CuHe will develop eco-friendly, smart and high-technological products for conservation and restoration of Cultural Heritage, taking advantages from the high density and diversity of touristic attraction in the local context. AGM for CuHe will have an impact on tourism industry, favouring the fruition of restored cultural heritages by using innovative materials and pursuing the local decision makers to improve more and more the attractiveness of the built historical environment thanks innovative tools. Tourists will have the possibility to follow all the research, experimental and applicative phases of the project, for a more responsible and conscious fruition of culture and cultural heritages. Moreover, the presence of a specific task devoted to mitigate seismic risk by formulating restoring products with high mechanical stress resistance aims to improve knowledge in this field and create transferable skills also in other geographical context dealing with seismic risk. The application will be forerun by a task devoted to modelling the long-term behaviour of both structures and products, as well their response to seismic stress. The achievement of the target objectives is supported by: a) the high qualified human resources and the presence of well-equipped laboratories for the development of researches in geology, biology, chemistry, engineering, physics, science of the material disciplines; b) the support of economic and juridical expertise of the sustainability assessment of the project; c) the presence of a well-experienced industrial leadership assured by companies involved in management, production, and restoration actions. AGM for CuHe will potential improve the employment rate, especially in southern Italian regions, with the formation of new experts in the field of a) extraction and recycle of wastes for the production of innovative material; b) production of nanoparticle-based products for restoration; c) standardised protocols for restoration; d) innovation in cultural heritage fruition. The target objectives of AGM for CuHe are in agreement with the European guidelines, in which actions focused on improving the impact of Cultural Heritages in economy, tourism market, societal challenges and employment rate is widely encouraged.

**FLY ASH-BASED ALKALI ACTIVATED MORTAR: PRELIMINARY STUDY OF THE FRACTURE ENERGY**

Lorenza Carabba<sup>1</sup>, Mattia Santandrea<sup>1</sup>, Christian Carloni<sup>1</sup>, Stefania Manzi<sup>1</sup>, Maria Chiara Bignozzi<sup>1</sup>

<sup>1</sup>Department of Civil, Chemical, Environmental and Materials Engineering, University of Bologna, Bologna, Italy

**Key Words:** Alkali activated materials, Fly ash, Fracture mechanics, Bond

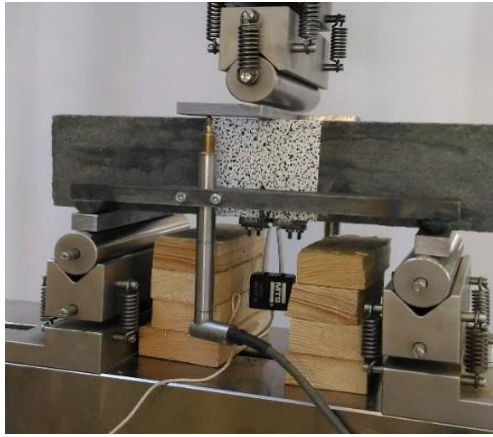


Figure 1 – Fracture mechanics set-up

Alkali activated materials (AAMs) are increasingly investigated as an emerging technology alternative to ordinary portland cement (OPC)-based mortars and concrete. In fact, if compared to OPC-based materials, AAMs demonstrated competitive properties and proved their strong potential as building materials [1].

Among the others fields of application, AAMs can find large use for strengthening and/or repair interventions of existing damaged structures. The systems currently employed for this purpose are the so called fiber reinforced polymer (FRP) composites and fiber reinforced cementitious matrix (FRCM) composites [2]. They are comprised of continuous high strength fibers embedded within a matrix, either organic like epoxy or inorganic like cement mortar, and externally bonded to the structure. AAMs can be considered as an alternative matrix suitable for this application. Indeed previous studies showed high compatibility between AAMs and concrete

substrates [3] and highlighted the remarkable temperature resistance of these materials [4]. Both these aspects make alkali activated mortars extremely competitive, especially if compared to FRP composites. Moreover, among aspects not extensively investigated yet in literature, there is the study of the fracture properties of AAMs. As OPC, AAMs present a brittle nature and for this reason, understanding the fracture properties of these materials is a topic of great interest, essential for their safe applications.

Considering these aspects, this study proposes the adoption of fly ash-based alkali activated mortar in substitution of cementitious mortar and epoxy to cast composites for strengthening applications of existing masonry structures. In addition, preliminary results of the fracture energy of the developed mortar are presented. The mortar is obtained by activating coal fly ash with sodium hydroxide and sodium silicate solutions at room temperature. Fine silica sand ( $d_{max} = 1$  mm) is adopted as aggregate. The mortar is used to embed continuous high strength fibers and bond the composite to masonry substrates in order to study the interfacial bond behavior of composite-substrate joints. Physical, mechanical, and microstructural properties of the mortar are determined to characterize completely the material. Single-lap direct shear tests are performed to study the stress transfer mechanism between the composite and the substrate. Finally, mortar prisms are tested by a three-point bending set-up (Figure 1) to evaluate the fracture energy of the material. The fracture energy,  $G_F$ , is evaluated as the area under the load-deflection responses.

Obtained results show that the developed mortars are suitable for casting fiber reinforced inorganic matrix composites. The adequate adhesion between the composite and substrate is proved by the observed failure mode. Indeed, a debonding (interlaminar failure) of the external layer of matrix rather than debonding at the matrix-substrate interface is observed. Furthermore, data of the fracture energy for mortar prisms, which are currently lacking in literature, are presented.

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**CORROSION RESISTANCE OF LIGHT-WEIGHT FLY ASH-BASED ALKALI ACTIVATED MORTARS FOR FIRE PROTECTION OF STEEL STRUCTURES**

Giulia Masi<sup>1\*</sup>, Lorenza Carabba<sup>1</sup>, Maria Chiara Bignozzi<sup>1</sup>

\*[giulia.masi5@unibo.it](mailto:giulia.masi5@unibo.it)

<sup>1</sup> Dipartimento di Ingegneria Civile, Chimica, Ambientale e dei Materiali, Università di Bologna, via Terraccini 28, 40131 Bologna (Italy)

**Key Words:** Alkali activated mortar; Porosity; Steel corrosion; Durability; Fire protection.

Fire protection of steel structure is an important requirement for structural components for several civil and industrial applications. Different technologies are currently developed as passive fire protective coatings. In particular, alkali activated materials (AAMs) have attracted a lot of attention as promising materials suitable for this kind of application, due to their remarkable resistance at high temperature exposure [1]. An optimised mix-design makes their amorphous structure more stable, when exposed to direct fire or heating from high temperatures, compared to cement-based materials. This is due to the fact that the ceramic-like structure of AAMs maintains its structural integrity at elevated temperatures, thus allowing to preserve the dimensional stability of the material. So, AAMs are considered strong candidates for applications requiring good resistance to high temperature or fire [2-5]. However, the durability of fire protective coatings is an open issue. It mainly depends on its adhesion on steel and its capacity to act as a barrier to mitigate steel corrosion phenomena. Therefore, the understanding of the corrosion resistance of steel coated with AAMs-based coatings is necessary for ensuring the service life of the structure.

This study aims at investigating the durability in terms of steel corrosion resistance of coal fly ash-based alkali activated mortars as passive fire protection system for steel structures. In particular, mortars were lightened by the combination of expanded perlite (lightweight aggregates) and hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>), as chemical foaming agent. The behaviour of the light-weight mortars (LWM) was compared to that of a normal-weight mortar (M). The characterisation of the electrochemical behaviour of carbon steel coated by the different mortars was carried out. In particular, samples were tested by accelerated ageing methods consisting of a partial immersion of the samples in an aqueous solution of sodium chloride (NaCl 3.5 wt%). During 30 days of exposure, corrosion potentials ( $E_{cor}$ ) were measured daily. In parallel, anodic polarization curves have been recorded at different stages of the exposure (1h and 30 days of partial immersion) to highlight the effect of chloride ingress in view of the corrosion resistance of the steel substrates. Finally, physical properties in terms of bulk density and porosity were measured to better understand the barrier effect of the different mortars on the carbon steel corrosion. In particular, porosity was measured in terms of water absorption in vacuum conditions and capillary water absorption tests according to EN 1015-18 [6].

Results show that alkali activated mortars provide protection for carbon steel in presence of an aggressive environment (i.e. presence of chlorides). However, corrosion resistance is strictly related to the physical properties of the developed mortars. In particular, the increased and more interconnected open porosity, which characterizes LWM sample, is responsible for a reduced corrosion protection of the steel substrate. Indeed, corrosion resistance is lower when carbon steel is embedded in LWM mortars and exposed to a 3.5 wt% NaCl solution compared to M samples. Finally, studies on the optimization of the pore size distribution and on the development of closed porosity instead of open one are still under investigation, in order to increase the protection provided by LWM.

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## POSTER

### MANAGEMENT AND VALORIZATION OF WATER POTABILIZATION SLUDGE THROUGH USE IN PRODUCING GEOPOLYMER MATERIALS

C. Ferone<sup>1\*</sup>, I. Capasso<sup>2</sup>, A. Bonati<sup>2</sup>, G. Roviello<sup>1</sup>, R. Cioffi<sup>1</sup>

\*[claudio.ferone@uniparthenope.it](mailto:claudio.ferone@uniparthenope.it)

<sup>1</sup> Dipartimento di Ingegneria, Università di Napoli "Parthenope", Centro Direzionale, Isola C4, 80143 Napoli, Italy

<sup>2</sup> Construction Technologies Institute, National Research Council, Via Lombardia, 49 – 20098 San Giuliano Milanese, Milano, Italy

**Key Words:** Geopolymers, Water potabilization sludge, waste reuse, sustainable building materials

Alumina-containing water potabilization sludge (WPS) is one of the residues produced by reservoir management activities. This waste, accumulated after treatment processes for water potabilization, which are based on flocculation-clarification using alumina-based coagulant, can be used as starting raw material in the building materials industry. Recently, the building sector has focused a great attention on geopolymers, emerging binding materials with the potential to form a substantial element of an environmentally sustainable construction and building products industry. Furthermore, the use of waste deriving from industrial processes, such as fly ash and slags, as raw materials to form geopolymers, has been widely proven and contributed to increase their reputation as "environmentally friendly binders". In this study, the use of WPS as main aluminosilicate source for the synthesis of geopolymers has been investigated. In particular, two different potabilization sludge deriving from the water treatment plants of two different artificial water reservoirs located in the south of Italy (Liscione, Molise and Camastra, Basilicata) have been selected. For both of these WPS, mineralogical (XRD analysis), physical-chemical (FTIR analysis), thermal (TGA analysis), porosimetric (BET analysis) and morphological (SEM analysis) properties have been evaluated. These wastes resulted to be particularly interesting for the geopolymerization reaction as they are rich in silica and alumina and the molar Si/Al ratio is about 1. In addition, the effect of calcination at 650°C on the starting materials was evaluated. Then, the geopolymeric samples produced by the alkaline activation of the WPS in an aqueous sodium silicate solution ( $\text{SiO}_2/\text{Na}_2\text{O}=2$ ) have been widely characterized from chemical, mechanical and microstructural points of view. In particular, the samples obtained using the calcined Liscione sludge, resulted to be more compact and showed the highest values of compressive strength. However, the FTIR analyses performed on the samples produced from both WPS, confirmed that the geopolymeric reaction effectively took place for all the samples. In general, the mechanical performances reached by the geopolymeric specimens, obtained from both the calcined WPS, suggest the possibility of a promising reuse of these WPS as raw materials for the synthesis of geopolymers and in the building sector.

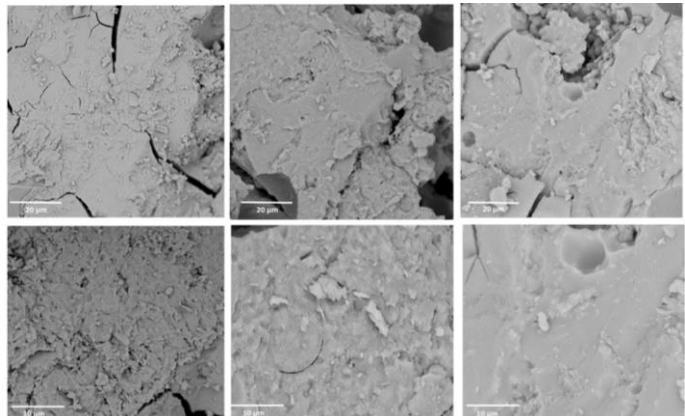


Figure 1. SEM micrographs of geopolymer samples

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## POSTER

### GEOSYL: GEOPOLYMER HYBRID MATERIALS FOR HIGH ENERGY EFFICIENT SYSTEMS

Giuseppina Roviello, Laura Ricciotti\*

[\\*laura.ricciotti@uniparthenope.it](mailto:laura.ricciotti@uniparthenope.it)

Dipartimento di Ingegneria, Università di Napoli "Parthenope", Centro Direzionale, Isola C4, 80143 Napoli, Italy

**Key Words:** Energy efficiency, geopolymer, hybrid materials, fly ash, thermal insulation

Over the past decades, considerable progresses have been made in the energy efficiency field, mainly for residential, commercial and office buildings. In fact, a conscious use of energy can bring environmental and economic advantages, since energy efficiency can become a strategic choice able to supports the world of industries and utilities to strengthen its long-term competitiveness.

One of the most important market demand concerns the possibility of constructing energy efficient buildings by using different kind of systems that can limit heat exchange between the inside and outside of the buildings.

In particular, this research project, (subjected to a proposal for a university Spin-Off constitution), deals with a large-scale development and industrialization of the production process of expanded elements for thermal and acoustic insulation, characterized by excellent performance, lower cost and higher eco-compatibility features than the autoclaved aerated concrete competitors (AAC) on the market.

In particular, innovative organic-inorganic geopolymer-based hybrid foams were prepared (Fig.1) by reacting fly ash and an alkalisilicate solution with mixtures of dialkylsiloxane oligomers. [1].  $\text{Si}^0$  powder as in situ foaming agent was used in different amounts to obtain foams with densities ranging from 0.4 to 0.6  $\text{g/cm}^3$ . [2, 3]. These new lightweight materials were characterized by remarkable mechanical properties, good fire resistance and low thermal conductivity, significantly better than those shown by neat geopolymer foams reported in the literature and comparable to AAC materials.

Substantially, these expanded systems, in addition to valorizing a waste (fly ash worldwide production: 750 Mt / year), are prepared by using a simpler, economical and with a lower environmental impact (temperature of 60 ° C for 12h at atmospheric pressure) if compared to their major market competitors such as aerated autoclaved concretes (AAC) that require expansive and high environmental procedures for the production of cement clinker and high temperatures and pressures (190 ° C for 12h at about 4-5 bar) for the realization of the expanded systems.



Figure 1: GeoSyl expanded systems.

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## POSTER

### SELF-SUPPORTING ZEOLITE BY GEOPOLYMER GEL CONVERSION (GGC)

Barbara Liguori<sup>1\*</sup> Paolo Aprea<sup>1</sup> Giuseppina Roviello<sup>2</sup>, Claudio Ferone<sup>2</sup>

\*[barbara.liguori@unina.it](mailto:barbara.liguori@unina.it)

<sup>1</sup> Applied Chemistry Labs-Department of Chemical, Materials and Industrial Engineering, University of Naples Federico II, Naples, Italy

<sup>2</sup> Materials Science and Engineering Research Group MASERG, Department of Engineering, University of Naples Parthenope, Naples, Italy

**Key Words:** zeolite, membrane, geopolymer gel conversion

Many industrial applications need supporting or shaping powdery zeolites. Different strategies were carried out to overcome this technological limit, such as porous ceramic binders, polymer foams and permeable bags, but complexity and cost of the final product were strongly affected [1]. Structuring a porous powder, as a zeolite, permits to obtain an optimized structure with high mass transfer, low pressure drops and high mechanical and chemical stability [2]. Since geopolymers can be considered the amorphous counterpart or precursor of crystalline zeolites, it is possible to promote zeolite crystallization inside the geopolymeric matrix by tuning pH, temperature and time of the geopolymerization reaction. This paper aims at designing a multifunctional porous material combining the microporosity of the zeolites and the mesoporosity of the geopolymer matrix. To widen the structure of the alkali-activated materials also to the macro range, a cellular structure was obtained by in situ inorganic foaming approaches. A one-step procedure was selected allowing to realize simultaneously geopolymerization and crystallization using mild operating conditions, without secondary processing. The geopolymer gel precursors were prepared by mixing metakaolin, silicon powder and sodium hydroxide solution. Different mixture compositions were investigated in order to study the effect of the silicon content, relative humidity (RH%), and synthesis time on the zeolitic content in the porous material. The experiments planning was carried out using the Design Of Experiments (DOE) approach. DOE technique enables designers to control simultaneously the individual and interactive effects of many factors that could affect the output results for any design. The space of the experiments was explored using a fractional factorial design (an orthogonal array has been used) considering two factors that can vary on two levels (silicon w%, time), and one factor that can vary on three levels (relative humidity of curing). Every sample obtained at synthesis time higher than two days, regardless relative humidity and silicon content, contained zeolite A [LTA] and X [FAU] as the only crystalline phases. A positive correlation of both silicon addition and relative humidity on the 13X growth was demonstrated, while it seems that the growth of Na-A negatively depends on both the added silicon and relative humidity. From a morphological point of view, the crystallization of zeolite phases occurred preferentially along the pore walls, whereas the struts are completely constituted by an amorphous geopolymer matrix (Figure 1).

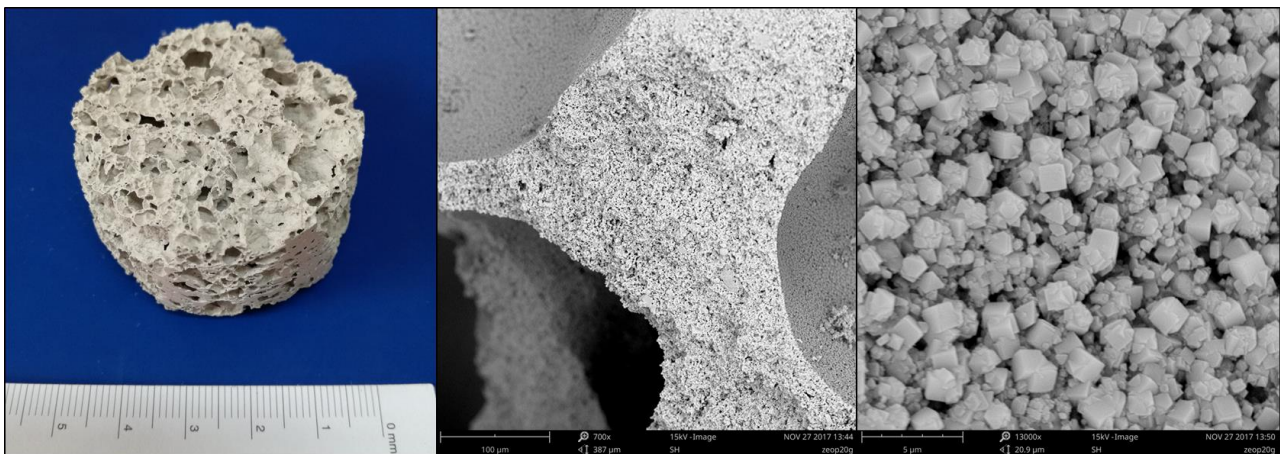


Figure 4 – Self-supporting zeolite at different magnifications

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## POSTER

### NANO-STRUCTURED CARBON FIBER MATERIAL FOR HIGH TEMPERATURE APPLICATIONS

Emanuele Apollo\*, Cristiano Bordignon  
\* [e.apollo@aeronauticalservice.com](mailto:e.apollo@aeronauticalservice.com)  
Aeronautical Service S.r.l., Fiumicino, Italy

**Key Words:** High-temperature; Composite; Carbon fiber; Nano-structured material

In the last few years, fire protection has become a safety key aspect to ensure an optimal relationship between passive protection and weight containment. Innovative architectures based on reinforced composite materials with nanometric inclusions enable the creation of highly engineered complex geometry structures with almost fully customizable properties. This work shows new nano-structured carbon fiber material, called AS-HT, with service temperature up to 1200°C.

This composite material can be used as structures in the aeronautic, naval, rail field, replacing the most common materials as Inconel or Titanium superplastic alloy. The comparison with the conventional materials highlights the unique property in terms of no-smoke emission, low thermal expansion coefficient, very low thermal conductivity and good mechanical properties. Lightness and low life cycle cost are its main advantages: for this reason it can be considered as a valid technological solution to replace heavy structural elements. When fire proofness and high working temperature are requested, AS-HT allows to save up to 30% in weight respect to all the conventional materials. Thanks to the possibility to realize complex shapes easily, AS-HT nano structured carbon fiber material is an excellent candidate for the manufacturing of engine bay, engine exhaust, heat shield, nacelle, leading edges, nozzles and upper duct of helicopter.



*Figure 5 – Engine nacelle*

#### ACKNOWLEDGMENTS

This innovative material is designed thanks to the collaboration with ISTECCNR, Faenza, and manufactured by Aeronautical Service.

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## PROJECT: VALORIZATION OF RED MUD WASTE IN THE SUSTAINABLE PRODUCTION OF ADVANCED GEOPOLYMER COMPONENTS

Claudio Ferone<sup>1\*</sup>, Mira Vukcevic<sup>2</sup>, Giuseppina Roviello<sup>1</sup>, Ivana Boškovic<sup>2</sup>, Veselinka Grudic<sup>2</sup>, Slobodan Radusinovic<sup>2</sup>

\*[claudio.ferone@uniparthenope.it](mailto:claudio.ferone@uniparthenope.it)

<sup>1</sup> Dipartimento di Ingegneria, Università di Napoli "Parthenope" – Centro Direzionale, Isola C4 – 80143 Napoli - Italy

<sup>2</sup> Faculty of Metallurgy and Technology, University of Montenegro - Cetinjski put bb, 81000 Podgorica - Montenegro

**Key Words:** red mud; geopolymers; composites.

Geopolymers are aluminosilicate materials characterized by excellent mechanical properties, micro- and nano- porosity, low shrinkage, thermal stability, fire resistance, frost and aggressive environment resistance and therefore they are very attractive for use in many applications. They are synthesized by a polycondensation reaction in highly alkaline environment starting from reactive aluminosilicate precursors. Many aluminosilicate materials have been used for the synthesis of geopolymers, mainly metakaolin, fly ash and slags, but any aluminosilicate of adequate reactivity is a potential precursor. This is the reason why a lot of natural and industrial waste have been successfully tested for the synthesis of geopolymers. The whole Western Balkans have great environmental problems with the red mud waste deriving from the Bayer

process of alumina production. Two basins of red mud in the Alumina Factory in Podgorica contain about 6.000.000 tons of red mud. Studies about valorization of red mud in the production of materials or products exist, but the results depend on the chemical and mineralogical features of red mud used in the process of synthesis. The main problem of red mud is the large presence of hydrated iron oxides, which can give rise to dehydration/rehydration phenomena, potentially affecting the dimensional stability of the obtained components. On this basis, this project aims at define the most favorable conditions and appropriate methods under which red mud, a mixture of raw sludge and white bauxite, could be used as the starting material in the synthesis of geopolymers. The geopolymerization parameters will be defined: the S/L ratio, the chemical parameters of activators and the Si/Al ratio in raw mixture. For this purpose, other aluminosilicate precursors could be added. The obtained materials will be thoroughly characterized in terms of structural, microstructural and mechanical properties. The second phase of the project will focus on the utilization of red mud based geopolymers in the production of geopolymer matrix organic-inorganic composites and hybrids and porous lightweight components. Innovative procedures will be applied to the optimized red mud geopolymer in order to obtain novel composites and hybrid materials in bulk and porous form. Potential applications may be as binders in construction materials, for structural components, composites, foams, filters, insulators, adhesives and thermal isolators. Developed materials will be tested in the production of full-scale high-added-value components.



Figure 6 – red mud lake near an aluminum processing plant in Podgorica

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## JECS TRUST Best POSTER Award Competition

### DEPENDENCE OF THE GEOPOLYMERIZATION AND END PRODUCTS FROM THE NATURE OF THE SOLID PRECURSORS

Elie Kamseu<sup>1,2</sup>, Valeria Alzari<sup>1</sup>, Daniele Nuvoli<sup>1</sup>, Davide Sanna<sup>1</sup>, Alberto Mariani<sup>1</sup>, Cristina Leonelli<sup>2</sup>  
\*[kamseuelie2001@yahoo.fr](mailto:kamseuelie2001@yahoo.fr)

<sup>1</sup>Department of Chemistry and Pharmacy, and INSTM Unit, via Vienna 2, 07100, Sassari, Italy

<sup>2</sup>Department of Engineering Enzo Ferrari, University of Modena and Reggio Emilia, Via  
Pietro Vivarelli 10, 41125 Modena, Italy

**Key words:** Geopolymer, solid precursor, performance, sustainable process, durability, classification, standards

This work presents an evaluation of the parameters that govern the geopolymerization focusing to keys facts that can enhance the scientific and technological evolution of the new binder and related composites. It is demonstrated that the processing -from activation to curing cycle- should be, for the best performance of the final products, determined by the characteristics and properties of the solid precursors. A deep investigation regarding many assumptions as the temperature of 700°C for the calcination of the clayey materials, the comparison of metakaolin and fly ash or the increase of the alkalinity of the activating solution to improve the final strength are “theories” with limitations. We proved, once more, that the nature of the solid precursor defines univocally the process and the final product performance. Additionally, the thoroughly knowledge of the solid precursor can establish fundamentals for the definition, the process, the classification and standards, the durability, economy and sustainability of geopolymers.

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## INFLUENCE OF PARTICLE SIZE ON MECHANICAL PROPERTIES OF SLAG BASED ALKALI ACTIVATED MATERIALS

Katja Traven<sup>1</sup>, Mark Češnovar<sup>1,2</sup>, Vilma Ducman<sup>1</sup>  
[katja.traven@zag.si](mailto:katja.traven@zag.si)

<sup>1</sup> Slovenian National Building and Civil Engineering Institute, Dimičeva 12, 1000 Ljubljana, Slovenia

<sup>2</sup> Jozef Stefan International Postgraduate School, Jamova cesta 39, 1000 Ljubljana, Slovenia

**Key Words:** alkali activated materials, electric furnace steel slag, mechanical properties, circular economy

Alkali activated materials (AAM) are recognised as promising alternatives to technical ceramic and building materials due to the lower energy demands for the production of AAM. Additionally for their production inorganic wastes or industrial residues can be recycled and consequently disposal of waste materials in landfills is avoided. AAM are inorganic systems which consist of, firstly, a reactive solid component (precursor) which contains sufficient amounts of  $\text{Al}_2\text{O}_3$  and  $\text{SiO}_2$  in their reactive semi crystalline to amorphous phases and, secondly, an alkaline activation solution (activator) which (apart from water) mainly contains alkali hydroxides and silicates. Among various precursors such as metakaolin, fly ash, and slag, ferrous slag present one of the potential sources due to their abundant availability and the presence of reactive aluminosilicates. [1]

Slags are by-products in steelmaking processes where the metallic and non-metallic constituents are separated from its raw ore. There are several kinds of slag obtained after the different processes of steelmaking such as blast furnace slag (air-cooled (ABS) or granulated (GBS)), basic oxygen furnace slag (BOS), electric arc furnace slag (EAF), secondary metallurgical slag (e. g. ladle furnace slag) and other (e. g. de-sulphurisation slag). In this study the EAF slag from metallurgical industrie in Slovenia was used in order to prepare AAM. EAF slag is produced in the primary refining stage by the electric arc furnace during the manufacture of crude steel. In this process the fluxes (lime[stone] and/ or dolomite) are heated to a liquid state by the means of an electric current. The chemical composition of slag differs according to the type of metallurgical treatment. [2]

Mechanical and chemical properties of AAM are strongly dependent on the type and amount of precursor, activator, the curing regime, particle size etc. It has been shown that the mechanical activation has a positive effect in the improvement of mechanical strength of AAM. Mechanical activation is usually carried out by high-energy milling devices. Changes induced in material during the milling process include particle size reduction, increase in specific surface area, particle morphology changes, structural defects, increase in amorphous phase degree and implying structural rearrangement. The most important consequence of transformation that occur in material during the mechanical activation is its enhanced reactivity and thus improved mechanical properties. [3, 4]

The objective of this study was to evaluate the influence of particle size (after mechanical activation) on mechanical properties (bending and compressive strengths). Therefore three sets of AAM pastes were prepared and evaluated with different particle sizes ( $d < 63$ ,  $63 < d < 90$  and  $90 < d < 125 \mu\text{m}$ ), under the same curing regime and fixed precursor, activator and water mass ratio. The results of the bending and compressive strength measurements reveal, that the influence of particle size on the bending strength shows no specific trend, whereas the compressive strength is significantly increased, e.g. for three times when the particle size is reduced by half. Additionally microstructural evaluation was performed by means of Mercury intrusion porosimetry (MIP) and scanning electron microscopy (SEM).

### ACKNOWLEDGMENTS

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## AGEING AND CURING OF ALKALI ACTIVATED SLAG BASED MATERIALS

Mark Češnovar<sup>1,2</sup>, Katja Traven<sup>1</sup>, Vilma Ducman<sup>1</sup>  
[mark.cesnovar@zag.si](mailto:mark.cesnovar@zag.si)

<sup>1</sup> Slovenian National Building and Civil Engineering Institute, Dimičeva 12, 1000 Ljubljana, Slovenia

<sup>2</sup> Jozef Stefan International Postgraduate School, Jamova cesta 39, 1000 Ljubljana, Slovenia

**Key Words:** alkali activation, ladle slag, electric arc furnace slag, ageing, curing conditions.

The alkali activation technology offers possibility to utilize large amounts of aluminosilicate rich secondary materials from metallurgical processes into a new group of useful building products. Steel slag based alkali activated materials have high mechanical strength, good fire and high thermal resistance at elevated temperatures and in case of low density also low thermal conductivity. [1]

In order to achieve hardened structure of alkali activated materials (AAM), beside the amount of reactive aluminosilicates phases in slag, the particle size distribution, the addition of alkali solution and also ageing and curing conditions are important factors. The development of strength in a slag based AAM in terms of ageing and curing regimes was extensively studied and presented in various publications. Although several researches in the field of curing have been done, and usability of specific approaches for AAM systems was confirmed there is no comprehensive model available so far. Altan et.al., suggested that alkali activated slag mortars, using alkali hydroxide and sodium silicate activator, cured at room temperature (23 °C) for sufficiently long time (70 days) attained equal or higher strengths in comparison to those cured at 80 °C for 4 days. [2] A Fernandez-Jaimenz et.al., reported the effect of curing conditions of alkali activated slag and fly ash based materials. The trend of compressive strength values for fly ash based alkali activated with 8 M NaOH, was increasing nearly linearly with elevated temperatures (45, 65 and 85 °C) and with curing time (5, 12, 24 h and 7 days). On contrary, for the slag mortars activated with 4 M NaOH lower compressive strength after curing at elevated temperature (45 °C) was reported in comparison to those cured at room temperature. The difference was ascribed to the fact that the solubility of Al and Si increases significantly with temperature but it is not so with calcium compounds which were present in slag. [3]

In the present research we have firstly focused on identification of suitable precursors for alkali activation. Two types of slags, the electric arc furnace steel slag (EAF) and the ladle furnace basic slag (LF) from different metallurgical industries in Slovenia were selected. Different ratio of selected precursors was prepared and activated in order to identify the proper ratio of selected precursors which would provide optimal mechanical properties. The values of bending strength measurements were in the range from 3 to 7 MPa whereas compressive strength measurements were between 30 and 63 MPa (for the EAF/LS ratios 1/0, 0/1, 1/3, 1/1 and 3/1). Finally, with the selected optimal mixture (EAF/LS mixed in ratio 1/1 and activated with K<sub>2</sub>SiO<sub>3</sub> with the activator/slag ratio 0.6) the influence of curing in a heat-chamber at room temperature (R. T.), 50, 70 and 90 °C, and ageing for 1, 3, 7 and 28 days was further studied. Linear trend on mechanical strength dependence on elevated curing temperatures at early age has been confirmed. It has also been found out that curing at 70 °C for 3 days resulted in practically the same (bending/compressive) strength as for specimen cured at room temperature for 28 days. Microstructure evaluation of input materials as well as differently cured samples was performed by means of XRD and FTIR.

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## JECS TRUST Best POSTER Award Competition

### NEW GEOPOLYMERS BASED ON WASTES OF CERAMIC INDUSTRY AS INNOVATIVE PRODUCTS FOR TILE INDUSTRY

Lorenzo Valeriani<sup>1,2,\*</sup>, Maria Chiara Bignozzi<sup>1</sup>, Claudio Beneventi<sup>2</sup>

\* [lorenzo.valeriani4@unibo.it](mailto:lorenzo.valeriani4@unibo.it)

<sup>1</sup> Dipartimento di Ingegneria Civile, Ambientale e dei Materiali, Università di Bologna, Italy

<sup>2</sup> Marazzi Group S.r.l. a socio unico, Italy

**Key Words:** Geopolymer, Ceramic Tile, Process waste, Flooring materials

The large amount of waste yearly disposed to landfill, the global impoverishing of natural resources and environment pollution, the emergency of carbon dioxide emissions, are some of the motivations driving this research. The aim of this project is to develop low cost and energy saving methods for manufacturing tiles by using geopolymerization as an innovative processing technique. Geopolymers are non-crystalline aluminosilicate polymers that are formed by mixing of alkali-silicate solutions with reactive aluminosilicate materials. This research wants to search a way to move towards sustainable solutions for tile industry. Geopolymer based tile may eliminate the main energy intensive processing step like the high temperature firing process. Moreover, the intention is to reach the total substitution of the natural raw materials with waste and sludge from the ceramic process. The research started with an accurate monitoring of waste produced during the ceramic cycle and currently disposed to landfill and those available for reintroduction into the production cycle. These materials were characterized from the physico-chemical point of view in order to obtain their chemical and mineralogical composition and grain size distribution. Ceramic wastes considered in this study as geopolymer precursors were: i) sludge resulting from rectifying process, ii) sludge resulting from lapping process, iii) mixed sludge resulting from lapping and rectifying process, iv) fired ceramic waste, v) powder from dry rectifying process. Many innovative mix designs of geopolymers were initially tested in order to determine the waste capability to be alkali activated. Casting and pressing methods were both used for shaping as well as granulometry studies were carried out in order to reach an optimized grain size distribution of the investigated precursors.

Preliminary results show that dry rectifying powder is the most promising waste to develop the best geopolymer mix design, minimizing water absorption and maximizing mechanical strength of the hardened product. Moreover potassium hydroxide and K,Li-silicate reagent resulted more efficient in the activation step leading to geopolymers with lower water absorption, greater mechanical strength and lower efflorescence than those activated by sodium hydroxide. Thermal characterization by DTA-TG analysis, optical dilatometer and heating microscope as well as microstructure analysis with XRD and SEM techniques have been carried out on the final products, which exhibit interesting properties comparable with those usually required by ceramic tiles. Indeed, the obtained pressed geopolymers may be classified as B1b and B11a classes according to tile requirements reported in ISO 13006 and they can be considered potentially appealing materials for floor and wall covering applications.



**EFFECT OF METAKAOLIN ADDITION ON AAM BASED ON VOLCANIC ASH AND GHIARA (ETNA-SICILY)**

Barone G.<sup>1</sup>, Finocchiaro C.<sup>1\*</sup>, Mazzoleni P.<sup>1</sup>, Stroschio A.<sup>1</sup>, Sgarlata C.<sup>2</sup>, Lancellotti I.<sup>2</sup>, Leonelli C.<sup>2</sup>

\*claudio.finocchiaro@unict.it

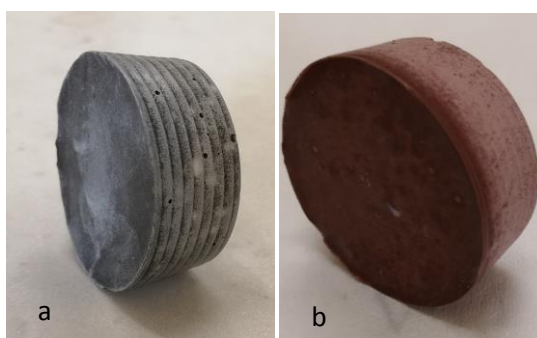
<sup>1</sup> Dipartimento di Scienze Biologiche, Geologiche e Ambientali, Università di Catania, CATANIA (Italy)

<sup>2</sup> Dipartimento di Ingegneria "Enzo Ferrari", Università di Modena e Reggio Emilia, MODENA (Italy)

**Key Words:** volcanic ash, geopolymers, eco-friendly, metakaolin, reactivity

Etna volcano offers large quantity of volcanic products: immense deposits of volcanic ash and paleo-soils, both characterized by a high amorphous content, were largely used as aggregate in historical buildings in the nearby of Catania town. The paleo-soils have undergone transformations due to contact with Etna's lava flows and they are indicated with the local name "ghiara" (Belfiore et al., 2010).

The goal of our research is to esteem the possibility of their use as aluminosilicate sources in the alkali activation process for eco-friendly materials with a low production cost and environmental impact. Based on preliminary results already published on alkali activation of volcanic ash and ghiara from Etna volcano (Kamseu et al., 2009), in this work, we added metakaolin to increase the reactivity of volcanic aluminosilicate powders to avoid the thermal treatments necessary to increase the mechanical resistance and chemical stability of AAM based solely on volcanic raw materials. These last have low reactivity, long setting time and low compressive strength (Tchakoute et al., 2013), for these reasons they, generally, require thermal treatment to enhance mechanical properties (Kamseu et al., 2009). Therefore, the main aim concern to mix with different percentage (5-25%) metakaolin to raw material powder at room temperature ( $25 \pm 3^\circ\text{C}$ ) to limit environmental impact, maintaining good results in terms of resistance (Diobo et al., 2014, Kani and Allahverdi 2009; Tchakoute et al., 2012, Robayo-Salazar et al., 2016). Different mixtures of sodium silicate ( $\text{Na}_2\text{SiO}_3$ ) and sodium hydroxide (NaOH) were used to activate the aluminosilicate materials. In detail, after a detailed mineralogical and chemical characterization of raw materials and metakaolin performed by XRD and XRF (with portable equipment) analyses, we used these volcanic powders as aluminosilicate source in the formulation of geopolymeric pastes. Seven days after alkali activation, we performed integrity test to all AAM samples with different amount of metakaolin added for both volcanic material, to check the chemical stability in water and mechanical resistance, obtaining good results for samples with 10-25% of metakaolin (Figure 1). The mineralogical and microstructural properties were evaluated by XRD and Raman analyses on samples with different metakaolin percentage with the aim to investigate the extent of aluminosilicate network reticulation. Finally, this work shows that despite the low reactivity of these volcanic raw materials, they can still be used to synthesize alkali activated materials with low metakaolin amount, avoiding thermal treatments and achieving good physical and mechanical properties at ambient temperature for different application fields.



*Figure 7 – (a) Geopolymer based on volcanic ash and 20% of metakaolin; (b) Geopolymer based on ghiara and 20% of metakaolin*

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## JECS TRUST Best POSTER Award Competition

### DEVELOPMENT OF ALKALI ACTIVATED CEMENTS BY USING MUNICIPAL SOLID WASTE INCINERATION (MSWI) WEATHERED BOTTOM ASH

A. Alfocea-Roig<sup>1</sup>, A. Maldonado-Alameda<sup>1,\*</sup>, J. Mañosa<sup>1</sup>, J. Giro-Paloma<sup>1</sup>, J. Formosa<sup>1</sup>, JM Chimenos<sup>1</sup>

\*[alex.maldonado@ub.edu](mailto:alex.maldonado@ub.edu)

<sup>1</sup> Departament de Ciència de Materials i Química Física, Universitat de Barcelona, C/Martí i Franquès, 1-11, 08028, Barcelona, Spain.

**Key Words:** Waste management; Alternative cements; Alkali-Activated Cements, Weathered Bottom Ash, Sustainability

The increase of energy demand and the Municipal Solid Waste (MSW) management are some of the environmental problems that have generated more interest in modern society. These conflicts have as main effects the growth of greenhouse gases (GHG) emissions, the global warming and the pollution of the environment and the natural resources. Regarding the building sector, the Ordinary Portland Cement (OPC) is the most polluting material, responsible for 2-3% of the global primary energy consumed and 5-7% of global CO<sub>2</sub> emissions [1,2]. Therefore, it is necessary to seek an OPC alternative and one of the most advanced candidates are Alkali Activated Cements (AAC). AAC has become an ideal alternative to Ordinary Portland Cement (OPC) because of its properties [2]. Moreover, the main benefit exhibited by AAC relies on the ecological side. If it is compared with OPC, the AAC use reduces the CO<sub>2</sub> emissions and the energy consumption associated with the manufacturing of cement [3]. In addition, as most of these new cements are produced by using industrial by-products or wastes, it also makes the AAC embrace the zero waste principle. Consequently, the AAC are considered as new sustainable and eco-friendly cements. The present study consists on the development of AAC by using incinerated Bottom Ash (IBA) from MSW as unique raw material precursor. IBA is the main by-product obtained from the incineration of MSW [4]. It is stabilized outdoors for 2-3 months to obtain the Weathered Bottom Ash (WBA) [5], which is currently used as secondary aggregate material in construction and in civil purposes. Due to its composition rich in calcium oxide, silica and alumina [6], WBA can be considered a suitable precursor for obtaining AAC. The main goal of this research is to evaluate the viability of developing different AAC formulations by using WBA as unique precursor and characterize them from mechanical, environmental and physical point of view. In addition, a physicochemical characterization by means XRF, XRD, FT-IR, SEM among others of the WBA and the final material obtained was conducted. It has been developed AAC formulations by using a mixture of commercial sodium silicate and sodium hydroxide with different molarities (2M, 4M, and 8M) as activators. Then, the AAC structure and properties obtained regarding the sodium hydroxide concentration effect have been analysed. The final results show the viability to formulate AAC by using WBA as unique precursor. In addition, it is observed an influence of sodium hydroxide concentration on the final properties of AAC developed, being better from mechanical point of view on AAC elaborated with 6M solution.

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## GEPOLYMERIC STRUCTURES FOR WATER PURIFICATION

Karine Goulart de Oliveira<sup>1</sup>, Luca Martone<sup>2</sup>, Maria Concetta Bruzzoniti<sup>2</sup>, Paolo Colombo<sup>1</sup>

\*[karine.goulartdeoliveira@studenti.unipd.it](mailto:karine.goulartdeoliveira@studenti.unipd.it)

<sup>1</sup>Department of Industrial Engineering, Università di Padova, Padova, Italy.

<sup>2</sup>Department of Chemistry, Università di Torino, Torino, Italy.

**Key Words:** Geopolymer; spheres; water treatment; ion exchange; surface modification.

With the increasing industrial development, the world's limited amount of available drinking water is susceptible to various contaminants of industrial waste waters, such as pesticides, heavy metals, antibiotics, perfluoroalkyl substances (PFAS), among others. This situation demands the development and enhancement of advanced water treatment, aiming at greater efficiency of contaminants removal at a low cost. The use of geopolymers (GP) for ion exchange has been increasingly used in the water treatment area, due to their ability to charge-balance (exchange) cations. However, the great proprieties for ion exchange that GP possess, suggest that it may be used also as an anion exchange system, if a surface chemistry modification is performed. For this work, the aim was to produce geopolymeric spheres for removal of contaminants from water, as well as a surface modification with the objective of removing also anions. To obtain the GP spheres, the suspension was prepared by adding metakaolin in a K based ( $K_2O/SiO_2=1.3$ ,  $SiO_2/Al_2O_3=4$  and  $H_2O/K_2O=18.5$ ) and in a mixed Na-K based ( $K_2O/SiO_2=0.65$ ,  $Na_2O/SiO_2=0.65$ ,  $SiO_2/Al_2O_3=4$  and  $H_2O/K_2O=18.5$ ) activating solutions and mixed at 2200 rpm for 10 minutes. Afterwards, 0.8% of SDS diluted 10x in water was added and mixed for another 10 minutes. To obtain the spheres the GP suspension was dripped into PEG 400 and 600 heated to 75°C in a water bath. When the GP drops came in contact with the PEG, they immediately solidified, forming the spheres. After approximately 3 minutes, the beads were withdrawn from the PEG and dried in room temperature for 30 min. They were then washed with ethanol several times to remove the remaining PEG. After this process, the spheres were dried in room temperature for 24 h and then transferred to an oven at 60°C for another 24 h. For the surface modification, the spheres were coated by immersion in a silane-Cl chemical, N-[3-(Trimethoxysilyl)propyl]-N,N,N-trimethylammonium chloride in 50% methanol, for 30 min. They were then dried for one hour in room temperature and heat treated at 60°C for 2 hours. The spheres were characterized by pycnometry, to obtain porosity data, which resulted in an open porosity of 62.86% and a total porosity of 63.51% for the K-based spheres, and an open porosity of 75.13% and the total 75.35% for Na+K-based spheres. However, the SEM images suggest that porosity is mostly closed, although it is possible to observe Cl residues in the internal structure of the coated spheres, suggesting a full infiltration probably through the mesopores in the GP. Preliminary data for SSA were also acquired, giving a value of 34.9 m<sup>2</sup>·g<sup>-1</sup> for the K-based spheres and 6.1 m<sup>2</sup>·g<sup>-1</sup> for Na+K-based spheres. The corresponding, computed pore size distribution was bimodal, with pores with a radius of 1.9 nm and 1.5 nm and a broad range of pores with a size centered around 7 nm and 30 nm for K and Na+K spheres, respectively. Adsorption tests with as-received and ground spheres (coated and uncoated) were performed by putting the GPs in contact with solutions containing different contaminants at a known concentration for at least 24 h. So far, the obtained results were negative for adsorption of anionic herbicide glyphosate, while cationic ion exchange occurred, as expected. Further tests as well as other types of coating for surface modification are going to be performed. Some 3D printed structures are also being produced for testing in this application.